THE JOURNAL

OF THE

Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 6.—Vol. XX.] **JUNE 1901.** [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6, all prepaid.

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NOTE.—The names in *italics* are those of Members of Committee who retire at the end of the current session.

NOTICES.

Annual General Meeting.

The Annual General Meeting will be held in Glasgow, on Wednesday, July 24th, and following days. A programme will be found in the May number of the Journal. A ticket of membership is enclosed herewith.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in *italics* in the list of Council will retire from their respective offices at the forthcoming Annual Meeting.

Mr. Ivan Levinstein has been nominated to the office of President; Mr. Jos. W. Swan, F.R.S., has been nominated a Vice-President under Rule 11; and Mr. Robert Irvine, Dr. R. Messel, and Mr. J. M. C. Paton have been nominated Vice-Presidents under Rule 8.

The Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Dr. Edw. Divers, F.R.S., Prof. W. R. Eaton Hodgkinson, Ph.D., Mr. David Howard, and Mr. Max Muspratt have been nominated, under Rule 18, to fill four vacancies among the Ordinary Members of Council. No Ballot will be required.

Changes of Address.

When notifying new addresses, members are requested to write them distinctly, and state whether they are temporary or permanent. Multiplication of addresses is also to be avoided as tending to create confusion. When sending subscriptions, the use of the form attached to the application helps in the verification of addresses, on which the safe delivery of the Journal depends.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

The Secretary would be glad to receive offers from members and others who may have complete copies of Vol. XVI., 1897, of the Society's Journal to dispose of.

Arsenic.

In view of the great public interest which has been aroused by the Arsenic Epidemic, it has been decided to republish, in pamphlet form, uniform with the Journal, the papers, discussions, and abstracts dealing with the detection and determination of Arsenic and other associated elements, which have appeared in the Journal since the beginning of this year. To these have been added extracts bearing on the subject from other sources, including a description, from Wanklyn's new book on Arsenic, of the Marsh test, in the inventor's own words, and dated 1836. Copies, price 1*s.* each, may be obtained from Messrs. Eyre and Spottiswoode.

LIST OF MEMBERS ELECTED 24th JUNE 1901.

Arundel, Arthur S. D., Penn Steelworks, Hoxton, N., Manufacturer.

Ayer, Harold O., c/o Southern Cotton Oil Co., Savannah, Ga., U.S.A., Chemist.

Browne, Arthur L., M.D., 13-15, North Street, Baltimore, Md., U.S.A., Analytical Chemist.

Cohn, Sigmund, 35, Frankfort Street, New York City, U.S.A., Metallurgical Chemist.

Dolden, Arthur G., Avondale, Dawlish Road, Leyton, Essex, Analytical Chemist.

Fairlie, Jas., Camelon Chemical Works, Falkirk, N.B., Manufacturing Chemist.

Ferrell, Jos. L., 2220, Race Street, Philadelphia, Pa., U.S.A., Mechanical Engineer.

Greaves, Albert E., Goole Alum Works, Goole, Yorks, Works Chemist.

Hartzell, Harry S., 22, North 8th Street, Allentown, Pa., U.S.A., Chemist.

Hershey, Aldus N., c/o Sharp and Dohme, Baltimore, Md., U.S.A., Chemist.

Hyman, Leonard W., New York Central and Hudson River R.R., West Albany, N.Y., U.S.A., Analytical Chemist.

Jacobsen, Rudolph C., 154, Lake Street, Chicago, Ills., U.S.A., Editor "Hide and Leather."

Ogden, Richard L., c/o Pittsburg Gas and Coke Co., Otto, Allegheny Co., Pa., U.S.A., Chemist.

Parker, Col. Richard H., N.Y. Testing Laboratory, Long Island City, N.Y., U.S.A., Analytical Chemist.

Redpath, Leon Wallace, U.S. Naval Torpedo Station, Newport, R.I., U.S.A., Chemist.

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Rogerson, John W., 101, Leadenhall Street, London, E.C., Maltster.

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Wren, E. Cecil, Glencoe, Eaglescliffe, R.S.O., Co. Durham, Vinegar Brewer.

CHANGES OF ADDRESS.

Annis, Dr. E. G., l/o Huddersfield; Public Health Dept, Town Hall, Greenwich, S.E.

Bloxam, W. Popplewell, l/o London; Presidency College, Madras, India, Professor of Chemistry.

Bolton, E. Richards, l/o Gateshead; 7, Leazes Terrace, Newcastle-on-Tyne.

Briggs, J. F., l/o Wandsworth; 1, Presburg Road, New Malden, Surrey.

Burls, F. B., l/o Stratford-on-Avon; 13, Langdale Road, Greenwich, S.E.

Chemical and Metallurgical Society of South Africa; Journals to Box 2596, Johannesburg, Transvaal.

Cohen, Dr. H., l/o North Spring Avenue; c/o S. Mandle, 3943, West Pine Boulevard, St. Louis, Mo., U.S.A.

Coleman, W. H., l/o Ashton New Road; 4, Sunnyside Terrace, North Road, Clayton, Manchester.

Crake, Wm., l/o 397; 394, Staniforth Road, Sheffield.

Cullen, Wm., l/o Gravesend; c/o South African Explosives Co., Modderfontein, Transvaal; and (Journals) Craig Knowe, Irvine, N.B.

Davidson, R. Holden, l/o Widnes; United Alkali Co., Ltd., Ammonia Soda Works, Fleetwood.

Dawson, Jas., l/o St. Vincent Place; British Dyewood and Chemical Co., 53, Bothwell Street, Glasgow.

Durant, H. T., l/o Salvador; retain Journals until further notice.

Fallon, J. M. H.; Journals to Box 771, Havana, Cuba.

Fehrlin, Dr. H. C., l/o Milwaukee; Belvidere House, 4th Avenue and 18th Street, New York City, U.S.A.

Fleming, Wm. P.; Journals to c/o Thos. Cook and Sons, Ludgate Circus, E.C.

Forbes, Paul R., l/o Boston; c/o Walsh, Hall, and Co., Yokohama, Japan.

French, Alf., l/o London; St. Bartholomew's Hospital, Rochester, Kent.

Galbraith, Wm., l/o Leeds; Mount Mine, Perranporth, R.S.O., Cornwall.

Green, J. Wilberforce, l/o Sydenham; 55, The Avenue, Kew Gardens, S.W.

Heileman, W. H., l/o Pullman; U.S. Dept. of Agriculture, Elwell, Iowa, U.S.A., Expert Field Assistant.

Hewitt, T. Lacey, l/o West Bromwich; Galen House, Malvern Link.

Huntington, Dr. H.; Journals to University Club, 1, West 54th Street, New York City, U.S.A.

Ichioka, T., l/o Stevenston; 15 Higashikatamachi, Hongo, Tokio, Japan.

Littlejohn, Jas.; Journals to c/o South African Banking Corporation, Johannesburg, Transvaal.

Lowe, Jas. S., l/o Edinburgh; Clemencia Estate, Bel Air Station, Mauritius.

McCrae, Dr. Jno., l/o Leeds; 7, Kirklee Gardens, Kelvinside, Glasgow.

Neilson, A. M.; Journals to 38, Brisbane Street, Greenock, N.B.

Orr, Robt., l/o Great Western Terrace; c/o Jas. Miller, Son, and Co., 79, West Nile Street, Glasgow.

Picard, H. F. K., l/o Gracechurch Street; 44, London Wall, E.C.

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Smith, J. Cruickshank, l/o Davenport Road; Tiri Tiri, Felday Road, Catford, S.E.

Sorel, E., l/o Avenue d'Orléans; 138, Boulevard Raspail, Paris, VI^{e}.

Stein, Sigmund, l/o Vauxhall Road; 214, Upper Parliament Street, Liverpool.

Strangman, J. Pim; retain Journals until further notice.

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Tennant, Jas., l/o Newcastle; Alex. Fergusson and Co., Ltd., 38, McAlpine Street, Glasgow, Lead and Colour Manufacturer.

Timmans, W. G., l/o Windmill Hill Lane; 108, Peel Street, Derby.

Uhlig, Wm. C., l/o South Brooklyn; c/o Hygeia Distilled Water Co., 349, West 12th Street, New York City, U.S.A.

Vreeland, C. D., l/o Chicago; Upper Montclair, N.J., U.S.A.

Webb, Wm. J., Journals to 90, Ash Street, Yonkers, N.Y., U.S.A.

Wilson, Ellwood, l/o Liberty; Saranac Lake, Franklin Co., N.Y., U.S.A.

Wilson, W. W., l/o Busby; The Grange, Carrbrook, near Stalybridge.

Wright, A. C., l/o Newland Avenue; 15, Northgate, Cottingham, Hull.

CHANGE OF ADDRESS REQUIRED.

Wyckoff, G. H., l/o Rochester, N.Y., U.S.A.

Death.

Truby, Charles; at Urmston, near Manchester. May 20.

STATEMENT OF REVENUE AND EXPENDITURE FOR THE YEAR 1900.

(Made up to the 23rd April 1901.)

REVENUE.

	£ s. d.	£ s. d.
Annual Subscriptions for 1900:—		
1 subscription received in 1898	1 5 0	
64 subscriptions received in 1899	79 16 0	
3,091 subscriptions received in 1900	3,863 10 0	
54 subscriptions received in 1901	67 10 0	
(Sundry odd balances of subscriptions)	3 4 1	
3,210		4,015 5 1
292 Entrance Fees	306 12 0	
Life Composition Fees	60 0 0	
Subscriptions on account of the "Collective Index"	..	38 0 6
Investments:—		
Interest on Metropolitan 3 per cent. Consolidated Stock	128 2 10	
Interest on Gas Light and Coke Company's 3 per cent. Consol. Deb. Stock.	17 6 6	
Interest on North British Railway 3 per cent. Consolidated Debenture Stock.	31 5 8	
Interest on Midland Railway 2½ per cent. Perpetual Preference Stock	17 14 2	
Interest on Great Eastern Railway 4 per cent. Guaranteed Stock	25 3 6	
Interest on New Zealand 3 per cent. 1945 Stock	28 15 0	
Interest on New South Wales 3 per cent. 1935 Stock	13 18 11	
Interest on South Eastern Railway 4½ per cent. Preference Stock	29 3 0	
Interest on Southwark and Vauxhall Water Company 3 per cent. Debenture Stock	14 5 0	
Interest on Deposit Account at Bank	30 14 1	336 8 8
Journal:—		
Advertisements	576 0 0	
Sales	422 19 1	
Loss by fire—Amount received from publishers	50 0 0	1,048 19 1
		£5,438 13 4

EXPENDITURE.

	£ s. d.	£ s. d.
Journal Sales:—		
Publishing	1,983 4 6	
Editorial:—	£ s. d.	
Editor's Salary	500 0 0	
Editor's Expenses	46 9 10	
Abstractors' Accounts	506 14 8	
Indexing Journal	89 2 0	
Patent Lists	71 10 3	
Portion of Secretary's salary	100 0 0	
Foreign Journals	9 6 6	
Sundry Journals	3 1 9	
	1,326 5 0	
Insurance of Stock	7 4 9	3,316 14 3
Printing Sundries		54 12 8
Sectional Expenses		368 8 9
Secretary's Salary (Portion not included in Editorial Journal Expenses)		350 0 0
Annual Meeting Expenses		160 6 3
Honorary Treasurer's Assistant		52 10 0
Stationery		71 14 6
Office Expenses		187 7 3
Library (Binding Books)		6 11 2
Auditors' Fee		10 11 6
Investment.—Purchase of 1,000*l.* Southwark and Vauxhall Water Company's 3 per cent. Debenture Stock	1,000 1 0	
Solicitors' charges		2 3 0
Sundries		2 1 7
Bank Charges		3 15 4
Treasurer's Petty Cash, Postage, &c.		40 6 7
Secretary's Petty Cash, Postage, &c.		61 7 7
Balance of Revenue over Expenditure		750 2 11
		£5,438 13 4

THE TREASURER—IN ACCOUNT WITH THE SOCIETY OF CHEMICAL INDUSTRY FOR THE YEAR 1900.

Dr.

	£ s. d.	£ s. d.
To Cash on Deposit (1st January 1900)	500 0 0	
Balance at Bank (1st January 1900).	221 1 7	
Cash in Treasurer's hands (1st January 1900)	13 3 0	
Cash in Secretary's hands (1st January 1900)	7 8 11	
Annual Subscriptions :—		741 13 6
2 for the year 1898	2 10 0	
54 for the year 1899	67 10 0	
3,091 for the year 1900 (less 5s. short paid)	3,863 10 0	
88 for the year 1901 (less 5s. 9d. short paid)	109 14 3	
1 for the year 1902	1 5 0	
Sundry odd balances and excess payments on Annual Subscriptions	3 4 1	
3,236		4,047 13 4
Entrance Fees (292 at 1l. 1s.)	..	306 12 0
Life Composition Fees (3 at 20l.)	..	60 0 0
Subscriptions received for the "Collective Index"	..	52 10 6
Interest from Investments :—		
Metropolitan 3 per cent. Consolidated Stock (£4,476 9 2)	128 2 10	
Gas Light and Coke Company's 3 per cent. Consolidated Debenture Stock (£600 0 0)	17 6 6	
North British Railway 3 per cent. Consolidated Stock (£1,084 13 4)	31 5 8	
Midland Railway 2½ per cent. Perpetual Preference Stock (£736 0 0)	17 14 2	
Great Eastern Railway 4 per cent. Irredeemable Guaranteed Stock (£654 0 0)	25 3 6	
New Zealand 3 per cent. 1945 Stock (£1,000 0 0)	28 15 0	
New South Wales 3 per cent. 1935 Stock (£485 4 8)	13 18 11	
South Eastern Railway 4½ per cent. Preference Stock (£673 0 0)	29 3 0	
Southwark and Vauxhall WaterCompany's 3 per cent. Debenture Stock (£1,000 0 0)	14 5 0	
(£10,709 7 2)		
Interest on Deposit Account at Bank.	30 14 1	336 8 8
Journal :—		
Advertisements	624 0 0	
Sales	415 10 9	
	1,039 10 9	
Amount received from Publishers for the loss of Journals destroyed by fire at their premises	50 0 0	
		1,089 10 9
		£6,634 8 9

Cr.

	£ s. d.	£ s. d.
By Journal Expenses :—		
Publishing	2,090 12 5	
Editorial :—	£ s. d.	
Editor's Salary	500 0 0	
Editor's Expenses	46 8 10	
Abstractor's Accounts	494 13 0	
Indexing Journal	88 2 0	
Patent Lists	71 2 3	
Foreign Journals	9 6 6	
Sundry Journals	3 17 5	
Portion of Secretary's Salary	100 0 0	
	1,313 10 0	
Insurance of Stock	7 4 9	
		3,411 7 2
Sundries Printing, &c.	..	57 14 7
Sectional Expenses :—		
Liverpool Section	28 6 1	
London Section	63 16 1	
Newcastle Section	17 17 6	
Manchester Section	49 6 0	
New York (U.S.A.) Section	123 0 0	
Nottingham Section	26 15 6	
Scottish Section	38 18 10	
Yorkshire Section	20 8 9	
		368 8 9
Secretary's Salary (Balance not included in Editorial Expenses)		350 0 0
Library (Binding Books)		6 5 4
Expenses in connection with Annual Meeting		160 6 3
Honorary Treasurer's Assistant		52 10 0
Stationery		47 1 9
Office Expenses, &c. :—		
Rent	102 7 0	
Cleaning, Attendance, &c.	12 18 8	
Lighting	4 14 1	
Furniture and Sundries; Fire Insurance	5 0 0	
Clerical assistance	62 7 6	
		187 7 3
Auditors' Fee	..	10 11 6
Solicitor's Charges	..	2 3 0
Purchase of 1,000l. Southwark and Vauxhall Water Company's 3 per cent. Debenture Stock	..	1,000 1 0
Sundries	..	2 1 7
Bank Charges	..	3 15 4
Treasurer's Petty Cash, and Postage	14 5 1	
Clerical assistance :—		
Writing up Subscriptions	15 11 6	
Writing up new Register	10 10 0	
		40 6 7
Secretary's Petty Cash and Postage	..	61 7 7
Cash on Deposit (31st December 1900)	650 0 0	
Balance at Bank (31st December 1900)	218 14 4	
Balance in Secretary's hands (31st December 1900)	4 6 9	
		873 1 1
		£6,634 8 9

We have compared the above Statement with the Vouchers, Counterfoils of the Receipts issued, and other records, and are of opinion it correctly exhibits the Cash transactions of the Society for the year 1900. The amounts of the Metropolitan 3 per cent. Consolidated, 3 per cent. New Zealand, and 3 per cent. New South Wales Stocks have been confirmed by the Chief Accountant to the Bank of England. Certificates for the remaining investments have been inspected, and the Bank Balances have been certified to us by the Bankers.

23, St. Swithin's Lane, London, E.C.,
16th April 1901.

(Signed) MIALL, WILKINS, RANDALL, & Co.,
Chartered Accountants.

London Section.

Meeting held on Monday, June 3rd, 1901.

MR. OTTO HEHNER IN THE CHAIR.

A DANGER INCIDENTAL TO GAS-FIRING IN SMALL GASWORKS.

BY G. CECIL JONES.

I HAVE lately had occasion to enquire into the circumstances attending a fatal accident, which occurred in a gasworks with an output of about 3½ million cubic feet per annum, and I am led to the conclusion that special precautions should be taken if gas-firing is to be safely introduced into works so small as this. The retort house of the works in question is small and ill-ventilated; the height of the walls to the springing of the roof is 16 feet; the distance between the front of the stack and the opposite wall is 12 feet, and the retort mouthpieces, &c., project two feet beyond the face of the stack, reducing the clear space to 10 feet, even when no part of the floor is occupied by coal or coke. High up in the roof are ample outlets for hot air, but no provision has been made lower down for the admission of cold air, which finds its way in as best it can under two small doors, the space below which is often much reduced by the presence of small coal and coke. Until two years ago the house held three settings of one, two, and three retorts respectively, all direct-fired; the two smaller settings were then replaced by a regenerative setting of five retorts. Regeneration is not carried to the greatest possible extent, and the generator is only about 3 feet 6 inches deep; coke is supplied by hand to the generator about twice as often as

the retorts are charged. The works are in charge of a highly intelligent working foreman, who in summer works unassisted, and in winter has one other man to do the night work. Neither of these men knew that the gases made in the generator of the new setting were more dangerous than the furnace gases of the older plant.

At 7 o'clock one morning in November last, the labourer who did the night work, a strong, healthy, and sober man, was found dead a few feet from the generator charging hole. The condition of the fire showed that this had been attended to about 4.30 a.m., but the retorts had not been drawn and recharged as they should have been at 5 o'clock. The man had clearly succumbed shortly after firing the generator. His appearance was consistent with poisoning by gas, that is by carbon monoxide, and the coroner allowed his jury to return a verdict to this effect. Unfortunately, no post-mortem examination was ordered, so that it is just possible that death was due to some other cause. The absence of absolute proof of the cause of death led to the failure of the widow's action for compensation for the loss of her husband. Had she succeeded, the fact and circumstances of the accident would have been widely reported, and there would have been no necessity for this paper. But even if proof were forthcoming that the man's death was due to some other cause than gas-poisoning, I should still hold that special precautions were necessary, where it was proposed to introduce gas-firing into small works.

There are several reasons why the use of gaseous fuel is attended with more danger in small works than in large. In large works a larger and deeper generator is generally used, and a shoot conveys hot coke to this from one or more of the retorts in the setting every five or six hours; the workman has no occasion to stand near the generator mouth, which is filled by the inrushing stream of coke during the short time that it is uncovered. In small works, with a small generator, it is necessary to add fuel more frequently, and this is done by hand, the man standing over the generator mouth. Further, in large works, even if there is some slight danger of a man being overcome by generator gases, he is never alone, but has someone near him who can carry him into the open air or render him other assistance.

The employment of a one-man shift is unavoidable in very small works, but it is easy to provide suitable inlets for fresh air, and not very difficult to devise some means whereby the workman would be prevented from inhaling carbon monoxide while firing the generator. Where generators of carbon monoxide are used in other industries, it is usual to provide a hopper with valve and cover, which prevents the outrush of gas when fresh fuel is added. The gas in these generators is usually at a pressure above that of the external air; it is assumed that the gases in a gasworks generator are at a slightly diminished pressure, but I am not sure that this is a safe assumption. For example, in a house constructed like that which I have described, there is a large volume of hot air under the roof tending to rise into the outer air, but prevented from doing so freely by the lack of inlets for the admission of cold air. When the generator cover is removed, I think it quite possible that the pull of the hot air in the roof might exceed the chimney draught, which, for reasons of economy, is always checked as far as possible. The importance of good ventilation where retorts are gas-fired has been insisted on repeatedly, because, where this has been overlooked, trouble with choked ascension pipes has been experienced; to this reason must now be added, in the case of small retort houses at least, that of safety. I think it necessary to repeat, what is already well known but too often forgotten, that no system of ventilation is good which fails to provide air inlets as well as vents.

The relative advantages of direct and gaseous firing have hitherto been considered solely from the point of view of economy, but I think gas engineers will be glad to have early warning of any dangers attending the use of gaseous fuel, so that they may take the necessary precautions before a crop of accidents causes a material rise in the rate of insurance of workmen employed in their industry.

ERRATA.

This Journal, May 1901.

Page 426, col. 1, line 32 from bottom: for "no" read "not."

Page 426, col. 2, line 25 from top: for "tannin" read "tanning."

Page 426, col. 2, line 34 from top: for "was" read "were."

Page 433, col. 2, line 19 from top: for "tonnage" read "tannage."

Page 433, col. 2, line 17 from top: after "oak bark" insert "tannage."

Page 437, col. 2, line 26 from top: for "sulphate" read "sulphide."

Newcastle Section.

Meeting held on Thursday, May 16th, 1901.

MR. W. L. RENNOLDSON IN THE CHAIR.

SUGAR IN SWEDES. PART I.—ANALYTICAL METHODS.

BY S. H. COLLINS, F.I.C.

It is well known and recognised that the food value of any root crop depends on the amount of dry matter contained in it. It is, perhaps, equally well known, but scarcely equally realised, that, of this dry matter, the sugar is the most valuable part.

In Table I. some figures are given showing how much greater are the variations of the amount of sugar than of any other constituent in the same variety of swede.

TABLE I.

Monarch Swedes.

—	Small.	Large.	Small.	Large.	Small.	Large.	Small.	Large.
Water (by drying at 60° C.)	88·59	90·34	89·36	90·01	89·23	89·63	89·45	90·41
Amides, &c.	0·57	0·57	0·61	0·48	0·36	0·34	0·52	0·46
Albuminoids.	0·47	0·57	0·56	0·58	0·54	0·48	0·63	0·63
Sugars (as $C_6H_{12}O_6$).	6·14	4·21	5·29	5·02	5·67	5·43	5·11	4·34
Pectins, &c.	2·70	2·68	2·49	2·32	2·60	2·48	2·67	2·72
Woody fibre.	0·96	1·07	1·09	1·03	1·07	1·11	0·95	0·82
Ash	0·57	0·56	0·60	0·56	0·53	0·53	0·67	0·62

In Table II. some figures are given showing how great is the variation of the amount of sugar in the individual swede, of the same variety, of about the same size, and grown in the same way. Between individuals the differences are greater than between varieties.

TABLE II.

—	Max.	Min.	Mean.
1st lot of 25 XL All Swedes.....	7·8	4·5	6·27
2nd " " "	7·4	4·7	6·22
3rd " " "	8·0	4·9	6·19
1st lot of 25 Monarch Swedes.....	8·0	3·7	5·40

Hence any investigations on the composition of swedes must embrace many varieties, and a multitude of individuals. If much work is to be done on this important subject, a rapid method of analysis becomes necessary.

As the differences in the amounts of sugar are so great, an error, which would ordinarily be considered excessive, becomes here immaterial. Since the feeding values of cane sugar, dextrose and lævulose, are nearly equal, it is unnecessary for the purposes of the present investigation to

distinguish between them. The objects of the usual methods of analysis of sugar beets and sugar cane are, on the other hand, to distinguish between these varieties of sugar.

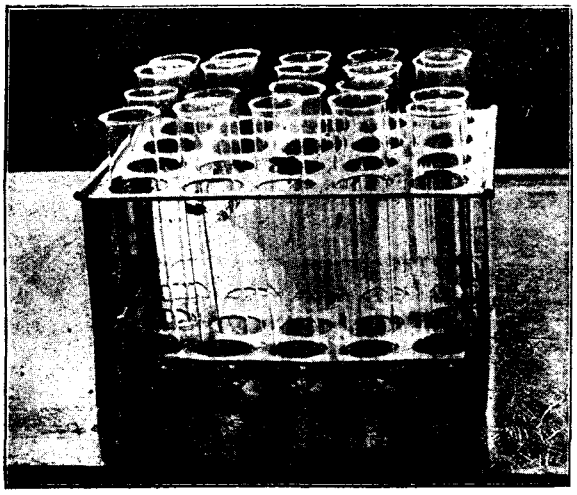
Not merely the objects of beet analysis, but the methods of polarisation, are also quite out of the question here. Since the sugars in swedes have very different rotary power, any method of analysis must depend on reduction. For rapid work gravimetric methods are of little use, and even the usual volumetric Fehling test is laborious. My attention was naturally directed to colorimetric methods, and, as I believe, no colorimetric modification of Fehling's method has ever been published, these notes are now given.

The general principles of the method to be described consist in causing the sugar to reduce Fehling's solution, and then measuring colorimetrically the excess of copper which remains unreduced.

In order to apply this method to turnips, it is necessary to remove other reducing substances, and to hydrolyse any cane sugar which may be present.

About 10 grms of swedes, 100 c.c. of water, and 1 c.c. of a concentrated solution of lead acetate are ground up together in a mortar and allowed to stand some hours to diffuse. Of the filtrate 50 c.c. are inverted by 0·5 c.c. of strong sulphuric acid. A simple apparatus for this purpose is a metal test-tube stand (Fig. A) fitted with

Fig. A.



tubes graduated at 50 c.c., and placed in a steamer such as is used for bacteriological work. Since the dilution is so slight, considerable errors in measurement would have no practical effect on the result. The whole of the lead is precipitated by the sulphuric acid. About 20 minutes in the steamer is, I find, ample for the complete inversion of the cane sugar. The tubes and their stand may be cooled rapidly by immersion in cold water, or left to cool of themselves.

An aliquot portion (2—5 c.c.) is removed by a pipette, care being taken not to touch any of the precipitated lead sulphate, and added to the Fehling solution. For this purpose I find the most convenient apparatus is a metal stand similar to the one used for inversion, but smaller, and fitted with test tubes, all carefully selected to be of the same diameter, say 16 mm. Each test-tube contains 10 c.c. of Fehling's solution of half the usual strength. For the method I am now describing, 250 c.c. of copper sulphate solution (69·3 grms. of crystallised copper sulphate per litre), and 250 c.c. of alkaline tartrate solution (100 grms. of caustic soda and 350 grms. of Rochelle salt per litre), and 500 c.c. of water form the standard solution, of which 10 c.c. are placed in each test-tube.

It will be noticed that the sugar solution used is acid, and will neutralise varying amounts of the soda in the Fehling's solution. That this has little or no effect will be seen by studying the results quoted later. On placing the test-tubes containing both copper and sugar in the steamer for 30 minutes, the precipitated cuprous oxide settles completely at the bottom. The stand and its tubes are allowed to cool for a few minutes, and the colour of the supernatant liquid judged by comparison with standard tints.

The standard tints contain 0·125, 0·250, 0·375, 0·500, 0·625, 0·750, 0·875, and 1·00 c.c. of the copper sulphate solution respectively, To this is added in each case 2·5 c.c. of the alkaline tartrate solution, and the whole made up to 14 c.c. with water.

The reason for this method of making the standard tints, is that the colour produced by the copper is much modified by varying amounts of tartaric acid. In the actual analysis the amount of tartaric acid is constant; it must also be constant in the standard tints. It is obviously convenient to dilute the copper sulphate solution to one-tenth the strength before making the standard tints. These tints are respectively 5, 10, 15, 20, 25, 30, 35, and 40 per cent. of the total amount of Fehling's solution used.

For most purposes 4 c.c. of sugar solution, will be a convenient amount, and for this reason 14 c.c. has been made the standard. If 3 c.c. be used, 1·14 must be added to the excess of copper as measured by the standards, and so on. It is evident that this method differs from that usually employed, and it becomes necessary to determine the ratio between the copper and the sugar. The results are set out in Tables III. and IV. The cane sugar was inverted under precisely the same conditions as the swedes.

TABLE III.

Anhydrous Dextrose.	Solution used.	Colour.	Corrected Colour.	Copper reduced.	Factor.
Grms.	C.c.			[Total=1]	
0·0215	2	9	7	0·93	0·0231
0·0193	2	17	15	0·85	0·0227
0·0171	2	27	23	0·77	0·0222
0·0149	2	38	33	0·67	0·0223
0·0223	3	5	5	0·95	0·0235
0·0203	3	10	9	0·91	0·0223
0·0183	3	23	21	0·79	0·0232
0·0163	3	30	28	0·72	0·0226
0·0143	3	40	37	0·63	0·0227
0·0217	4	7	7	0·93	0·0233
0·0192	4	17	17	0·83	0·0231
0·0177	4	24	24	0·76	0·0233
0·0162	4	30	30	0·70	0·0231
0·0147	4	37	37	0·63	0·0233
0·0220	5	3	3	0·97	0·0227
0·0203	5	11	12	0·88	0·0229
0·0184	5	16	18	0·82	0·0224
0·0166	5	25	27	0·73	0·0227
0·0148	5	32	34	0·66	0·0224
				Average	0·0228

TABLE IV.

Invert Sugar.	Solution.	Colour.	Corrected Colour.	Copper reduced.	Factor.
Grms.	C.c.			[Total=1]	
0·0200	2	8	7	0·93	0·0215
0·0182	2	17	15	0·85	0·0214
0·0173	2	25	21	0·79	0·0219
0·0164	2	30	26	0·74	0·0221
0·0146	2	40	34	0·66	0·0221
0·0207	3	6	6	0·94	0·0221
0·0193	3	12	11	0·89	0·0217
0·0181	3	18	17	0·83	0·0218
0·0207	4	5	5	0·95	0·0218
0·0189	4	11	11	0·89	0·0212
0·0172	4	18	18	0·82	0·0210
0·0193	5	8	9	0·91	0·0212
0·0172	5	17	18	0·82	0·0210
0·0151	5	30	32	0·68	0·0222
0·0129	5	36	39	0·61	0·0212
		Grammes of invert sugar to reduce all the copper added			= 0·0216

The conclusion to be drawn from these figures is that the reducing power is equal at all parts of the scale. No single

determination differs from the mean by more than 3 per cent.

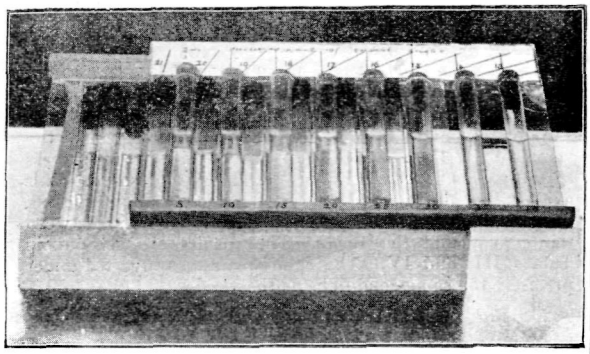
It will be seen that the sugar has a much greater reducing power than that usually assigned to it. This is due to the concentration and long period of heating.

For the purposes of the analysis of swedes, I propose to use the figures from invert sugar only.

If this method be used for fresh swedes the colours obtained match the standards well. If dried swedes be used for analysis, a yellow colour is imparted to the solutions, and various shades of greenish blue are obtained, to judge the depth of blue in which is difficult.

To get over this difficulty, I use the following simple form of colorimeter (Fig. B). On one pane of glass a row of

Fig. B.



test tubes containing alternately plain water and a graduated series of yellow solutions is placed; on another pane a row of test-tubes containing the standard blue solutions is placed over the yellow tubes, blank spaces being left over the plain tubes to take the tubes used for the actual analysis. The pane holding the blue tints slides over the pane holding the yellow tints, producing any desired combination, and the whole is placed in a wooden frame at an angle of 45°, with a white base board. A special scale enables one to read directly the milligrammes of sugar. A suitable material for the yellow solutions is Methyl Orange.

I now proceed to discuss the question of sampling. A swede is not homogeneous, the top half being richer than the bottom half. Owing to the ease with which they can be taken at the farm and forwarded, I have used borings taken either with a cork borer or an auger to form the sample. Table V. shows the percentage of sugar (as

TABLE V.

	Yellow Turnip.		Swede.	
	Large.	Small.	Large.	Small.
Short horizontal bore	4·73	4·02	6·32	6·76
Long " "	4·41	4·05	6·06	6·77
Vertical bore	3·90	4·50	6·58	6·71

"invert") in borings made in different directions, but passing through the centre in all cases. It will be seen that whilst there is some difference between the figures, for rough purposes the single boring is a true sample. There is no constant difference between borings in one direction or another, hence a number of borings would be a true sample of a number of swedes. In order to find out how many swedes it is necessary to take in order to obtain a true sample of a variety, the figures given in Table II. may be employed. If a swede of maximum percentage of sugar were replaced by one of minimum percentage, or *vice versâ* in the Monarch swedes in Table II., then the average of—

6 swedes would be altered from 5·40 to 4·70 or 6·10; of
12 " " " 5·04 " 5·76; of
25 " " " 5·23 " 5·57.

Even if 25 swedes be taken, the errors of sampling are greater than the errors of the method of analysis given above.

In conclusion it is clear that the greatest errors occur in taking a sample of a variety of swedes, the errors of sampling a single root being smaller, and the errors of the colorimetric method of analysis smaller still.

This colorimetric method might, with a few modifications, be utilised for the estimation of starch in potatoes, and other foods.

On the subject of the composition of swedes, data are accumulating, and I hope before long to be in a position to make a further communication on the matter.

DETERMINATION OF CALCIUM IN HIGH-GRADE FERRO-SILICON.

BY G. WATSON GRAY, F.I.C.

HIGH-GRADE ferro-alloys of late years, especially those produced in the electric furnace, have presented many interesting points to the metallurgical chemist, and at the same time some troublesome ones to the analyst.

Having recently come across a ferro-silicon containing calcium, and not having noticed this element recorded before in a ferro-alloy, I have ventured to read this paper so that its presence may be noted by users, and its good or ill effect on the steel observed.

I have noticed the presence of magnesium and aluminium in ferro-chromes for some time past, but calcium has been absent. The presence of magnesium and aluminium is not to be wondered at, seeing that chrome ores contain these elements in large amounts, and the reduction of the chrome ore being brought about in the electric furnace. The same, to some extent, may be expected with ferro-silicon as no doubt calcium compounds constitute a large proportion of the flux.

I append an analysis of ferro-silicon containing calcium:—

	Per Cent.
Silicon	44·05
Iron	45·28
Chromium	1·62
Manganese	3·51
Aluminium	0·47
Calcium	3·29
Magnesium	0·44
Carbon	0·52
Sulphur	0·045
Phosphorus	0·013
Copper	Trace.
Nickel	Trace.
Titanium	Nil.
Tungsten	0·42
	99·658

Regarding the analysis of ferro-silicon, the lower grades may conveniently be brought into solution by acids, and in some cases by bromine, but the high grades resist such treatment. They are, however, readily broken down by Stead's tribasic mixture, and as I use this reagent for ferro-chrome, I likewise used it with success for ferro-silicon, until the present case, where calcium and magnesium were to be looked for. (I may here mention that I have given up the use of sodium peroxide for such work, owing to the varying amounts of impurities it contains, some of which are very objectionable.)

In looking round for some means of decomposing the ferro-silicon without the introduction of calcium and magnesium, I made use of the reaction:—

$$K_2CO_3 + Si = K_2SiO_3 + C.$$

On trying the reaction I found it to take place, and not only with potassium carbonate but with ordinary fusing mixture. The mode of procedure is then similar to any ordinary analysis, but there are a few points of detail which require to be attended to.

One gramme of the ferro-silicon, somewhat finely ground, is mixed with 4 grms. of fusing mixture in a platinum crucible, covered, and heated over the Bunsen flame. Very

soon a strong reaction takes place. When this first part is complete, which is readily seen by removing the cover, the crucible is somewhat more strongly heated over the Bunsen flame until fusion is complete. The mass is then treated with water, and removed as completely as possible to a No. VII. porcelain dish, the crucible being cleaned by hydrochloric acid, which is added to the liquid in the dish, the dish being covered with a clock glass. Hydrochloric acid, however, fails to remove all the iron from the platinum crucible, so that it is necessary to dry the crucible in the flame and place 1 grm. of bisulphate of potash in it, and heat at a low red heat until the iron is removed from the sides of the crucible. This mass is then dissolved out of the crucible, and might be added to the liquid in the dish, but I prefer to precipitate the iron, &c. by ammonia and add the precipitate and filtrate to their respective places. The contents of the dish are brought to a boil. The iron is in the spongy condition, dissolving slowly. 10 c.c. of nitric acid are now gently added, and after the bulk of the reaction has taken place, the cover is washed and removed, and the whole evaporated to dryness, to render the silica insoluble. The dried and heated mass is then treated with 15 c.c. of hydrochloric acid, to redissolve the iron, &c. The silica is collected on an ashless paper, dried, the paper burnt off, and the residue finished in the muffle, weighed, evaporated with hydrofluoric acid and again weighed, and the loss in weight calculated into silicon.

The residue in the crucible may be removed by fusing with carbonates of soda and potash, and dissolving out with hydrochloric acid, or, better, by again heating with 1 grm. of bisulphate of potash and dissolving in water. If the sample contains chromium the bulk of it is likely to be found here, in which case the basic sulphate of chromium will require to be boiled and concentrated with hydrochloric acid to bring it into solution. If tungsten be present it will be found at this stage, and will require to be filtered off, washed with acid and water, burnt off, and weighed.

The ammonia precipitates, three in number, are all burnt off, transferred to a beaker, dissolved in hydrochloric acid, evaporated to dryness, and any silica or tungstic acid, which has escaped separation previously, recovered.

This may appear unnecessary, but silica, when present in large amounts in such analyses as those of silicious iron ores or clays, may go to augment the amount of alumina. The solution is then saturated with sulphuretted hydrogen to remove copper and any trace of platinum, as the platinum interferes with the estimation of the iron if performed by reducing with stannous chloride and titrating with bichromate. The separation of the iron, maganese, aluminium, chromium, calcium, and magnesium are proceeded with by the usual methods.

Sulphur and phosphorus may be obtained from the one portion. I use the same process as for ferro-chrome. Take 2 grms. of the sample and 15 grms. of tribasic mixture and heat in the muffle for 1½ hours. The oxidised mass is then transferred, in the case of a ferro-silicon, to an evaporating basin, treated with excess of hydrochloric acid, evaporated to dryness, and the silica removed. The iron and phosphoric acid are precipitated by ammonia and removed. The filtrate is rendered very slightly acid, and the sulphates precipitated with barium chloride. The ammonium hydrate precipitate is washed off the paper into the original beaker, and sufficient bisulphite of ammonia or soda added to reduce the iron. The filter paper is washed with sufficient hydrochloric acid to dissolve the iron, and the washings added to the ammonia precipitate and bisulphite. The remaining parts of the estimation are too well known to require further notice.

If only silicon, sulphur, and phosphorus are required, it is possible to get all these from the one portion.

The magnesia is prepared from the natural carbonate (magnesite) by first calcining and then pounding. This gives a magnesia very free from sulphur and phosphorus, but it is necessary to determine the several impurities in the mixture prepared.

The determination of the total carbon is also made by the same method as that used for ferro-chrome, that is, by grinding together in an agate mortar 0·5 grm. of the very finely powdered alloy with 5 grms. of ignited oxide of copper (this requires grinding, not merely mixing), and then 7 grms. of recently ignited fused chromate of lead mixed with it. The 12·5 grms. are then transferred to a suitable sized Meissen porcelain boat and heated for 2½ hours in Fletcher's No. 2 organic furnace in a current of oxygen, the carbon dioxide produced being collected in potash bulbs provided with calcium chloride tube, as is usual.

It is always desirable to do a blank test with each fresh stock of oxide of copper and chromate of lead.

New York Section.

Meeting held on Friday, May 24th, 1901.

MR. CLIFFORD RICHARDSON IN THE CHAIR.

ADDITIONAL NOTES ON CEMENT TESTING.

BY OTTO H. KLEIN AND S. F. PECKHAM.

WE had the pleasure of bringing before this Society at the meeting held 25th May 1900, the results of a research that had been in progress for more than a year, in the Physical and Chemical Laboratories of the Commissioners of Accounts, of the City of New York, upon the relations between physical tests and chemical analyses of cement.

Before proceeding to discuss the main proposition of this paper, viz., the merits of the method of analysis which was described in our former paper, we wish to call attention, first, to the "Progress Report of the Special Committee on the proper manipulation of tests of cement," made to the American Society of Civil Engineers, and published in the Proceedings of that Society, Vol. XXVI., No. 4, April 1900.

This report consists of the various replies to a long list of questions, offered by many persons and submitted by the Chairman of the Committee, Professor G. F. Swain, without comment.

In reply to question 5, "What elements or compounds should be determined?" Professor Henry Carmichael of Boston (who is an acknowledged authority as a cement expert) says, "Hydraulic cement consists of a double silicate of lime and alumina (including iron oxide) which is readily soluble in dilute hydrochloric acid, leaving little or no insoluble residue. In addition to the soluble silica and the oxides of calcium, aluminium and iron, good cement contains traces of the oxides of magnesium, sodium, and potassium, together with traces of carbonates, sulphates, chlorides, and combined water, and finally minute amounts of insoluble sand or cinder."

In reply to question 6, "What do you consider the best methods of determining these compounds with sufficient accuracy"? Professor Carmichael continues, "The sample is ground fine in an agate mortar. One gram is carefully weighed out in a shallow porcelain dish and well covered with a 3 per cent. solution of hydrochloric acid. After several hours the cement should completely dissolve in this acid with the exception of a small amount of sand, mostly black cinder from the fuel employed in making the cement. The residue, if any, is filtered off and determined. The clear solution is evaporated to dryness on a water bath in a flat dish. Hydrochloric acid is poured over the dry residue, and the acid is then evaporated. Add a few drops of same acid, again drive off acid. Moisten residue again with same acid and boil up with pure water. The silica is rendered insoluble by the above operation and can be filtered off and weighed. The silica which thus dissolves in the dilute acid, and is in turn rendered insoluble, is the silica which is available in the setting of the cement. The filtrate from silica is boiled with a few drops of nitric acid, and pure ammonia is then added, which precipitates the oxides of iron and aluminium. With the ammonia is added also ammonium chloride in sufficient quantity to retain the lime in solution. After boiling for some time, the oxides of iron and aluminium are filtered off, and after drying are ignited

and weighed." Here follow directions for separating the iron and aluminium. "To the filtrate from iron and aluminium oxides is added a slight excess of ammonium oxalate whereby the lime is precipitated as oxalate which is filtered off, ignited at a dull red heat in a platinum crucible and weighed as carbonate."

His scheme offers further details for the determination of the ingredients which he says are found in good cement in traces; for the determination of water and carbon dioxide by ignition; and for the determination of free lime by titration.

This scheme closely resembles our own, while differing from it in several important respects. It was worked out without any knowledge whatever on the part of either party concerning the other.

Following this scheme of Professor Carmichael is another scheme by R. L. Humphrey, of Philadelphia, who writes as follows: "One half grm. of the finely pulverised sample dried at 100° C., is thoroughly mixed with four or five times its weight of sodium carbonate, and fused in a platinum crucible until CO_2 no longer escapes; the crucible and its contents is placed in a beaker, and 20 or 30 times its quantity of water, and about 10 c.c. of dilute HCl is added; when complete solution is effected, it is transferred to a casserole and placed on a water bath, and evaporated to dryness several times. The mass is taken up with dilute HCl and water, heated for a short time and filtered, washing the residue on the filter thoroughly with hot water. The filter is dried, ignited, and weighed. This weight (less ash) gives the amount of SiO_2."

"The filtrate is brought to boiling, and ammonium hydrate added in slight excess; the boiling is continued until the odour of ammonia is no longer perceptible. Filter and wash. Re-dissolve in hot dilute HCl, again precipitate with ammonia and filter through the previous filter and wash with boiling water. The precipitate dried, ignited, and weighed (less ash) gives the amount of Al_2O_3 and Fe_2O_3."

Then follows a method of separating iron from alumina: "The filtrate from the iron and alumina is heated to boiling, and boiling ammonium oxalate is added until a precipitate is no longer formed. After boiling for a few minutes, it is set aside for a short time; when the precipitate has settled perfectly, decant the clear liquid through a filter, wash by decantation, dissolve the precipitate in hot dilute HCl, using as small a quantity as possible to effect a complete solution, heat to boiling and add ammonia, heat on a water bath for a few minutes; when the solution clears, filter through the previous filter, wash thoroughly with hot water. Dry the precipitate, ignite to constant weight, and weigh as CaO, or determine the lime volumetrically by titration with potassium permanganate." He then determines the ingredients occurring in small proportion. He determines SO_3 in a separate portion after removing the silica.

We wish to call attention secondly, to correspondence which we have lately held with a prominent manufacturer of cement, in which he made the following criticisms on our paper.

"(*a*) Experiments made by us show that the solubility of commercial Portland cements in dilute acid depends greatly upon the fineness of grinding. (*b*) We have found no cements which, if ground to extreme fineness in an agate mortar, show more than a fraction of one per cent. of insoluble matter. (c) It seems to us, therefore, that your separation of the components of cement into active and inactive constituents is not well grounded. (*d*) We think it probable that the active index would be somewhat reduced if the cement is dissolved as completely as possible. (*e*) Since even the monosilicate of lime, Wollastonite, is readily decomposed by acid, it is evident that the residue should contain practically no lime, and would consist of a minute amount of uncombined clay. (*f*) It is impossible that this should reach more than a fraction of 1 per cent. in a good cement. (*g*) Further, that the use of sufficiently dilute acid and fine grinding will give a clear solution without any separation of gelatinous silica. (*h*) In our opinion, no conclusion can be drawn from the amount of lime soluble in water. (i) This would also depend greatly on the degree of pulverisation. (ii) It is generally held, as you know, that all cements are decomposed by water into calcium hydrate and a hydrated mono-silicate. (iii) If this is true, the action of water would be progressive and prolonged action of sufficient water would dissolve out all the lime. (iv) In fact, Le Chatelier found this to be the case."

To which we replied, the first sentence of your letter furnished a key to the whole matter. You say "that experiments made by us show that the solubility of commercial Portland cements in acid depends greatly upon the fineness of grinding." Believing that this fact, as stated by you, has been proved beyond any question, we insist that every sample of cement should be analysed in exactly the condition in which it is brought to the laboratory; that is, that the specimen shall be neither dried nor pulverised, nor in any manner treated in such a way as to either lessen or increase the differences that exist between the samples as they are brought upon the works or are subjected to physical tests.

We have not yet found a weighable amount of lime in any of the residues from dilute acid that we have examined.

We think that if you read our paper carefully you will see that no conclusions are drawn from the amount of lime soluble in water. It is, however, an observed fact, that all good cements contain about 5 per cent. of such soluble lime.

If you would advocate the uniform grinding of samples of cement to an impalpable powder, in an agate mortar, in order that they may be more completly dissolved and brought into solution, we insist that we do not agree with you. We believe that cement of proper fineness for use is soluble in 10 per cent. hydrochloric acid without gelatinisation, and that any matter not so soluble, contained in commercial cement, is not cement at all, and is, and should be classed as "inert matter."

To which he replied, (*j*) "your separation of the constituents of cement into 'active and inactive constituents' by the action of 10 per cent. hydrochloric acid on the commercial cements appears to me to be without foundation. (*k*) The varying amounts of insoluble matter obtained by you on treating the same cement with acid of varying strengths and in various ways appear to show that the amount of insoluble matter depends upon the method employed, rather than upon the chemical character of the cement analysed. (*l*) I have found that most commercial Portland cements, if ground to great fineness, give scarcely any insoluble residue on treatment with sufficient quantity of 5 per cent. acid. (*m*) The high percentage of insoluble matter obtained by you simply results from the comparatively coarse grinding of the cement. (*n*) You certainly will not contend that the composition of the coarser particles is materially different from that of the fine ones, or that the chemical character of the cement is changed by grinding the coarse particles to uniform fineness with the rest. (*o*) Your choice of 10 per cent. acid, and method of mixing appear to me to be wholly arbitrary, and the conclusions drawn from the amount of insoluble matter obtained to be quite unjustified. (*p*) Since coarsely ground cement gives a considerable residue when treated with dilute acid, while finely ground cement gives practically none, and since this residue consists chiefly of silica, and contains, as stated by you, practically no lime, it appears to me evident that this insoluble matter results chiefly from local separation of silica contained in the coarse particles. (*q*) The lime and other constituents contained in these particles are, however, dissolved, and are included by you in the group of active constituents. (*r*) The injustice of this is apparent. (*s*) If the silica of the coarse particles is inactive, the lime must be so also. (*t*) It is undoubtedly correct to submit commercial samples of cement to physical tests as they are received, without grinding. (*u*) To submit these samples to chemical analysis, however, without making them homogeneous by grinding, is certain to lead to erroneous conclusions. (*v*) In burning, however, a disturbing factor enters, and this is the ash of the coal dust used as fuel. (*w*) This ash adds at least 2 per cent. to the silica, alumina, and iron oxide of the product. (*x*) It is, however, brought into

combination with the lime of the charge sufficiently to become wholly soluble in acid, but not uniformly enough to allow the lime of the raw material to be raised to a corresponding extent. (*y*) I believe fully that the best Portland cements are thoroughly homogeneous in character, and that the excess of silica, alumina, and iron over that called for by the formula is due to the ash of the fuel and to the general practice of carrying the lime a little below the maximum in order to offset possible fluctuations in the mixture."

We asked our correspondent to send us a sample of the fuel ash.

He replied, "(*z*) it will be impossible to send you a sample of the coal ash to which your refer, as this melts in with the clinker with which it comes in contact in the rotary kilns. (*za*) The amount of fuel used is about 150 lb. to the barrel of cement. (*zb*) The ash of this fuel is about 8 per cent., and if all absorbed by the clinker would add about 3 per cent. of silica, iron, and alumina to the latter."

We wish to discuss in association these methods of analysis and this correspondence, all of which comes from unquestioned authority and is strictly professional, for they all represent a purpose that is in fundamental opposition to our own purpose.

The purpose of the research described in our former paper was to ascertain whether or no any correspondence could be established between the results of a chemical analysis and the physical tests of cement. It was not to defend or defame any brand of cements, to contrast any one cement with another, or to contrast American cements with foreign cements; or one class of foreign cements with another class of the same, although some very unexpected results obtained from the research led incidentally to such comparisons. This object was kept clearly in view from first to last, and while the different brands of cement examined represented in nearly every case nothing but a name, still, the different samples were designated wholly by numbers that were purposely made different in the two laboratories. The cements first analysed were brought to the office from various works in progress in the city. They gave results that were extremely unsatisfactory apparently for various reasons that we do not care to take the time and space to discuss here. Keeping the object of our research in view we solicited samples of freshly ground cement from a number of manufacturers and dealers. We still found that, when these fresh, and in most instances high class, cements were analysed by the methods described in the books, no correspondence could be observed between the physical tests and chemical analyses.

Believing that nature could not contradict herself, we were convinced that there must be some defect in the question put to nature in the chemical analyses; in other words, that the chemical analysis was not properly conducted. This led us to conclude that the purpose of an analysis was not satisfied when an inadequate method, in some respects, of ultimate analysis was followed.

For no cement on the market consists of chemically pure hydraulic cement. Assuming the correctness of the researches that led up to and include those of the Messrs. Newberry, we have a right to further assume that hydraulic cement consists of tri-calcic silicate and ferro-aluminiate in the proportions indicated by the equation—

$$\frac{\text{CaO}}{(2{\cdot}8 \times \text{silica}) + (1{\cdot}1 \times \text{alumina and ferric oxide})} = 1$$

A commercial cement therefore consists of the above-named compound plus, not a trace, but an unavoidable percentage of the ashes of the coal employed as fuel, also of over-burned clinker, also of under-burned clinker, of uncombined clay, of magnesia, of sulphates, of alkalis, and of a small percentage of water and CO_2.

We assume that these impurities are unavoidable, because from the nature of the case no cement can be manufactured without them, and no cement that we have examined has been entirely free from them, and we believe that we have examined some of the best cements now made in the world.

With these is found in some commercial cements a variable amount of carbon, ranging in those we have examined from zero to nearly 2 per cent.; this carbon is not a proper constituent of a cement; no good cement contained more than a trace of it; it is, and must be from the nature of the case, a source of weakness, especially when it is in the form of an oily soot from imperfect combustion of oil used as fuel. These methods of analysis do not offer any opportunity for the determination of this impurity. There is also found plaster of Paris, introduced purposely to influence the setting of the cement; when moderate in amount this is not an injurious ingredient. Also a percentage of quartz sand; Stillman's and Humphrey's methods of analysis offer no opportunity for the determination of this ingredient. Also a percentage of unburned rock or clinker which results in the presence of carbonate of lime and which is inert, having no cementing properties; Stillman's method furnishes no opportunity for the determination of this ingredient.

Cements made from marl are liable to contain glauconite, which is a mineral containing a comparatively large amount of alkalis. Cements made from slag are also liable to contain alkalis. We have no reason to suppose, however, that alkalis exist in any injurious amount in any of the samples that we have examined, and they were therefore not determined.

The question, therefore, to be put to nature is, how much hydraulic cement does this mixture contain as it is brought to the laboratory? And what else does it contain that it should not?

In order to answer this question, it may first be determined how much of the sample of cement is volatile at a bright red heat. We have found by repeated examples that an ordinary Portland cement of good quality will give about 4 per cent. or less, of matter volatile at a red heat. A number of tests showed that this amount was not materially increased by the use of a blast lamp. In a few instances the increase amounted to a few tenths per cent., but in a majority of instances it was not appreciable. This loss at a red heat was found in a majority of instances to very nearly correspond with the amount of carbon dioxide determined directly, but in those instances where this loss was large, amounting to more than 10 per cent., the difference between these two factors increased as the loss increased, showing that in those cases where this loss is large, an increasing proportion of such loss is water. If more satisfactory, the percentage of carbon dioxide can be directly determined, but even when it is so determined, the matter volatile at a red heat should always be ascertained.

The problem of analysis is simply an ordinary problem in mineral analysis. The physical tests have been made upon the sample as brought to the laboratory, and in such a manner as to exhibit the greatest possible difference between this sample and all other samples. The samples were neither dried nor pulverised, because either drying or pulverising, or both, would make another and different sample to be tested from the one submitted. Our correspondent admits this, and if he did not, everybody else does; yet, referring to his sentence marked (*u*) he claims that to submit these samples to chemical analysis, without bringing them into homogeneous condition by grinding, is certain to lead to erroneous conclusions. Both schemes of analysis that we have quoted require that the sample shall be ground, and one of them that it shall be dried. We have found the loss that good cements sustain when dried at 100° C. is not of practical importance; with bad cements it is otherwise. We also believe that right here is the keynote to the controversy. We believe that in discussing any subject the disputants must be agreed as to their subject, or, they may argue for ever and both be correct in reaching diametrically opposite conclusions. In this case, the two chemists and our correspondent are agreed that the samples should be pulverised to an impalpable powder, and we on the contrary believe that as soon as a sample is pulverised it is converted into a new sample. This our correspondent practically admits (*see* sentences *a, b, d, f, g, l, m, p, q, s, y*). We claim that the sample should be analysed as received and thus made to

exhibit the greatest possible differences, as is the case in the physical tests. If the samples are pulverised and thus brought uniformly into solution, certain differences are lost, and it is not strange that the physical tests and chemical analyses do not correspond. The same result follows when the samples are fused with sodium carbonate, and when the sample is dissolved in concentrated HCl. In both instances the distinction between the silica that is combined into tri-calcic-ferro-alumino-silicate, or cement, and the silica that exists as sand or fuel ash or as unburned clay, is lost.

In our former paper we did not base any distinction of "active" and "inactive" constituents on the action of 10 per cent. HCl (*o, j*). We base those distinctions on the researches of the Messrs. Newberry, and we insist that, if those researches are conclusive, and we believe that they are, our conclusions follow from the nature of the case. There was nothing arbitrary in using 10 per cent. HCl. It is nothing but the ordinary dilute HCl found on every laboratory shelf. There is nothing arbitrary in using a sieve to scatter the cement upon the acid and prevent the development of sufficient heat to render a part of the silica insoluble.

Moreover, our correspondent admits that a theoretically perfect cement, will contain in spite of anything, 3 per cent. of fuel ash and 1 per cent. or less of unburned clay (*v, w, z, za, zb*). These materials are not cement. Why should they, or any part of them, be brought into solution with the cement if it could be avoided? Let us suppose that that an unskilful or dishonest manufacturer uses more coal than is necessary, or poor coal, and thus increases his coal ash to 6 per cent., and his unburned clay to 4 per cent., why should the 6 per cent. be dissolved by fine grinding, or the 10 per cent. be dissolved by fusing with sodium carbonate? In other words, why should a method of analysis be pursued that destroys all differences, while the physical tests are conducted in such a manner as to exhibit all differences?

That this contention is correct is proved by the results of our analytical work. No. 149 (Chemical Laboratory) was a cement that gave the highest figures in physical tests (Physical Laboratory, No. 88). They were, one day neat, 429 lb.; seven days neat, 784 lb.; seven days mortar, 209 lb. When analysed this cement gave:—

	Per Cent.	
Insoluble in 10 per cent. HCl	3·79	
Soluble silica	19·43	
Alumina and iron oxide	8·34	
Lime	63·44	91·21 %
Magnesia	Trace	
Sulphates (in terms of SO_3)	1·89	
Volatile at a red heat	1·39	
Undetermined	1·72	
Total	100·00	

$$\text{Active index} = \frac{63{\cdot}44}{2{\cdot}8 \times 19{\cdot}43 + 1{\cdot}1 \times 8{\cdot}34} = 1{\cdot}005$$

That is to say, we have here a commercial cement that consists of 91·21 per cent. of cement, 3 per cent. of fuel ash, 0·79 per cent. of unburned clay, 1·89 per cent. of sulphuric anhydride, 1·39 per cent. of carbon dioxide and water, and 1·72 per cent. of ingredients not determined. This is practically a perfect cement, and the other six best American cements so nearly approximated this cement in composition that with it they averaged as follows: one day neat, 259 lb.; seven days neat, 683 lb.; seven days mortar, 257 lb.

	Per Cent.	
Insoluble in 10 per cent. HCl	4·38	
Soluble silica	18·45	
Alumina and iron oxide	9·45	
Lime	61·89	89·80 %
Magnesia	1·78	
Sulphates (in terms of SO_3)	1·87	
Volatile at a red heat	1·79	
Undetermined	0·38	
Total	100·00	

$$\text{Active index} = \frac{61{\cdot}89}{2{\cdot}8 \times 18{\cdot}45 + 1{\cdot}1 \times 9{\cdot}46} = 0{\cdot}997$$

That is to say, these seven American cements are 89·8 per cent. pure, with 3 per cent. of fuel ash, 1·38 per cent. of unburned clay, 1·78 per cent. of magnesia, 1·87 per cent. of SO_3 as sulphates, 1·79 of carbon dioxide and water, and 0·38 per cent. undetermined. If a method of analysis can be used that will separate this unburned clay and fuel ash from the cement, why does it lead to erroneous conclusions? Why should a method be used that makes it impossible to distinguish between the silica and alumina in the unburned clay and fuel ash, and the remainder of those ingredients that are properly combined to form cement.

In the answer to these questions lies the whole matter in controversy.

In order to test these conclusions further, we wrote to our correspondent, and asked him to send us a sample of the coal he was using, the mixture that was burned, and the clinker that resulted from the burning. With great kindness he immediately complied with our request.

We labelled the samples: Coal, 449 B; Mixture, 449 C; Clinker, 449 D.

We found the clinker to consist of small nearly spherical masses, about the size of peas, that had been vitrified, and that were hard under the pestle, and also larger pieces that were more or less spongy and softer under the pestle.

449 B yielded 11·95 and 11·76 per cent. of ash, an average of 11·85 per cent., which, at 400 lb. to the barrel of cement, equalled 4·44 lb. to 100 lb. or 4·44 per cent. of ash instead of 3 per cent. Of this ash, 22·92 per cent. was soluble in 10 per cent. hydrochloric acid. The ash consisted of:—

	Per Cent.
Silica	42·94
Alumina and ferric oxides	41·41
Lime (CaO)	9·52
Sulphates (in terms of SO_3)	3·58
Undetermined	2·55
	100·00

The sulphates (see SO_3 above) were wholly soluble in dilute hydrochloric acid.

449 C contained 19·79 per cent. insoluble in dilute hydrochloric acid. This insoluble portion contained, of silica, 68·774 per cent., and alumina and iron oxide and lime, 31·226 per cent. These figures are equivalent to 13·62 per cent. and 6·17 per cent. of the amount taken. The soluble portion contained soluble silica 0·17 per cent., alumina and iron oxide 0·60 per cent., lime (CaO), 41·16 per cent. These results are equivalent in round numbers to: clay, 20 per cent.; marl, 80 per cent.

449 D, when finely pulverised, gave:—

	Per Cent.		Per Cent.
Insoluble in 10 per cent.	4·62	and	3·36
Soluble silica	18·24	„	19·84
Alumina and ferric oxide	8·74	„	8·65
Lime (CaO)	65·64	„	65·38
Magnesia (MgO)	..		..
Sulphates (in terms of SO_3)	Trace		Trace
	97·24	„	97·23
Undetermined	2·76	„	2·77
	100·00	„	100·00

These analyses were made on two separate portions pulverised separately. The insoluble portion did not contain a trace of lime.

449 D.—5·0334 grms. of the small unbroken clinker were placed in a flask with 250 c.c. of 10 per cent. HCl on March 15th, at 3 p.m. On March 16th, at 9 a.m., the action of the acid was very marked; each mass was covered with a shell of white silica. On March 18th, at 9 a.m., nothing remained of most of the spheroids but a white shell of silica. A few of them were but little acted on. On the morning of the 20th the residue of silica was wholly white, and the solution had the colour of proto-chloride of

iron. The contents of the flask were then analysed, and gave :—

	Per Cent.
Insoluble silica	20·95
Soluble silica	2·09
Alumina and ferric oxide	8·43
Lime	66·69
	98·16

449 D.—5·0908 grms. of the large porous pieces of clinker were placed in a flask with 250 c.c. of 10 per cent. HCl, on March 20th. The action of the acid was very unequal. Some of the pieces were completely decomposed in 48 hours; others, apparently containing more iron, resisted its action for several days. On March 26th, one piece was still brown, and was broken down with difficulty.

The contents of the flask were then analysed, and gave :—

	Per Cent.
Insoluble silica	22·01
Soluble silica with trace of iron	1·56
Alumina and ferric oxide	8·97
Lime	66·72
	99·26

These results show that our correspondent was mistaken in assuming that (*y*) "the best Portland cements are thoroughly homogeneous in character," as his own clinker is not homogeneous. Moreover, these analyses prove beyond question that the results of the action of acid out of the same bottle and acting on material from the same bottle, depends upon the size of the masses of cement. The two samples of clinker above set forth were purposely ground of different degrees of fineness.

We then asked our correspondent to send us samples of coal, mixture, clinker, and ground cement that would be as nearly as possible representative of the same batch of mixture. These were received by us, and numbered: Coal, 449 E; Clinker, 449 D; Mixture, 449 C; Cement, 449 B.

449 E yielded 10·63 per cent. of ash, which is equal to 3·99 per cent. of ash in the clinker. When analysed, this ash yielded :—

	Per Cent.
Silica	43·30
Alumina and ferric oxide	38·69
Lime	9·12
	91·11

There was undetermined CO_2 and a trace of H_2S.

449 D.—The clinker was finely pulverised. It contained a trace of sulphates and no magnesia.

Analysed it gave—

	Per Cent.
Insoluble in 10 per cent. HCl	2·81
Soluble silica	20·61
Alumina and ferric oxide	8·69
Lime	65·62
	97·73

449 C gave on analysis :—

Silica	14·62
Alumina and ferric oxide	9·21
Lime	41·05

The clay had absorbed some water.

This mixture is slightly different from the first, containing a little more clay.

449 B.—The physical tests were: 1 day neat, 369 lb.; 7 days neat, 720 lb.; 28 days neat, 756 lb.; 7 days mortar, 209 lb.; 28 days mortar, 326 lb. The cement was analysed as usual. It gave :—

	Per Cent.	Per Cent.	Per Cent.
Insoluble in 10 per cent. HCl	3·73		
Soluble silica	19·65		95·60
Alumina and ferric oxide	7·95		
Lime (CaO)	64·27	91·87	
Sulphates (in terms of SO_3)	1·54		
Magnesia (MgO)	..		
Volatile at a red heat	3·11		
	100·25		

$$\text{Active index} = \frac{64·27}{2·8 \times 19·65 + 1·1 \times 7·95} = 1·008$$

This sample when analysed by Humphrey's method gave :—

	Per Cent.	Per Cent.
Silica	22·36	
Alumina and ferric oxide	7·52	95·92
Lime	66·04	

449 F.—A mixture of pulverised fire-brick and lime was made, having approximately the ultimate composition of a cement. When analysed by our method the results were as follows :—

	Per Cent.
Insoluble in 10 per cent. HCl	33·25
Soluble silica	0·11
Alumina and ferric oxide	0·28
Lime	60·89
	94·53

The insoluble portion contained :—

	Per Cent.		Per Cent. of whole.
Silica	58·52	=	19·45
Alumina and ferric oxide	26·98	=	8·97
Lime	11·98	=	3·98
Magnesia, undetermined	..		..
			32·40

Analysed by Humphrey's method it gave :—

	Per Cent.
Silica	19·82
Alumina and ferric oxide	8·68
Lime	66·44
Magnesia, undetermined	..
	94·94

Humphrey's method is not a proper method of mineral analysis.

Whatever portion of the material is soluble should be dissolved without fusion. Fusion in the manner described introduces a large amount of sodium chloride into the assay to no purpose, and should therefore be avoided. So much sodium chloride makes it difficult to separate the silica completely, and prolongs the scheme to no purpose. Moreover, the results given above speak for themselves.

Believing that the results obtained by the method of analysis used by ourselves, and the interpretation of which they are capable, furnish the strongest possible argument for its general use, we submit the following suggestions respecting it :—

The samples should be taken and preserved in a well-stoppered bottle.

The sample for analysis should be exactly like the sample submitted to physical tests. It should be subjected to no treatment whatsoever that will change its properties in any manner. It should be neither dried nor pulverized.

Five grms. should be weighed out and gradually introduced into 250 c.c. of not stronger than 10 per cent. HCl, in such a manner as to avoid any appreciable rise in temperature. The solution should be vigorously stirred at intervals for half an hour. If effervescence follows it indicates the presence of carbonates. Traces of H_2S are to be expected. A black residue indicates carbon or soot, which may be estimated by gathering the residue on a balanced filter, weighing, burning off the carbon and again weighing.

Our correspondent regards this residue as the coarse particles of the cement deprived of lime by "local separation" whatever that may mean. There is not a particle of evidence to support any such contention. The residue from 10 per cent. HCl is not coarse. It is fine as ashes, and is ashes. He says that it is not safe to add to the raw material lime enough to convert the silica and alumina of this fuel ash into cement; hence it is properly to be inferred that the lime and ash will not unite to form cement. That being the case, the cement is dissolved away from the ash and unburned clay, the former of which can only be brought into solution by prolonged action of the acid on the material in very fine powder. To us, this means, which we have discovered of separating the cement from that which is

not cement, seems extremely fortunate, and we cannot understand why anyone else who desires to distinguish between good and bad cements should think differently.

The filtered solution is evaporated to dryness over a water bath, the residue carefully and completely desiccated, drenched with concentrated HCl, taken up in water, the silica filtered off, dried and ignited as "soluble silica." This is a proper designation to be applied to this material, as it has been wholly dissolved by the action of the weak acid upon the cement. It is also that silica which is in combination to form cement, provided that it is not augmented by the silica that forms the bulk of the fuel ash, and, in Humphrey's scheme, also by the silica of the unburned clay.

The filtrate from the silica is made up in a graduated flask to 1 litre, and two portions of 100 c.c. each, are precipitated with ammonium hydrate, the ammonia boiled off and the precipitate brought upon a filter and filtered while hot. Mr. Humphrey very properly dissolves this precipitate in dilute HCl and reprecipitates, passing the filtrate through the same filter. In no other way can the iron and alumina be freed from lime and magnesia, if either exists in considerable quantity.

The united filtrates are brought to a temperature near boiling, and the lime is precipitated as oxalate. After being allowed a sufficient time to settle, the lime may be filtered off and determined, either as oxide or carbonate, or titrated with potassium permanganate, as suits the convenience of the operator.

If extreme accuracy is required, the filtrate from the lime will be evaporated to dryness, ammonium salts expelled, and the magnesia precipitated with hydrogen ammonium phosphate. For practical purposes this evaporation is not necessary, but the magnesia may be determined in the filtrate from the lime.

For practical purposes too, the sulphates may be determined in the filtrate after removing the magnesia, after it has been acidulated, but the original solution is better. To test this point, the sulphates were determined (*a*) in the original hydrochloric acid solution of a cement freed from silica, (*b*) in the same solution from which the iron and alumina had been precipitated, (*c*) in the same solution from which the iron and alumina had been precipitated and the ammonia had been boiled off, (*d*) from the solution after removing magnesia. The determinations (*a*) and (*c*) varied by 0·03 of 1 per cent., (*d*) was one-tenth of 1 per cent .greater, while (*b*) was very much less than either of them. According to our experience, barium sulphate thrown down in presence of iron and alumina always contains traces of these oxides.

With the determination of the matter volatile at a red heat, these results furnish all of the analytical data necessary to form a judgment concerning the quality of any cement, either Portland or Rosendale, and with slight modifications the method may be applied to concretes. To cast these results into the form of an "active index" or "inactive constituents" will not change the results in any respect, but may be useful in comparisons, as are many wholly artificial devices.

It was originally our purpose to discuss the significance of results obtained by us in the determination of the lime and alumina of cements soluble in water, both gravimetrically and by titration, but the length of this paper leads us to defer this till another occasion.

THE MANUFACTURE OF NITRIC ACID.
PART I.

BY C. H. VOLNEY, PH.D.

THE increase in the consumption of strong nitric acid which has taken place in the last 30 years, has necessarily led to improvement in the process of manufacture of the acid.

The process of producing the acid from sodium nitrate and sulphuric acid had always been considered a simple matter, and as late as 1870, the condensation was effected by surface air cooling in large Woulfe's bottles, or tourilles, of stoneware. Later on, gradually the condensation by water cooling was introduced, and the principal improvement in this industry for quite a long time related to the perfecting of the apparatus for condensing the acid vapours.

Reviewing the literature on this subject, we find that the perfection of these condensing apparatus kept pace with the progress in the manufacture of chemical stoneware, in two directions: the apparatus, consisting of combinations of tubes and coils cooled by water, large receivers, towers for condensing and regenerating acid vapours, were turned out with a perfection which had not been previously considered possible; and the material of which these vessels were made was improved with a view to resist the effects of acid vapours and heat in a most remarkable degree, so that at the present time the old form of Woulfe's bottle, or tourille, which formerly occupied the most conspicuous place in the still house, has almost completely disappeared from a modern plant, and is only used as a receiver of condensed acid, and not any more exclusively as a condenser.

From the time that condensation by water cooling was found to be superior to air cooling, the forms of apparatus, the providing of tubes and receivers, in fact all the details necessary to increase the efficacy of the whole apparatus underwent gradual changes, and thus the apparatus of to-day, used for the condensation of nitric acid vapours, has assumed a different appearance, caused by the adoption of water cooling, and the regeneration of nitrous vapours.

In general, the principles here mentioned have been acted upon in all modern plants; we find in the respective literature data which result from the working of different systems. These have, for the past 10 years, been discussed and illustrated in detail, so that a discussion would be only a reference to the literature of the day on that subject.

It may, then, at present, be accepted that the modern nitric acid plant consists of a water-cooling system in connection with surface cooling and a regenerating tower; the latter is usually provided with lifting pumps, whereby the liquid is repeatedly brought to the top of the tower and then comes over and over again in contact with the acid vapours. Hereby an acid of 1·38 sp. gr., and even higher, may be obtained.

The retorts have in principle undergone hardly any change. The material is still cast iron, and the shape and size is somewhat varied according to the ideas of the constructor of the apparatus. They are usually made now of greater dimensions than formerly, so that one-half ton, or more, of nitre may be used at one time. They are also provided with an outlet for the liquid bisulphate.

In relation to the process of decomposition, very few improvements have been developed during the last 30 years. The quantities of material, that is, the proportion between sodium nitrate and sulphuric acid, as well as the concentration of the latter, and the treatment of the mixture in the retort, have undergone little, if any, change.

When I later undertake to discuss, beyond mere mention, proposed processes, it is done for the importance given to one or the other improvement, either by patent literature or by investigations and arguments given in a number of articles and journals or hand-books.

Since 1890 considerable material in reference to the condensation apparatus has accumulated. The proposals made by Oscar Guttmann,* to substitute tubes for the tourilles, were gradually developed, and after applying therewith an efficient system of cooling for a sufficient number of tubes by water,† and connecting his system with the Lunge Rohrmann condensing towers, he could report on the workings of this system in a number of factories erected on large scales in Europe. The published results account for a surprising yield, viz., 98 per cent. monohydrate, and 2 per cent. from the condensing towers as acid of 1·40 sp. gr. The economical results of the work were equally satisfactory, as he reports that for 3·5 kilo. of monohydrate, only 1 kilo. of coal was consumed. As the towers condensed almost all the nitrous and other gases, the system thus fulfilled the purpose in an excellent manner.

The production of concentrated acid, as well as an acid free from nitrous oxides, chlorides, &c., has been proposed and effected by partial condensations‡ by Frasch, Diterle and Rohrmann, and others. The removal of nitrogen

* Zeits. angew. Chem. 1890, pp. 507 and 700.
† O. Guttmann, Zeits. angew. Chem. 1893, p. 37.
‡ Frasch, Ger. Pat. 82,573, 1894; Diterle and Rohrmann, Ger. Pat. 85,240, 1896.

peroxide (N_2O_4) always takes place on addition of water to the monohydrate, and the end results depend upon the degree of heat to which the liquid or the gases are subjected. But they will consist of monohydrate and gaseous nitrogen oxide, thus :

$$N_2O_4 + H_2O = HNO_3 + HNO_2$$
$$3NO_2 + H_2O = 2HNO_3 + NO$$

The latter is converted in the presence of atmospheric oxygen again into peroxide, so that in the presence of water ultimately HNO_3 is obtained. It is however assumed that by water cooling and quick and efficient condensation, the monohydrate condenses without absorbing any considerable amount of these lower oxides of nitrogen, and at the present time the systems of Guttmann, Lunge, Rohrmann, Bettenhausen, Griesheim, &c., fulfil these purposes satisfactorily according to published results. The intricate combinations, as well as the nature of material in these apparatus, should however be seriously considered, and certainly will form some drawback in practical working.

It may be safely assumed at present, that by the use of efficient water cooling in connection with sufficient condensing and regenerating apparatus, a concentrated and practically pure acid can be produced economically.

The material, which forms the source of all nitric acid consumed for use in arts and manufactures, is still sodium nitrate, and the old theory of its decomposition of sulphuric acid into acid sulphate and nitric acid is still generally accepted. Some progress in the right direction is being made in this process of decomposition, indicated by the proposals to use polysulphate, formed by the action of sulphuric acid on the nitrate. I shall return to this subject when discussing the processes themselves.

Improvements in apparatus which are supposed to effect this theoretical decomposition more or less, have been projected by Guttmann and Valentiner. The former* inserts, in the tubes leading from the retorts to the condensing apparatus, a hot air blast, which not only exerts an oxidising influence on the acid vapours coming from the retorts, but would also necessarily produce more or less of a vacuum in the retorts themselves; the latter† proposes the distillation of the acid *in vacuo*.

Although Guttmann himself recommends the introduction of air in connection with the hot air blasts, inserted between the retorts and condensing apparatus, I am inclined to assume that thereby an influence is produced on the process by the quickened removal of the acid vapours by the suction of the air blast, which will necessarily lessen the pressure in the retorts, and also protect the monohydrate from contact with the hot surfaces of the apparatus. I have been unable to find in the technical literature of to-day, so far, any data which might throw light on the process in the retorts during the distillation in this apparatus, and no direct experiments and determinations of temperature seem to have been made or published. We have, therefore, to accept the favourable results obtained therefrom as the effects of the hot air on the distilling acid, in connection with the system of water cooling and regeneration.

The specification of the Valentiner patent describes the distillation of nitric acid from dry saltpetre and sulphuric acid of 66° Bé. in a vacuum. It states also, that the monohydrate commences to distil as soon as all the air is exhausted, but that the distillation is assisted "essentially" by heating.

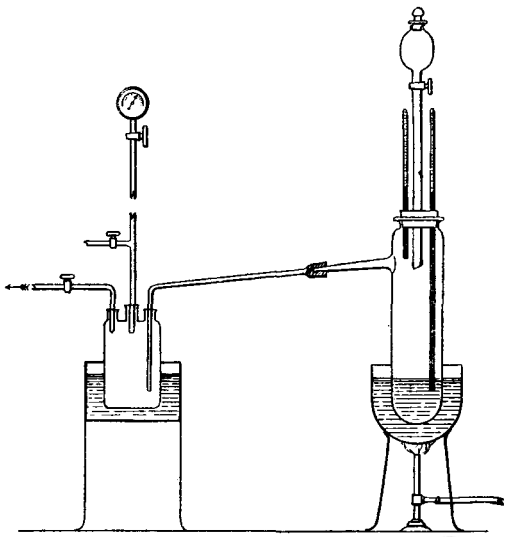
Later‡ he recommends the distillation *in vacuo* also for the manufacture of monohydrate from diluted nitric acid and concentrated sulphuric acid. We find detailed reports on the practical working of this system in periodicals for the last five years, giving results of the working on larger scales of the system. §It appears, however, that now the fact may be accepted, and that the distillation of concentrated acid from dry sodium nitrate and sulphuric acid of 66° Bé., has been abandoned, and that weaker, or chamber acid is used. This is evidently owing to practical difficulties, such as excessive foaming and running over of the salt mixture, &c. The result is, that at the present time, a sulphuric acid of 60° Bé. is used, furnishing diluted nitric acid of some 40° Bé., and that for the production of stronger acid a second distillation of dilute nitric acid and stronger sulphuric acid is necessary.

The reports which have been published on the practical working of the Valentiner system are very favourable, both in view of quality and quantity of the acid, and it appears that the handling of the apparatus with the air pumps and manipulation of vacuums can be done effectively by attentive and trained workmen. Lately Guttmann* has offered a lengthy comparison between the systems of Guttmann-Rohrmann, and Valentiner. From the statements furnished it would appear that 100 parts of saltpetre yield in the Guttmann apparatus 99 parts acid of 1·502 sp. gr., to 83 parts of 1·500 sp. gr. in the Valentiner apparatus.

It is claimed to be one of the special advantages of the vacuum distillation that it takes place at a proportionately low temperature; that consequently the apparatus does not suffer as much as in the usual practice; it may be also supposed that the resulting nitric acid is less subjected to the decomposing effects of the heated surface of the retort.

In the publications to which I have had access I find nothing definite in regard to the effect of the vacuum on the chemical process in the retort; that is, on the decomposition of sodium nitrate by sulphuric acid. Dr. Bergmann† states that the retort is provided with a long thermometer for the control of the temperature, and that the distillation of the nitric acid begins at 85° C. Evidently, this thermometer indicates the temperatures of the distilling vapours, and not that of the salt mixture in the retort. In the descriptions of the apparatus and of the working of the same, which have come to my knowledge, this is the only reference to the temperatures, and an investigation of the process itself, as it takes place in the retort *in vacuo*, appears not to have been given in any of the publications. If we consider the distillation of a mixture of two such liquids as nitric acid and sulphuric acid *in vacuo*, we can understand that monohydrate, which boils under 760 mm. pressure at 81·5° C., will distil at a lower temperature than that under less pressure. It is presupposed, however, that the nitric acid, in this case, exists as free acid.

But when we have a mixture of sodium nitrate and sulphuric acid, the nitric acid must be produced first in a free state before it can be distilled; that is, the sodium nitrate has first to be decomposed by the sulphuric acid. The question therefore, is whether this decomposition,



* O. Guttmann, Zeits. angew. Chem. 1890.
† F. Valentiner, German patent, 63,207, 1892.
‡ Valentiner, German patent, 88,321, 1895.
§ *M. Bruley, Mémorial des poudres et salpetres*, 1897 (France). Franche, Zeits. anal. Chem. 1899 12. Bergmann, *ibid.* 1899, 42.

* Zeits. angew. Chem. 1901.
† Zeits. angew. Chem. 1899, 42

in vacuo, takes place at a lower temperature than under the ordinary atmospheric pressure. As I cannot find that this question has been investigated, I have made the following experiments in a retort of strong glass, of the shape shown in the attached sketch, connected with the receiver, air pumps, vacuum gauge, and provided with thermometers to measure the temperatures of distilling vapours, of the residues in the retort, and of the oil bath. I subjected 100 grms. of dry sodium nitrate and 110 grms. of concentrated sulphuric acid to distillation.

The series of observations, made under a vacuum of 300 mm., show the following:—

Time.	Temp. of Oil Bath.	Temp. of Salt Mixture.	Temp. of Distilling Acid.	Remarks.
PM.	° C.	° C.	° C.	
1.35	..	20	..	.. ..
1.50	75	36	25	Little gas.
1.55	110	40	35	Quietly boiling.
	145	55–65	45	Distilling.
2.00	146	100	75	Distilling violently.
2.02	127	104	70	Foaming.
	108	102	62	
	120	106	78	All nitre not yet dissolved.
	130	..	78	Foams.
	147	..	78	Foams violently.
2.12	160	111	78	Nitre dissolved.
		115	78	„ „
	125	115	67	Distilling lessens.
2.17	125	115	66	„ „
2.22	136	116	66	Light yellow vapors.
	148	118	70	Distilling.
	150	124	71	„ foaming.
2.34	153	127	76	„ „
	144	128	76·5	Quiet, distilling.
2.40	132	124	72·5	Salt separating.
2.50	144	124	76	Foaming ceases, distils quietly.
	149	127	79	Distilling lessens.
	150	128	76	„ „
3.00	151	129	76	„ ceases.

The temperature of the oil bath was regulated as the distillation required. It follows from the series of observations, that at a temperature of 65°—100° C. of the retort contents, the monohydrate distils, showing a temperature 45°—75°, ending the first phase of the action of sulphuric acid on the sodium nitrate.

In the retort is then a quantity of undecomposed nitrate and a liquid polysulphate of sodium. This latter, when the temperature of it has risen to 100° C., commences to further decompose the remaining sodium nitrate, entering thus on the second phase of the process, and its temperature rising from 100° to 129°, the rest of the nitric acid—to the greatest part clearly consisting of monohydrate, distils over, showing under 300 mm. pressure a boiling point of 70° in average. Comparing the temperatures thus obtained with those appertaining to the distillation under 760 mm., we find that the latter furnishes the monohydrate by distilling from the salt mixture in the retort at 97°—122° C., the boiling point of the acid under that pressure lying between 77°—90°. The second phase takes place at a temperature of the salt mixture of 130°—165° C. The second portion of the nitric acid, also practically a monohydrate of 1·505 sp. gr., shows a boiling point of 94°—100°, under 760 mm. pressure; and the rest of the acid results at a temperature above 165° of the retort contents, and is the yellow acid containing the decomposition products by heat of the monohydrate. It will be seen from this that the effect of the polysulphate of sodium on the rest of the sodium nitrate in the retorts commences under 300 mm. pressure at 100° C. and is finished at 129°, whereas under the higher pressures this process commences at 130° and is not finished at 165°.

As Dr. Bergmann (this Journal, 1899, 1122) states that the distillation of nitric acid from sodium nitrate and sulphuric acid takes place in the Valentiner retorts at a vacuum of 600 mm. I shall present in the continuation of this paper the results of my investigations at that pressure, and also of the distillation of mixtures of nitric and sulphuric acids.

THE IMPROVEMENT OF INSTRUCTION IN TECHNICAL CHEMISTRY.

BY ALBERT LACHMANN.

In attempting to discuss so broad a subject as the methods of teaching technical chemistry employed in this country, one is met at the outset by numerous difficulties of interpretation. No two of the thirty odd institutions claiming to prepare students for the practice of technical chemistry seem to agree on the topics necessary for study, the order in which these should be taken up, the extent to which anyone should be cultivated, or the actual subject-matter of courses given under the same name, to say nothing of the non-chemical subjects in the curriculum, such as mathematics, literature, analytic mechanics, and other "strains and stresses." In some colleges, chemical engineering seems to mean a mixture of less chemistry and less engineering than is required of either chemists or engineers; whereas in others "analytical chemists" are turned out after one or two years experience on "unknown and complex ores." It is plain, therefore, that in order to arrive at any comprehensive view of actual and of desirable conditions, it will be necessary to define, in a manner somewhat more precise than usually customary, the fundamental aims of technical education with reference to chemistry.

The Unity of Chemical Practice.—Stated in its baldest terms, the aim of such technical education must necessarily be, adequate preparation for professional practice. To be sure, this definition merely re-states the problem itself; we must immediately ask, what do we mean by adequate preparation, and what by professional practice? Let us consider the latter question first. At first sight, the professional practice of the chemist appears as an exceedingly complex affair, incapable of closer statement; a chemist may be a mineral analyst, a food analyst, a metallurgist, a manufacturer of heavy or of fine chemicals, a gas chemist, an electro chemist, a pharmaceutical chemist, a dyer, a manufacturer of coal tar products, a fermentation chemist, to leave out further subdivisions, and last but not least, a teacher of chemistry, perhaps the most technical of all. It will be said that this is far too broad a picture of the chemist's activity; and that the whole of technical practice may be summed up under three distinct heads—analysis, manufacture, instruction. Indeed, it may be safely said that this represents the opinion of the majority of American and English chemists. And yet, the analyst, the manufacturer, the teacher, are merely chemists, thinking by the same mental processes, applying the same general laws, attacking very similar problems, differing only in the accidental circumstances of the materials they work with. The analyst sacrifices time and money to the cause of accuracy, the manufacturer gives up accuracy for the sake of time and money, the teacher wears himself out in the effort to be accurate without wasting either time or money.

The fundamental unity of chemical practice is not a new discovery. It was "made in Germany" some forty years ago, and, although not patented, the Germans enjoy an exclusive monopoly of its use. The analyst, the manufacturer, and the teacher are continually exchanging their experiences through a sort of chemical clearing-house. Young docents frequently spend two or three years in a factory for the purpose of broadening their knowledge; a factory sends its problems to the university laboratory to be solved. An investigator in need of expensive substances, or of mechanical appliances to handle large bulks of material, need only apply to the nearest factory to have its machinery placed at his disposal, or its wares furnished at little or no cost. The larger factories have their own research laboratories, in which a hundred and more university graduates and professors spend months and years on single investigations, and are paid liberal salaries, even if their results are technically worthless. One factory recently purchased the entire scientific library of Kekulé. The manufacture of nearly all the numerous coal-tar products consists merely of laboratory methods on a large scale; and the scientific problems solved in this connection have

been of the utmost importance and benefit to "pure" science. Certain large establishments prefer their chemists to have had training in pure science only, and then give them from three to six months of technical training in their own works and at their own expense. The specifications of chemical patents constitute an important section of scientific literature, and the German Chemical Society spends large sums of money for the purpose of abstracting and indexing them. If we also take into consideration the unquestioned pre-eminence of Germany in all branches of chemical practice, what better demonstration can we give of the fundamental unity of the profession.

Present Status of Instruction.—We are now in a better position to return to the former question: What do we mean by "adequate preparation"? For no matter what may ultimately be agreed upon, it will be identical in its first two or three years for all classes of students. It is a great comfort to have this important point definitely settled in advance; for one of the main difficulties in arranging our college curricula has been the supposed necessity of providing two or more co-ordinate sets of courses in chemical instruction. This has been a great strain on the teaching staff as well as upon the financial resources of the laboratory.

An examination of a number of college catalogues brings out the fact that at present all students do follow essentially the same courses for about two years. These courses usually consist of one or two terms of general inorganic chemistry and simple laboratory work, one or two terms of qualitative, and one or two terms of quantitative analysis; all these combined so as to occupy from two to three years at the rate of 8 to 15 hours per week. It would seem, then, that we have the present American conception of "adequate preparation"; for the subsequent courses are almost invariably special short ones in various branches of work. We may well ask: Is this preparation really adequate? I think not; but before considering it in detail as the main business of this paper, a few words must be said concerning these special addenda, the "finishing courses."

Even in some of our best institutions, these final courses come perilously near the standard of the "polite deportment" and "philosophy" of young ladies' seminaries. There is an unfortunate lack of caution in the claims published in their catalogues. Lack of space prevents citation of many of the choice extracts I have found; two or three will suffice, however, for purposes of illustration. One college offers a course of 44 lectures on the following topics: "Metallurgy, glass, ceramics, chemicals, illuminating gas, bleaching, photography, petroleum, brewing, wines and liquors, vinegar, fats and oils, essential oils and resins, sugar, starch, glucose, milk, distillation of wood, paper, tanning, &c., &c." In order to cover the ground, these lectures must be illustrated with a kinetoscope. Another institution claims to prepare its students for: "Metallurgy and mining, chemical manufactures, dyeing, bleaching, tanning, sugar-refining, &c., and for work as analytical chemists, assayers, or teachers of chemistry." The claim is based upon three lectures per week for two years, and two laboratory courses of 12 hours per week each, which may be taken in one year. A third college offers a course of three hours per week for one semester in: "Qualitative and quantitative examination of air, water, food, disinfectants, baking powders, flour, bread, tea, coffee, cocoa, spices, milk, butter, lard, beer, and other subjects."

It may be objected to the above that college catalogues are notoriously optimistic. The fact would seem to be demonstrated; none the less, such exaggeration is of very questionable value, and should be discouraged for the benefit of the "raw graduate," if for no better reason. It would appear, moreover, that the majority of colleges consider training in analytical methods equivalent to complete technical training; nearly all of them give several courses in water analysis, analysis of fuels, iron and steel, &c. The importance of such analytical training is undoubtedly over-estimated; a student who must continually neglect the factors of time and cost in his work receives too one-sided a training. The same objection is to be raised to many of the so-called courses in "technica work." If they do not consider time and cost as the essential factors, they have no better claim to a "technical" nature than the ordinary beginner's preparation of hydrogen or chlorine. But more of this later.

Let us now examine more closely the nature of that preliminary preparation which we have found to be so nearly uniform throughout the United States. In the way of lectures, there is always a course on inorganic chemistry, occasionally a short one on organic, infrequently a very brief treatment of "general" or theoretical chemistry incorporated with the above or as a separate course. Modern theories, and the details of organic chemistry, are usually left for advanced and optional courses. The time spent on these lectures varies greatly; but perhaps a fair estimate is three hours per week for two years (of about 35 weeks each). In many, in fact in most, colleges this average is not maintained. The laboratory training includes one, very infrequently two terms of general introductory work, ranging from three to six hours each week. On the whole, this course may be described as satisfactory. Then follows a course in qualitative analysis, averaging 12 hours per week for one or two terms. Recitations accompany it in many instances, mainly for drill in writing equations, it would seem. In a few of the better institutions, but only in very few, the subject is made to serve as a practical demonstration of the theory of solutions. Finally, from one to three terms are spent in quantitative analysis, also averaging perhaps twelve hours a week. The subject matter of this last course also varies greatly with the college; at the best, there is included training on a few alloys, sulphide ores, silicates, and a number of volumetric methods on technical products. The feeling cannot be repressed that in this course results call for a disproportionate expenditure of time. It is with the training afforded by the above that the student proceeds to follow his natural bent, and to acquire the special technical skill needed for his professional activity.

Outline of the Proposed Improvements.—The technical preparation of the teacher need not concern us further. Every college and university is practically a technical school for training teachers first of all, and for training technologists only secondarily. Not that the training of teachers of chemistry is incapable of great improvement; but on the whole it is so much more satisfactory, that the improvement of instruction in other technical branches is more imperative. Moreover, taking as our major premiss the essential unity of chemical practice, be it that of analyst, manufacturer, or teacher, it follows that the main deficiency in the present training of our teachers is exactly the lack of knowledge we are deploring and endeavouring to remedy. Should it be possible, therefore, to generate within the college walls the mental atmosphere of the busy world where things must be mastered as well as ideas, we shall also have ministered to the wants of the budding preceptor.

Having thus wearied you by a circuitous return to our starting point, in order to eliminate possible objections based upon differences in the point of view, let us again take up the question: "What do we mean by adequate preparation?" If, in the following discussion, the proposals I have to make shall appear a trifle too radical, I trust you will bear in mind that they spring from a conscious and deliberate idealism; and should the views here expressed really prove to have a basis of truth, any merely practical difficulties will yield as surely as the difficulties of manufacturing Indigo yielded to the idealism of the German professor.

I take it, then, that an adequate preparation for the technical chemist has been secured when (1) a sufficiently broad general foundation of inorganic, organic, and physical chemistry has been laid in the class-room and in the laboratory; (2) when the "chemical instinct," *i.e.*, the ability to think in chemical terms, has been developed; (3) when sufficient analytical skill has been attained to ensure accuracy in following new methods; (4) when enough preparative skill has been acquired to make any compound with maximum purity in maximum yield, at the lowest possible expense under given conditions; (5) when

speaking acquaintance with current chemical thought, both pure and applied, has been reached; and (6) when time has been found to accomplish all this.

The Broad Foundation of Chemical Knowledge.—First, then, as to the general foundation of inorganic, organic, and physical chemistry in the lecture-room and laboratory. I think that the present equipment of our students in this line is too meagre. The plan seems to be to give very elementary courses in all three subjects, and then to assume that this information will multiply by cell-division in the laboratory atmosphere. Another very prevalent view among teachers is that if you only give the student fundamental ideas, the facts will take care of themselves. My own experience is that, for the amount of time spent in study, the outfit of actual information about chemical substances is unreasonably small. The unfortunate policy of feeding students on peptonised and malted facts may avoid mental indigestion in college, but it predisposes them to colic afterwards. Certain very prominent text-books have had a bad influence in this direction, by seeking to eliminate all possible difficulties of comprehension and any reference to partially solved problems. It is not uncommon for students to ask if they "have to remember the names of those substances" in their lessons, after a few weeks with those books; indeed, the question is not unreasonable, if we consider that the names constitute the sole remaining difficulty. Again, both text-books and teachers seem afraid that the students will know too much, and prune out all except matter of the first importance. To be sure, excess of detail must also be avoided if clearness of presentation is sought; but is not the present tendency somewhat too violent a reaction from the methods of our forefathers? One very prominent teacher told me some years ago that each year he takes up less material in his lecture courses; what will become of them in 10 years more? It is the function of the teacher to guide students through the maze of facts as through a crowded city, that later they may find their own way about; not to whisk them around in a closed trolley-car, on the globe-trotter principle.

One difficulty lies in the use of text-books which are, really, only elaborated outlines of a lecture course. The result of using such books is that the student's horizon is bounded by the cover-boards. A lecture must be straightforward and consistent if it is to have any value at all; but the printed page can be read over and over again, and its details mastered gradually. The text-book alone can provide the large number of facts that must be assimilated; the lecturer's syllabus is properly a key to the text-book, and no more. The teacher thus has a perfect right to demand of his students a greater knowledge of detail than he himself presents in his lectures. Another obstacle to the absorption of the proper number of facts lies in the almost universal attempt to treat a subject once and for all. This does not seem rational, nor do the student's mental limitations make it feasible. To take up the metaphor of the crowded city, the first efforts of a good guide result in a general survey, pointing out the topography, main thoroughfares, and most important activities. Then come historical landmarks, and those sights which distinguish this city from all others; finally, a detailed study of each quarter, of special industries, and of prominent people. We can follow no better plan in teaching a new science, giving a broad survey first, then repeat, filling in many new details; finally going over it a third and a fourth time if necessary. In this way the student's memory will be aided rather than overtasked; the relationship of parts to the whole, one of the most difficult of problems to him, will certainly be clearer; and the relative importance of various topics will stand out prominently. Then we shall avoid the necessity of turning out "chemists" whose sole acquaintance with chemistry as distinguished from analysis was formed in the freshman year, and discontinued immediately thereafter. Chemical facts ought to be systematically studied each year of the college course.

But little need be said in addition to the above concerning the laboratory work to be correlated with this plan. I should merely wish to emphasise that substances should be studied from the preparative, analytical, and physico-chemical sides simultaneously; the artificial division of the science for purposes of classification should influence the course of instruction little, if at all, during the first two years.

The Chemical Instinct.—Chemistry is a science which reasons about facts through a medium of abstractions. We observe colours, smells, and precipitates, and we talk about atoms, molecules, and space configurations. The thinking chemist must continually bridge the gulf that lies between fact and fancy; if he can do this freely, and avoid metaphysics, he possesses the chemical instinct. To develop this instinct in the student is the most important and most difficult problem of the teacher. Aside from intuitive inherent teaching power, I know of but one plan for fostering this instinct; every topic should be presented in the form of a problem. Chemistry has advanced to its present proportions because of the problems presented to it; all research work is a series of correlated problems; the installation of every manufacturing plant, of every new process, the regular operation of every established factory, is a series of problems; every analysis for whatsoever purpose is a problem; teaching is one vast problem. The main preliminary to the solution of any problem is a clear and complete realisation of its nature. What is aimed at, what are the difficulties, what means are available? These questions should be as definitely in the mind of the student at every point of his college work as they must necessarily be in his later professional activity.

Analytical Skill. — Perhaps no portion of chemical instruction is better given than training in analytical work. Methods have been worked out with such precision, and mechanical aids are so perfect, that given time, patience, and care, anybody may become a fairly skilled analyst. Moreover, teachers seem agreed that intoductory work, both in qualitative and in quantitative analysis, should precede all special analytical courses. Perhaps it may seem superfluous to offer any suggestions for the improvement of this portion of chemical training. I shall discuss this later. At this point I wish to speak of these special courses. It has already been shown that they constitute the crux of the "technical" training of this country. It seems to me that they tend to destroy the unity of analytical practice by inducing the student to specialise far too narrowly and far too soon. Students often spend the whole of their third and fourth years at college on these courses alone, and thus deliberately sacrifice the sole opportunity of their lives to acquire a broad and thorough training for all future emergencies. I have known students at one of the largest universities of the land to avoid all courses on theoretical and organic chemistry, on the grounds that they would have no use for them at a blast furnace—that college sending most of its graduates into the iron industry. Surely no college should thus encourage its students to neglect their opportunities. A little of the wise and far-sighted co-operation practised by German employers would furnish immediate relief from this state of affairs. Experience has shown that the employer does not suffer by choosing broadly-trained chemists in place of stall-fed analysts.

Besides, most of the "technical methods of analysis" taught in our colleges have a way of getting antiquated. Each year witnesses some new committee of technical societies for the purpose of improving analytical methods. By the time these methods get into the text-books (copper-plates being valuable) another committee is under way. If such analytical instruction is reserved until the last term at college, and then based upon the reports of these committees, the student will be more likely to acquire really useful knowledge, and have more time for broader study.

Skill in making Preparations.—Until quite recently, the only training in the preparation of chemical substances was afforded by organic chemistry; latterly, a number of colleges have introduced courses in inorganic preparations as well. These courses constitute excellent discipline, as far as they go; they do not go far enough. The actual preparation of chemical substances may serve three purposes. It may be intended to place the substance in the student's hands for study; if no more is sought, it is often cheaper and always quicker to furnish it out of laboratory stock. It may be intended to illustrate the reaction. In combination with

the first purpose, this end is eminently desirable for beginners; for advanced students it wastes too much time in proportion to the result, such illustration would be met as well by the use of chalk and blackboard. And finally, it may be intended as a study of chemical technology. As such, it must inevitably take cognizance of the aims of technology, which are to prepare a substance in given grade of purity from the available "Ausgangsmaterialien" at the smallest cost of time and money. It involves a sufficiently complete knowledge of the materials employed, strictest economy of time, labor, and reagents, the demonstration of the required purity, and a calculation of cost and value. If a substance be prepared along these lines, to the amount of one grm. or of one ton, it constitutes an exact duplicate of technical methods. The factory may employ cast-iron vats in place of flasks, and filter-presses in place of funnels; it is not the more "technical" by virtue of its appliances.

It will be objected that no university can make soda-ash or sulphuric acid on technical lines. To be sure; but it need not attempt to. The first condition of success in any undertaking is a clear understanding of one's limitations. The college cannot do much more than teach the factory spirit; if it does that much well, enough will be accomplished at present. Moreover, chemical industry is not limited to the production of heavy chemicals at the rate of fifty tons a day. Innumerable substances are manufactured in relatively small quantities, and by methods which do not differ widely from standard laboratory manipulations. These are wholly within the power of the college. I would propose that each college inaugurate as a part of its curriculum, required of all its graduates in chemistry, a full year's course in the actual economical preparation of laboratory supplies. It can manufacture most of its own chemically pure reagents, ammonia, and its salts, the products of the rarer minerals, either much more cheaply than they can be bought or imported under existing revenue laws, and practically all its organic preparations from a few technical products. Even if it should cost a little more to make these substances than to buy them, the gain in actual experience to instructor as well as to students is worth the extra cost. The equipment for this work need not be expensive. For the chemically pure reagents, for example, the same outfit of large evaporators and crystallising dishes, solution tanks and filter presses can be used. The need of great care in cleaning these out for use on different materials would be an excellent feature of the work, to be controlled by analysis. By systematic planning, the laboratory could manufacture from 30 to 60 substances a year, in quantities to last five years; at the end of that period it would have supplied all its wants, and could begin the cycle over again. In one year of such work the student would gather more experience than a factory would yield in ten; for no factory can undertake to slow down its procedure for the benefit of a novice.

In connection with this laboratory instruction, the usual lectures on chemical technology will certainly be more fruitful of results. Such lectures should not be omitted, nor anything else likely to broaden the student's acquaintance with facts. Indeed, these lectures are able to supply information not obtainable in the factory, viz., the comparison of factory methods, and the deeper principles that underlie all technical work and are taken for granted—the business world drives them home with a club. Finally, it need hardly be said that frequent visits to every establishment within reach should be a constant feature of technological training. In my own college course the "frequent visits" materialised just once, when three large factories were visited in the course of two hours—another illustration of the optimism of college catalogues.

Acquaintance with Current Chemical Thought.—The necessity of familiarising students with new facts at first hand is self-evident, and realised by all conscientious teachers. The main difficulty would appear to be the accomplishment of the task. I know of only one method—to weary not of well-doing, and to keep everlastingly at it. Frequent meetings of students and instructors on an informal basis, be it a "seminar" or a "chemical society," where new facts are discussed without reference to their classification, comprehensive lectures on recent progress, essays by the students themselves, the current numbers of journals laid out in a cosy reading-room in the laboratory (the librarian must be overcome by fair means or foul)—all of these methods persisted in for two or three years will solve many a "complex unknown" cerebral obstruction. *Ad astra per aspera.*

Where to Find the Time.—The ambitious programme I have outlined now calls for the consideration of a purely practical problem: How can we find the time to accomplish all this? Even as our courses stand, there is barely time within the four years at college to complete the minimum of chemical work; where is there room for all the extra lectures and laboratory exercises that a really thorough technical preparation would seem to call for? I am afraid that my suggestions will contain many heresies.

For one thing, our college authorities must be made to realise that the main essential of training in technical chemistry is a knowledge of chemistry. This somewhat axiomatic doctrine is by no means universally accepted. Thus, the chemical engineers in two prominent institutions (Columbia, Pennsylvania) take considerably less chemistry than other students in chemical branches. Now while other topics are certainly necessary and valuable assets for chemical engineers, there must be a limit somewhere. The main problems before even the chemical engineer are chemical; those of the teacher and analyst almost wholly so. I will not presume to outline just how much or how little of these extraneous courses should be incorporated in the curriculum for technical chemists; but I should like to venture upon the principles which may fitly guide those more directly concerned with the task. I should say, then, that the question should be considered upon its own merits: no inherited prejudices, no educational theories should stand in the way of the prime fact that in studying chemistry a knowledge of chemistry comes first. The problem of general education and culture must not be allowed to interfere in any way; where cultural education is also sought, the time needed for it must be debited to its own account, and not written off the technical calendar. The two problems are absolutely distinct, and have no relation to one another. This must be insisted upon, since college faculties are only too prone to ignore it altogether. Better for a college not to give any technical courses at all, than to play at makebelieve and ruin the careers of its graduates. If four years at college are not enough for both general and technical education, take six eight, or ten—but take enough to do the work thoroughly. I would say, also, that the non-chemical subjects should be reduced to the lowest possible figures, and chemistry be given the benefit of every doubt. Wherever feasible, these subjects should be a part of the general education, and thus serve both ends; such would be, *e.g.*, German and French, physics, first-year chemistry, mathematics, &c. The cardinal rule should always be kept in mind, that it is better to know one thing well, than to have a smattering of many and command of none. We must not expect to see in each of our students a Helmholtz or a Ludwig Mond; if any of them are destined for such versatility they will have little need of our poor instruction.

One solution of the time problem, then, is to insist that enough time must be granted, and all extraneous matter reduced to a minimum. By the same token, however, it behoves us as conscientious chemists to do our best to shorten the time required for our own subject for the benefit of the student, it might be observed, not for the benefit of the college faculty. By the aid of one further heresy, I feel able to indicate where an important saving of valuable time may be accomplished. I would abolish from the curriculum the study of qualitative analysis, the archtype of anachronisms. We owe a tender feeling to the kindly nurse who brought us up carefully, and taught us the dark ways and vain tricks of the phosphates; but our nurse is old and decrepit, and no longer able to guide the toddling steps of the beginner. It will not be difficult to prove this. The study of qualitative analysis is intended to give knowledge of a useful art, and specific exercise in chemical thinking. It achieves neither purpose.

Systematic Qualitative Analysis as a Useless Art.—The problem of systematic qualitative analysis, as taught in

our schools, is to recognise all the ingredients of a given mixture. As a matter of fact, however, how much of this art have we achieved? We are able to recognise a limited number of inorganic acids and bases under special circumstances; and the instructor must exercise great self-restraint not to make his unknowns "too hard." As for the rarer acids and earths, to say nothing of the vast bulk of organic compounds, as well as for the commoner acids and bases in the presence of these latter substances, we must admit our inability to follow any comprehensive "scheme" of analysis. The analysis of such mixtures resolves itself into a series of special tests; and our only check upon the correctness of the analysis comes through the quantitative necessity of finding 100 per cent. of the ingredients. This limitation is clearly recognised by the professional analyst. Thus, the chemists of the United States Geological Survey never carry out qualitative analyses of the rocks they investigate; they assume that all of some 20 or 30 ions are or may be present, and check the absence of any one during the progress of the quantitative analysis. Nor do they undertake to analyse one single sample for all of these 30 ingredients; two or more possible ones constitute a group that is examined by itself, without reference to the other contents. Again, the analyst is seldom, if ever, called upon to make a complete analysis of an absolutely unknown brew; on the contrary, he is usually asked to estimate some two or three ingredients, whose presence is either known or whose absence is to be demonstrated. The assayer never makes other than a quantitative analysis of gold and silver ores. For the food analyst, all is grist that comes to his mill—moisture, fats, carbohydrates, proteids, and ash.

Where then is our boasted art of qualitative analysis, and where the need of dragging every chemist through the wearisome unknowns, so fearfully and wonderfully made, the like of which man never saw before nor will again? Why spend from two hundred to four hundred valuable hours to teach an art which does not exist? At the same time, it will be objected, the numerous qualitative tests referred to must be learned, and as well this way as any other. Not so; the important qualitative reactions of all the important substances should be studied in the first instance, when the substances themselves are studied, not be kept on ice for a "systematic" course. In the laboratory instruction in elementary chemistry time can be found for the methods of recognising the acids and the metals the student works with, while he is working with them. We must counteract our mania for subdivisions and classifications, and teach chemistry as a unit. To be sure the regular "scheme" for the metals and acids is a useful thing occasionally, and students ought to be familiar with it; but it can be taught in one week to any student having a fair supply of analytical reactions among his mental baggage. I would teach these reactions by the side of a course in chemical preparations, rather than in a course by themselves.

Qualitative Analysis as an Inferior Discipline.—The intrinsic value of qualitative analysis is thus seen to be small. Its pedagogic merit is not much greater. As a matter of fact, teachers know only too well that it requires herculean exertions on their part to prevent students from rushing through the course mechanically. The majority of text books are the merest skeletons of outlines, omitting a vast bulk of details "because they interfere with a clear grasp of the subject." One is strongly reminded of the way in which Latin is, or used to be, taught; the object being to reproduce its literature and culture, the literature and culture are left out to have more time for the syntax. So with qualitative analysis, the object being to train analysts, the analytical facts are left out to have more time for the system. Nor are we alone in our troubles; permit me to quote from the recent vice-presidential address of Professor W. H. Perkin to the British Association:—

"It has always seemed to me that the long course in qualitative analysis which is usually considered necessary, and which generally precedes the quantitative work, is not the most satisfactory training for a student. There can be no doubt that to many students qualitative analysis is little more than a mechanical exercise; the tables of separation are learnt by heart, and every substance is treated in precisely the same manner; such a course is surely not calculated to develop any original faculty which the student may possess. I question whether any really competent teacher will be found to recommend this system as one of educational value or calculated to bring out and train the faculty of original thought in students."

With this quotation I am content to rest my case.

What shall we substitute for it?—One important question remains. The art of testing unknown substances must always be an integral portion of the chemist's outfit; if the present course, designed for that very end, fails to teach it, what alternative have we to offer? The plan I venture to suggest may be found worth a trial.

I propose, first of all, to annul the divorce of chemical analysis and chemical preparation. Many colleges now introduce quantitative experiments into their beginner's course, such as (I quote in part from a circular issued by a conference of teachers at Chicago in 1896) definite proportions by volumetric methods, multiple proportions from the oxygen evolved from potassium chlorate and perchlorate, equivalent weight of zinc, weight of a liter of air, water of crystallisation in copper sulphate (both stages), neutralisation of normal acids and bases, &c. If along with these quantitative experiments the student is also taught the descriptive features and qualitative tests of the substances studied, as well as the fundamental facts of physical chemistry (also recommended and elaborated by the Chicago conference), I should say that there we had a course eminently satisfactory. If time permits, a year might be spent with great profit on this work. Then should come a course of the same general order, but more difficult. Starting with a metal, a mineral, or some technical product, the student should prepare a series of salts or other compounds of some ten or more metals. He should study the problem of obtaining the desired compounds; submit his plan to the instructor for criticism; prepare his substances, and analyse them qualitatively and quantitatively. The analyses should not be complete, but merely for effective purity and undesirable impurity; for economy of labour should be taught, and no work done that is not of direct value under the given circumstances. By a proper selection of material, the teacher will be able to present to his pupils, during two or three terms, all of the important qualitative and quantitative methods of separation. Nor is it necessary for each student to do exactly the same work; indeed, I should call it undesirable. Students would then learn from each other as well as from the teacher, and a laboratory "atmosphere" would then be created where students may learn by a process of cutaneous absorption, as they seem to do in Germany. The work, moreover, should be reported regularly in the accompanying "seminar"; current and older periodical literature should be searched for additional information bearing on each student's topics; innumerable little opportunities for research will present themselves, and the most ought to be made of them; a well-planned course of lectures should run parallel with the laboratory work and expand its horizon, for the whole field of chemistry cannot be reviewed in the laboratory; and then, I venture to believe, the instructor will have a class of interested if not enthusiastic students. Special schemes of separation, the "systematic" analysis now so widely current, the examination of milk and honey can then be taught as the special things they are, and made to take their proper places in the economy of chemistry.

Conclusion.

In conclusion, allow me to summarise the propositions I have tried to maintain:—

(1) The practising chemist, be he teacher, analyst, or manufacturer, is of one kin.

(2) For that reason, the training for these professions ought to be identical for several years at least.

(3) At present this training is inadequate.

(4) There is needed a much broader foundation in pure chemistry.

(5) The "chemical instinct" needs cultivation.

(6) Analytical training should be general rather than special.

(7) The college should establish *bonâ fide* courses in preparations, on a working scale.

(8) Acquaintance with current thought must be fostered.

(9) Time must be made for this programme by cutting off all but the most important non-chemical topics.

(10) Time can also be saved by eliminating qualitative analysis, because it is useless as an art, and inefficient as a discipline.

(11) The place of qualitative analysis should be taken by properly organised laboratory courses.

Perhaps it will be best to leave the twelfth and last conclusion to the charity of my hearers.

THE DETERMINATION OF MANGANESE IN FERRO-MANGANESE AND NICKEL IN STEEL.

BY GEORGE L. NORRIS.

THIS method is based upon the separation of iron from the other metals by the solubility of ferric chloride in ether, and in its details is very similar to the well-known ether method for the determination of nickel in steel.

The details of the method are as follows :—½ grm. of ferro-manganese is dissolved in 15 c.c. of nitric acid of 1·42 sp. gr. in a beaker, evaporated to dryness, and heated for a few moments to drive off all the nitric acid. The residue is then dissolved in 30 c.c. of dilute hydrochloric acid (equal parts of water and acid). This solution is then cooled to ordinary atmospheric temperature, and transferred to a 200 c.c. separating funnel, rinsing out the beaker with a little hydrochloric acid. To the solution in the separating funnel add a drop or two of bromine and 40 to 50 c.c. of acetone, shake hard for a moment to mix the contents, then add 75 c.c. of ether and shake again for two or three moments. Allow the solutions to separate and draw off the aqueous layer into a 600 c.c. beaker, washing out the traces remaining in the funnel with a little dilute hydrochloric acid. Add to the solution in the beaker about 300 c.c. of hot water, which will drive off most of the acetone and ether present. Bring to boiling point, add about 5 grms. of sodium acetate, and then precipitate the manganese in the boiling solution as phosphate, by the addition of 20 c.c. of a 10 per cent. solution of sodium ammonium phosphate and an excess of ammonia.

After the precipitate has been ignited and weighed, it is re-dissolved in a little dilute hydrochloric acid, and the small amount of silica present is filtered off and weighed. This usually only amounts to a few tenths of a milligram. There is seldom any iron present. In case chromium is present in the ferro-manganese, it can be determined in the hydrochloric acid solution of the ignited phosphate. This is most conveniently done by reprecipitating the chromium as phosphate in the slightly acid solution in the presence of sodium acetate.

The use of acetone in the ether separation is of distinct advantage, as being miscible in both ether and water, it permits the ether to more readily remove the ferric chloride from the aqueous solution. The bromine is used to prevent the reduction of any ferric chloride during the separation.

The method is equally applicable to manganese ores and manganese in steel. In the case of ores these can be dissolved direct in strong hydrochloric acid, then diluted with an equal amount of water, and the separation made as above.

This method has been in constant use for over a year, during which time it has been very thoroughly checked. A sample of ferro-manganese containing 81·12 per cent. as determined by several prominent chemists, gave by this method 81·13 per cent. and 81·16 per cent., the time required for a determination being about 45 minutes.

In determining nickel in steel by means of this acetone ether separation, 1 grm. of steel is dissolved in 20 c.c. of nitric acid of 1·20 sp. gr., evaporated to dryness, taken up in 30 c.c. of dilute hydrochloric acid (equal parts of water and acid), transferred to a 200 c.c. separating funnel, and one or two drops of bromine and 40 c.c. of acetone added. After thoroughly mixing, 50 c.c. of ether are added and the solution violently agitated for a moment or two. The aqueous solution is then drawn into a second separating funnel and a second separation made with 50 c.c. more of ether.

The aqueous solution contains, besides the nickel, manganese and traces of iron, any chromium, aluminium, and copper that may have been present in the steel. Draw this solution from the funnel into a 600 c.c beaker, add about 300 c.c. of hot water, bring to boiling point, add 10 grms. of sodium acetate and 10 c.c. of a 10 per cent. solution of sodium ammonium phosphate, and after boiling for a moment or two, add an excess of ammonia and continue boiling for a few moments. All the metals present excepting nickel and copper are precipitated and readily filtered. No nickel is lost in this precipitate. The filtrate is made acid with hydrochloric acid and the copper precipitated with hydrogen sulphide and filtered. The filtrate is then nearly neutralised with ammonia, brought to boiling point, and the nickel precipitated as sulphide, filtered, ignited, and weighed as oxide.

When chromium is present, unless sodium acetate is used, the precipitated phosphates have a tendency to go through the filter.

The results obtained by this method are a little higher, about two-tenths of a per cent., than when acetone and bromine are not used. By the use of acetone the ether separation is more quickly made. Three recent determinations by this method gave 3·81 per cent., 3·88 per cent. and 3·87 per cent.

THE SYNTHESIS OF INDIGO.

BY J. MERRITT MATTHEWS, PH.D.

I. *Introductory.*

UNTIL within quite recent times Indigo had its chief source in the plant raised principally in India and surrounding countries and islands. If we glance into the history of the dyer's art, we find that Indigo was one of the first dyestuffs of common use; its application in India and Egypt dates from times too remote to be reached by authentic history. Its introduction into European countries was of more modern date, this being accredited to Marco Polo, who carried it back with him from his journeys in India about 1300. Previous to this time, however, Indigo dyeing had been carried out to quite an extent in Europe by the use of woad, a plant which contains a certain amount of Indigo, and is capable of being utilised in a manner similar to that of the indigo plant itself. In fact, there appears to have been quite a conflict at the time between woad and indigo, resulting in the latter triumphantly establishing its superiority. At this point it may be well to call attention to the fact, in view of the present conflict between natural and synthetic Indigo, that Indigo, on its first introduction into Europe, was anathematised as a pernicious drug, and was spoken of as the "devil's food"; and furthermore, was regarded as a very inferior dyestuff, by no means the equal to woad in fastness of colour nor beauty of shade. In fact, so bitterly was its adoption opposed that laws were passed in several countries forbidding its use. However, as the natural law of the survival of the fittest will overcome all statutory edicts, true superiority must in time work out its own justification.

Although Indigo has been so well known as a dyestuff from antiquity, it is interesting to find that, notwithstanding its large and almost universal use throughout the world, very little was known of its chemical nature and constitution until comparatively recent years. It is also a highly significant point that this natural product was really the starting point for the artificial colour industry. Aniline, which may be said to form the basis of the vast majority of these products, was first discovered, we are told, by Unverdorben in 1826, who obtained it by the destructive distillation of Indigo. This, indeed, is a remarkable coincidence when considered in relation to the synthetic reconstruction of this dyestuff from aniline products. In fact,

if we look into the derivation of the word aniline itself, we will find that it comes from anil (and this in turn from the Sanscrit word mila, meaning Indigo, and also signifying dark blue), the Portuguese name for Indigo; hence, etymologically considered, so called aniline dyestuffs are in reality Indigo dyestuffs. Again, it appears that Picric Acid, probably the oldest artificial dyestuff known, was first prepared by Haussmann in 1788, by the action of nitric acid on Indigo.

As Indigo has always been the most important dyestuff in practical use—its consumption probably exceeding that of any other—it is a matter of considerable surprise to find that its production has been regulated so little by what may be termed scientific supervision. The fact that it has always been in such constant demand probably accounts for this lack of care, as the Indigo producer could always find a ready market and a good price for his article, he was not much given to worrying about improvements or progress until of late years. Or, it may be, that like most natural processes, the raising of Indigo is not capable of any pronounced scientific refinement. Since the introduction of the artificial coal-tar dyestuffs, however, chemists naturally endeavoured to obtain a substitute for this all-important vegetable dyestuff, as they had in mind several considerations:—

In the first place, Indigo is comparatively difficult to obtain in a uniform condition, so many are the influences of weather, soil, season, cultivation, preparation, &c., in determining the quality and quantity of the dyestuff produced from year to year; it may be said to fluctuate with the varying whims of nature, the conditions of which are but little understood, and hence not capable of proper control.

Secondly, Indigo is a peculiar dyestuff, in that its application radically differs from all others, its dyeing being carried out by what are to be regarded as clumsy and laborious methods in comparison with the ordinary processes of dyeing.

Thirdly, the commercial side of the problem was, of course, an attractive one; Indigo dyeing was high priced, and stood for a high standard of excellence. Substitutes for this dyestuff would, in consequence, have a large demand. This striving to replace Indigo in many of its applications by cheaper products and those more easily applied, has led to the result that there are more blue dyestuffs known than any other colour, and this is especially true of those which have their chief application to cotton.

We may be sure that it was not long after the artificial dyestuffs had been firmly established, that the attention of colour chemists was focussed on Indigo, with the certain result that the chemical nature and constitution of this body was soon ascertained. The next step was, naturally, its synthesis and its preparation by this means on a commercial scale. The solution of the problem relating to the constitution of Indigo is a fascinating subject to the student of organic chemistry, and the results which have been obtained form a beautiful demonstration of the truth and validity of chemical theory and philosophy. The successful synthesis and commercial preparation of this dyestuff, which soon followed the knowledge of its chemical constitution, has been one of the most brilliant achievements of technical chemistry, and exemplifies in a most striking manner the superiority of scientific methods.

II. *The Constitution of Indigo.*

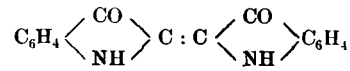
A full discussion of the chemical studies involved in the demonstration of the proper constitution of natural Indigo would be a too involved and lengthy task for the scope of this paper; but in order to understand and appreciate the working out of its synthesis, it will be necessary that we review in a cursory manner the leading points which resulted in the establishment of the chemical formula of Indigo.

Indigo, as it exists in the plant, appears to be combined in the form of a glucoside, which is known by the name of *indican*. This substance under the hydrolysing influence of acids or of an enzyme is resolved, like other natural glucosides, into a glucose, there also being formed Indigotin, which is Indigo Blue. The reaction proceeds according to the following equation:—

$$\underset{\text{Indican.}}{2C_{26}H_{31}NO_{17}} + 4H_2O = \underset{\text{Indigotin.}}{C_{16}H_{10}N_2O_2} + \underset{\text{Indiglucin.}}{6C_6H_{10}O_6}.$$

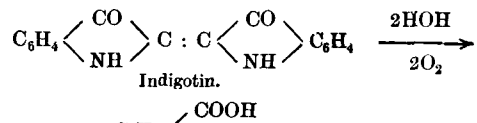
The formula of Indigo here given was established by Sommaruga by analysis and determination of its vapour density, its empirical formula, C_8H_5NO, having been previously ascertained by Crum and Laurent. In 1865 Baeyer undertook a systematic investigation in reference to the chemical constitution of Indigo, and, after fifteen years of patient labour and research, this chemist and his collaborators solved the problem, and effected the synthesis of this dyestuff. The greater part of the work was of theoretical interest, and had no industrial application, and it was not until almost twenty years more had passed that the problem received its practical solution. But it was the work of Baeyer, in ascertaining the constitutional formula of Indigo, that laid the basis of all the future chemical work with respect to this dyestuff, and for this reason it will be well to review the main points of the problem.

The constitution which Baeyer finally assigned to Indigotin was the following:—

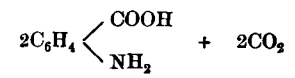


Now, bearing this in mind, we may understand how it was deduced from the following reactions and decomposition products which Indigo was found to give.

1. Indigotin is converted into anthranilic acid by oxidation in the presence of alkalis:—



Indigotin.

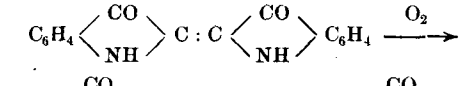


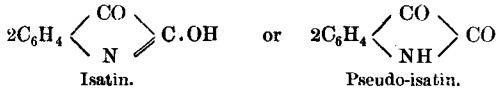
Anthranilic acid.

From this it is easy to understand the formation of aniline by the distillation of Indigo with caustic alkalis, the anthranilic acid being further decomposed:—

Aniline.

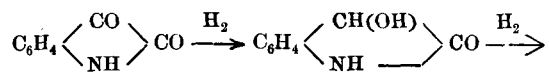
2. Isatin is formed by the oxidation of Indigo:—



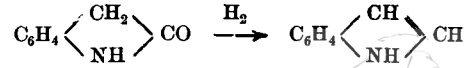


Isatin. Pseudo-isatin.

At first isatin was given the formula of its tautomeric body, but from reactions which were afterwards observed, the first formula was assigned to it. This peculiar wandering of the hydrogen atom from the imido group of the Indigo to the hydroxyl group is also to be observed in other compounds possessing the nature of inner anhydrides like isatin. The successive reduction of isatin yields in turn, dioxindole, oxindole, and finally, indole, as follows (using the tautomeric formula for isatin):—

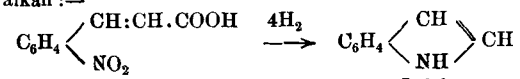


Dioxindole.



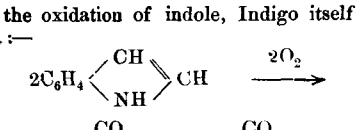
Oxindole. Indole.

3. This last body, indole, was prepared from ortho-nitro-cinnamic acid, by heating with iron filings and caustic alkali :—



o-nitrocinnamic acid. Indole.

4. By the oxidation of indole, Indigo itself was again produced :—



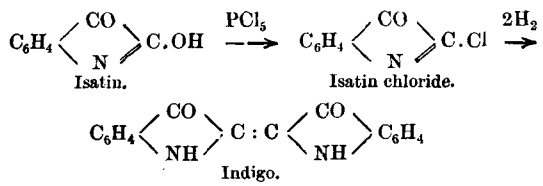
This, in fact, was one of the first syntheses of Indigo.

5. By proving oxindole to be an inner anhydride of ortho-amidophenylacetic acid, the formula of isatin was reasoned out in a logical manner; and then, from the derivation of Indigo by the reduction of isatin chloride, the formula of Indigo itself was deduced.

3. *Syntheses of Indigo.*

The first synthesis of Indigo was effected in 1870 by Baeyer, and since then numerous chemists have accomplished this by a widely-different series of processes, and the more important of these it will be our purpose to indicate.

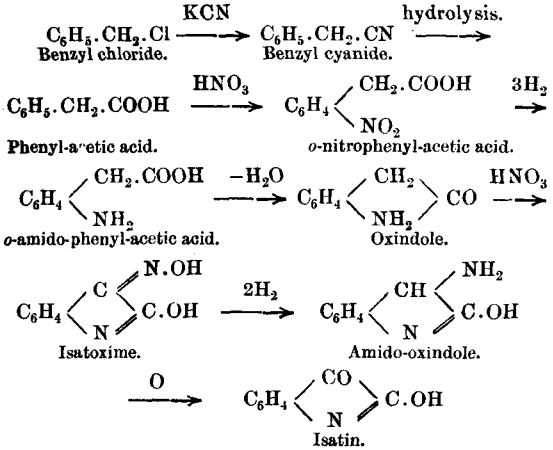
1. ***From isatin*** (Baeyer and Emmerling, 1870).—This body, treated with phosphorus pentachloride, gives isatin chloride, which on reduction is converted into Indigo :—



Isatin. Isatin chloride. Indigo.

Isatin itself was synthesised as follows :—

(*a*) ***From benzyl chloride*** (Baeyer, 1878). —When benzyl chloride is treated with potassium cyanide, benzyl cyanide is formed, which on hydrolysis with alkalis gives phenyl-acetic acid; this, by reaction with nitric acid, furnishes an ortho-nitro-compound. The latter on reduction is converted into the corresponding amido-body, which on condensation with dehydrating agents gives oxindole. Nitrous acid acting on oxindole forms a nitroso-compound, which, on reduction, is converted into the amido-body, and isatin is finally formed by the oxidation of this latter compound :—



Benzyl chloride. Benzyl cyanide.

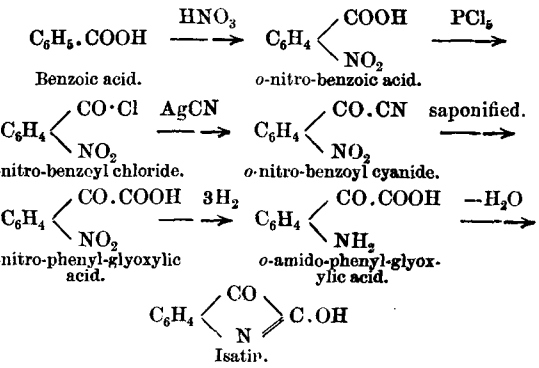
Phenyl-acetic acid. *o*-nitrophenyl-acetic acid.

o-amido-phenyl-acetic acid. Oxindole.

Isatoxime. Amido-oxindole.

Isatin.

(*b*) ***From benzoic acid*** (Claisen and Shadwell).—Benzoic acid yields on nitration an ortho-nitro-derivative; by the action of phosphorus penta chloride on this, the corresponding nitro-benzoyl chloride is produced. The chlorine is replaced by the cyanide radicle by means of silver cyanide, and then on saponification the nitro-phenyl-glyoxylic acid is formed. This latter body on reduction is converted into the amido acid, which condenses with the elimination of a molecule of water to form isatin :—



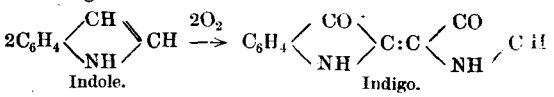
Benzoic acid. *o*-nitro-benzoic acid.

o-nitro-benzoyl chloride. *o*-nitro-benzoyl cyanide.

o-nitro-phenyl-glyoxylic acid. *o*-amido-phenyl-glyoxylic acid.

Isatin.

(2) ***From indole*** (Nencki, 1875).—When indole is oxidised by means of ozone under suitable conditions, it is converted into Indigo :—

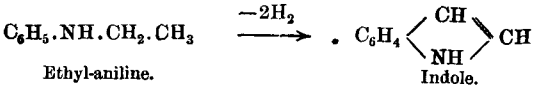


Indole. Indigo.

Indole itself was synthesised as follows :—

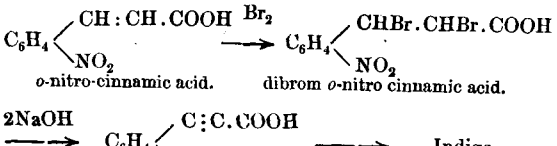
(*a*) ***From ortho-nitro-cinnamic acid***, as has already been indicated above.

(*b*) ***From ethyl-aniline*** (Baeyer and Caro, 1877).—The vapours of ethyl-aniline, on being passed through a red-hot tube, condense to the closed indole ring :—



Ethyl-aniline. Indole.

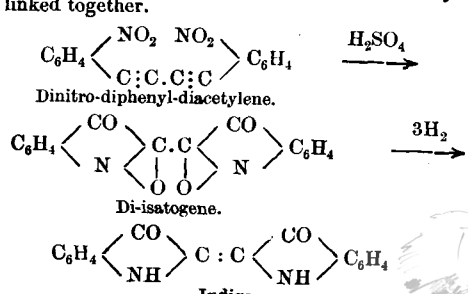
(3) ***From ortho-nitro-cinnamic acid*** (Baeyer, 1880).—This was the first synthesis which was patented. The propiolic acid compound was also used in printing in connection with sodium xanthate for the production of Indigo directly on the fibre. Ortho-nitro-cinnamic acid, being a body containing two unsaturated carbon atoms, combines with two atoms of bromine forming a dibrom-derivative. This latter compound, on treatment with caustic alkali, loses two molecules of hydrogen bromide, whereby the corresponding phenyl-propiolic acid is produced; and this substance, on being warmed with an alkali, is converted into Indigo :—



o-nitro-cinnamic acid. dibrom *o*-nitro cinnamic acid.

o-nitro-phenyl-propiolic acid.

(4) ***From dinitro-diphenyl-diacetylene*** (Baeyer).—The production of Indigo from this body furnished chemists with a proof that all of the carbon atoms in this dyestuff were linked together.

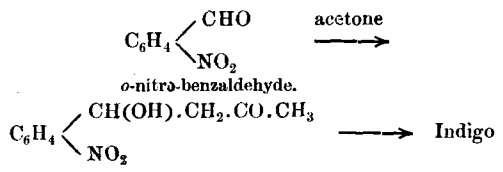


Dinitro-diphenyl-diacetylene.

Di-isatogene.

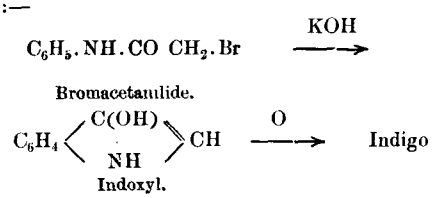
Indigo.

(5) *From ortho-nitro-benzaldehyde* (Baeyer and Drewsen).—When acetone is allowed to condense with nitrobenzaldehyde, the usual ketone body is formed, which in turn suffers an inner condensation with the elimination of a molecule of water and the splitting off of acetic acid, resulting in the formation of Indigo :—



o-nitro-benzaldehyde.

(6) *From brom-acetanilide* (Flimm) (this Journal, 1890, 281).—This body is formed by the action of aniline on bromacetyl bromide; by treatment with caustic alkalis it is converted into indoxyl, which on oxidation gives rise to Indigo :—

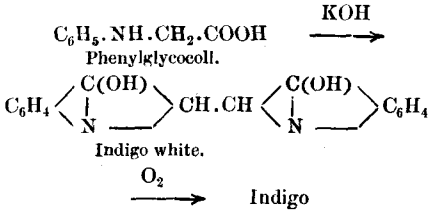


Bromacetanilide.

Indoxyl.

In carrying out this reaction, it is recommended to heat equal parts of bromacetanilide and caustic potash, until a homogeneous melt is obtained. This is dissolved in dilute acid, and ferric chloride is added, which causes the precipitation of Indigo. The yield of the dyestuff, however, is very small, being only about 4 per cent.

(7) *From phenyl amido-acetic-acid* (Heumann) (this Journal, 1890, 1121).—This substance, otherwise known as phenyl-glycocoll, is fused with two parts of caustic potash out of contact with the air. After cooling, the melt is dissolved in water, and a current of air is conducted through the solution, which causes the precipitation of pure Indigo. By using phenyl-glycocoll-*o*-carboxylic acid, the Indigo is formed at a lower temperature of fusion. In the reaction it is probable that Indigo-white is first formed, and this body, by the oxidising influence of the air, is converted into Indigo :—

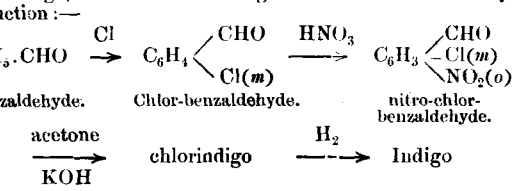


Phenylglycocoll.

Indigo white.

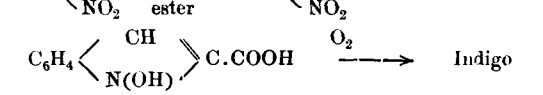
A variation of this synthesis, is to heat a mixture of equal molecules of aniline and monochloracetic acid, with three to four parts of caustic soda until the fusion becomes orange in colour. The melt is dissolved in water and a current of air is passed through the solution, whereon Indigo is precipitated. The yield is about 9·5 per cent. In this synthesis the aniline and chloracetic acid first react to form phenyl-amido-acetic acid, which is then converted into Indigo as already indicated above (this Journal, 1891, 827). By the action of fuming sulphuric acid on phenylglycocoll, Indigo Carmine may be formed; one part of phenylglycocoll is mixed with 20 parts of sand and then added to 20 parts of sulphuric acid, containing 80 per cent. of anhydride, the temperature being kept below 30° C. A yellow liquid is obtained, which is mixed with sufficient concentrated sulphuric acid to combine with the excess of sulphuric anhydride present. The liquid assumes a blue colour, and yields Indigo Carmine on being salted out. The yield is about 60 per cent.

(8) *From Benzaldehyde* (Muller, 1883).—The chlorination of benzaldehyde yields meta-chlorbenzaldehyde; on nitration this yields the ortho-nitro-derivative, which on heating with acetone and caustic potash, is converted into chlorindigo, from which Indigo itself is obtained by reduction :—

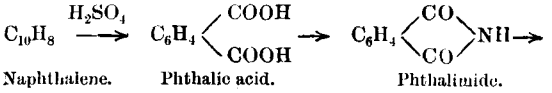


Benzaldehyde. Chlor-benzaldehyde. nitro-chlor-benzaldehyde.

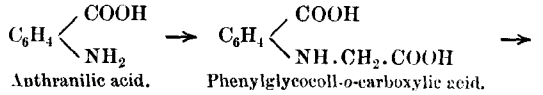
(9) *From Nitrotoluene* (Reissert).—On heating ortho-nitrotoluene with oxalic ester a nitrophenyl-aceto-formic acid is formed; on reduction this yields an inner anhydride which on oxidation gives Indigo. The yield is said to be 23 per cent. :—



(10) *From Naphthalene.*—This synthesis represents the latest development of the subject, and is the one on which is principally based the commercial success of synthetic Indigo. It really includes Heumann's process of using phenyl-glycocoll-*o*-carboxylic acid as has been described above, but it derives this latter body from naphthalene in a manner that allows of its practical commercial application. It has been found that if naphthalene be treated with fuming sulphuric acid it is decomposed into phthalic acid; this body is then converted into phthalimide, and thence into anthranilic acid. By the action of chloracetic acid the latter is transformed into the phenylglycocoll compound, from which Indigo is then made in the manner already shown—



Naphthalene. Phthalic acid. Phthalimide.



Anthranilic acid. Phenylglycocoll-*o*-carboxylic acid.

The early syntheses of Indigo were of little or no value to the manufacturer, as the yield of dyestuff was insufficient, and the materials employed for its production were too expensive to allow of its being manufactured on a commercial scale, in competition with the natural dyestuff. At first, the most promising synthesis appeared to be that which used ortho-nitrobenzaldehyde as a starting point, and the methods for making this compound were so highly perfected, as to bring it within the limit of cost for the industrial preparation of Indigo. But at this point another difficulty arose which was impossible to surmount. The raw material for the preparation of the nitrobenzaldehyde is toluene, and it appears that for every part of Indigo finally produced by this method, there is required at the start four parts of toluene. As the annual consumption of Indigo for the entire world is estimated at about 11,000,000 pounds, and as the total production of toluene itself does not much surpass this figure, it means that the world's output of toluene would have to be quadrupled in order to satisfy the demand of this chemical for the manufacture of Indigo alone, to say nothing of its other uses, supposing that in order to successfully compete with the natural Indigo the artificial product must be capable of being made in a quantity sufficient to supply the world's present demand for that dyestuff. As toluene is derived from the coal-tar industry in connection with benzol, and since every part of toluene obtained means also four parts of benzol, it would be necessary for the manufacturer to create a demand for

that extra amount of the latter product in order to make the toluene commercially available, and such a task would be far greater even than making Indigo. It was then that the derivation of the phenylglycocoll compound was discovered from naphthalene, and the known method of making phthalic acid by oxidation with fuming sulphuric acid was made use of. This method furnished a raw material which was both cheap and abundant; in fact, there was a surplus of naphthalene, and a quantity sufficient to supply all the demands of the Indigo trade. It is interesting to note that in this connection, another important development in industrial chemistry was effected, and one which will eventually have considerable influence on other lines of work besides synthetic Indigo. It was found that the lead chamber process for the manufacture of sulphuric acid was not adequate to the production of the enormous quantities of fuming acid required for the preparation of the phthalic acid from naphthalene, so the new sulphuric acid process, known as the "contact" method, was perfected. The Badische Company, to whom this valuable work was due, have become in consequence, the largest producers of sulphuric acid in the world, and are using 50,000 tons of the fuming acid annually for the preparation of their phthalic acid. In the manufacture of the other chief chemical employed, the monochloracetic acid, large quantities of pure chlorine were demanded. The processes of Weldon and Deacon proved to be unsatisfactory, and the attention of the manufacturers was turned to the new electrolytic process for the preparation of chlorine from alkali chlorides, with the result that the method was so far perfected that the necessary conditions were realised. Hence the chlorine is now prepared electrolytically, and is used in a liquid condition.

It may be said that synthetic Indigo became a commercial reality in 1897, for it was in that year that it began to appear in sufficient quantity to seriously compete with the natural dyestuff. Since then the amount manufactured has been growing with great rapidity—a fact which may be made apparent by a glance at the following statistics :—

Imports of Natural Indigo to Germany.

	Lb.
First six months of 1899	1,771,805
„ „ 1900	961,837

showing a decrease of about 45 per cent.

Exports of Synthetic Indigo from Germany.

	Lb.
First six months of 1899	1,191,700
„ „ 1900	2,085,447

showing an increase of about 75 per cent. Of the above amount of synthetic Indigo exported from Germany during the first six months of 1900, the United States took 695,149 lbs.

Imports of Natural Indigo to England.

—	Lb.	Value, Dols.
In 1899	5,897,700	4,930,450
„ 1900	3,351,800	2,610,445

showing a decrease of about 40 per cent. in one year.

According to a recent publication of the Indian Government, the exports of Indigo from the provinces of Calcutta and Madras have been as follows :—

	Lb.
1895—1896.....	17,242,000
1899—1900.....	9,777,900

showing a decrease of about 43 per cent.

These figures are very significant in their meaning. We can but behold in them the rapid extinction of the natural Indigo industry, which forms one of the bulwarks of eastern trade and commercial activity. It is the case of the madder root *versus* synthetic Alizarin over again, and we can have no doubt as to the outcome of the strife. It is merely one more step in the progress of industrial chemistry along the lines of human ingenuity, and this problem having been solved, the time is ripe for the development of other fields.

ERRATUM.—This Journal, May 1901, page 445, col. 2, line 30 from top: after "Ceylon" insert "graphite."

Nottingham Section.

Meeting held at University College, on Wednesday, June 5th, 1901.

PROF. F. S. KIPPING, F.R.S., IN THE CHAIR.

THE CONSTITUTION OF GLASS.

BY W. JACKSON, A.R.C.S., AND E. M. RICH, B.SC.

THE constitution of glass has been the subject of a great deal of work at different times by many workers. The result of these labours is that there are several views held on the subject. Sometimes one hears the chemical formula of a glass used with the same degree of assurance as that of sulphuric acid, the inference being that a glass may be considered as a definite chemical compound.

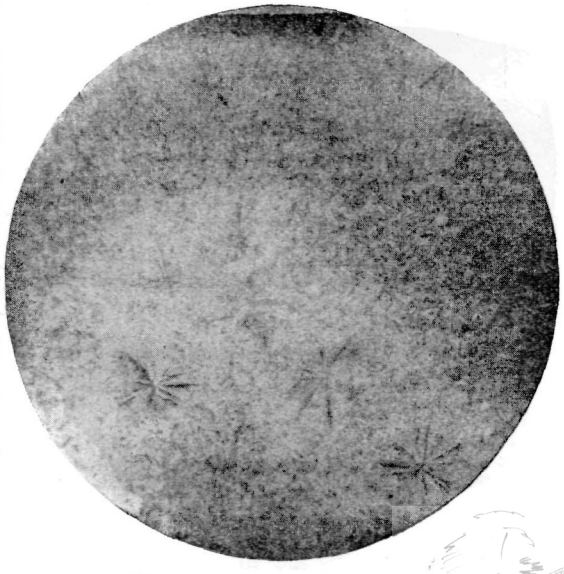
The view that glasses are "mutual solutions" of silicates has found much favour. To quote Benrath (Die Glasfabrikation), p. 25: —

"If one considers that the contents of silica in commercial glasses is very variable, and that at a higher temperature more of this acid can be taken up than can be retained by the glass as the temperature falls; that further, felspathic minerals may be taken up and again separated by a molten glass; finally, also sulphates, phosphates, borates, metallic oxides and metals (gold) may enter into the constitution of glasses, it would appear that these facts allow of no other explanation than that commercial glasses are quickly solidified solutions, not only of different silicates in each other, but even solutions of basic oxides or silica and other salts and metals, in molten silicate. Whether in all glasses some fundamental silicate, a compound, say—

$$Na_2O.CaO.6SiO_2 \text{ or } Na_2O.CaO.4SiO_2,$$

is to be taken as the solvent or not, must remain a question so long as nothing more definite is known than at present;

Fig. A.



NEGATIVE GLASS. Etched × 50

yet many of the observations appear to speak for such an assumption."

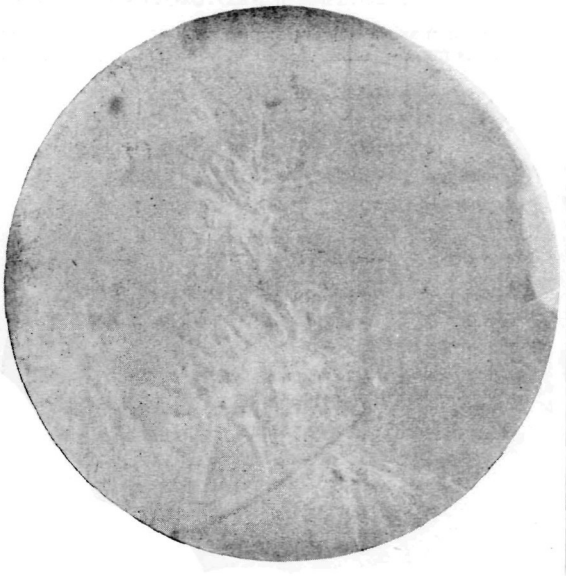
On the other hand, Dumas (Comptes Rend. 1855, **40**, 321), speaks of glass as being "an indefinite mixture of definite silicates"; and in support of this contention many

Fig. B.

CLOCK GLASS. Etched × 50.

acts may be cited. The phenomena of devitrification afford strong evidence; for it has been shown that this change in the character of a glass is due to crystallisation of one or more of its constituents. To quote again from Benrath (*ibid.* p. 20): "It appears that the composition of the

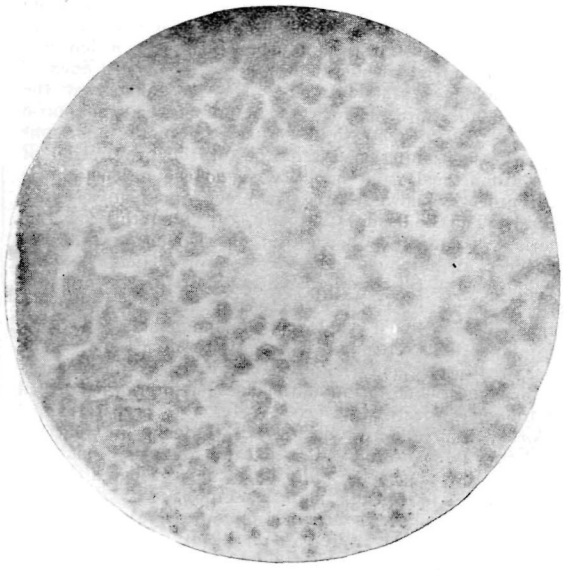
Fig. C.



SHEET GLASS (Chance Bros.). Etched × 50.

crystalline portion of a devitrified glass differs from the amorphous matrix. The difference is, however, not great; nor could one expect it to be so, for the devitrified glass consists almost entirely of an entanglement of fine needles or six-sided plates which do not lie in close contact. The spaces between are filled with the undevitrified matrix, which we may consider as the solidified mother liquor of the separated crystals. A mechanical separation of these portions is not possible, and so the analysis of a mixture of at least two unknown bodies gives no clear insight into the constitution of either."

Fig. D.

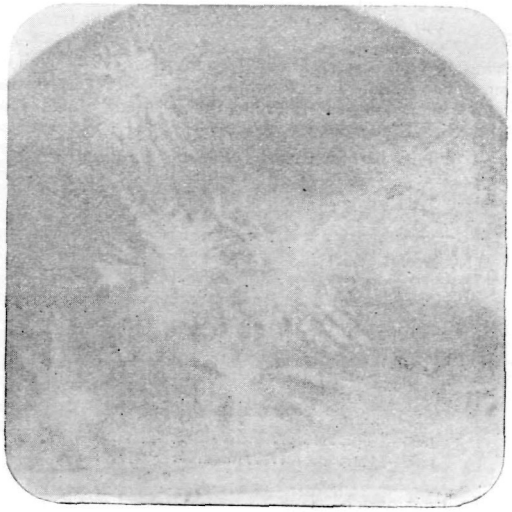


SHEET GLASS (Chance Bros.). Etched, dil. HF. × 50.

By suitable chemical means—fractional solution—it has been possible to separate a devitrified glass into distinct constituents. In one case free silica, and in another felspathic minerals have been recognised.

It would thus appear that a devitrified glass is a heterogeneous mixture, and while support is lent to the theory that the original undevitrified material is likewise a

Fig. E.



WATCH GLASS. Etched × 50. STARS.

mixture it cannot be accepted as conclusive proof. One can conceive the formation of such a heterogeneous mixture, when a glass is devitrified by being kept for a long

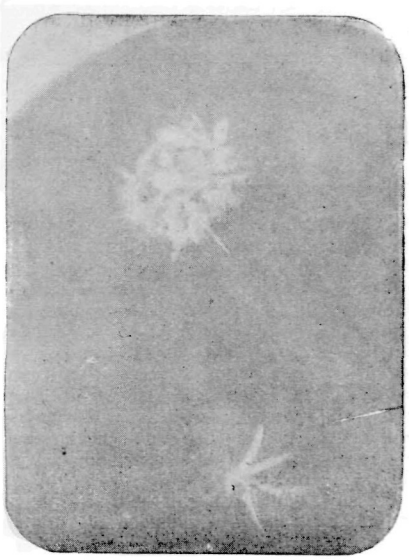
time at a high temperature, on the assumption that one is dealing with a mixture, more or less homogeneous, and that an agglomeration or segregation of the constituents takes place giving rise to crystalline forms. But on the other hand it is also conceivable that there is a real solution, as described by Benrath, supersaturated at a high

Fig. F.

[Watch Glass. Etched × 50. Rod.

temperature with some definite silicate or other compound which gradually crystallises out. Hence the facts of devitrification may lend support to either theory

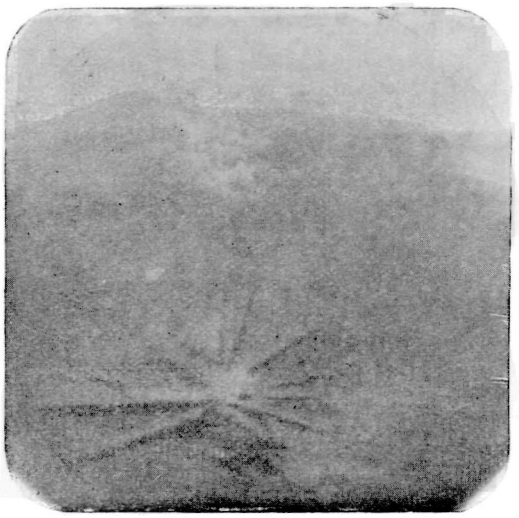
Fig. G.



Hard Crown (Chance Bros.). Etched × 50.

Work by Leydholt (Comptes Rend., 1852, **34**, 565), is of interest in this connection. He found that if a clear surface of glass, in the condition in which it solidified from fusion, be subjected to the action of concentrated hydrofluoric acid it is etched, and microscopic crystalline forms may be detected. Such forms are clearly indicated in the micro-photographs shown below, and which have been prepared according to Leydholt's method. It will be seen that common sheet glass (Chance Bros.), the glass of a

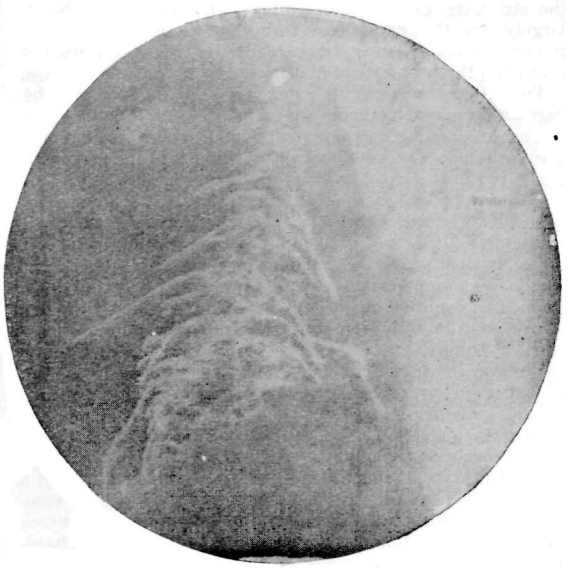
Fig. H.



Hard Crown (Chance Bros.). × 50.

photographic negative and cover-glasses used in the laboratory all show well-developed crystal forms or at any rate forms strikingly similar to those detected in sections of obsidian, pitchstones, &c., under the microscope. In every case the forms are starshaped, or rods with crystalline radiations on both sides, and are particularly well developed

Fig. K.



Dense Flint (Chance Bros.). Etched × 50.

in the glass of a sensitive plate. There is also shown the appearance of the glass surface when acted upon by dilute hydrofluoric acid. In this case the surface is irregularly broken up, with no sign of crystalline structure.

The forms developed on the surface of a lead glass are very different. In every case examined—dense optical flint glass, light optical flint glass and soft crown glass (containing oxide of lead) from Messrs. Chance Bros., the same characteristic forms have been developed with no trace of

Fig. L.

Light Flint (Chance Bros.). Etched × 50.

the forms common in ordinary glasses or optical glass free from lead. They consist of sets of parallel-curved filaments extending on both sides of an axis to varying distances. In different glasses the curves are surprisingly similar and may in many cases be actually superposed. It would hence appear that the formation is not due to accidental irregularity in texture of the glasses.

It would thus appear that the ideas now prevalent as to the structure of steels and alloys, and which are based largely on the crystalline forms developed by etching polished surfaces, must be extended to glass, for we see that even the best optical glass presents similar phenomena.

To further elucidate this point it would appear that the regular chemical methods are bound to fail, for the only means of opening up these compounds result in their decomposition. The proximate constituents of the glass are destroyed in the effort to separate them.

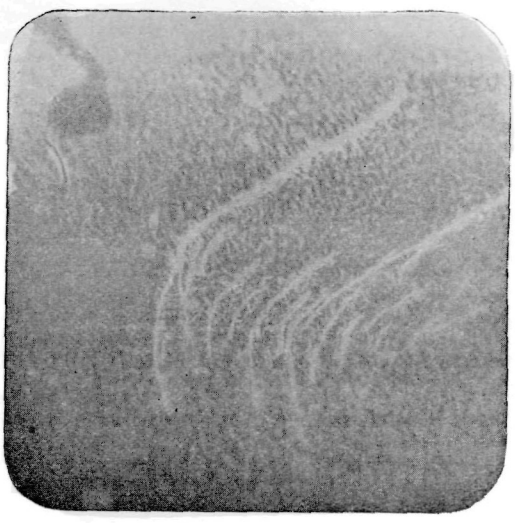
Certain physical methods, however, seemed to us worthy of trial. We argued that if glass is a definite chemical compound all parts of it should be possessed of exactly the same physical properties, no matter how small the pieces of glass examined may be. The same would be expected if glass is a "mutual solution" of silicates. Applying the term "solution" in its strict sense, the substances are in such a condition that unless the degree of division is molecular in its fineness, all particles must be possessed of the same properties, and have the same composition. Hence, whether glass is a definite chemical compound or a mutual solution, physical methods would be expected to fail in bringing about a separation of the material into distinct proximate constituents. If, however, glass is but a more or less uniform mechanical mixture of various compounds, it appeared to us that a physical method of attack might be successful. Differences in specific gravity, hardness, and toughness, would be likely to exist between the different members of the mixture, and taking advantage of these differences it might be possible to effect at any rate a partial separation.

Experiments on these lines have been carried out, and the results seem to show that the theory of mechanical mixture is likely to be correct. Necessarily the homogeneity of the mixture will be different in different glasses, according to the processes adopted in the manufacture. The more complete and prolonged the fusion of the batch, and the more vigorous the agitation of the glass, the greater will be the homogeneity of the mixture. With the very best glasses, for instance, optical glasses, therefore one would not expect to find this physical method of separation to be so successful as with commoner glasses. Yet the circumstance that a separation in such a case might be impossible would not necessarily condemn the theory. The fact would be indicated that the components have been so intimately mixed, or so finely scattered through each other, that physical means fail to get between them for their separation.

Faraday, by examining the higher and the lower portions of a flint glass batch, has been able to show a difference in chemical composition. The lower layer of glass was richer in lead oxide, and therefore heavier, than the upper layer.

In our experiments, however, we have sought to determine whether such difference might not also be detected in different portions of a small piece of glass, not exceeding 2 c.c. in volume. In Faraday's experiments the differences

Fig. M.



Soft Crown (Chance Bros.). Etched × 56.

would be explained by assuming the heaviest portion of the batch had settled out. Using, however, such a small volume of glass as we have used, this settling is out of the question.

The problem was investigated as follows:—

Several lead fritts—which are glasses used by potters for glaze-making—and which were quite clear except for the presence of a few small bubbles, were ground in a mortar and the lightest or finest material was floated off from time to time with water into a Schone's Elutriation Apparatus. From this ground material the finest and lightest particles were separated by a current of water having a velocity of about 0·7 m.m. per sec. The maximum diameter of the particles would not exceed 0·01 m.m. The portion carried over by the water and the coarser remainder in the apparatus were dried at 100° C. and the contents of lead oxide determined. The method of analysis was as follows. The fritt (0·5 grm.) was decomposed at a gentle heat in a platinum dish with 5 c.c. of re-distilled hydrofluoric acid. A few drops of concentrated sulphuric acid were added and the whole evaporated to dryness and gently ignited until all silicon fluoride and acid fumes were driven off. The residue was moistened with hydrofluoric acid and again evaporated with sulphuric acid to ensure complete decomposition. The residual sulphates were dissolved in dilute hydrochloric acid and the lead precipitated as sulphide by sulphuretted hydrogen. This was filtered, washed, and

ignited in a porcelain crucible with a drop of nitric and sulphuric acids and weighed as lead sulphate.

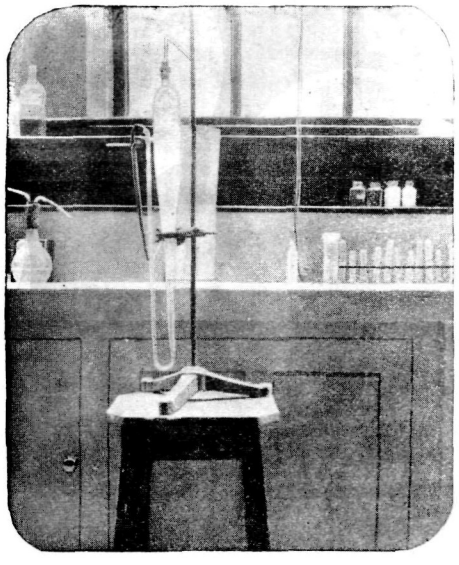
The following table shows the results obtained with various fritts :—

Material Carried over by Velocity of Water 0·7 mm. per Second.	Material left in Elutriation Apparatus.
Per Cent. PbO.	Per Cent. PbO.
32·24	33·9
32·69	35·24
31·92	34·8
31·26	35·5
30·5	34·9
47·7	48·6
30·4	33·8
31·1	37·38

From these results it is plain that in every case the finest portion has a lower percentage of lead oxide than the remainder. The average difference is about 10 per cent. of the lead contents of the fine fraction.

These variations in the analyses may be due either to real differences in the composition of the two portions of the fritts or to contamination of the finely ground portions with material ground off the mortar and pestle used in their preparation. In the latter case the contamination would be in an exceedingly fine condition and would therefore be found associated with the finest fractions of the fritts, in which it would cause a reduction of the lead contents. This point was examined as follows :—One of the fritts was ground in a weighed agate mortar and the finest portion floated off with distilled water. This and the coarse remainder were dried, and the lead oxide present was found to be 31·6 per cent. in fine fraction, 34·6 per cent. in coarse fraction. No loss of weight was observed when the

Fig. 1.

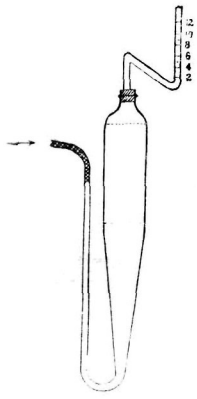


View of Elutriation Apparatus.

agate mortar and pestle were re-weighed. Hence it appears established that the finest portions were not contaminated by material from the mortar, and that therefore there is a real difference in the chemical composition of different portions of these glasses when separated in this way.

The physical properties taken advantage of are probably toughness and hardness—which would affect the ease with which fine material may be obtained—and also specific gravity. In fact it would appear to us that the last-mentioned property may have been the most important from this point of view; for decrease in the contents of lead oxide in a glass is associated with a decrease of the specific gravity. [Except of course in such as thallium glasses containing elements of greater atomic weight than lead]. Hence, if all the particles of glass submitted to elutriation were of the same size and shape, such as possessed the lowest lead contents would be the first to be carried over. This agrees entirely with the results obtained. We doubt, however, whether the specific gravity was the sole property taken advantage of; for it is well known that glasses vary in elasticity or resiliency according to lead contents. The glasses containing little or no lead oxide are more brittle than those with higher contents of that oxide. Hence it might be easier to obtain a fine powder from the more brittle constituents of a glass than from the tougher. It is, hence, difficult to decide whether the specific gravity, the toughness or both properties were the agents of the separation.

Fig. 2.



Elutriation Apparatus. Sectional Diagram.

The following are the complete analyses of the two portions into which one of the fritts used was divided :—

——	Finest or Lightest Portion.	Coarse Residue.
Silica	48·78	46·06
Lead oxide	31·00	34·10
Alumina and ferric oxide	6·82	6·00
Lime	11·12	11·46
Alkalis	2·28	2·36
	100	100

These results may be expressed in formulæ as follows :—

$$\text{Fine...} \left\{ \begin{matrix} 0{\cdot}37\ PbO \\ 0{\cdot}53\ CaO \\ 0{\cdot}10\ (NaK)_2O \end{matrix} \right\} 0{\cdot}018\ Al_2O_3 . 2{\cdot}16\ SiO_2.$$

$$\text{Coarse} \left\{ \begin{matrix} 0{\cdot}38\ PbO \\ 0{\cdot}52\ CaO \\ 0{\cdot}10\ (NaK)_2O \end{matrix} \right\} 0{\cdot}015\ Al_2O_3 . 1{\cdot}96\ SiO_2.$$

From these formulæ it would appear that the finest portion is almost exactly a disilicate. Assuming Al_2O_3 to be a base, the ratio of basic to acidic oxygen is 1 : 4·09, whereas for a disilicate the ratio should be 1 : 4·00.

It seems therefore to be established that potters' lead fritts consist merely of a more or less heterogeneous mixture of at least two distinct compounds.

Our attention was now turned to commercial glass, for it appeared to us that what was found to be true of fritts might be found to apply, to some extent at any rate, to ordinary glass.

The first glass examined was a piece of a clock glass. It was prepared by grinding and elutriation as previously described. The fine and coarse fractions gave the following analyses :—

——	Fine.	Coarse.
Silica	71·03	71·18
Alumina and ferric oxide	3·54	2·00
Lime	16·23	15·08
Magnesia	0·72	0·65
Alkalis	8·68	11·83
	100·2	100·74

The equivalent of the alkalis was 33·25.

These figures give the following chemical formulæ :—

$$\text{Fine...} \left\{ \begin{matrix} 0{\cdot}66\ CaO \\ 0{\cdot}04\ MgO \\ 0{\cdot}30\ (NaK)_2O \end{matrix} \right\} 0{\cdot}08\ Al_2O_3 . 2{\cdot}70\ SiO_2.$$

$$\text{Coarse} \left\{ \begin{matrix} 0{\cdot}584\ CaO \\ 0{\cdot}034\ MgO \\ 0{\cdot}382\ (NaK)_2O \end{matrix} \right\} 0{\cdot}04\ Al_2O_3 . 2{\cdot}56\ SiO_2.$$

The fine part is richer in the lime and silica principally.

Daubrée (Ann. des Mines, V. Serie, 1857, **12**, 297) has shown that by digestion in a sealed tube with superheated steam at a high temperature, ordinary glass is decomposed into quartz, wollastonite, and alkaline silicate. It would appear that a similar separation has been attained by our physical method. It can be shown that the fine part may be considered to consist of 76 per cent. of the original glass, 10·7 per cent. of wollastonite or similar minerals, 12·5 per cent. of free silica. (See Appendix.)

Similar experiments have been carried out with English flint glass. Results as follows :—

——	Finest or Lightest Fraction.	Coarse Fraction.
Silica	55·94	53·74
Lead oxide	33·70	34·50
Alumina and ferric oxide	0·90	0·48
Alkali	9·44	11·24
	100·00	100·00

Equivalent of alkalis: fine, 37·5; coarse, 43.

and these give the formulæ—

$$\text{Fine or light} \left\{ \begin{matrix} 0{\cdot}54\ PbO \\ 0{\cdot}46\ (NaK)_2O \end{matrix} \right\} 3{\cdot}35\ SiO_2.$$

$$\text{Coarse or heavy} \left\{ \begin{matrix} 0{\cdot}542\ PbO \\ 0{\cdot}458\ (NaK)_2O \end{matrix} \right\} 3{\cdot}13\ SiO_2.$$

The difference appears to be in the silica contents.

But here, again, one cannot say what particular property has been taken advantage of. Mr. W. J. Pope, to whom our best thanks are due, suggested that we should try the effect of methylene iodide on our separation and so get rid of all effects but those of variation in specific gravity. The glass was finely powdered and was then placed in methylene iodide in which it floated. Benzene was added until some portion of the glass settled out. The liquid and suspended glass were then decanted. More benzene was now added to the suspension until the whole of the glass settled out, then methylene iodide until some portion was re-suspended. This was decanted and constituted the lightest fraction of the glass. On analysis the light portion was the same as that separated by elutriation, but the heavy part had the following composition :—

Heavy Flint Glass.

Silica	52·96
Lead oxide	35·03
Alumina and ferric oxide	0·78
Alkalis	11·23
	100·00

Formula :—

$$\left\{ \begin{matrix} 0{\cdot}547\ PbO \\ 0{\cdot}453\ (NaK)_2O \end{matrix} \right\} 3{\cdot}06\ SiO_2,$$

from which it seems the heaviest constituent of the flint glass is practically a trisilicate.

That this glass as a whole is not a definite compound nor a mutual solution of silicates seems to be plain; for it will be observed, on comparison of the formulæ, that the ratio of the bases to each other is nearly the same in all three cases (*viz.*, 1·207; 1·195; 1·181), but the silica contents vary much more. The degrees of saturation are 3·35, 3·13, 3·06 in the lightest fraction, the glass as a whole, and the heaviest fraction respectively. It would seem that the lightest fraction may contain 95 per cent. of heaviest fraction, with 5 per cent. of free silica.

The specific gravities of the fine portion and the glass as a whole were 3·14 and 3·19 respectively.

We have attempted to get a similar separation of the constituents of certain optical glasses received from Messrs. Chance, both the methylene iodide method and elutriation being adopted. But to the present we have not succeeded in getting any appreciable difference in the various fractions. Though the development of crystalline form on the surface of these glasses by the action of hydrofluoric acid betokens some want of homogeneity, it is not sufficiently pronounced to enable our method to detect it. This fact was, however, quite in accordance with our expectations on the grounds previously mentioned.

It would hence appear that glass must be considered to be a mixture of various silicates and oxides more or less homogeneous according to the method of manufacture. In ordinary crown and flint glass the mixture is sufficiently imperfect to allow the constituents to be readily separated by grinding; but the optical glasses show a much greater degree of homogeneity. The particles of the individual constituents forming the mixture appear to have been reduced to such a small size, and to be so uniformly distributed by the processes of manufacture, that grinding fails to separate them. Still, we can conceive that by some refinement of the method we have adopted, it may be possible, even in these cases, to bring about a separation or fractionation of the mixture. Possibly, however, the degree of sub-division of the constituents may be molecular. If so, the hope of effecting physical separation would have to be abandoned.

As to whether such a molecularly fine mixture is to be considered a "solution" or merely a mechanical mixture, is a question we will leave to the physicist.

Appendix.

Clock glass :—

$$\text{Fine portion} \left\{ \begin{matrix} 0{\cdot}66\ CaO \\ 0{\cdot}04\ MgO \\ 0{\cdot}30(NaK)_2O \end{matrix} \right\} 0{\cdot}08 Al_2O_3 . 2{\cdot}70 SiO_2 \quad (1)$$

$$\text{Coarse portion} \left\{ \begin{matrix} 0{\cdot}584\ CaO \\ 0{\cdot}034\ MgO \\ 0{\cdot}382(NaK)_2O \end{matrix} \right\} 0{\cdot}04 Al_2O_3 . 2{\cdot}56 SiO_2 . (2)$$

Multiplying each coefficient in formula for fine by $\frac{382}{300}$, so as to obtain a formula having 0·382 $(NaK)_2O$, they become—

$$\left\{ \begin{matrix} 0{\cdot}84\ CaO \\ 0{\cdot}05\ MgO \\ 0{\cdot}382\ (NaK)_2O \end{matrix} \right\} 0{\cdot}1 Al_2O_3 . 3{\cdot}438\ SiO_2 \quad (3)$$

Deducting the formula of coarse (2) from (3), the result is—

$$\left\{ \begin{matrix} 0{\cdot}256\ CaO \\ 0{\cdot}016\ MgO \end{matrix} \right\} 0{\cdot}06\ Al_2O_3 : 0{\cdot}878\ SiO_2,$$

which may be represented as, molecularly,—

0·27 (CaMg)O . SiO_2 Wollastonite or similar pyroxenic mineral)

0·60 free silica,

or, in percentages,—

76 per cent. of glass as a whole,
10·7 per cent. of pyroxenic mineral,
12·5 per cent. of free silica.

The small amount of alumina is neglected.

NINTH JUBILEE OF THE UNIVERSITY OF GLASGOW.

The celebration of the 450th Anniversary of the foundation of the University of Glasgow took place on June 12th last and following days. On next page will be found a reproduction of the address presented on behalf of the

Council of this Society by Mr. George Beilby and Prof. G. G. Henderson, D.Sc., the appointed delegates on this occasion. The address was engrossed on vellum by Mr. W. Graily Hewitt of the County Council School of Arts and Crafts, 316, Regent Street, London, W. The large capitals and scrolls were in raised gold.

TO THE CHANCELLOR, THE COURT, AND THE SENATE OF THE UNIVERSITY OF GLASGOW

THE President & Council of the Society of Chemical Industry offer to the University of Glasgow their most hearty congratulations on the attainment of its Ninth Jubilee & they have nominated, to represent them on this happy occasion, George T. Beilby, Past President & George G. Henderson, D.Sc., Vice President.

AT no time in the history of experimental Science has there been any hard & fast line dividing Science from its applications, & the President & Council of the Society of Chemical Industry recognise with pride that among the sons of the University of Glasgow have been found some of the most brilliant examples the world has seen, of the genius, which, while it aims after & grasps the highest generalisations, is, at the same time, intensely alive to the bearing of scientific laws & analogies on the improvement of the conditions of daily life & work. As the home of such men of genius as Kelvin, Lister, or Watt, the University of Glasgow arouses the sympathy & admiration of all followers of applied Science, a sympathy and admiration which the President & Council of this Society feel they can confidently express for the followers of applied Chemistry all over the world.

WHILE men of like genius have appeared at all periods in the history of this & other universities, the organised & systematic application of Science to Industry is essentially a modern development, which is still far from complete. To the University of Glasgow belongs the credit of having established the first Chair of Engineering Science; & the pioneer work of Rankin, the second holder of that Chair, in research as well as in teaching, has had a very important influence on the later developments of the teaching of Engineering. It must be the earnest wish of all who are truly interested in the systematic application of Physics & Chemistry to Industry, that the University of Glasgow may in the near future become a great centre of research in these Sciences & in their application to industrial problems.

THE President & Council of the Society of Chemical Industry cannot forget that the Scottish Section of the Society owed much of its early success to the helpful energy of members of the Chemical Department of the University of Glasgow, & that on at least one occasion the whole Society has been the guest of the University. In wishing for the University continued & increasing prosperity the President & Council trust that the cordial relations existing between the University & the Society of Chemical Industry in the past may be maintained & extended, & result in a more & more complete cooperation for their common aims.

PRESIDENT

Journal and Patent* Literature.

* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office Southampton Buildings, Chancery Lane, London, W.C.

I.—PLANT, APPARATUS, AND MACHINERY.

PATENTS.

Incrustation in Steam-Generators; Mixture for Preventing ——. A. von Fritz, Vienna. Eng. Pat. 19,339, Oct. 29, 1900.

A MIXTURE is made of about 30 parts of "Japan earth," 24 parts each of "barium chlorate" and of potash, and 22 parts of myrobalans, which mixture, after powdering, is digested in 40 litres of water. The insoluble residue may be separated, or stirred with the liquid before use. Or the solid mixture may be added to the feed water in the proportion of 1 kilo. to each cubic metre of the water. The "barium chlorate" may be replaced by any other barium salt.—E. S.

Stone, Artificial; Manufacture of ——, and Apparatus therefor. W. Dünkelberg, Steinhausen, Germany. Eng. Pat. 20,639, Nov. 18, 1900.

THE invention relates to a press in which the opposite sides of a block are subjected to pressure by plungers, "differentially"; the upper plunger being moved twice, whilst the lower one is moved once, or *vice versâ*.—J. W. H.

Vacuum Filters or Apparatus for Separating Liquid from Materials, and for Washing and Saturating Materials with Liquid. C. B. Symonds, Wirksworth, Derbyshire. From T. Breakell and W. Hopwood, both of Bolwar, Venezuela. Eng. Pat. 7723, April 26, 1900.

THE apparatus is applicable for separating liquids from slimy materials, such as clays, fine sands, chemical precipitates, &c., for washing such materials, or for saturating them with water or chemical solutions. It consists of a revolvable cylinder or barrel having a periphery adapted for carrying the material to be treated, means for withdrawing air and liquid from the interior of the barrel, and means for applying the material to the periphery of the barrel and for removing it therefrom. The periphery of the barrel is covered with wire gauze, felt, or the like, and a filter cloth, &c., and takes up the material from a suitably arranged container, the removal of the material from the periphery being effected by a counterbalanced scraper. The barrel is carried by a hollow perforated axle, which is connected to a suction device. Water or other liquid may be applied to the material on the periphery of the barrel, between the points where the material is taken up and removed.—R. A.

Filter Applicable for Use in Sewage Treatment, Ore Reducing, and the like Processes. J. I. Pullon, Leeds. From E. C. van Blarcom, Mexico. Eng. Pat. 7884, April 28, 1900.

A SELF-CLEARING or continuous filter, consisting of a screen cylinder carrying the filtering medium. This rotates so as to produce the desired fluid pressure. There is also a spiral scraper rotating at a different speed, to produce a scraping action on the surface of the filtering cylinder. With this there is also a hollow trunnion, forming an inlet pipe of suitable dimensions for the admission of the material to be filtered. Externally, there is an outer fixed casing and cross diaphragms suitably to direct the incoming material and to separate the solid portions from the liquid effluent.
—L. A.

Tanks, Boiling Vessels, and similar Receptacles; Manufacture of ——. L. Nobis and A. Wenzel, both of Vienna. Eng. Pat. 11,514, June 25, 1900.

THE receptacles are constructed of an iron framework covered with concrete, the framework consisting of frame bars bent to the shape of the vessel required, and two reticulated or lattice-work partitions, which are attached one to the outer side and the other to the inner side of the frame bars. Each partition consists of two layers of round, square, or flat iron bars, and these partitions are attached by inserting them in slotted strips fixed to the frame bars, the reticulations of one partition being arranged in alternation with those of the other. The partitions are also secured to the frame bars and to each other by interwoven iron or steel wire. To ensure tightness of the joints at the caps, nozzles, manhole covers, &c., the iron at these places is covered with red lead and sand, and then coated with soluble glass.—R. A.

Evaporating Vessels for Concentrating Liquids. J. Foster Glasgow. Eng. Pat. 12,190, July 6, 1900.

THE evaporating vessel, in which the liquid to be concentrated is heated by hot air or furnace gases, &c., is provided with a baffle plate adapted to deflect and retard the currents of hot air or other heating medium. The baffle plate is arranged vertically at the side of the vessel opposite to the hot-air inlet, extending across the vessel and nearly to the top and bottom thereof. A transverse rib or partition connects the baffle plate to the "down-comer" circulating duct, and the space above this partition communicates with the hot-air outlet, which the heating air or gas reaches by passing through suitably arranged apertures in the partition and baffle plate, and through the spaces above and below the latter. In a modification the baffle plate is replaced by a channel or annular space, which branches or extends towards the hot-air inlet from the opposite side of the vessel, apertures being formed in the bottom and ends of the walls of this channel for the passage of the heating medium to the outlet.—R. A.

Steam or Vapour of any Desired Pressure from Liquids Heated under Pressure but not Boiling; Process and Apparatus for Producing ——, and the Application of the Process for Concentration of the Liquids. C. Steffen, Vienna. Eng. Pat. 12,805, July 16, 1900.

THE liquid to be vaporised, while not boiling, is transferred from the vaporising space, or receptacle, at a temperature corresponding to the desired pressure, through a heater under higher pressure, where it is heated to a higher temperature, still without boiling. The liquid is then returned to the vaporising space, where, under the lower pressure maintained in such space, steam or vapour separates. The energy of the returning liquid is applied to work a motor, such as a Pelton wheel or turbine, which actuates a pump for forcing the liquid through the heater. This process may be employed for the concentration of liquids, such as sugar juice, &c. Several arrangements of apparatus for carrying out the process are described in the specification.
— R. A.

II.—FUEL, GAS, AND LIGHT.

Coals; Effect of Washing Certain Cape Breton ——. H. S. Poole. Proc. and Trans. Nova Scotian Inst. Science, 1899—1900, **10**, 246.

THE results are given of a series of tests as to the effect of washing coals.

(1.) Tests on 50-ton samples from each of the following mines:—

——	Raw Coal.		Washed Coal.	
	Ash.	Sulphur.	Ash.	Sulphur.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Hub	7·50	3·24	4·37	2·38
Caledonia	15·00	3·02	7·05	2·87
Stirling	11·09	4·23	5·50	3·12
Gowrie	11·55	5·26	6·01	3·15

(2) A test of 10,000 tons of coal from the Dominion Coal Company gave the following average results:—

——	Raw Coal.	Washed Coal.	Coke from Washed Coal.
Moisture	2·10	1·97	..
Volatile combustible matter.	31·00	33·21	1·86
Fixed carbon	56·83	60·00	88·98
Ash	10·07	4·82	9·16
Sulphur	2·38	1·79	1·62

(3) In order to test the efficiency of the coal washer, a laboratory trial was made. The results obtained in the laboratory are compared with those obtained by means of the coal-washer, in the following table, the samples being dried at 212° F.:—

——	Raw Coal.	Laboratory Test.		Treated in Coal Washer.		Coke from Washed Coal
		Washed Coal.	Shale.	Washed Coal.	Shale.	
Moisture	..	..	..	..	..	0·40
Volatile combustible matter.	33·06	33·79	31·43	34·07	30·82	1·60
Fixed carbon	55·93	61·33	15·33	61·26	23·21	89·82
Ash	11·01	2·89	48·08	4·67	41·22	8·18
Sulphur	2·41	1·64	5·16	1·70	4·48	1·65

—A. S.

Fuels; Determination of the Calorific Power of ——. O. Rebuffat. Gaz. chim. ital. **31**, [1], 78—82. Chem. Centr. 1901, **1**, [17], 971.

After a short critical description of the methods for the determination of the calorific value of fuels, the author, on the ground of his own experiences, and the results obtained by other investigators, rejects the proposals of Antony and Di Nola concerning Berthier's method (see this Journal, 1900, 1092). In order to obtain accurate results, it is always necessary to employ a calorimeter or calorimetric bomb. In making calorimetric determinations, commercial compressed oxygen is now generally used, and in this connection, attention is drawn to the fact that electrolytic oxygen contains small quantities of hydrogen, and, consequently, may give high results.—A. S.

Gas Producer; Riché ——. P. Corbier. Génie Civil, 1900, **37**, 149—151. Proc. Inst. Civil Eng. **143**, [1], 72—73.

The author describes the Riché wood-gas producer for producing gas suitable for driving gas-engines and for heating purposes (see this Journal, 1900, 1092). In each retort, 10—12 kilos. of wood can be distilled per hour, making 70—90 cb.m. of gas having a calorific value of 3,000 calories per cubic metre. The composition of Riché wood-gas is: carbon dioxide, 20; carbon monoxide, 20; ethylene, 15; and hydrogen, 45 per cent. by volume. The flame is of high temperature, owing to the absence of nitrogen.

In practice, 1,000 cb. m. of Riché wood-gas are produced by distilling 140 kilos. of wood, and burning 56 kilos. of coal of average quality, or 160 kilos. of wood; the residue consists of 26 kilos. of wood charcoal. The value of the charcoal varies with the kind of wood. The presence of moisture in the wood necessitates more fuel in the furnace, but does not affect the quality of the gas produced.—A. S.

Aërogen Gas (Carburetted Air); Production of ——. De Perrodil and De Morsier. Mém. de la Soc. des Ingén. Civ. de France, 1900, 403. Proc. Inst. Civil Eng. **143**, [1], 48—50.

The author gives a description of the van Vriesland apparatus, comprising a carburettor, motor, feeder, and regulator, for the production of "aërogen gas" or air impregnated with the vapour of gasoline or light petroleum oil. The carburettor, which acts also as a compressor of the aërogen gas produced, is a horizontal hollow drum, closed at both ends, which rotates within a stationary cylindrical wrought-iron casing, also closed at both ends; the drum is carried on a central longitudinal shaft, which passes out through stuffing-boxes in the ends of the casing. The drum itself is double, having an inner cylindrical shell within an outer; and the annular space between them is subdivided by means of four helical partitions into a succession of helical channels of rectangular section, which wind round the drum like the threads of a quadruple-threaded screw. At the front end of the drum, each channel has an inlet aperture through the drum head. The clear space between the drum and the casing is kept filled with gasoline from a feeder, up to one-third the height or diameter of the casing; the space of the upper two-thirds is occupied by air admitted through a controlling valve on the top of the casing. As the drum revolves, the inlet aperture of each channel in succession dips beneath the surface of the liquid, and a certain quantity of the latter enters; during the remainder of the revolution, air enters the channel, and when the orifice again dips into the liquid, there is a portion of air entrapped between the liquid now entering and that which entered in the preceding revolution. Thus the air above and the gasoline below are being continuously forced along the helical channels from the front to the back end of the drum, and the air is passing over surfaces always freshly wetted with gasoline. Each helical channel is, in effect, an Archimedean screw working horizontally, partly in liquid and partly in air. At the back end of the drum, each helical channel is turned radially inwards, to deliver into the enlarged hollow end of the central shaft, through which both the gasoline and the aërogen gas pass into a closed chamber; the gasoline collecting at the bottom returns thence through a U-tube into the bottom of the carburettor casing, whilst the aërogen gas passes off through a pressure regulater on the top of the chamber, and is delivered for consumption. The two discharge pipes are provided with throttle valves, so that the aërogen gas becomes compressed in the chamber, and exerts a back pressure on that in the helical channels.

The apparatus is now being used at Montgeron, near Paris, France, at Brocklesby and Ulceby, Lincolnshire, England, and in Holland.

In the apparatus which has been in constant use since June 1898, at Breukelen, Holland, and which is capable of supplying 250 burners, the carburettor or drum, is 0·8 m. (31½ ins.) diameter inside and 0·9 m. (35½ ins.) outside, and 1·36 m. (53½ ins.) long; the casing containing the drum is 0·98 m. (38⅝ ins.) diameter and 1·4 m. (55⅛ ins.) long; the total length, including the chamber at the back end, is 1·79 m. (70½ ins.). The section of the helical channels is 45 mm. (1¾ ins.), parallel to the driving shaft, by 50 mm. (2 ins.) radially. The gasoline used has a sp. gr. of 0·65 and distils over completely at 80° C.—A. S.

Hydrogen Sulphide; Determination of ——, *in Illuminating Gas.* C. C. Tutwiler.

See under XXIII., *page* 621.

PATENTS.

Combustible Material or Compound; more especially intended for use in the Manufacture of Briquettes of Artificial Fuel; Manufacture and Production of ——. Petolite Fuel Syndicate, Limited, and E. Johnson, both of London. Eng. Pat. 10,055, May 31, 1900.

To an absorbent material containing moisture (such as sawdust), are added in succession petroleum, resinous matter, finely and freshly powdered unslaked lime, and pitch. When the mass is cold, it is crushed or ground, and mixed with small coal, town refuse, dried sewage sludge, or other briquette material.—R. S.

Fuel, Liquid; Apparatus for Burning ——. H. H. Lake, London.—From B. Rein, Rochester, U.S.A. Eng. Pat. 5329, March 13, 1901.

Vapour burners for boilers or steam generators, especially for motor vehicles, are provided with means for starting the generator and burner without the use of a torch. The vapour generator is arranged at the side of the main burner, and consists of a metallic shell, the shell being provided with air inlets and a number of "retorts drilled in its wall" one of the retorts supplying the main burner and another an auxiliary burner. The latter heats the retorts, and a portion of the flame passes through an opening in the side of the burner into the main burner for lighting it. The supply and discharge pipes are separately governed and regulated.—R. S.

Vapour Burning Apparatus and Systems. E. Philipson, H. M. Baker and W. B. Sabel, all of Brooklyn, U.S.A. Eng. Pat. 19,195, Oct. 26, 1900. (Internat. Convent., date claimed April 3, 1900.)

The burner is provided with a vaporising tube having a nozzle, the orifice of which regulates the flow of hydrocarbon vapours from the vaporising tube, and which is kept clean automatically while in operation, by a plunger working within the vaporising tube and an extension thereof. The plunger is moved in one direction by oil pressure, and in the other by vapour pressure assisted by a spring: or the plunger may be operated by electrical means. The hydrocarbon vapour mixes with air in a special tube. The air and the oil supplies are regulated by valves provided with means for operating them both by one continuous movement in one direction, but during the reversal of that movement, only the oil supply valve is operated. An automatic device is supplied for measuring the charge of oil used, this device being situated between the fluid reservoir and the cup, which is provided with an igniter.—R. S.

Carburetted Air Producer with Automatic Regulation. G. Fischer, Risa, Germany; M. Richter, Dresden, Germany; H. Mestern, Vienna; F. Woda, Vienna; and P. Pallester, Vienna. Eng. Pat. 9609, May 24, 1900.

THE production of carburetted air is regulated automatically by the demand. The apparatus for doing this consists of a combination of an improved carburettor, a pressure-reducing device, an air reservoir or accumulator, and a hot air or other suitable motor of any construction. The air to be carburetted passes first through the motor jacket so as to cool the motor cylinder and warm the air. The air then passes into a reservoir provided with a loaded escape valve, from which it either passes into the carburettor or escapes into the outside air without being carburetted according to the amount of carburetted air required. To prevent any irregularities in the pressure of the air supplied to the carburettor, the air next passes through a pressure-equalising device. It then enters the carburettor through a central tube, and passing downwards, escapes through a number of small holes at the bottom into the carburetting liquid. To keep these small openings clean, the entering air is caused to act upon rotating vanes mounted on a vertical shaft provided at the bottom with brushes which rotate in contact with the small openings. A portion of the produced carburetted air is led to the motor for use therein.—R. S.

Compressed Gas for Lighting or Heating Purposes; Production of ——. F. Hoffmann, Altona, Prussia. Eng. Pat. 4982, March 8, 1901.

THE gas is compressed by a rotary pump inserted in the gas conduit and having a cylinder eccentrically arranged in a casing, the cylinder being provided with radially movable slides pressed outwards by springs so as to press tightly against the walls of the casing. To equalise the pressure on either side of the pump, in the gas conduits, a weighted elastic branch tube is provided; or instead of this, a spring valve may connect the compressed gas conduit with the suction conduit.—R. S.

Electric Furnaces of great Power. O. Imray. Eng. Pat. 10,580, 1900.

See under XI. A., *page* 588.

Coke-kiln Gases; Process for Recovering the by-products from ——. W. Heinemann, Bochum, Germany. Eng. Pat. 5232, March 12, 1901.

THE cool raw gas from the kilns is treated with steam, preferably by means of a steam injector. The condensing steam separates the particles of tar and absorbs the ammonia. The steam may be introduced into the main conduit from all the kilns belonging to one battery.—R. S.

Gas Purifying Apparatus. F. Sasse, Cologne-on-the-Rhine. Eng. Pat. 6735, March 30, 1901.

TO the cistern of a gas scrubber or washer is fixed an up-standing ring, the upper edge of which is, according to the pressure employed, more or less above the lower rim of the washer. By this means the washer may be cleaned during use without allowing air to enter.—R. S.

Acetylene Gas; Apparatus for Generating ——. G. Seagrave, Walthamstow. Eng. Pat. 7751, April 26, 1900.

THE bell of a gasholder, which receives the gas for distribution through a purifier to the burners, actuates a valve on a water service, so that, when the bell is nearly at its lowest position, water is admitted to a vessel in which it operates a piston or distends a diaphragm, whereby, through a ratchet and pawl mechanism, a cylinder containing carbide is dropped into a generating tank containing water. The gas evolved passes into the gasholder, and through the rise of the bell causes the flow of water to be cut off, and that which has operated the piston to be discharged into the generating tank.—J. A. B.

Acetylene Gas; Means for Generating ——, *and for Lighting by Means of such Gas.* G. W. Johnson, London. From La Compagnie Française de l'Acetylène Dissous, Paris. Eng. Pat. 7859, April 27, 1900.

A RECEIVER containing carbide has walls consisting partially of felt or other capillary substance, through which water gains access to the carbide when the receiver is immersed in water in an outer vessel. The gas evolved passes through a drying chamber direct to the burner or burners of the twin-jet type. In order to arrange a number of these burners in close juxtaposition for search-light or similar uses, they are set obliquely to the base plate or pipe, so that the flames are at an angle of about 45° to its axis and parallel to one another.—J. A. B.

Acetylene Gas Generators or the like. J. H. Ross, Birmingham. Eng. Pat. 9537, May 24, 1900.

MEASURING spouts attached to the outlet of a hopper containing carbide by means of a swivel joint, are actuated according to the movements of a bell gasholder, so that a measured quantity of carbide is discharged into water in a generating chamber as required. A distributing cone or inclined plane, immersed in oil floating on top of the water, is used to prevent escape of gas through the direct contact of the carbide with the water.—J. A. B.

Acetylene Gas; Apparatus for Automatically Generating ——. M. Martin, Saint-Michel de Maurienne, France. Eng. Pat. 1283, Jan. 19, 1901.

CARBIDE is contained in a basket, supported within a water-sealed chamber attached to the top of a bell floating in a tank of water. A prolongation of the chamber dips constantly into the water. A pipe, connecting the upper part of the chamber with the gas space of the bell, may be closed, when it is necessary to refill the basket with carbide, by means of a cock a handle of which locks with the lid of the chamber, so that the latter can be opened only when the cock is closed.—J. A. B.

Acetylene and other Gases; Apparatus for Generating and Burning ——. J. J. Hendler and E. K. Reeves, both of Kansas City, U.S.A. Eng. Pat. 2944, Feb. 11, 1901.

A GENERATING chamber, in which carbide rests on a perforated shelf, is surrounded by a tank containing water, with which it is in communication through holes near its base. The tank is supplied, through a narrow pipe extending nearly to its bottom, with water from a superposed reservoir, the upper part of which communicates through a wide pipe with the upper part of the tank. The gas evolved from the carbide forces water back from the tank to the reservoir through this wide pipe. When equilibrium of pressure is established, the water required to replace that consumed is supplied from the reservoir through the narrow pipe. A burner is mounted on the top of the generating chamber.—J. A. B.

Acetylene Gas Machines and Regulators. E. R. Cook, Sacramento, Cal., U.S.A., and G. F. Heusner, Portland, Oregon, U.S.A. Eng. Pat. 4954, March 8, 1901.

A GENERATING chamber, containing receptacles for trays of carbide and water-distributing troughs, is surmounted by a tank the bottom of which is connected by a pipe with the lower part of the generating chamber, to which water thereby gains access from the tank. On reaching the level of a pipe delivering into one of the distributing troughs, the water flows into the latter and on to the carbide until the gas evolved drives back the water into the tank. The gas passes through a purifier and regulator of special construction to the burner. The apparatus is designed specially for the supply of locomotive head-lights.—J. A. B.

Incandescent Lighting; Production of Inflammable Mixture especially Adapted for ——. J. St. C. Legge, Dublin. Eng. Pat. 8635, May 10, 1900.

THE inflammable mixture is produced by passing air under pressure through a carburetting mixture composed of benzoline or gasoline or other light hydrocarbon, and

camphor in approximately certain proportions, and then causing the mixed air and gas or vapour to impinge on naphthalene.—R. S.

Incandescence Oil Burners. F. Richter, Vienna. Eng. Pat. 9359, May 21, 1900.

This describes a flame-spreader for a circular-wick incandescence lamp. A perforated cylindrical shell, closed at its upper end by a plate and supported by a central rod, is attached at its lower end to an annular metallic disc, of slightly conical cross-section, the outer (and higher) edge of which has a downwardly projecting lip a little less in diameter than the interior of the outer wick tube. The inner edge of the annular disc may have a cylindrical flange, to prevent the flame from entering the head of the spreader. It is claimed that with this flame-spreader, perfect combustion is attained, with no formation of soot. —H. B.

Incandescing Media for Incandescence Lighting; Manufacture of ——. R. A. Nielsen, Copenhagen. Eng. Pat. 9785, May 28, 1900.

Reference is made to Eng. Pat. No. 2672 of 1900 (this Journal, 1900, 335). As it has been found that in fusing the oxides in the electric arc, preparatory to drawing them out into threads, troubles arise owing to the formation of carbides and reduction products through the action of the carbon vapour in the arc, the patentee now avoids the direct application of the arc. This he effects by placing the substances under treatment, in crucibles or the like and introducing them therein into the electric furnace. From the molten mass obtained, threads are to be produced by suitable mechanical means. Incandescing media made from such threads may be provided with a pulverulent coating of incandescible substances; as, for instance, by immersing the medium in a solution of the salts and afterwards raising the medium to incandescence.—H. B.

Electric Lamps; Incandescent ——. B. M. Drake and The Nernst Electric Light, Ltd., Westminster. Eng. Pat. 9944, May 30, 1900.

It has been found that in lamps of the Nernst type, the anode contact becomes hotter than the cathode contact, the heat tending to cause breakage near the anode contact. This invention consists in sufficiently dissipating the heat at the contact by providing the latter with radiating surfaces of suitable forms and dimensions. For example, round the end of a Nernst filament the contact-making wire is wound closely, some of the convolutions being formed with radially-extending loops. Each loop is then twisted so as to form a more or less rigid arm, and the wire coils are coated with a paste (which may have the same composition as that of the filament), the radially-arranged arms either being wholly covered by the coating, or preferably, projecting beyond its outer surface. The contacts at both ends of the filament may be constructed in this way.—H. B.

Incandescent Oil and Spirit Lamps. W. P. Thompson, Liverpool. From J. Rubenstein, Berlin. Eng. Pat. 11,128, June 19, 1900.

In the lamp described, which is of the circular-wick type, those metallic parts which are in contact with the flame are made of German silver or other poor conductor of heat. The wick, which may consist wholly or partly of asbestos, is tightly woven at its upper part, less tightly woven at the middle part, and loosely woven at the lower part. The glass chimney has the form of two truncated cones, joined at their bases, an inward "shoulder" being produced, at their junction, a short distance above the top of the mantle. The outer wick tube, which extends above the inner wick tube, has its upper edge bent inwards slightly to prevent the wick being raised too far, and is attached to the lamp by means of a bayonet joint, so that it may be removed to facilitate the fixing of a fresh wick in the lamp. By removing the gallery ring the incandescent lamp may be converted into an ordinary lamp.—H. B.

Incandescence Oil Lamps. L. Denayrouze, Neuilly, France. Eng. Pat. 11,665, June 27, 1900.

This relates to burner fittings, which can be applied to the reservoir of an ordinary oil lamp. A bundle of cotton wicks is bound tightly in a tube of badly-conducting metal, such as German silver, which can be screwed into the top of the lamp reservoir, the lower parts of the wicks being immersed in the oil. At the top of the wicks, where they are tightly bound, is formed a vaporising chamber, having small holes at the top, by which the oil vapour passes into an air-mixing tube, afterwards burning within a mantle as usual. A central heat-conducting rod extends upwards into the region of the flame and downwards through the vaporising chamber and a short distance into the bundle of wicks. The vapour produced by the heat of the rod is prevented by the closely packed wicks from getting down into the reservoir. From the vaporising chamber there is a by-pass pipe, furnished with a cock, leading to the upper part of the reservoir. To extinguish the lamp this cock is opened, when the vapour passes into the reservoir and condenses. To start the lamp, the vaporising chamber is heated in any convenient way.—H. B.

Incandescent Gas-Burners. F. Fuerstenheim, Berlin. Eng. Pat. 6262, March 25, 1901.

Within the mixing tube of a Bunsen burner is fixed a central tube *p*, up which air alone passes, gaining access through the lateral perforations *n*. Gas enters the annular mixing tube through vertical perforations *h* in the head *g*, drawing in air at the inlets *k*. The air issuing from the funnel-shaped top of the tube *p* forces the annular flame against the interior surface of the mantle, and as it is claimed that a much larger area of the mantle than usual is thus rendered incandescent, "mantles of exceptional size may be employed." To prevent the flame firing back, a constriction is made in the mixing tube—such as that shown at *r*, where two opposed metallic rings are inclined towards each other, forming an annular slot. —H. B.

Fig. 1.

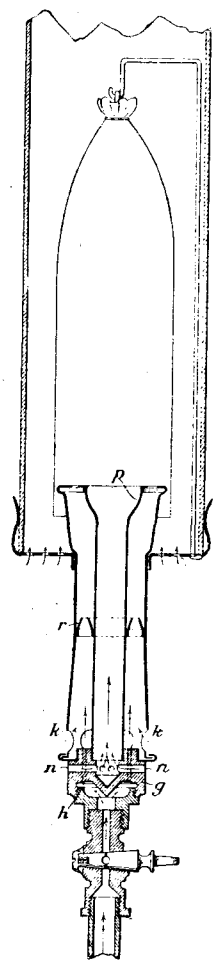
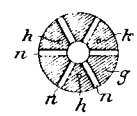


Fig. 2.



Incandescent Bodies for Gas Lighting; Process for Strengthening ——. W. P. Thompson, Liverpool. From A. Wasmuth, Hamburg. Eng. Pat. 2005, Jan. 29, 1901.

THE process consists in adding "magnesium sulphate or its constituents" to the solution of salts used in impregnating a purified fabric, more particularly a ramie fabric, in the proportion of about 0·3 to 1 part of magnesium sulphate to 100 parts of the water employed for making the solution. For example, the solution may be composed of: thorium nitrate, 30 parts; cerium nitrate, 0·4—0·5; yttrium nitrate, 0·03—0·05; erbium nitrate, 0·01—0·025; magnesium sulphate, 0·3—0·8; and water, 100 parts.—H.B.

Incandescent Bodies for Gas and other Lighting, Manufacture of ——. K. Schultze, Berlin. Eng. Pat. 5352, March 13, 1901.

FABRICS are to be impregnated with a solution composed of about 0·25 part of uranium nitrate, 0·75 part of erbium nitrate, 250 parts of thorium nitrate, and 500 parts of distilled water, the fabric being burned off afterwards. —H. B.

Incandescent Gas Lighting. C. G. Richardson, New York. Eng. Pat. 6860, April 2, 1901.

A CONCENTRATED solution of celluloid is thoroughly mixed with a solution of suitable incandescence salts, and after evaporation to a pliable mass, the mixture is moulded by pressure into the desired form—preferably that of a hollow perforated cone or mantle. The mantle is then denitrated by means of a suitable reagent, such as ammonium sulphide. Instead of first evaporating the liquid mixture to a pliable mass, it may be "injected under pressure into a mould having the desired configuration. As the liquid hardens in the mould it acquires the desired shape." The mantle is then denitrated.—H. B.

Nernst [Electric] Lamps, and Heaters therefor. A. J. Wurts, H. N. Potter, E. Bennett, and M. C. Beebe, Pittsburgh, U.S.A. Eng. Pat. 11,561, June 26, 1900.

IN making electric heaters for Nernst lamp glowers, the heating wire is wound upon a thin, light support, composed preferably of ground talc mixed with a binder, such as gum-tragacanth; the support is bent into suitable shape, and the form is rendered permanent by baking. A suitable shape for the composite insulated heater thus obtained is that of a helix, centrally within which the glower may be arranged. Both heater and glower are fixed, the convolutions of the former interfering to an immaterial extent with the light emitted by the glower. A number of methods of constructing the heaters, arranging them relatively to the glowers, connecting them into the circuits, and fitting them detachably into Nernst electric lamps, are described and claimed. —H. B.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, PETROLEUM.

Benzols; Prices, Imports and Exports, Composition, Examination, and Application of——. F. Frank. Chem. Ind. 1901, **24**, [8], 237—239, and [9], 262—266.

THE reduction in the prices of benzol since 1882 is shown in the following table, which also gives the German imports (from England and Belgium) and exports:—

	Marks per 100 kilos.		Marks per 100 kilos.
1882.....	175—400	1896.....	50—120
1885.....	50—90	1897.....	65
1890.....	100—125	1898.....	25
1892.....	40—60	1899.....	20
1895.....	25—60	1900.....	20

Year.	Imports.	Exports.
	Tons.	Tons.
1891	7,316	516
1892	5,683	916
1895	8,317	1,966
1896	11,305	1,539
1897	10,185	1,270
1898	5,915	1,611
1899	5,291	2,755

At the present time Germany produces upwards of 28,000 tons of benzol annually, of which at least 22,000 tons are consumed in the colour works, the remainder being used for the enrichment of gas and other purposes.

The fall in prices was accompanied by the demand for a purer product, stress being laid on freedom from paraffins, light hydrocarbons, carbon bisulphide, and thiophen.

The ordinary method of testing benzols by observing the volume of distillate obtained at certain temperatures is no guarantee as to their true character. For example, although the following mixtures fulfil the requirements of 90 per cent. benzol, *viz.*:—

(1) 82·0 per cent. benzol + 18·0 per cent. toluol;
(2) 92·2 per cent. benzol + 7·8 per cent. xylol;
(3) 90·0 per cent. benzol + 5·0 per cent. toluol + 5·0 per cent. xylol;
(4) 84·0 per cent. benzol + 13·0 per cent. toluol + 3·0 per cent. xylol;

only the last can be regarded as a typical 90 per cent. benzol. The annexed interpolation curve table by Spilker best expresses the variations in such mixtures, and sufficiently explains itself. See page 567.

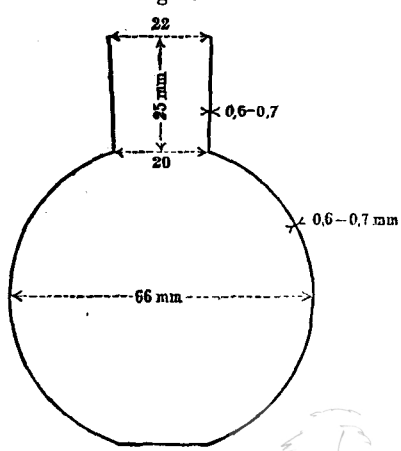
Solvent Naphtha:—90 per cent. at 160° C. is the strength usually claimed, but it is pointed out that it would be better to limit the end-point of the distillation to 150° C., so as to exclude coumarone and indene, and thereby improve both odour and stability to light. Another type of solvent naphtha, consisting chiefly of cumol, and yielding 90 per cent. of distillate between 140° and 175° C., is obtained by washing heavy naphtha with sulphuric acid and distilling with live steam.

For the proximate composition of commercial benzols as used in colour works, Kraemer and Spilker give the following data:—

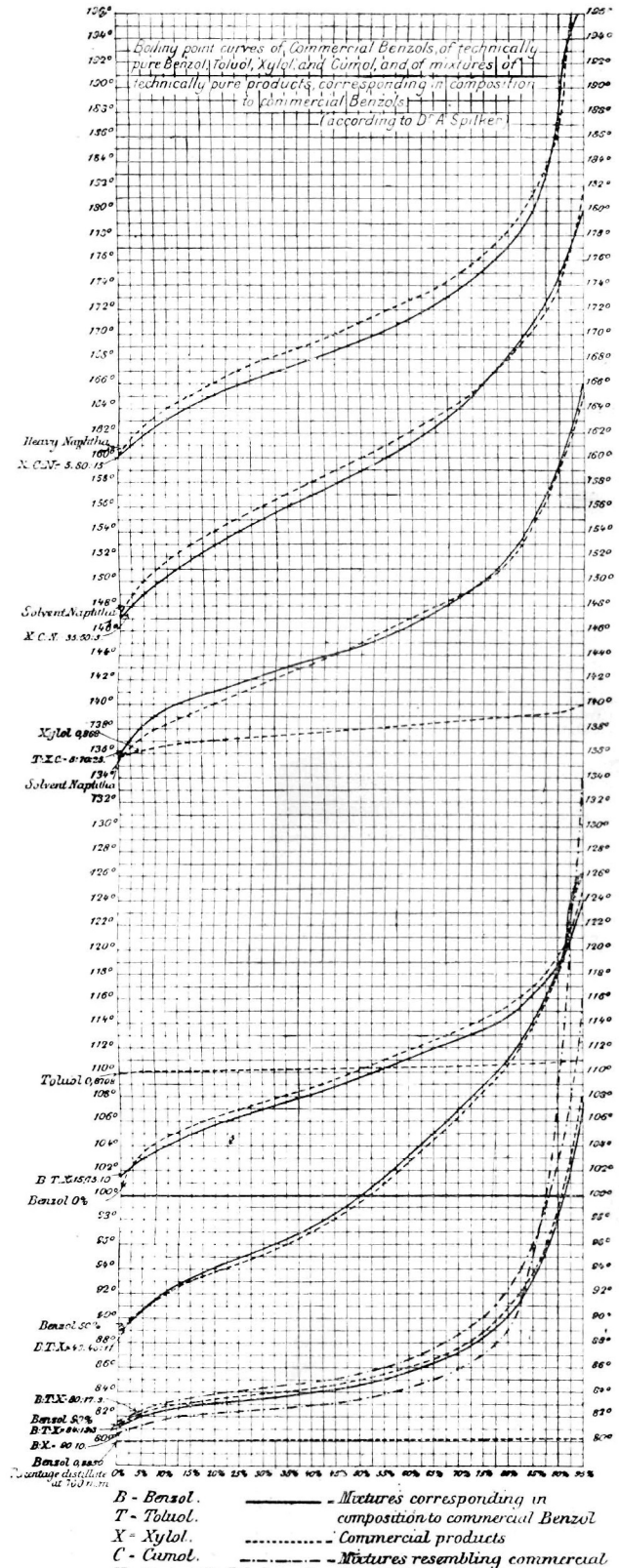
——	90 per Cent. Benzol.	50 per Cent. Benzol.	0 per Cent. Benzol.
	Per Cent.	Per Cent.	Per Cent.
Water	0·060	..	..
Paraffin.....	0·100	0·25	0·50
Carbon bisulphide	0·686	0·39	..
Substances absorbing bromine, including thiophen and the like.	1·202	1·23	0·82
Thiophen *per se*.....	0·264		
Benzene	80·922	45·37	13·54
Toluene.....	14·850	40·32	73·42
Xylene.....	2·180	12·44	11·69

With regard to the examination of benzols, it is stated that, with but few exceptions, the ordinary retort method, with the thermometer immersed in the liquid, has been replaced by the following system:—The distilling vessel is a globular copper still of 150 c.c. capacity, the bottom being flattened as shown in diagram, Fig. 1. It is 66 mm.

Fig. 1.

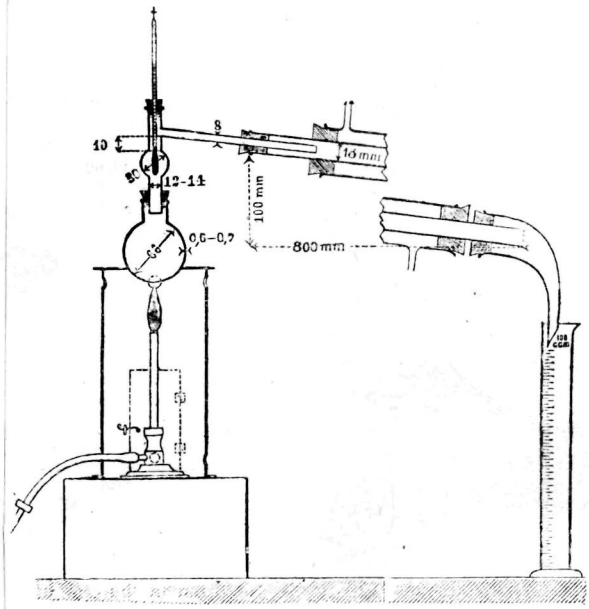


indiameter and 0·6 to 0·7 mm. thick. The neck is 25 mm. long, 20 mm. wide at the bottom, and 22 mm. wide at the top. The glass still head (see Fig. 2) is 150 mm. long and



14 mm. wide, with a globular enlargement in the centre, and, 10 mm. above this, a side tube 8 mm. wide, branching off almost at a right angle. The still rests on an asbestos disc with a circular hole 50 mm. in diameter, and is heated by the colourless flame of a Bunsen burner. The burner is surrounded by a metal cylinder with four holes 10 mm. from the top, for the escape of the products of combustion. The Liebig condenser is 800 mm. long, and inclined at such an angle that the outlet is 100 mm. lower than the inlet. The charge is 100 c.c., the rate of distillation 5 c.c. per minute (about 2 drops per second), the operation being finished when 95 per cent. have passed over. The thermometer is made of thin glass about half the thickness of the diameter of the still head, the mercury bulb being fixed in the centre of the globular enlargement. It is graduated to tenths of a degree for pure products, and to half a degree for commercial benzols, and should be tested occasionally against a Government standard. The variations in barometric pressure are allowed for by making the necessary corrections (compare Lenders, Chem. Ind. 1889, 169), or may be disregarded by employing a thermometer with an adjustable scale, and distilling 100 c.c. of pure water through the apparatus, fixing the 100° C. point the moment that 60 c.c. have passed over. The complete apparatus is shown in Fig. 2.

Fig. 2.



The following figures give the average specific gravities of commercial benzols:—

90 per cent. benzol	0·880—0·883
50 per cent. benzol	0·875—0·877
0 per cent. benzol	0·870—0·872
Solvent naphtha, 90 per cent. at 160° C.	0·874—0·880
Solvent naphtha, 90 per cent. at 175° C.	0·890—0·910
Heavy naphtha	0·920—0·945
Pure benzene	0·883—0·885
Toluene	0·870—0·871
Xylene	0·867—0·869

The estimation of carbon bisulphide is effected in the following manner:—50 grms. of benzol are mixed with 50 grms. of a solution of potash (11 grms.) in absolute alcohol (90 c.c.), and kept at the ordinary temperature for a few hours. The mixture is then shaken up with 100 c.c. of water, the aqueous solution is separated from the benzol, and the washing operation repeated three or four times. The potassium xanthate is determined in the washings by

acidifying with acetic acid and titrating with a solution of copper sulphate obtained by dissolving 12·475 grms. of the crystallised salt in a litre of water, 1 c.c. = 0·0076 CS_2. The end-reaction is ascertained by the red colour produced when a drop of the liquid, taken out with a glass rod, is brought into contact with a drop of a solution of potassium ferrocyanide on a filter paper. 90 per cent. benzol contains from 0·2 to 1·0 per cent., 50 per cent. benzol up to 0·5 per cent. of carbon bisulphide.

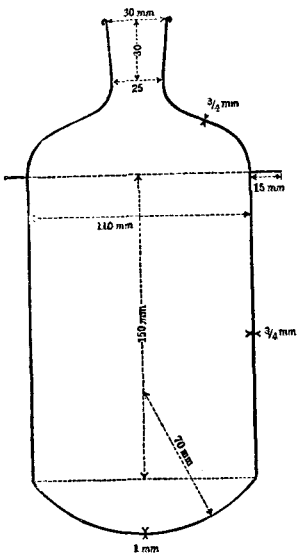
Thiophen can be detected by the indophenine test (blue colour). For its quantitative estimation the method proposed by Denigès is recommended, which depends on the formation of the compound $SO_4(HgO)_2:Hg.SC_4H_4$, by acting upon thiophen with mercuric sulphate (this Journal, 1896, 746).

For the quantitative estimation of paraffins, 200 grms. of the benzol under examination are thoroughly mixed for 15 mins. in a separating funnel with 500 grms. of fuming sulphuric acid (20 per cent. anhydride), taking care to avoid heating. The mixture is allowed to stand for two hours, the sulphuric acid solution being then drawn off, and the treatment with fuming sulphuric acid repeated a second and third time. The residual paraffins are then separated, whilst that portion dissolved in the acid is recovered by diluting the acid with three volumes of ice water, and distilling the solution in a flask of three litres capacity until, in addition to oil, 50 c.c. of water have passed over. The distillate, after separating the water, is added to the original oil, and the mixture treated several times with fuming sulphuric acid (30 grms.) until its volume ceases to diminish. The quantity of paraffin in 90, 50, and 0 per cent. benzols seldom exceeds 1 per cent., toluol is mostly free from this impurity, whilst xylol frequently contains as much as 3 per cent.

To ascertain the presence of unsaturated compounds, 5 c.c. are shaken vigorously for 5 mins. with 5 c.c. of strong sulphuric acid in a stoppered bottle holding 15 c.c., and the coloration, if any, is compared with a solution of potassium bichromate in sulphuric acid (50 per cent.). The coloration imparted to 90 and 50 benzol should not exceed the colour of a bichromate solution containing at the most 2·5 grms. of $K_2Cr_2O_7$ in 1 litre of sulphuric acid, whilst pure products should remain colourless.

For the purpose of determining the bromine absorption, 5 c.c. of benzol are mixed in a stoppered glass cylinder of 50 c.c. capacity, with 10 c.c. of dilute sulphuric acid (20 per cent.). A decinormal solution of potassium bromide and bromate (9·9167 grms. KBr + 2·7833 grms. $KBrO_3$ per litre, 1 c.c. = 0·008 grm. Br) is then added from a burette until there is at the end of five minutes' continuous shaking a permanent coloration, and the oil gives a blue colour to freshly prepared zinc iodide starch paper. The amount of bromine assimilated by 90 and 50 per cent. benzol is usually 0·6 per cent., and rarely exceeds 1 per cent.

Fig. 3.



The following are the mean flashing points of benzols:—Pure benzol, − 8° C.; 90 per cent. benzol, under − 4° C.; toluol, + 5° C.; xylol, + 21° C.; solvent naphtha (90 per cent. at 160° C.), + 21° C.; solvent naphtha (90 per cent. at 175° C.), + 28° C.; and heavy benzol, + 47° C.

The percentage composition of commercial benzols is ascertained by distilling 1 kilo. of the sample in a copper vessel, as shown in Fig. 3, fitted with a Le Bel-Henniger dephlegmator 60 cm. in length. The distillate is collected in tared bottles. It is divided into the following fractions:—

For 90 and 50 per cent. Benzol.

Up to 79° C.	First runnings.
79° to 85°	Benzol.
85° to 105°	Intermediate fraction.
105° to 115°	Toluol.
Residue	Xylol.

For Toluol.

Up to 109° C.	First runnings.
109° to 110·5°	Toluol.
Residue	Last runnings.

For Pure Benzol.

Up to 79° C.	First runnings.
79° to 81°	Benzol.
Residue	Last runnings.

For Xylol.

Up to 135° C.	First runnings.
135° to 137°	Paraxylol.
137° to 140°	Metaxylol.
140° to 145°	Orthoxylol.
Residue	Last runnings.

Percentage Composition.

—	90 per Cent. Benzol.	50 per Cent. Benzol.
First runnings	1·0	0·3
Benzol	78·8	18·3
Intermediate fraction	10·0	47·5
Toluol	8·0	23·7
Xylol	2·0	10·0
Loss by distillation	0·2	0·2

Pure Benzol.

First runnings	0·5
Benzol	98·0
Last runnings	1·2
Loss by distillation	0·3

Toluol.

First runnings	0·3
Toluol	97·3
Last runnings	2·2
Loss by distillation	0·2

Xylol.

First runnings	1·3
p-Xylol	15·0
m-Xylol	76·5
o-Xylol	5·0
Last runnings	2·0
Loss by distillation	0·2

—D. B.

Petroleum; Composition of Californian ——. C. F. Mabery and E. J. Hudson. Amer. Chem. J. **25**, [4], 253—284.

The following conclusions are drawn by the authors from the results of their investigations:—

The relatively low proportion of distillates below 225° C. is an essential characteristic of Californian oil. The bulk of such crude oils as distil below that temperature consists of methylenes, similar, as regards specific gravity and boiling point, to those from Russian oils, except in the case of undecanaphthene (b. pt. = 195° C.), dodecanaphthene (b. pt. = 216° C.), and tridecanaphthene (b. pt. = 230°—232° C.). The aromatic hydrocarbons are present in larger amount than in Russian oil, the benzene homologues being abundant, especially in the lighter fractions; the distillate 221°—222° C. from Puente oil contained so much naphthalene as to solidity at 0° C.

Another point of difference is the absence of members of the C_nH_{2n+2} series, as well as the presence of large proportions of oxygen and nitrogen compounds. In some of the high-boiling distillates, *e.g.*, from Summerland oil, hydrocarbons of the C_nH_{2n-2} and C_nH_{2n-4} series were identified.—C. S.

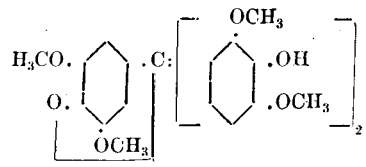
Petroleum; Composition of Japanese ——. C. F. Mabery and S. Takano. Amer. Chem. J. **25**, [4], 297—307 (See also this Journal, 1900, 1003.)

THE results show that Japanese oil mainly consists of methylene hydrocarbons, the very heavy oils also probably containing hydrocarbons with two or more methylene rings of the series C_nH_{2n-2} or C_nH_{2n-4}. Solid paraffins were found in some of the oils (Amaze, Miyagawa, and Hiyama). The proportion of benzene derivatives is smaller than in Californian oils, and the nitrogen and sulphur compounds present vary considerably in amount.—C. S.

IV.—COLOURING MATTERS AND DYESTUFFS.

Eupitton and Pittakall. C. Liebermann. Ber. **34**, [6], 1026—1030.

THE author, in 1876, separated from wood tar an orange-red dyestuff, which he identified as the parent substance of a product obtained by v. Reichenbach in 1833, termed pittakal. The latter, on investigation, proved to be a metallic salt of an acid dyestuff, to which the name Eupitton was given. This product has the formula—



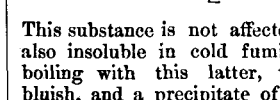
and, as its constitution shows, is not a mordant dyestuff. The same is also true of its sodium salt. Reichenbach's statements as to the dyeing properties of his pittakal appear to have been exaggerated, as the author was only able to obtain dyeings with difficulty by the use of strong dye-baths. For this purpose a solution of Eupitton sodium is precipitated in the cold with glacial acetic acid, then saturated with ammonia, and the fabric (calico or linen) mordanted or unmordanted, is dyed at the boil. The shade produced is a bluish violet, approximating to that of Indigo. The dyed fabric stands water and lukewarm soap lye, but acids turn the shade yellow; the original bluish-violet colour, however, is restored on passing the fabric through lime water.

—T. A. L.

Eupitton Derivatives. C. Liebermann and F. Wiedermann. Ber. 1901, **34**, [6], 1031—1040.

THE determination of the number of methoxyl groups in Eupitton by Zeisel's method gave numbers agreeing with Hofmann's formula, $C_{19}H_8(OCH_3)_6O_3$. Moreover, Eupitton forms salts with mineral acids, and of these, the hydrochloride, like that of Aurin, crystallises with alcohol, which, however, it loses after standing some considerable time in a desiccator, and the salt then has the formula $C_{25}H_{26}O_9HCl$. When Eupitton (3 grms.) is heated to 110° C. for half an hour with concentrated sulphuric acid (100 c.c.), it loses its methyl groups and is converted into hexahydroxyaurin, to which the name Eupitton Black, or Noreupitton, has been given. The product, when free from acid and before drying, is readily soluble in hot water and alcohol to dark brown solutions. It is an excellent mordant dyestuff, giving deep blacks fast to soap, and does not appear to be identical with N. Caro's hexahydroxyaurin (Ber. **25**, 2675).

The derivative obtained by Hofmann from Eupitton and ammonia at 160° C., and termed Eupitton triamine or hexamethoxyrosaniline, is not a mordant dyestuff, but dyes wool blue shades, and gives an acetate having a distinct metallic sheen. It is noteworthy that this salt, when repeatedly moistened and evaporated on the water-bath, loses the whole of its acetic acid, leaving the base almost colourless. In determining the number of methyl groups in this product by Zeisel's method, it was observed that the hydriodic acid solution deposited, on cooling, crystals of the de-methylated compound. The hydrochloride of this compound, hexahydroxyleucaniline, has the formula $C_{19}H_{19}N_3O_6 3HCl$. The aqueous solution, after adding a few drops of potash lye, is carefully oxidised by air to a blue dyestuff. After saturating with glacial acetic acid, the mixture is evaporated on the water-bath with the addition of alcohol or acetic acid, when the oxidation is complete. The product dyes wool a fine blue, and is without doubt hexahydroxyrosaniline, from which it follows that the blue colour given by Eupitton triamine on unmordanted animal fibres is not due to methoxyl groups only, but that the presence of six hydroxyls is sufficient to produce a blue dyestuff from Rosaniline. When hexahydroxyleucaniline hydriodide is boiled for 10 minutes with fused sodium acetate and acetic anhydride, it yields ennea-acetylhexahydroxyleucaniline, which crystallises from alcohol in colourless needles melting at 173° C., and having the formula—



This substance is not affected by cold alkali lye, and it is also insoluble in cold fuming hydrochloric acid, but on boiling with this latter, the solution becomes slightly bluish, and a precipitate of the trihydrochloride of hexahydroxyleucaniline is obtained. In order to determine the effect on the shade by the introduction of further methyl groups into hexamethoxyrosaniline, the authors heated Eupitton with methylamine in alcoholic solution to about 160° C. for 2½ hours. A leuco base is formed, which, on oxidation in alcoholic solution in presence of acetic acid with hydrated manganese peroxide (from permanganate by reduction in alkaline carbonate solution), gives most probably hexamethoxydimethyl rosaniline. The deep blue solution dyes wool blue in the cold, but the colour can be stripped by boiling with acetic acid or alcohol. Hofmann stated that Eupitton, when heated with water to 270° C., gave, together with the dimethyl ethers of pyrogallol and methylpyrogallol, a product crystallising in white needles and soluble in alkalis. The authors have identified this as leuco-eupitton, or hexamethoxyleucaurin,—

$$HC : [.C_6H_2(OCH_3)_2OH]_3,$$

and have obtained it by reducing Eupitton with zinc dust and acetic acid. The product is readily soluble in alcohol, and melts at 198° C. It dissolves to a colourless solution in alkali, which, on boiling with strong potash, turns blue. Triacetylleuco-eupitton, $C_{25}H_{25}(C_2H_3O)_3O_9$, obtained in the usual manner by acetylating leuco-eupitton, crystallises from dilute alcohol in colourless needles melting at 236° C., and dissolves in concentrated sulphuric acid with the red colour characteristic of Eupitton.—T. A. L.

Colour Change; Theory of ——. C. Liebermann. Ber. 1901, **34**, [6], 1040—1042.

EUPITTON triamine, according to Hofmann, is hexamethoxyrosaniline. The entrance of six methoxyl groups has determined a change of colour from red to blue. This might be thought to be due simply to the methyl, which produces a similar change when it replaces the amino hydrogen in Rosaniline, giving rise to violets and blues. But in the present case the change must be attributed to the entire methoxyl, the methyl itself only playing a subordinate part, since hexahydroxyrosaniline is also blue, and here the colour change can only be due to the hydroxyl. Eupitton itself—hexamethoxyaurin—is only a feeble dyestuff, and hardly differs in shade from Aurin. But whereas the alkaline solution of Aurin is red, that of Eupitton is blue, again showing the influence of methoxyl groups. When the methyl is removed from these methoxyl groups, the colour of the sulphuric acid solution changes from magenta to blue; the new compound, the Noreupitton (Eupitton Black) of

the preceding abstract, dyes mordants so deeply that its essentially violet colour can only be recognised at the first moment of its application. The effects here attributed to hydroxyl or methoxyl groups, *i.e.*, a deepening of colour and a shifting towards the blue end of the spectrum, can also be obtained in other classes of dyestuffs, such as the hydroxyanthraquinones. In this class, with successive additions of hydroxyl, there is a change of colour from the yellow or orange of hydroxyanthraquinone and of the corresponding dihydroxyanthraquinone having one hydroxyl in each nucleus, through the red and violet of Alizarin and Purpurin to the blue of alkaline solutions of penta- and hexa-hydroxyanthraquinone (Cyanin). The position, however, as well as the number of the hydroxyl groups is important, for the alkaline solutions of Alizarin, Hystazarin, and Quinizarin are bluer than that of Purpurin, whilst in the anthragallols the colour change is towards green instead of blue. On an alumina mordant the changes of colour are from the red of Alizarin and Purpurin through the Bordeaux of Quinizarin to the blue of Cyanin, whilst, on the other hand, with different positions of the hydroxyl groups, the anthragallols and rufigallic acid are brown. In the aurin hydroxycarboxylic acids similar changes from red to brown appear to take place with increasing hydroxyl groups (Ber. **25**, 949), providing that the substances there described were pure.

The two series of colour changes—yellow, orange, red, violet (ponceau), blue, black—and yellow, red, brown, black—which in the foregoing instances mark successive additions of hydroxyl to the molecule, are also well known in the class of azo colours. Here, indeed, there are few examples of an aggregation of hydroxyl groups, but an increase in the number of azo groups or of amino groups has the same effect. Lastly, conjugation with phenol, naphthol, and anthrol respectively (this Journal, 1882, 181 and 226) is marked by a similar colour change, progressing with the increasing number of benzene rings in the molecule. It is remarkable that certain groups, such as sulphuryl, have no influence on colour. Bromine, however, as exemplified in the case of the Eosines, tends to deepen the colour.

It must be remembered that the colours of dyed materials and of solutions are not spectrum colours, and little is known about them spectroscopically; still it is noteworthy that the heaping up of atomic groups only tends to facilitate the passage of rays of shorter wave length.—T. A. L.

Picric Acid; Process for Manufacturing ——. — Wenghöffer. Rev. Prod. Chim. **4**, [3], 35.

A THIN paste is made of 100 kilos. of sulphanilic acid and water, this being then mixed with 40 kilos. of sodium nitrite in a dilute solution, and filtered to separate undissolved impurities. The filtrate is treated with 28 kilos. of sulphuric acid, which throws down the diazobenzene sulphonic acid formed. After filtration, the mass is introduced into nitric acid, the concentration of which is such that the liquid has a final density of about 40° B. Heat is applied until nitrogen begins to come off, and when the evolution of gas diminishes, heating is discontinued and the solution is left at rest for about 36 hours, to allow of the separation of the picric acid. The latter is washed and dried, the washing being simpler than in the usual method of manufacture, owing to the nitration being effected in the absence of sulphuric acid.

The method may be modified by stirring the above quantity of sulphanilic acid into a solution of 40 kilos. of sodium nitrite in the same weight of water, the resulting yellow paste being then introduced into such an amount of nitric acid as to obtain 320 kilos. of acid of 40° B.; the final stages are the same as before.

By these methods, 110 kilos. of picric acid can be obtained from 50 kilos. of aniline.—C. S.

Nitrated Azo Dyestuffs; Reduction of ——. A. Rosenstiehl. Comptes Rend. **132**, [16], 985–988.

THE author, in conjunction with Noelting, patented, in 1887, a red dyestuff obtained from meta-azoxyorthotoluidine by diazotising and coupling the diazo compound with α-sulpho-α-naphthol. The meta-azoxytoluidine was obtained by acting with zinc and caustic soda on meta-nitro-orthotoluidine, but as the nitramine is insoluble in alkali the action was very slow and the yield bad. The author now finds that if the nitramine be diazotised and the diazo compound then coupled with α-sulpho-α-naphthol, the substance so formed is soluble in alkali, and may be reduced in alkaline solution by stannous chloride, arsenious acid, formaldehyde, or glucose, at 50°—60° C. No change of colour occurs during the reduction, but while the nitramine derivative dyes wool only, and only in an acid bath, the azoxy-amine derivative does not dye wool, but dyes cotton in an alkaline bath.

This reaction is general. Metanitramines yield azoxy derivatives of the same colour but different dyeing powers, like the "St. Denis Red," which is their type; paranitramines yield azoxy derivatives differing both in colour and in dyeing power; orthonitramines give rise by a different reaction to colourless compounds which are not azo derivatives.

The reduction may be pushed further by raising the temperature and increasing the amount of glucose; the derivative of the azoxy-amine may then yield the free azo-amine, or ultimately the diamine. The author has thus prepared para-azo-orthotoluidine, from which, by diazotising and treating with sulphonaphthol, he has obtained a dyestuff giving bluer shades of red than the corresponding compound from the azoxy-amine.—J. T. D.

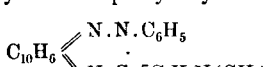
Amino-Azo Compounds. R. Möhlau and M. Heinze. Ber. 1901, **34**, [6], 881—888.

IN studying the differences between *o*- and *p*-amino-azo compounds, it is noteworthy that although the latter behave in all reactions like primary amines, still in view of the reaction of tetramethyldiaminodiphenylcarbinol with *p*-quinonoid compounds (Ber. **31**, 2351, and **32**, 2146; this Journal, 1899, 35 and 1010), and especially with *p*-hydroxyazo compounds (Ber. **33**, 2858), it appeared to be of interest to investigate the behaviour of *p*-amino-azo compounds towards this reagent. The authors find that the products obtained are not quinone derivatives, but leucauramines, and in view of their experiments they conclude that whereas *p*-amino-azo compounds form leucauramines within a wide range of temperatures, *o*-amino azo compounds only form leucauramines at temperatures up to 40° C., whilst at higher temperatures (up to 80° C.) Auramines are produced. For the preparation of benzene-azo-phenylleucauramine, equimolecular proportions of *p*-amino-azobenzene and tetramethyldiaminodiphenylcarbinol are dissolved in 96 per cent. alcohol and cohobated on the water-bath for about five hours. The condensation product crystallised from benzene-petroleum spirit separates in brownish-yellow prisms melting at 156·5° C. with an 80 per cent. yield. The action of acetic anhydride yields acetyl-*p*-amino-azobenzene. Dimethylamino-azobenzene does not react with the carbinol, proving that the reaction occurs with an amino hydrogen. Benzene-azo-α-naphthylleucauramine, obtained in a similar manner, separates in yellowish-brown prisms melting at 235° C. When *o*-azo-*p*-aminotoluene is dissolved in alcohol, together with an equimolecular proportion of tetramethyldiaminodiphenylcarbinol, and allowed to stand for about 12 hours with occasional agitation, it is converted into the corresponding leucauramine, which crystallises from benzene-petroleum spirit in red needles melting at 174·5° C. It is completely decomposed into its components by warming with dilute mineral acids. When boiled for some time in alcoholic solution until carbinol can no longer be detected by the blue colour with acetic acid, the product is converted into *p*-toluene-azo-*p*-tolylauramine, which separates in orange-yellow plates melting at 190° C. The same product can be obtained by boiling the components for some time in alcoholic solution. Benzene azo-β-naphthylamine readily condenses with the carbinol in alcoholic solution at a temperature not exceeding 40° C. The leucauramine separates from hot acetone in scarlet needles melting at 184° C. When boiled with seven times its weight of alcohol until completely dissolved, it loses two atoms of hydrogen, and forms a dark yellowish-red crystalline powder soluble in most organic solvents and melting at 74° C. The new

product is tolerably stable towards somewhat dilute hydrochloric acid, and it is therefore doubtful whether the compound is Benzene-azo-β-naphthyl-auramine—

$$C_6H_5.N_2.C_{10}H_6.N : C[C_6H_4N(CH_3)_2]_2,$$

or tetramethyldiaminotriphenyldihydronaphtho-α-triazine—

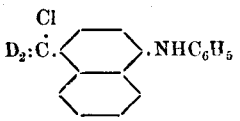


The same compound can be obtained directly by boiling together, in alcoholic solution, equimolecular proportions of benzene-azo-β-naphthylamine and the carbinol, but the product cannot be so readily isolated and purified.

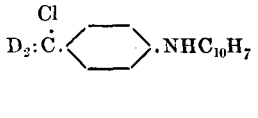
—T. A. L.

Substituted Aminobenzophenones and Aromatic Amines in Sulphuric Acid Solution; Reaction between ——. P. Lemoult. Comptes Rend. 1901, **132**, [14], 885—888.

In the presence of condensing agents, such as phosphorus oxychloride, substituted *p*-aminobenzophenones react with equimolecular proportions of a secondary or tertiary aromatic amine, forming triphenyl—or diphenylnaphthyl—methane derivatives. If, however, concentrated sulphuric acid be employed as the condensing agent, only amines of the diphenylamine type react. When 100 grms. of pure sulphuric acid, 30 grms. of tetramethyl-di-*p*-aminobenzophenone, and 17 grms. of diphenylamine are heated together at 130°—150° C. for about three hours and the melt is poured into ice water, a dyestuff is precipitated having the formula of a tetramethylphenylsulphotri-*p*-aminotriphenylmethane with a 90 per cent. yield. Sulphonation can be avoided by moderating the reaction, but condensation then only takes place very slowly. No condensation at all occurs with secondary or tertiary amines containing only a single aromatic nucleus, whilst, on the other hand, the following substances do react: methyl-, ethyl-, and benzyl-diphenylamine, phenyl-*p*- and *o*-tolylamine, but not di-*p*-tolylamine. In the case of the secondary and tertiary naphthylamines, phenyl-α- and phenyl-β-naphthylamine, *o*-tolyl- and methylphenyl-β-naphthylamine react, whilst *p*-tolyl-α and *p*-tolyl-β-naphthylamine, as well as α-β-dinaphthylamine, do not. The difference between the dyestuffs formed from phenyl-α-naphthylamine according to the condensing agent employed is illustrated as follows, where D represents $(CH_3)_2:N.C_6H_4$—



With $POCl_3$

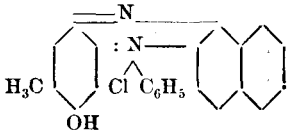


With H_2SO_4

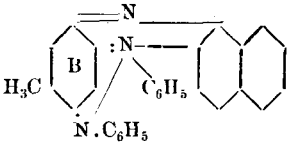
A similar reaction takes place with sulphonated tetramethyldiaminobenzophenone, the dyestuff obtained giving very similar shades to those from the unsulphonated product. The general conclusions arrived at as a result of these investigations are that substituted *p*-aminobenzophenones in sulphuric acid solution give dyestuffs with certain aromatic amines which contain at least two aromatic nuclei directly attached to nitrogen. One of these must be a phenyl group in which the position para to the nitrogen is free.—T. A. L.

Isorosinduline and Isorosindone Reaction. O. Fischer. Ber. 1901, **34**, [6], 940—949.

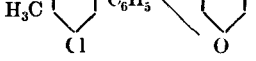
In determining the limits of the reaction between nitroso compounds (nitroso-aniline, -dimethylaniline, -phenol, -α-naphthol, &c.), and β-alkyl- or alphylnaphthylamines, resulting in the formation of isorosindulines and isorosindones, the author finds that the nitroso-*o*- and *m*-toluidines, as well as the nitroso-*o*- and -*m*-cresols, readily react with β-methyl- or -phenylnaphthylamine, but homologues of nitroso-aniline or nitroso-phenol containing two alkyl groups, whatever their position, do not form isorosindones. Such, for example, are nitroso-*p*-xylenol, -thymol, and -carvacrol. B-*o*-methylisorosindone is obtained from nitroso-*o*-cresol and phenyl-β-naphthylamine according to the method already given for isorosindone (Ber. **29**, 2755; this Journal, 1897, 132). The base crystallises from 70 per cent. alcohol in long reddish-brown metallic needles melting at 258° C., and having the formula—



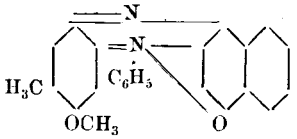
It is only a weak base, and its well-crystallised salts are dissociated by water and alcohol. By the action of phosphorus pentachloride it is converted into B-*o*-methylchlorophenylnaphthophenazonium chloride, in which, like isorosindone chloride, the chlorine atoms are unequally bound, the azonium chlorine being readily replaced in cold aqueous or alcoholic solution by I, Br, or NO_3, &c. On boiling the alcoholic solution of the chloride with two molecular proportions of aniline for several hours under an inverted condenser, it yields B-*o*-methylphenylisorosinduline—



The base separates from benzene in coppery crystals, and dissolves with a blue colour in concentrated sulphuric acid. The homologous B-*o*-methyl-*p*-tolylisorosinduline from the chloride and *p*-toluidine melts at 226° C., and dissolves to a greenish-blue solution in concentrated sulphuric acid. The chloride gives with β-naphthylamine, B-*o*-methyl-β-naphthylisorosinduline, a reddish-violet dyestuff, giving a deep blue solution in concentrated sulphuric acid, becoming reddish-violet on dilution. On boiling the aqueous solution of the dichloride with two molecular proportions of sodium acetate, it is converted, like isorosindone chloride (Ber. **33**, 1485; this Journal, 1900, 654), into B-*o*-methylchlororosindone—



On heating B-*o*-methylisorosindone with an excess of methyl iodide under pressure at 100° C. for several hours, and treating the iodide obtained with alcoholic potash, there is obtained the corresponding *o*-methylmethoxyrosindone—



which separates from alcohol in shining brownish-red needles, melting at 288° C. By condensing nitroso-*m*-cresol with phenyl-β-naphthylamine, it is converted into *m*-methylisorosindone, which crystallises from dilute alcohol in golden bronzy needles. The base gives magenta coloured solutions in alcohol, benzene, and chloroform, whilst the salts are readily dissociated by water. It dissolves to a reddish-violet solution in concentrated sulphuric acid, which on dilution becomes brown and then reddish-yellow, depositing on cooling, the sparingly soluble sulphate.

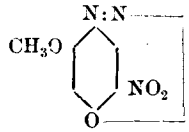
In conclusion, the author characterises some new products. 2-nitroso-1.3.5-xylenol is obtained by known methods from *s*-xylenol, melting at 64° C. It crystallises from dilute alcohol in yellowish prisms, melting at 175° C., and is readily soluble in alcohol, ether, benzene, and ammonia,

but almost insoluble in cold water. 6-nitroso-1.2.3-xylenol is obtained from 1.2.3-xylenol. It forms yellowish needles, melting at 166° C., and has a similar solubility to the foregoing. 2-nitroso-1.3.5-ethylxylidine is produced from *s*-xylidine, boiling at 221° C. by heating with ethyl bromide, conversion into the nitrosamine and transformation into the nitroso base, which separates from benzene in steel blue prisms, melting at 138° C.—T. A. L.

Dinitroanisidine and the Constitution of the Resulting Product; Diazotisation of ——. R. Meldola and J. V. Eyre. Proc. Chem. Soc. **17**, [238],

A PAPER just published by Freyss (this Journal 1901, 356), anticipates some conclusions at which the authors have arrived as the outcome of an investigation upon which they have been engaged since last October. They are led, therefore, to place upon record the results of those experiments which appear to give independent support to the conclusions of the author above named. This work is an extension of that already published (Meldola and Wechsler, Trans. Chem. Soc. 1900, **77**, 1172; Proc. 1898, **14**, 226, this Journal, 1898, 1138).

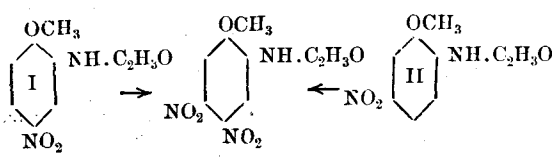
The crystalline diazo-compound, obtained by the action of a nitrite on dinitroanisidine in acetic acid solution, is a diazoxide of the formula—



The proof of this constitution is given by several considerations, some of which appear from the experiments of Freyss, the most cogent, perhaps, being its ready convertibility into nitromethylresorcinol, $C_6H_3.NO_2.OH.OCH_3 = 1:2:4$. The authors find the most convenient method for bringing about this conversion is to dissolve the diazoxide in alcoholic sodium hydroxide. The decomposition takes place at the ordinary temperature, with the evolution of nitrogen and the formation of aldehyde. On diluting the alkaline alcoholic solution with water and acidifying, the nitroresorcinol methyl ether separates in a crystalline form, and is best purified by steam distillation.

The diazoxide is remarkably stable towards acids; it can be boiled with dilute hydrochloric or sulphuric acid and can be crystallised from boiling glacial acetic acid or from acetic anhydride without undergoing decomposition. It is decomposed on boiling with hydriodic acid solution, and the product is iodonitroresorcinol methyl ether (Meldola and Wechsler, *loc. cit.*, 1173), $C_6H_2.OCH_3.OH.NO_2.1 = 1:3:4:6$. The azo-β-naphthol derivative described in the former note (*loc. cit.*) has the formula $C_6H_4.NO_2.OCH_3.OH.N_2.C_{10}H_6.OH$. In addition to the properties already assigned to this compound, it may be added that it is distinctly phenolic in character, dissolving in cold aqueous alkali with a dull claret-red colour and being precipitated unchanged by acids. The phenolic character is due to the hydroxyl group in the para position. An examination of the product of the action of ammonium sulphide on the azo-compound has shown that no amino-azo-compound is formed, and this is in accordance with the view that the nitro-group is not in the para-position with respect to the azo-group (Meldola, Trans. Chem. Soc., 1883, **43**, 425).

The constitution of dinitroanisidine (m. pt. 188°) first described (Meldola and Wechsler, *loc. cit.*) follows from the fact that it is obtainable by the further nitration of *both* the nitroacetanisidides resulting from the nitration of *o*-acetanisidide—

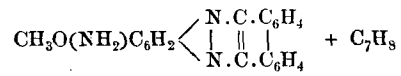


The authors have proved this directly by the nitration of carefully purified specimens of the mononitroacetyl derivatives. No. I. was prepared by the reduction of dinitroanisol and acetylation of the nitroanisidine (Meldola, Woolcott, and Wray, Trans. Chem. Soc. 1886, **69**, 1330). No. II. was prepared from the corresponding nitroanisidine, the latter being obtained pure, by a modification of the method formerly described (this Journal, 1898, 1138). The mixture of nitroacetyl derivatives resulting from the nitration of *o*-acetanisidide is boiled with dilute alkali till completely hydrolysed. The mixed nitroanisidines on being dissolved in hot dilute sulphuric acid deposit, on cooling, silvery scales of the sulphate of the *p*-nitro-compound, $C_6H_3(NO_2)(NH_2)OCH_3 = 1:4:5$, the sulphate of the isomeric compound remaining in solution. The crystalline sulphate gives pure *p*-nitro-*o*-anisidine on treatment with dilute alkali and crystallisation of the product from hot water. In order to characterise further the two nitroanisidines and the dinitroanisidine, the benzoyl derivatives have been prepared: $C_6H_3(NO_2)NH(C_7H_5O).OCH_3 = 1:4:5$, white silky needles from alcohol, m. pt. 149°—150° C.; $C_6H_3(NO_2)NH(C_7H_5O).OCH_3 = 1:3:4$, slender ochreous needles from alcohol, m. pt. 160°—161°; $C_6H_2.(NO_2)_2.NH(C_7H_5O).OCH_3 = 1:2:5:4$, ochreous scales from acetic acid, m. pt. 185°—186° C.

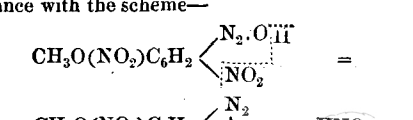
The constitution of the *p*-nitro-*o*-anisidine was proved by two methods differing from those adopted by Freyss, as well as by conversion into the *p*-nitro-guaiacol of m. pt. 104°. The latter compound, of which the discovery is assigned to Rupe (Ber. 1897, **30**, 2446), was first described by one of the authors (Meldola, Proc. Chem. Soc. 1896, **12**, p. 125). The *p*-nitro-*o*-anisidine when diazotised and the amino-group replaced by iodine in the usual way, gave an iodonitroanisol which crystallised in straw-coloured needles melting at 127°—129°.

The constitution of the nitroanisidine of Cahours (Meldola, Woolcott, and Wray, *loc. cit.*) was further proved by converting it into the corresponding iodonitroanisole by replacing the amino-group by iodine by the diazo-method. The iodonitroanisol, $C_6H_3(NO_2)I.OCH_3 = 1:3:4$, melts at 95°—96° and is identical with that described by Reverdin (Ber. 1896, **29**, 998; this Journal, 1896, 534).

Experiments were in progress to obtain direct evidence of the constitution of the dinitroanisidine at the time of appearance of the paper by Freyss. The triaminoanisol obtained by reducing the dinitroanisidine with tin and hydrochloric acid forms a crystalline hydrochloride, but it is very unstable, passing readily into a deep violet colouring matter on exposure to the air. The dinitroacetanisidide does not give an anhydro-base on reduction, an observation which is in harmony with the view that the acetamino-group is not in the ortho-position with respect to a nitro-group. On the other hand, that the triaminoanisol contains two amino-groups in the ortho-position is proved by the formation of an azine with phenanthrenequinone. On heating together equimolecular weights of the triamine and the quinone in glacial acetic acid, the aminoazine is gradually formed, and on adding hydrochloric acid the hydrochloride is completely thrown out on cooling as a dull red precipitate. The free base is an ochreous powder which dissolves in alcohol with a magnificent green fluorescence. It does not readily crystallise, but separates from boiling toluene in the form of brown nodules having a melting point of about 237° C., but softening below this temperature. The base thus obtained contains one molecule of toluene and has the formula—



The result of this investigation thus confirms the conclusion that on diazotising 3:4-dinitro-*o*-anisidine in acetic acid, it is the 4-nitro-group which is eliminated, probably in accordance with the scheme—



Detection of Dyestuffs; Application to Indophenols. C. Camichel and P. Bayrac.

See under XXIII., *page* 621.

PATENTS.

Black Disazo Dyestuffs; Manufacture of ——. C. D. Abel, London. From The Actien-Gesellschaft für Anilin Fabrikation, Berlin. Eng. Pat. 10,844, June 14, 1900.

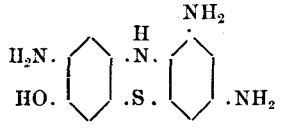
This specification is supplementary to Eng. Pats. 24,527 of 1897, 2360 and 6583 of 1899 (this Journal, 1898, 916; 1900, 42 and 238), which relate to the manufacture of black disazo dyestuffs by combining the diazo compounds of *p*-aminodiphenylamine sulphonic acids, *p*-aminophenyl-β-naphthylamine sulphonic acids or *p*-aminodiphenylamine carboxylic acids or their homologues or substitution products with α-naphthylamine, diazotising the products so obtained, and combining them with suitable dyestuff components. The patentees now find that dyestuffs of a similar character are obtained by using as first components, sulphonic acids of *p*-aminodiphenylamine which contain both a hydroxyl and a carboxyl group. These products are formed by the action of *p*-nitrochlorobenzene *o*-sulphonic acid on *p*-aminosalicylic acid or on *p*-aminocresotic acid, and subsequent reduction of the nitro-products obtained. Since the new products contain hydroxyl and carboxyl groups they are suitable for dyeing on chromium mordants. The process employed is analogous to that described in Eng. Pat. 24,527 of 1897 (*loc. cit.*). The dyestuffs give directly violet-black shades, and on chrome mordanted fibres produce a deep black.—T. A. L.

Sulphurised Leuco-Compound; Manufacture of a ——. C. D. Abel, London. From the Actien Gesellschaft für Anilin Fabrikation, Berlin. Eng. Pat. 10,843, June 14, 1900.

According to Ger. Pat. 103,301, leuco-thionoline is produced by the action of sulphur on mixtures of equimolecular proportions of certain disubstitution products of benzene. The patentees now find that another sulphurised leuco-product, containing two more amino groups than the above compound, can be obtained by the action of sulphur and sodium sulphide on diaminophenol—

$$OH : NH_2 : NH_2 = 1 : 2 : 4.$$

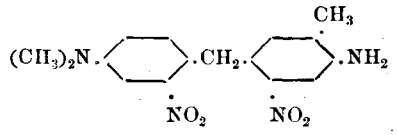
The new product has the formula—



and the following method gives a satisfactory yield. A solution of 124 kilos. of diaminophenol in 750 litres of hot water is mixed with 60 kilos. of crystallised sodium sulphide and 32 kilos. of sulphur in 90 litres of water, the whole being boiled under a reflux condenser for some time, when diaminoleucothionoline separates in shining leaflets and is filtered off. The new product forms shining yellowish leaflets, scarcely soluble in water, alcohol, ether, or benzene, readily soluble in dilute acids, caustic alkalis, and sodium sulphide solution. It is converted by the action of oxidising agents into the corresponding thionoline which combines with acids and alkalis to form violet and blue coloured salts respectively.—T. A. L.

Colouring Matters [*Yellow*] *of the Acridine Series; Manufacture or Production of New* ——. H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 11,035, June 18, 1900.

Unsymmetrically dialkylated or trialkylated derivatives of tetra-aminodiphenylmethane or their homologues, obtained by nitrating and reducing the corresponding unsymmetrically dialkylated or trialkylated *p*-diaminodiphenylmethane bases, or their homologues, are heated with agents capable of removing ammonia, and are then oxidised to the new dyestuffs. For example, 24 kilos. of dimethyldiaminophenyl-*o*-tolylmethane (Ger. Pat. 107,718) dissolved in 200 kilos. of sulphuric acid (66° B.) are nitrated below 5° C. with a mixture of 21·5 kilos. of nitric acid (40° B.) and 50 kilos. of sulphuric acid monohydrate. The melt is poured into water and neutralised with sodium carbonate when dinitrodimethyldiaminophenyl-*o*-tolylmethane—



separates in brick-red crystals. The product, after recrystallisation from acetone, forms red leaflets melting at 160° C. In order to reduce it, it is dissolved in 5 times its weight of hydrochloric acid (36·5 per cent.) and treated in the cold with twice its weight of zinc dust. After filtering from excess of the latter, the diamino product is precipitated by caustic soda lye. On recrystallisation from chloroform, it yields colourless leaflets melting at 177° C. In order to obtain a dyestuff, 1 kilo. of the product so formed is mixed with 10 kilos. of sulphuric acid (20 per cent.) and boiled for seven hours, in a lead-lined vessel. The resulting solution is mixed with about 1·25 kilos. of calcined sodium carbonate, and oxidised with the necessary quantity of ferric chloride, after adding salt and zinc chloride. The dyestuff so produced forms a reddish-brown resinous mass, which soon solidifies to a red crystalline product, easily soluble in cold water, to an orange-yellow solution. It dyes leather level yellow shades.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Oxycelluloses; Researches on ——. A. Nastukoff. Ber. 1901, **34**, [5], 719—723.

The author has oxidised Swedish filter paper with (1) chloride of lime solution as in previous experiments (Ber. 1900, **33**, 2237, this Journal, 1900, 733); (2) permanganate solution followed after 36 hours by the introduction of sulphur dioxide gas, and finally the addition of weak and lukewarm sulphuric acid. The products in both cases appeared to be α-oxycelluloses. By heating these on the water-bath with 10 volumes of sulphuric acid solution (5 per cent.), washing, and again heating with a like amount of sodium carbonate solution (10 per cent.), a new class of oxycelluloses, characterised by solubility in water, were obtained in 60—80 per cent. yield. The oxycellulose resulting from the action of chloride of lime required to be heated with acid for three hours; that from the permanganate oxidation for only one hour. A small quantity of sugar was formed during this acid hydrolysis and a larger quantity if the time of heating was extended; it gave a hydrazone, possibly mannose-hydrazone. The alkaline hydrolysis need be continued for only 10—30 minutes at a temperature of 70°—100° C. For the new soluble oxycelluloses the author proposes the name γ-celluloses as he considers them to be different to the class already distinguished by the prefix β- (Cross and Bevan, J. Chem. Soc. 1883, 22. See also this Journal, 1884, 206 *et seq.*).

Their properties are briefly as follows:—The aqueous solution when dilute is opalescent or milky, and yellow in transmitted light, is quite filterable and does not alter on standing or when heated. Stronger solutions (5—10 per cent.) resemble glycerin or viscose, and when dried in a desiccator or over a water-bath deposit silky transparent scales or plates. The addition of various metallic salts, of acids or alcohol, produces precipitation. When precipitated by an acid, the solubility gradually diminishes as the substance dries, but is restored by the action of warm sodium carbonate solution. If washed with almost any dilute acid, the oxycellulose becomes insoluble once more, and this cycle may be repeated as desired. Drying at 110° C. does not however destroy the solubility.

γ-Oxycellulose reduces Fehling's solution when heated and forms a yellow hydrazone which is insoluble if prepared from the insoluble oxycellulose, but like it, becomes soluble

by the action of alkali; also its solutions are yellow, opalescent, and deposit lustrous gold-coloured scales. Iodine produces no coloration; the pentosan reaction does not take place; and the ash has an alkaline reaction.

γ-Oxycellulose, in its soluble form, would appear to be the sodium salt of a soluble acid which, when dried, becomes an insoluble anhydride or lactone.—R. L. J.

PATENTS.

Fatty Matters from Wool, Sheepskins, and other Textile Fibres; Process and Apparatus for Extracting ——. C. D. Abel. Eng. Pat. 2360, 1900.

See under XII., *page* 591.

Mercerizing of Yarn; Machines for Use in ——. F. Shuman, Philadelphia, U.S.A. Eng. Pat. 3568, Feb. 19, 1901.

A MACHINE, chiefly applicable to the mercerising of loose yarn. The latter is taken up on a moving frame composed of two endless chains which travel through the mercerising fluid. By means of cam discs the yarn is stretched during mercerization and washing.

(See also this Journal, 1898, 149, 240, 345, 452, 573, 756, 840, and 1141; 1899, 36, 136, 267, 49, 680, 911, and 1122; 1900, 43, 144, 240, 821, and 1011.)—C. M.

Textile Fabrics and the like, Non-Inflammable and Waterproof; Process and Composition for Rendering ——. C. Baswitz, Berlin. Eng. Pat. 8267, May 4, 1900.

THE following composition, namely:—34 parts of "amphibolin, a fire-resisting natural earth found near Baden," 39 parts of size or other gelatinous material, 2 parts of chrome alum, 2 parts of ammonium sulphate and 53 parts of water, is thoroughly mixed and then applied to the fabric by a brush, or priming machine. After each coat the fabric is exposed to light. The number of coats vary with the purpose for which the fabric is wanted.—C. M.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Fog in Dye- and Bleach-Works; Prevention of ——. F. Milius. Färber Zeit. 1901, **12**, 117—118.

IN order to prevent the condensation of steam in a dyehouse, the air must be changed 8—10 times hourly, and the air which is blown in should be heated 22°—30° C. above its original temperature. Air heated, for example, from − 15° C. to + 15° C. is capable of taking up 11·42 grms. of water vapour per cubic metre, and the author describes a dyehouse of 2,630 cb. m. into which 26,300 cb. m. of air thus warmed are introduced hourly. To heat this air through 30° C. there are required 236,700 cal., or allowing 200,000 cal., for machines, walls, roof, etc., a total of 436,700 cal. In the works described, this heat is provided by the waste flue gases which are drawn off at the foot of the chimney into a heating apparatus consisting of a series of copper pipes, where they give up a portion of their heat to cold air blown in from outside. This air, when warmed, passes to the dyehouse, entering it by a number of openings about 2½ metres above the floor.

The temperature of the flue gases is 225° C. at their entrance into the copper pipes. The boiler fires burn 800 kilos. of coal per hour, which, at N.T.P., will give 16,800 cb. m. of gas, or at 225° C., 30,408 cb. m. In the pipes the temperature of the gases is lowered to 100° C. and 1,178,310 cal. are thus provided hourly, a far greater amount of heat than is required as shown above, and the cost of working is practically nothing. If the heat were supplied by direct steam at, say 75 lb. pressure, about 775 kilos. of steam would be required hourly. For the production of this heat there would be used 103 kilos. of coal at a cost per day of 10 hours of 23·7 marks (23*s.* 6*d.*).

The arrangement is thus very cheap, and it works satisfactorily. The workmen are not exposed to draughts, owing to the height of the inlets above the floor. The admission of this large volume of air at a comparatively high temperature keeps the air always far above the dew point, and it can therefore undergo a considerable degree of cooling without the deposition of water. Outlets should be provided in the roof to the extent of 40 sq. cm. for every 25 sq. m. of floor. —R. B. B.

Dyeing; Effect of Temperature in ——. R. B. Brown. J. Soc. Dyers and Col. 1901, **17**, [4], 92—99.

IT may be stated broadly that wool requires a higher temperature in mordanting and dyeing than cotton; while silk occupies an intermediate place in this respect (see this Journal, 1901, 226).

The basic dyestuffs should never be dyed at the boil. This is true not only for wool, to which fibre they are seldom applied, but also for tannin-mordanted cotton. The temperature in dyeing with basic dyestuffs should not exceed 80° C., and even at this temperature some of them become pale and dull. Thus Chrysoïdine, which at 60° C. yields a bright orange, at 80° C. dyes only a dull orange yellow. With Auramine, the difference is still more marked, this dyestuff being decomposed on boiling, as is also Turquoise Blue. When it is absolutely necessary to apply the basic dyestuffs at the boil, as in straw dyeing, such colours as these must be strictly avoided. Other colours, again, can be boiled for a short time, but will not withstand prolonged boiling. An instance of this class is Brilliant Benzo Green B, which dyes a bluish-green after half an hour's boiling, but on continuation of the boiling turns to a dull red or brown, and finally to a grey colour. The Sulphocyanine group of dyestuffs, it is stated, suffers decomposition in the same way.

In some instances it is necessary to dye at a low temperature in order not to injure the material which is being dyed. This is the case with leather. If ordinary "vegetable-tanned" leather is steeped in a solution heated to above 50° C., it is practically reduced to the condition of skin again, and on drying it shrinks and becomes extremely brittle. Such leather is therefore dyed at a temperature not exceeding 45° C., and a considerable amount of dyestuff as a consequence is usually left in the dyebath. Dyestuffs, *e.g.*, those of the Alizarin group, which are not dissolved at this temperature, cannot be employed. "Chrome" leather, on the other hand, can be dyed at 80° or 90° C., or even at 100° C., without injury, so that the dyestuffs available in the dyeing of this are much more numerous. With sheepskins, the wool does not become dyed at a low temperature, and the leather would be destroyed at a high temperature. The difficulty is met by chlorinating the wool, which may then be dyed full and even colours at a temperature of 45° C., without injury to the skin.

The property of chlorinated wool may be applied in another way. Woollen materials are printed with bleaching powder and then dyed at a low temperature, the effect being produced of a dark pattern on a paler ground.

In the dyeing of artificial silks made from nitrocellulose, the fibre is greatly weakened by treatment with hot solutions. It is, therefore, necessary to dye in a lukewarm bath, for which purpose the basic dyestuffs are the most suitable. The so-called Vanduara silk (this Journal, 1899, 16) is dyed prior to the spinning process, owing to the tendering action of water upon it.

In the manufacture of straw hats an artificial substitute for straw is frequently plaited together with the natural product. This material, which is known as satin-straw or satin-chip, is employed on account of its high lustre. It is made from fine threads of natural or artificial silk, or sometimes of hemp or cotton, placed side by side and caused to adhere together in the form of a band, by being pasted over with a solution of gum or gelatin. It is not, as a rule, dyed after manufacture, as the gelatin, on immersion in water, is dissolved, and the plait then falls to pieces. It may, however, by dyed, after being subjected to the action of formaldehyde vapour to render the gelatin insoluble. The "chip" (shavings of willow wood), with which the satin-straw is usually plaited, is dyed at 60°—70° C. by certain basic dyestuffs, like the satin-straw itself.

A wide diversity of behaviour in dyeing wool and cotton unions is met with in the direct cotton dyestuffs. Some of these, *e.g.*, the Sulphone group, in a boiling bath, dye the wool only, and merely tint the cotton; others, *e.g.*, the

Mikado dyestuffs, act in the opposite way. It is therefore possible by mixing dyestuffs of opposite tendencies to produce "shot" effects in a single boiling bath, but the shades are not remarkable for brilliancy or purity of tone. The method might be used in dyeing "solid" shades, by mixing two dyestuffs of similar colour but opposite properties, but this is seldom done, since there are now a large number of direct dyestuffs which yield "solid" colours in a single dyebath.

To produce "shot" effects, it is sometimes desirable to dye the cotton in the cold with a direct dyestuff, after the wool has been dyed with an acid dyestuff. The following dyestuffs have been selected as giving full shades upon cotton in the cold, while leaving the wool but little affected: Diamine Rose B extra, Chloramine Orange, the Mikado Oranges and Yellows, Direct Yellow R (Bayer), Diamine Fast Yellow C, Columbia Green, Chicago Blue 4 B, Columbia Black B, Diamine Black R M W, Diamine Milling Black B, Direct Grey B (S.C.I. Basle), Toluylene Brown R, Cotton Dark Brown B M, Congo Brown R, Benzocyanine 3 B. Certain of the Blacks are now employed in place of tannic acid and iron salts in the so-called "burl-dyeing" process for unions.

The Janus colours are applied in the dyeing of cotton and wool unions in an acid bath. Some of them, *e.g.*, Janus Claret Red, give "solid" colours at the boil; others, *e.g.*, Janus Yellow G, at 80°—90° C., while Janus Red B does so at 60° C. The makers recommend, instead of dyeing below the boil, first to boil the dyebath to cause the wool to take up colour, and then to cool down the bath in order to dye the cotton.

In dyeing the unions of cotton and silk, the direct cotton dyestuffs are chiefly employed. The temperatures at which the two fibres are dyed alike, varies with the dyestuff, but it is frequently about 80° C. Many of the direct dyestuffs are, however, quite unsuitable for the purpose. With Geranine G the two fibres are similarly dyed up to a temperature of 60° C.; beyond that, to 80° C., the silk is more deeply dyed, while the cotton is less deeply dyed as the temperature is increased. The same dyestuff at 80° C. dyes wool and cotton alike. Direct Yellow G (Kalle) at all temperatures dyes cotton more strongly than it does the animal fibres. Diamine Green B is an example of a dyestuff which is suited for dying tissues composed of silk, wool, and cotton, as at 80° C. it gives "solid" colours on unions of all these fibres.—E. B.

Potassium Permanganate in Dyeing; Application of —— [for Manganese Brown]. M. G. Saget. Monit. Scient. **15**, May 1901, 319—320.

Potassium permanganate may be employed in cotton dyeing for the production of pale brown or drab shades. Its oxidising action is made use of to destroy a colour already on the fabric with the production of manganese peroxide in the interior of the fibre. Metallic tannates form the most suitable preliminary colours, and the final depth of shade will vary according to the amount of tannin fixed; excess of permanganate is therefore not to be feared. In applying the permanganate any rise of temperature must be avoided or uneven dyeing results; reduction takes place very rapidly even in the cold. The grey obtained with tannin and iron is a useful starting point for brownish drab (Beige) shades. The cotton is padded in chestnut extract, catechu, etc., and subsequently in pyrolignite of iron; after some hours' exposure to air it is washed and passed through a cold dilute solution of the permanganate; at the end of 15 minutes the grey has completely disappeared and given place to the required drab. The iron may be replaced by other metals, *e.g.*, Pb, Cu, Cr, Al, Mn, and the colours of their oxides are added to that of the manganese brown. The acetates of the metals are to be preferred to any other salts. Calcium plumbite fixed on the fibre also reduces permanganate with production of a reddish-brown, a mixture of the peroxides of lead and manganese.

The shades of this kind produced with permanganate are very fast to the action of chlorine, but boiling soap impoverishes the colour and it fades very rapidly in sunlight, the colour being restored by chlorine. The fastness to light is increased by an after treatment with sodium carbonate at 60° C., which does not influence the shade. The same improvement is effected by a treatment with bleaching powder solution at $\frac{1}{2}$° B. in the cold, followed by a thorough rinsing.

Steaming makes the colour much paler, and acids destroy it, even when very dilute; it is also discharged by bisulphite of soda and by other mineral and organic reducing agents.

On oil-prepared cotton the shades are much faster, especially if the goods are soaped at 60° C. after the permanganate bath; the colour then resists the action of light and steaming and is fairly fast to soap. The cotton may either be previously prepared with oil or the oil may be added to the tannin bath rendered alkaline with ammonia. The goods in this case must be dried before applying the metallic salt. The shade is greyer than on goods which have not been oiled. A drab dyed on cotton which has not been oil-prepared is made faster to acids and soap, but not to light, by padding in acetate of alumina and then drying and steaming without pressure; the resulting shade is also yellower and brighter in tone.

Peroxide of manganese fixed on the fibre in this way can act as a mordant for various colouring matters and also as an oxidising agent for certain aromatic amines.—R. B. B.

Dyeing Wool Black by means of Nitrosulphide of Iron. M. Prud'homme. Monit. Scient. 1901, 251—252.

The nitrosulphides of iron discovered by Z. Roussin in 1860 are prepared by adding gradually a solution of 350 grms. of ferrous sulphate in 2 litres of water to a solution of 210 grms. of potassium nitrite and 150 grms. of sodium sulphide also in two litres of water. After some minutes' boiling, the liquid is filtered; on cooling, the filtrate deposits black crystals of a composition corresponding to the formula $Fe_3S_3K_2(NO)_4$. These compounds have been studied by O. Pawell, who recommends for their production the addition of 40 grms. of sodium sulphide dissolved in 300 grms. of water to a boiling solution of 40 grms. of potassium nitrite in 600 grms. of water. Into this mixture is poured with continual stirring a solution of 70 grms. of ferrous sulphate in 300 grms. of water. The formula of the black crystals so obtained after suitable purification would be $Fe_7S_5K_2(NO)_{12}$.

The solution of nitrosulphide of iron prepared by either of the above methods possesses the property of dyeing unmordanted wool from a neutral bath, giving a black with a brownish reflection. If the wool is previously mordanted with iron, or still better with copper, the black is somewhat less brownish. The potassium nitrite in the above recipes can be replaced with an equivalent quantity of sodium nitrite.

The author has found that the preparation of a solution for dyeing is best effected by dissolving 14 grms. of ferrous sulphate in 150 grms. of boiling water, and pouring the solution with constant stirring into a mixture of the requisite amounts of nitrite and sulphide of sodium dissolved in 200 grms. of water. The liquid is boiled for several minutes and filtered, the residue being washed with hot water until the filtrate amounts to 500 c.c. This filtrate, diluted with water, forms the dyebath without any addition, and the solution made from 14 grms. of ferrous sulphate is sufficient to dye 25 grms. of wool.

The wool at first acquires a yellowish-brown colour, and only becomes black after dyeing for $\frac{3}{4}$—1 hour at 90°—95° C. The bath is perfectly exhausted, and since it was found to contain sodium nitrite the author diminished the quantity of this substance in Pawell's recipe, which is better than that of Roussin. The proportions finally adopted were $FeSO_4$, 14 parts; Na_2S, 8 parts; and $NaNO_2$, 5 parts.

The black obtained by the use of this mixture is very brownish when viewed "overhand," and to remedy this defect the wool may be previously dyed with logwood or indigo, or with a green, blue, or violet coal-tar dyestuff. A still more satisfactory black is produced by first dyeing the wool Prussian blue with 10 per cent. of potassium ferricyanide and 10 per cent. of sulphuric acid for $\frac{3}{4}$ hour at 95° C., washing well, and re-dyeing with nitrosulphide of iron. After dyeing the Prussian blue, the shade becomes deeper and more violet if the wool be passed through water containing 10 grms. of ammonia per litre.

The colour resists the action of dilute acid and of SO_2, and the fastness to alkalis is also good. In boiling water it bleeds a little, giving a brown tinge to the water, and it rubs off a little when wet, producing a pale blue stain which is due to the Prussian blue. The fastness to light is satisfactory. The cost of the materials used for dyeing this black amounts to about 2*d*. per pound of wool dyed.

—R. B. B.

Sulphur Colours; Employment in Dyeing and Printing of a New Class of ——. A. G. Green. J. Soc. Dyers and Col. 1901, **17**, [4], 89—91.

THE dyestuffs in question are known in commerce as *Clayton Fast Blacks* (Eng. Pat. 21,832 of 1898, 5,039 and 18,658 of 1899; this Journal, 1899, 909; 1900, 141 and 816). The commercial preparations are the undiluted dyestuffs, free from all inorganic sulphur compounds. They are black powders, which are quite insoluble in water, but dissolve freely in aqueous solutions of sodium sulphide or of sodium sulphite; some also dissolve in caustic alkalis.

Their application in dyeing is based upon their becoming reduced to leuco compounds, which are absorbed by the cotton fibre, and re-oxidised and fixed upon this in an insoluble form. In the case of most of the "sulphide" colours, this reduction is commonly effected by means of sodium sulphide, which is either present in the dyestuff itself, or is added for the purpose to the dyebath. The same method can be employed with the new dyestuffs, but it is not recommended owing to (*a*) the tendency under certain conditions, at present not clearly ascertained, of the tissues so dyed becoming "tender," more particularly after they have been stored for some time, or when undergoing subsequent steaming or hot pressing (see this Journal, 1901, 439); (*b*) the liability to give uneven dyeings through precipitation of insoluble colour on the surface of the fibre, necessitating, in consequence, great care to avoid exposure to air during dyeing; (*c*) the necessity for excluding all copper or brass parts (steam pipes, rollers, &c.), from the dye-vessels; (*d*) the unpleasant odour arising from the waste liquors when run into the drains.

With the new dyestuffs these difficulties have been overcome in the following way (Eng. Pat. 15,413 of 1900):—The dyestuffs are dissolved by boiling in a concentrated solution of neutral sodium sulphite, with which they form easily soluble sulphite compounds (probably salts of thiosulphonic acids). The latter are stable compounds of little colour, and devoid of dyeing power, but by reducing agents they are readily converted into leuco compounds (probably sulphhydryl derivatives), which are possessed of dyeing properties. Dyeing occurs with these as they are oxidised in the air, the sodium sulphite present combining with that portion of the re-formed dyestuff which has not been fixed upon the fibre, and thus holding it in solution for later use and preventing uneven dyeing. The reduction is effected preferably by glucose and an alkali, which are added in portions during the dyeing in amount sufficient to maintain the dyestuff in a satisfactorily reduced condition. After dyeing, the cotton materials are thoroughly washed, and the dye is then fixed by metallic salts, preferably copper sulphate and an alkali bichromate, the latter treatment being necessary when absolute fastness to soap is required.

This process has given good results with mercerised tissues, which can be dyed upon the ordinary jigger without any special precautions, such as keeping the tissue below the surface of the liquor, &c. All that is necessary is that a squeezing roller shall be attached to the machine.

The colour obtained, for instance, from Clayton Fast Black M, compares well in depth and fastness with Aniline Black, over which it has the advantage of not greening and of not weakening the fibre, while it is easier to produce. Moreover, in the case of mercerised cotton it does not diminish the gloss.

In calico printing, the dyestuffs are applied, either in the insoluble form or as sodium sulphite compounds, in conjunction with a thickening agent and caustic soda (Applic. for Eng. Pat. 17,193 of 1900). A suitable printing mixture is prepared from 100—200 parts of Clayton Fast Black B P, paste, 10 parts of starch, 170—70 parts of water, and 220 parts of caustic soda-lye at 70° Tw. (making in all 500 parts). The tissues printed with this are steamed without pressure for ¼—1 hour. By preparing the tissues with a 5 per cent. solution of glucose, the duration of the steaming operation may be shortened to 3—10 minutes. After steaming, the tissue is well washed to remove caustic soda, and, if a high degree of fastness be required, it is passed through a boiling bath containing 6 parts of copper sulphate, 6 parts of acetic acid, and 2 parts of sodium bichromate per 1000 parts of water. As no special reducing agent is necessary in the printing process, it is considered probable that under the influence of steam and caustic soda, the cotton fibre itself fulfils this function. The addition of glucose to the printing mixture has no effect.

By employing Clayton Fast Brown P in the above manner, dark brown prints are obtained.—E. B.

White and Coloured Resists for Paranitraniline Red. Textile Colorist, 1901, 77.

THE following mixtures are printed upon cotton tissues, prepared as usual with sodium β-naphtholate. After being dried, the tissues are passed through a solution of diazotised paranitraniline, and are then rinsed and soaped, or otherwise treated as described below.

The *white resist* mixture is composed of 1 kilo. of a 50 per cent. solution of caustic soda, and 160 grms. of British gum solution (65 : 100), or of 1 litre of caustic soda solution (5 : 4), and ¼ litre of dextrin solution (65 : 100).

The *blue resist* consists of 1 kilo. of one of the above mixtures, to which are added 150 grms. of caustic soda, ⅓ litre of Indigo paste (20 per cent.), 40 grms. of glycerin, 300 grms. of glucose, and 40 c.c. of water. This is printed and steamed for about a minute with wet steam, before developing the Red, the tissue being afterwards soured, washed, soaped, washed and dried.

1 kilo. of the white resist, along with 250 grms. of lead nitrate, 100 grms. of glycerin, and 100 c.c. of water, forms the *yellow resist*. After the development of the red, which is effected with the addition of 30 grms. of chalk to the litre of diazotised paranitraniline solution, the yellow is "raised" in a 5 per cent. solution of sodium bichromate, and the tissue is rinsed and dried.

A *blue resist* may also be obtained from a mixture of 1,200 grms. of the white resist, 900 grms. of a solution of 500 grms. of potassium ferrocyanide in 1 kilo. of the white resist, and a solution at 50° B. of 375 grms. of "nitrate of iron" and 375 grms. of glycerin. The tissues printed with this are passed successively through baths of chalk water, dilute (5 per cent.) sulphuric acid and water, after going through the diazotised paranitraniline bath.—E. B.

White Discharge Effect on a Blue Ground; Production of —— [*in Calico Printing*], *by Means of Diazine Blue.* H. Scherdel. Färber Zeit. 1901, **12**, [8], 118—119.

SAFRANINE-AZO-β-NAPHTHOL dyed on tannin-mordanted cotton has proved a useful Indigo substitute, and is sold under the names Indoïn, Diazine Blue B, Naphthindone, Indone Blue, &c. In calico printing it is less satisfactory, since it cannot be discharged to a perfect white, but Messrs. Kalle and Co. have recently introduced a dyestuff of this class, Diazine Blue A E, which is well suited for the production of white patterns on a blue ground. The colour is equal to Indigo in fastness to light and washing, and superior to it in fastness to rubbing. It has the defect of dyeing the unmordanted cotton to a slight extent, but this tint can be removed by chlorine, and the following process is recommended:—The goods are padded with a tannin solution containing 20—30 grms. per litre and fixed in the usual manner with an antimony salt. They are then printed with an alkaline discharge consisting of caustic soda and dextrin, and made so thin that it only just refrains from running. After printing, the material is quickly dried, steamed, aged in the open width, washed, soaped, washed with bran, and rinsed in warm water. For dyeing, 2 per cent. of dyestuff is required. The solution of the dyestuff is acidified with acetic acid and divided into two portions. The dyebath is made up with one portion and 1—2 per cent. of alum; the temperature is raised to 38° C.,

when the remainder of the dyestuff is added, and the heating continued to 80° C., the whole dyeing operation occupying 1½ hours, after which the goods are washed, soaped, and dried. Dyeing is followed by one or, if necessary, two passages through a bath made by diluting 100 litres of bleaching powder solution at 7° B. with 500 litres of water. The goods pass through this bath at the rate of 50 pieces in an hour, and are then led through squeezing rollers to a drying cylinder. After the chlorine bath, the blue appears fuller, brighter, and a shade redder than before.—R. B. B.

PATENTS.

Kiers for Bleaching Textile Fabrics. C. Rigamonti and G. Tagliani, Milan, Italy. Eng. Pat. 228, Jan. 3, 1901.

This kier consists of a casing made of boiler plate; from one end a trunk projects, and inside the casing is a U-shaped frame. Means are described by which endless aprons descend the trunk and are then piled up in the U-shaped frame, finally leaving it and returning to the entrance of the trunk. The feeding of the aprons and fabrics is separate and independent. Arrangements are also described whereby the U-shaped frame may be kept automatically rocking if desired; there is also a valve at the top of the trunk for drawing off the impure liquor.—C. M.

Dyeing or Drying Yarns; Machines for ——. W. P. Thompson, Liverpool. From G. Mallinson, Carolina, U.S.A. Eng. Pat. 12,020, July 3, 1900.

This machine is an improvement on that described in U.S. Patent No. 620,470, Feb. 28, 1899, and consists of a frame mounted on a shaft, which is journalled into a vat. On the hub of this frame are mounted slides, each pair rigidly connected together and slotted for the attachment of the yarn.

Devices are described whereby the yarn, during the rotation of the frame in the dye-bath, is moved in every part and stretched or slackened as may be desired.—C. M.

Aniline Black; Process for Dyeing Wool or other Animal Fibre with ——. G. Bethmann, Leipsic, Germany. Eng. Pat. 21,236, Nov. 23, 1900.

Previous processes devised for dyeing Aniline Black on wool have been based on the principle that the chief obstacle to success is the reducing action of the fibre. An equally important obstacle, however, is the alkaline reaction of wool, and to remove this the wool is treated with a dilute acid before padding with the Aniline Black mixture. The material is boiled for about one hour in dilute acid, *e.g.*, HCl or H_2SO_4, under an additional pressure of 20 lb. per sq. in., or it may be subjected to the action of an acid vapour.

The padding mixture should contain somewhat more than the usual quantity of sodium chlorate or similar oxidising substance, so that, after squeezing or wringing, the wool may still retain about 3 per cent. of chlorate. The colour is developed by steaming or ageing, and the material then treated with a bichromate as usual.

An alternative process consists in saturating the wool with dilute acid and then adding an alkali chlorate, or the acid may be wholly or partially added to the padding mixture.—R. B. B.

Dyeing Yarns, and Apparatus therefor. R. F. Schule, Kircheim, Germany. Eng. Pat. 12,976, July 18, 1900.

On the bottom of the dye-vessel is arranged a heating pipe, the heating surface of which must amount to about six or eight times the area of the bottom of the vessel. The dyeing liquor is placed in the vessel, but only up to about one-third of the height of the goods, which may be in the form of hanks, cops, or warps. By means of steam passed through the heating pipe, the liquor is made to lather so that it foams vigorously and forms small bubbles, in which condition it is capable of permeating the goods right through with the dyeing liquor. The goods are exposed for about two hours to the lather in this vessel, and are then dried in the usual manner by centrifugal action.

To ensure even dyeing, the frames or rollers which carry the cops, warps, &c., can be subjected to a continuous to-and-fro or revolving motion.—R. B. B.

Dyeing and Printing [with Sulphide Dyestuffs]. C. D. Abel, London. From The Actien Gesellschaft für Anilin Fabrikation, Berlin. Eng. Pat. 11,042, June 18, 1900.

Instead of using sodium sulphide the dyestuffs are dissolved in caustic soda, with the addition of glucose, lactic acid, milk sugar, or dextrin. The injurious action of sodium sulphide on the hands of the workmen, and on metal dye-vessels or printing apparatus when working with the sulphide dyestuffs, is thus entirely avoided. An addition to the dyebath of normal sodium sulphite is, in most cases, of great advantage.—R. B. B.

Colour-Printing of Textile Fabrics. C. H. Hope, Providence, Rhode Island, U.S.A. Eng. Pat. 10,709, June 12, 1900.

On a piece of cloth having a glossy calendered surface are printed lines of a different colour from that of the cloth, and sufficiently fine and near to each other to simulate the threads of the fabric. The pigment or colouring matter of the printed lines is retained above the glossy surface of the fabric, so that the effect of a changeable colour will be produced when the cloth is viewed at different angles of light and shade. The coloured lines may also be combined with a figured design for the production of a changeable ornamental fabric.—R. B. B.

Printing of Indigo, or Indigo and other Colouring Matters. G. W. Johnson, London. From Kalle and Co., Biebrich-on-Rhine, Germany. Eng. Pat. 9587, May 24, 1900.

In the usual (Schlieper-Baum) process for printing Indigo, if the steaming be too prolonged, by even a few seconds, the colour is rendered quite useless. This objection to the process is removed if the cotton fabric, after being prepared with glucose, be printed with the strongly alkaline Indigo printing colour, dried in the absence of air, and then steamed with dry steam, at a temperature not below 106° C. This temperature varies from 106°—116°, according to the intensity of the blue. In this case no ill-effect results from prolonged steaming, but the time should not be less than 10 minutes. Since the steaming period is not limited, it is possible to combine the Indigo with steam colours, *e.g.*, Alizarin Red, Gallocyanine, Safranine, &c.

Except with very strong printing colours, it is advantageous to replace a portion of the thickening material by Turkey red oil, which prevents the colour from penetrating too far into the fabric and produces more intense and brighter shades.—R. B. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Sulphur Trioxide. R. Schenck. Annalen, **316**, [1], 1—17.

The existence of two forms of sulphur trioxide, the ordinary or asbestos-like form, which vaporises without melting, and the colourless prismatic crystals, which melt at 15°—18° C., has long been known. Weber showed that the latter form was produced when moisture was most carefully excluded, and regarded its conversion into the ordinary form as occurring only in presence of moisture (some of Weber's specimens are still, after many years, unconverted, and on cooling yield only the colourless crystals); hence, he looked on the ordinary form as a hydrate. The author has repeated Weber's experiments, and finds that though the conversion of the colourless into the ordinary form is connected with the presence of moisture (or of monohydrate), yet it occurs in the presence of an amount of moisture so small that one cannot look upon it as forming a hydrate, and must regard its action as catalytic. There remains the possibility of polymerism, as explaining the relationship of the two forms.

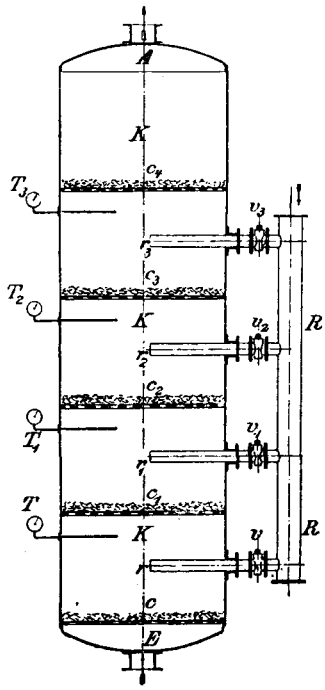
The author has investigated the subject, and as a result of molecular weight determinations and also determinations of the coefficient of expansion of the liquid under different conditions, he concludes that liquid sulphur trioxide is to be looked on as a mixture, in or tending towards equilibrium, of

simple and polymeric trioxide. At a high temperature the simple form exists alone, but if the liquid be cooled, formation of the polymer sets in and goes on till a state of equilibrium between the two forms is reached. If even traces of monohydrate be present, the rate of conversion is comparatively rapid; but if there be none, the rate, even at low temperatures, may be almost immeasurably slow, so that, as in the case of Weber's specimens, years may elapse without any notable amount of the polymer having been formed.

Or it may be looked on as a solution of the polymer in the simple liquid, and at any temperature there will be two possible limits; the limit of equilibrium between the two forms, mentioned above, and the limit of saturation of the simple liquid solvent and the polymeric solute. At a high temperature, probably, the concentration of the polymer in saturated solution is greater than its equilibrium-concentration, and consequently an unlimited amount of the solid polymer can be dissolved, for before saturation is reached it is converted into more of the solvent; while at a low temperature, the saturation-concentration is lower than the equilibrium concentration, and as the amount of the polymer formed, oversteps the saturation concentration it separates in the solid form, so that ultimately the whole is converted into the solid polymer Between these two temperatures must be one at which these concentrations will be identical—at which, therefore, there is equilibrium between the solid polymer and the liquid; above this the solid polymer will ultimately be completely liquefied; and below it the liquid will ultimately be completely converted into the solid polymer. This temperature (if the ordinary asbestos-like form be the polymer) according to the experiments of Schultz-Sellack, seems to be about 25°—27° C. If the specimen be carefully freed from the catalytic agent, so that the rate of conversion on cooling of the simple form into the polymer be immeasurably slow, then saturation of the solution with the polymer can never occur, and on cooling the solvent sufficiently—the transparent, prismatic form—separates out.—J. T. D.

Sulphur Trioxide; Production of ——. Ger. Pat. 119,505, Jan. 3, 1899. Meister, Lucius und Brüning, Höchst-a/M. Zeits. angew. Chem. 1901, **16**, [18], 445.

In the contact-space K, of the apparatus shown, the contact-substance (c, c_1, c_2, c_3, and c_4) lies on the perforated stages. The main supply of warmed sulphur dioxide and air enters at E, but subsidiary supplies, not warmed, enter by the pipes r, r_1, r_2, r_3. These supplies are so regulated by the valves v, v_1, v_2, and v_3, that the mixture reaching any stage (of gas heated by the reaction at the stage below and of cold gas introduced) has approximately the temperature of the original gas at E. This is indicated by the pyrometers or thermometers T, T_1, T_2, T_3. The gas leaves the uppermost stage, and A, hot enough to raise the gas entering at E to the requisite temperature. Dissociation of sulphur trioxide through excessive rise of temperature is avoided by this arrangement.—J. T. D.



Monopersulphuric Acid (*Caro's Acid*). A. Baeyer and V. Villiger. Ber. 1901, **34**, [6], 853—862. (See also this Journal, 1900, 172, 278, 777.)

Caro's acid ($HO.SO_2.O.O.SO_2.OH$) is obtained (1) by treating a persulphate with strong sulphuric acid (Zeits. angew. Chem. 1898, 845); (2) by the electrolysis of tolerably concentrated sulphuric acid (Ger. Pat. 110,249, 1898, of the B.A.S.F.); (3) by the action of strong sulphuric acid on hydrogen peroxide (this Journal, 1900, 278). In the third process much hydrogen peroxide escapes reaction; it has not, therefore, been further studied. In the first two methods, persulphuric acid is the starting point; it is formed at the anode as the primary product of the electrolysis of moderately strong sulphuric acid by the union of two HSO_4 ions. The non-formation of hydrogen peroxide at the anode, although a derivative of it is produced, is explained by the relative stability of hydrogen peroxide and persulphuric acid towards oxidising agents; the former is at once oxidised by the liberated oxygen, whilst the latter is remarkably stable towards oxidising agents.

The primary product of the electrolysis, persulphuric acid, in contact with sulphuric acid, is hydrolysed, producing Caro's acid and sulphuric acid, $(OH)(SO_2).O.O.(SO_2)(OH) + H_2O = (OH).SO_2.O.OH + (OH)_2SO_2$, the Caro's acid finally, by further hydrolysis, giving sulphuric acid and hydrogen peroxide.

In the presence of persulphuric acid and monopersulphuric acid (Caro's acid), hydrogen peroxide may be estimated by means of permanganate in the ordinary manner, at a sufficient degree of dilution. Monopersulphuric acid liberates iodine from an acidified solution of potassium iodide much more rapidly than persulphuric acid; thus, in titrating with thiosulphate, a colourless solution is obtained, which in a short time again becomes blue; this further reaction is due to the persulphuric acid, and is complete in 12—24 hours; in the estimations the result of a blank experiment was deducted. This method gives sufficiently accurate determinations of the monopersulphuric and persulphuric acids.

Monopersulphuric Acid.—The acid could not be isolated in the free state or as a salt. A solution was obtained by triturating 10 grms. of potassium persulphate with 20 grms. of strong sulphuric acid, allowing to stand for one hour, pouring on ice, precipitating sulphuric acid with a solution of monobarium phosphate (baryta at once decomposes Caro's acid), filtering, and subjecting to a current of air *in vacuo* until the smell of ozone and bleaching powder had disappeared. The solution (1·5 litre) was very stable; it deposited only traces of barium sulphate on standing for months; hydrogen peroxide was absent. About 16 per cent. of unaltered persulphuric acid was still present. The product of the action of strong sulphuric acid on potassium persulphate has the same composition as Traube's electrolysed sulphuric acid.

Persulphuric Acid was obtained by exact precipitation of a solution of barium persulphate with dilute sulphuric acid. On standing, the acid gradually changes to sulphuric acid and Caro's acid. The decomposition is almost complete in eight days; hydrogen peroxide is not formed.

In the presence of sulphuric acid of 8 per cent. strength, Caro's acid is as stable as in the presence of phosphoric acid; after seven days, hydrogen peroxide could be detected by means of titanic sulphuric acid. Persulphuric acid is converted into Caro's acid almost completely in a few days by 40 per cent. sulphuric acid; hydrogen peroxide is formed. The electrolysis of sulphuric acid of different concentrations was studied, using platinum electrodes and a current density

of 2—2·5 ampères. Persulphuric acid is the first product, which is converted into monopersulphuric acid during the electrolysis of acid of 55 per cent. strength; with acid of 40 per cent. strength the conversion into monopersulphuric acid only takes place to a small extent during the electrolysis. With 20 per cent. acid the formation of the per-acids takes place only to a limited extent.

The substance with an odour resembling bleaching powder, produced by Caro's acid on standing, may be S_2O_8, which may also be the substance obtained by the action of the silent electric discharge on a mixture of sulphur dioxide and oxygen.—A. C. W.

Potassium Ferricyanide; Action of Hydrofluosilicic Acid upon ——. J. Matschek. Chem. Zeit. 1901, **25**, [31], 327—328.

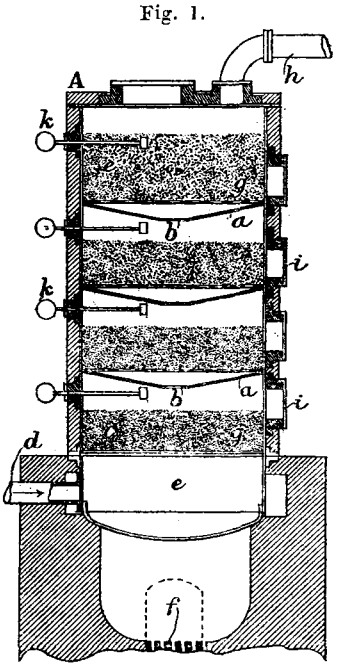
HYDROFLUOSILICIC acid acts upon potassium ferrocyanide (this Journal, 1901, 363). The action upon potassium ferricyanide is to produce a blue precipitate, which was shown to be formed in the absence of air. The smallest quantity of ferricyanide which can yield the reaction is 0·002514 grm.; this amount, when dissolved in 2 c.c., gives a greenish-blue coloration after shaking with 50 c.c. of hydrofluosilicic acid (1 c.c. = 0·584 grm. of H_2SiF_6), whilst 0·0013 grm. of potassium ferrocyanide in 50 c.c. gives the same depth of colour with 0·6 c.c. of hydrofluosilicic acid. The precipitate was shown to contain iron and cyanogen in the proportions required by Prussian blue, $[Fe(CN)_6]_3Fe_4$; it contains soluble Prussian blue. The quantities of the reagents entering into the reaction agree with the equation $7[Fe_2(CN)_6.6KCN] + 21H_2SiF_6 + 3H_2O = 21K_2SiF_6 + 2Fe_4[Fe(CN)_6]_3 + 48HCN + 3O$.—A. C. W.

Sulphuric Acid; Determination of Water or of Sulphur Trioxide in Concentrated Fuming ——. H. Rabe.

See under XXIII., *page* 619.

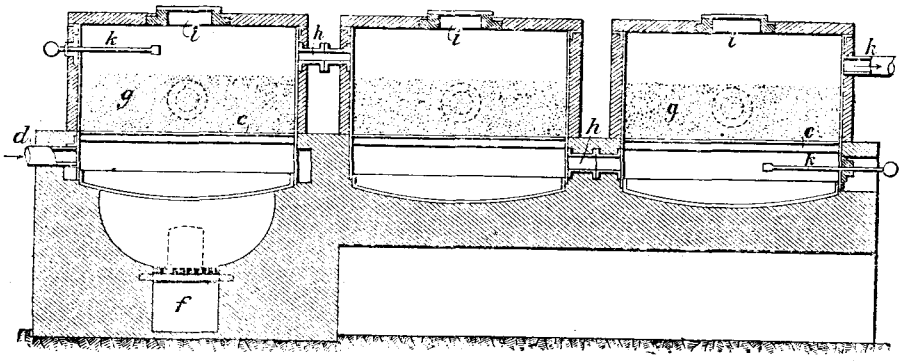
aperture *b* in the curved partition plate *a*. In like manner the gases pass through each of the superposed chambers, having exit at *h*, whence they are led to the absorbing apparatus for the SO_3 formed. The pyrometers *k* indicate the temperature. It is stated that, "in using the above-described partitions, the gases have to find their way from time to time through comparatively narrow openings, and in doing so they thoroughly mix together, even in contact vessels of very large diameter."

Fig. 1.



For working on a large scale, the apparatus shown in Fig. 2 is preferred, in which the chambers are arranged horizontally, the gases entering at the nipple *d*, and after traversing the contact mass, pass through the

Fig. 2.



Nitric Acid in Alkali Nitrates; Detection and Determination of ——. E. P. Perman.

See under XXIII., *page* 619.

Chlorine; Production of ——, *from Ferrous Chloride by Electrolysis.* Roubertie and Pepin.

See under XI. A., *page* 588.

PATENTS.

Sulphuric Acid; Apparatus for Making ——, *by Catalysis.* Actien Gesellschaft für Zink-Industrie vormals W. Grillo and Max Schroeder, both of Neumühl-Hamborn, Germany. Eng. Pat. 17,034, Sept. 25, 1900.

IN the contact apparatus shown in Fig. 1, the sulphurous gases, after preheating in any suitable apparatus to 260°—280° C., pass in a steady stream through the nipple *d* into the vessel *e*, set over a fireplace, for maintaining the temperature, if necessary; the gases then ascend through the perforated plate *c* into the contact mass *g*, above which they become mixed, as the only exit is through the central exit *h* into the next chamber, where they descend through the contact mass, and find exit in the bottom compartment into the base of the following chamber, and so on, The contact mass may be that described in Eng. Pat. 25,158, 1898 (this Journal, 1899, 584), in which a platinum salt is dried with a water-soluble salt, such as magnesium sulphate, or a soluble phosphate.—E. S.

Gas Liquors; Treatment of ——. H. Koppers, Carnap, near Essen-Ruhr, Germany. Eng. Pat. 6272, March 25 1901.

THE claims are: "(1) A method for reducing the amount of noxious waste liquors in the treatment of ammoniacal water, characterised by drawing off a quantity of the ammoniacal liquor, after the separation of the free ammonia and before the separation of the combined ammonia, from the purifying apparatus, and using it instead of fresh water for the ammonia washers"; and "(2) In the treatment of ammoniacal liquors described, supplying the lime necessary for the separation of the combined ammonia to the purifying apparatus," not as milk of lime, "but as marshy lime in a pulpy condition."—E. S.

Cryolite; Process of Treating ——. C. A. Doremus, New York, U.S.A. Eng. Pat. 18,969, Oct. 23, 1900.

CRYOLITE is heated in an iron retort, through which steam is passed, to obtain hydrofluoric acid and a residue of sodium aluminate, or sodium fluoride and alumina, or a mixture of these. Or steam is caused to impinge on cryolite fused on the hearth of a reverberatory furnace; as HF escapes, the mass stiffens, "through the production of aluminate, or of sodium fluoride and alumina, or a mixture of these," which may be further treated with heat and steam, or with products of combustion containing steam. Another process, intended to obtain an aluminate with much alumina, consists in heating cryolite, mixed with alumina or bauxite, in "a mechanical furnace by a fuel consisting of or containing hydrogen." This process may be modified by making up briquettes of cryolite and bauxite, and heating them, with or without steam passing, with the fuel described. A fifth process consists in melting cryolite in a muffle furnace, suitably lined, and passing steam as long as the mass remains molten. In all these processes the temperature is kept below a "white heat."

—E. S.

Nickel Salt, and Process of Making Same. H. A. Frasch, Hamilton, Canada. Eng. Pat. 284, Jan. 4, 1901.

SOLUTION of a nickel salt, such as the sulphate or chloride, is treated with ammonia in excess, and sodium chloride is added as long as a nickel-ammonium salt separates. In the case of a solution containing, besides nickel chloride, other chlorides, as of iron, copper, and cobalt, the iron is removed by oxidation and precipitation by calcium carbonate, and the solution is then saturated with ammonia; on addition of sodium chloride (or potassium chloride), nickel-ammonium chloride separates, and may be filtered off, washed with a saturated solution of sodium chloride containing ammonia, and dried. The salt is stated to have the formula "$Ni(NH_4)_2Cl_2 + 4NH_3$, or $NiCl_2 + 6NH_3$," and is described as "forming a crystalline mass of violet to purple colour, very hygroscopic, freely soluble in water with a blue colour, emitting a strong odour of ammonia, and, upon boiling, giving off two-thirds of its ammonia contents." "It differs from the normal nickel-ammonium chloride or sulphate in the excess of ammonia it contains, there being about six equivalents of ammonia to every one of nickel."

—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Glasses; Phosphatic ——. F. Cedivoda. Chem. Zeit. **25**, [33], 347—350.

THE author summarises the results of his researches as follows:—When glasses composed of the metaphosphates of calcium and sodium are fused together, true double compounds are formed, as in the case of silicates, of greater resistance than is possessed by the component glasses,—the resistance increasing with the acid ratio, though it is in all cases inferior to that of the corresponding silicate glasses. In the case of silicate glasses, the principal constituents removed by the action of water are alkalis, whereas the phosphate glasses also yield up a considerable amount of phosphoric acid to this solvent. Again, other conditions being equal, the phosphate glasses are more powerfully affected by acids than are the silicate glasses; they are also more readily fusible than the latter, their fusibility increasing with the acid ratio. The fact that phosphate glasses can be fused in a porcelain crucible without appreciably attacking the latter, is regarded as a proof that these glasses are soluble with difficulty in silicate glasses.—C. S.

The Ceramic Stoneware of the Sèvres Porcelain Works. G. Vogt. La Céramique, **3**, [133], 127—131.

THE manufacture of ceramic stoneware at the Sèvres works was introduced for the purpose of supplying this material for structural purposes at the Paris Exhibition. The body was composed of the following ingredients, the figures representing the proportions before and after the elimination of moisture and combined water in the kiln:—

Clay from St. Amand-en-Puisaye (Nièvre)	56	50
Clay from Randonnai (Orne)	27	25
Sand from Decize (Nièvre)	26	25
Totals	109	100

the chemical composition, after firing, being: silica, 76; alumina, 21; alkalis, 3 per cent.

The firing temperature employed was that of the fusing heat of Seger cone No. 9, *i.e.*, 1,270° C. The colour of the ware is yellow when fired in an oxidising atmosphere, but pale bluish-grey in a reducing atmosphere. The body is of very fine grain, is easily moulded or thrown on the wheel, and can probably be shaped by means of the usual mechanical appliances. Care is necessary to dry the pieces slowly, to prevent cracking or deformation; and it is advisable, in the case of large pieces, to add to the body 20—25 per cent. of powdered stoneware shards. The degree of shrinkage is such that pieces intended to measure 1 metre when baked must be 1·125 m. in the fresh state. This body can be worked up in conjunction with the "new porcelain" body prepared at Sèvres.

Glazing.—The new stoneware can be glazed with salt, added in the kiln, in the proportion of 200—800 grms. per 1 cb. m. of kiln capacity, or by the glazes described below.

Glazes: Colourless Glaze.—Pyrenees felspar, 42·1 parts; Nemours quartzose sand, 27·2; dry argillaceous kaolin, 13·0; Bougival chalk, 17·7 parts. The felspar contains: silica, 65·45; alumina and ferric oxide, 19·52; and potash, 11 per cent., whilst the sand is nearly pure silica, and the kaolin is an almost pure hydrated aluminium silicate.

Coloured Glazes.—These are prepared by simple mixing, without previous fusion, and are laid on the unbaked ware with the brush after stirring up in water thickened with gum tragacanth. The following recipes are given:—

Uranium Yellow.—Uranium oxide, 5·0 parts; chalk, 17·7; Pyrenean felspar, 42·1; Nemours sand, 29·3; dry kaolin, 8·5 parts.

Brown-Yellow.—Pure red ferric oxide, 5·0 parts; chalk, 13·0; felspar, 45·0; quartzose sand, 28·5; dry kaolin, 11·0 parts.

Chrome-Green.—Green chromium oxide, 1·0 part; chalk, 17·7; felspar, 42·1; sand, 28·0; kaolin, 11·3 parts.

Copper-Green.—Cupric oxide, 4·0 parts; chalk, 12·7; felspar, 42·1; sand, 27·2; dry kaolin, 13·0 parts.

Cobalt Blue.—Cobalt oxide, 3·0 parts; chalk, 14·1; felspar, 42·1; sand, 27·2; dry kaolin, 13·0 parts.

Manganese Violet-Brown.—Brown manganese oxide, 5·0 parts; chalk, 13·0; felspar, 45·0; sand, 28·5; dry kaolin, 11·0 parts.

Nickel Red-Brown.—Nickel carbonate, 3·0 parts; chalk, 15·3; felspar, 42·1; sand, 27·2; dry kaolin, 13·0 parts.

Flesh Tint.—Equal parts of the foregoing manganese and colourless glazes.

Violet-Grey.—Nickel glaze, 10 parts; cobalt glaze, 3; colourless glaze, 87 parts.

Bluish-Green.—Chrome glaze, 30 parts; cobalt glaze, 3; colourless glaze, 67 parts.

Golden-Brown.—Equal parts of manganese and iron glazes.

Reds.—Chrome-pink being found unsuitable for the desired shades of red for frieze of the Palais des Beaux-Arts, the following underglaze masses were prepared:—

——	Pale Red.	Red.
Randonnai clay	57	57
Decize sand	41	36
Ferruginous sand from Thiviers	5	10
Pegmatite	5	5

The shade was modified, when required, to orange by covering with yellow glaze, or to violet by the aid of blue glaze. An oxidising atmosphere is necessary in firing all these coloured glazes, to maintain their purity of tone.

Semi-opaque Glazes: Colourless.—Pegmatite, 30 parts; pure kaolin, 40; Nemours sand, 28·5; chalk, 20 parts.

Ivory Yellow.—Pegmatite, 35·7 parts; kaolin, 13·7; Nemours sand, 43·6; chalk, 15·9; natural rutile, 9·6 parts.

Yellow.—Pegmatite, 53 parts; kaolin, 14; sand, 14·1; chalk, 25·5; rutile, 9·6; colcothar, 2·4 parts.

Reddish-Yellow.—Same as above, but with 4·8 parts of colcothar.

Violet (Yellow Tinge).—Pegmatite, 33·6 parts; kaolin, 12·9; sand, 47; chalk, 15; rutile, 6·6; colcothar, 6 parts.

Crystalline Yellow-Brown.—Same as above, except that the proportions of rutile and colcothar are each increased to 9·6 parts.

Crystalline Golden-Brown.—Pegmatite, 53 parts; kaolin, 14; sand, 14·1; chalk, 25·5; rutile, 22; colcothar, 17·5 parts.

Crystalline Dark Green.—Pegmatite, 30·85; kaolin, 25·35; sand, 36; chalk, 28; rutile, 18; cobalt oxide, 12 parts.

Greenish Blue-Grey.—Pegmatite, 53 parts; kaolin, 14·1; sand, 14; chalk, 25·5; rutile, 12; cobalt oxide, 1·2 parts.

Red.—A frit is made of: Pegmatite, 108 parts; sand, 126; zinc oxide, 15·5; barium carbonate, 36; fused borax, 45; dry sodium carbonate, 16·5 parts; and this is incorporated with 2 per cent. of copper oxalate and 1 per cent. of calcined tin oxide.

This glaze must be fired in a reducing atmosphere, since in an oxidising fire it turns green; the reduction, however, should not be prolonged beyond the point of incipient fusion, or the ware will become black.

Crystalline Glazes.—Like the above red, these glazes must first be fritted, the following fluxes being prepared in order to permit the proportions of potash and zinc oxide to be modified so as to furnish the best results in different cases:—

——	Flux 1.	Flux 2.
Dry potassium carbonate	138	69
Zinc oxide	162	202·5
Quartzose sand	360	350

The proportions found to give the best results at Sèvres were: No. 1, 85 parts; No. 2, 15 parts. The glaze must be applied to biscuit ware, and laid on thick enough for an excess to run down the ware in the firing. An oxidising atmosphere and a temperature of 1,270° C. are necessary; and slow cooling facilitates the production of good crystals.

Yellow.—The above proportions of fluxes are mixed with a frit (No. 3) of: dry potassium carbonate, 138 parts; zinc oxide, 162; sand, 300; rutile, 82 parts.

Another variety of *colourless glaze*—so far as the crystals are concerned—is obtained by replacing the rutile in frit No. 3 by pure titanic acid.—C. S.

PATENTS.

Glass Annealing Furnaces. F. T. Brearley, St Helens, Lancs. Eng. Pat. 7854, April 27, 1900.

Two annealing chambers and one annealing kiln or "leer" are arranged together in the shape of a Y, and, if desired, the stowing openings can be arranged in such a position that the same turntable feeds both of the annealing chambers, the casting tables in such event being situated radially with regard to the turntable.—C. S.

Enamelling the Surfaces of Refractory Materials; Apparatus for ——. S. S. Bromhead, London. From C. H. Waterman, New Jersey, U.S.A. Eng. Pat. 5204, March 12, 1901.

The apparatus consists of an electrical heater of large superficial area, and of means for presenting the material to be enamelled in a plane parallel and in close proximity to the heater, so that the entire surface of the material will be heated simultaneously, immediately, and uniformly, a non-oxidisable shield being interposed between the heater and the material in order to protect the enamel from contamination by contact with emanations from the heater. —C. S.

Muffle Kilns or Ovens [Ceramic or Enamelled Ware]; Continuous Gas ——. H. H. Lake, London. From M. Souvero and Co., Turin. Eng. Pat. 6921, April 12, 1900.

The kiln is of annular shape and the goods to be baked are conveyed by means of a support extending right through the kiln. Gas is generated by the distillation of coal in an adjoining hearth, and is ignited in presence of air, heated by the waste heat of the same hearth, the resulting flame being directed through arches and lateral passages about the central chamber, thus baking the goods without being brought into contact therewith. The kiln is combined with a cooling chamber, an open charging and discharging aperture being situated between the two.—C. S.

Porcelain and the like; Fusing ——. G. Ott, Paris. Eng. Pat. 14,286, Aug. 9, 1900.

The burner claimed, consists of an internal pipe, through which gas is discharged under pressure, and of an external pipe supplying oxygen, the resulting flame generating a sufficiently high temperature to fuse pure porcelain in a platinum mould.—C. S.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Portland Cement from Blast Furnace Slag. C. Steffens. Stahl u. Eisen, **20**, 1170 (Zeits. angew. Chem. **14**, 472).

Experience shows this process to be simple, cheap, and regular. The yields of rotary furnaces are specially satisfactory. The raw material is wet granulated slag (not powder) and hard limestone; these are dried, intimately mixed, and finely ground. The slag, mixed with the requisite amount of small limestone, is brought into a calcining furnace heated by the waste gases of the rotary furnace. The slag is dried and is pulverised by the limestone during the rotation of the furnace, whilst the limestone is partly calcined and rendered brittle so as to be easily powdered. From the calcining furnace it passes into a mill, whence the finely ground substance goes direct into the rotary furnace. This acts quietly and regularly, and needs very little looking after. It will yield on an average 150 casks per day. The whole of the fuel needed, amounts to 18 or 20 per cent. of the slag burnt.—J. T. D.

Portland Cement and Sulpho-Aluminate of Calcium; Action of Sea Water on ——. O. Rebuffat. Gaz. Chim. Ital. **31**, [1], 55; Chem. Centr. 1901, **1**, [17], 927.

The author has made an investigation as to the action of magnesium salts, sodium chloride, and sea water on sulpho-aluminates, aluminates, and silicates of calcium. He concludes from his results, that in the case of cements immersed in sea water, the formation of sulpho-aluminates is, at the most, transient, and can only take place to a small extent, and therefore, contrary to the view of Le Chatelier, sulpho-aluminates can scarcely have any influence on the stability of cements. It is pointed out, however, that in considering the question of the stability of cements, it has been quite overlooked, that aluminates as well as sulpho-aluminates are strongly attacked by sodium chloride. 200 c.c. of a 30 per cent. solution of sodium chloride, when left in contact with 1 grm. of calcium sulpho-aluminate, contained even after 12 hours, small quantities of alumina and large quantities of SO_3 and CaO. Calcium aluminates are attacked in a similar manner by 30 per cent. sodium chloride solution, lime and aluminium passing into solution. —A. S.

Wood with Tar-Oils; Economical Saturation of ——. F. Seidenschnur. Zeits. angew. Chem. **14**, 437—441.

An ordinary sleeper, 2·7 m. by 0·26 m. by 0·16 m., needs 35 to 40 kilos. of creosote to saturate it thoroughly. To lessen the cost of this, attempts have been made to dilute the creosote, and to introduce it into the wood in solution in a volatile solvent, the solvent being afterwards removed by heat; but in all these attempts the loss of solvent has been so great that no saving of cost has been effected. The

author has succeeded, however, by emulsifying the oil, and introducing the dilute emulsion; the emulsion is made with a resin-soap, as follows:—*Creosote rid of phenolic constituents;* 22·5 kilos. of resin and 3 kilos. of caustic soda (98 per cent.) are dissolved in water, heated by steam, and diluted to 150 litres. To this 450 kilos. of creosote (washed free from phenols with NaOH) are added, the whole thoroughly agitated, and water added till the required percentage of creosote is reached (say 15 per cent.). The emulsion holds for a very long time. Even after a week the oil will have only settled out as a creamy layer, which is readily mixed again by mere shaking. The drops are very minute and penetrate wood with ease; by a water pump vacuum of 40 mm., there was driven through a piece of beech, 12 cm. in diameter and 15 cm. long, in half an hour, 350 c.c. of the emulsion, containing at entry 15, at exit 11 per cent. of creosote. *Creosote containing Phenols;* to emulsify this, much larger quantities of resin-soap are needed, and the resulting emulsion is not so permanent, so that on all grounds the substance free from phenols is to be preferred. The author's experiments seem to show that the permanence of the emulsion depends on the smallness of the globules of oil; but it is also improved if they contain minute quantities of substances which, like free resin, are absolutely insoluble in water, and increase the surface-repulsion between oil and water, while it is lessened if they contain constituents soluble in water.

To ascertain how far the creosote penetrates the wood when used in this way, pine sleepers were impregnated with a 15 per cent. emulsion, prepared as above described. They were steamed for half an hour at a pressure of 1½ atmospheres, exhausted for half an hour at a pressure of 690 mm., and submitted for half an hour to a pressure of 7 atmospheres. A sleeper, weighing originally 68·6 kilos., took up 36·5 kilos. of emulsion, or 5·5 kilos. of creosote. A diagram is given showing the distribution of the oil, the figures being percentages of creosote reckoned on the dry wood. A second diagram gives the result of similarly impregnating beech sleepers with 30 per cent. emulsion, when sleepers of 80 kilos. weight absorbed 31 kilos. of emulsion, or 9·3 kilos. of oil.—J. T. D.

PATENTS.

Tiles, Roofings, Linings, and the like; An Improved Composition or Material more especially intended for Use in the Manufacture of ——. P. Sohège, Paris. Eng. Pat. 10,865, June 14, 1900.

The composition claimed consists of ground tan, hemp waste, fine sand, or powdered pumice, and sizing materials, such as gelatin, resin soap, &c.—J. W. H.

Marble, Imitation; Manufacture of ——. J. Tuckwell, Glasgow. Eng. Pat. 15,830, Sept. 6, 1900.

Keene's marble cement, white silver sand, and ground alum, are mixed in certain proportions with water and allowed to set; the block obtained is stove-enamelled, polished with rottenstone and water, varnished, grained, and again stoved, and finally polished with a composition of beeswax, turpentine, and precipitated chalk.—J. W. H.

Insulating and Packing Material; New or Improved ——, *and a Method of Manufacturing the same.* M. Raphael and L. Elias, Breslau. Eng. Pat. 16,010, Sept. 8, 1900.

Small plates of mica are made to adhere by means of moistened asbestos; subsequent pressure and drying complete the preparation of the material.—J. W. H.

Iron Constructions; Process of and Compositions for Protecting ——, *from Incandescence in case of Fire.* I. Koslowsky, Mulhouse, Alsace, Germany. Eng. Pat. 6241, March 25, 1901.

The elements of iron constructions are brushed over with a solution of 2½ parts of borax, 3 parts of alum, 2½ parts each of sodium tungstate and ammonium sulphate, 3 parts of magnesium sulphate, and 2 parts of ferrous sulphate, in 100 parts of water. Then a thick paste is repeatedly applied, obtained by mixing 15 parts of the described solution with 50 parts of sodium silicate, 30 parts of asbestos, 10 to 20 parts of wood ash, and 15 to 25 parts of chalk. Adhesion can be secured by embedding a metallic tissue in the protective coating.—E. S.

Lime, Slaked; Production of ——. H. H. Lake, London. From D. Wachtel and Co., Berlin. Eng. Pat. 1479, Jan. 22, 1901.

Slaked lime paste is mixed with dry calcium hydrate and submitted to the action of high pressure steam; perfect slaking is claimed.—J. W. H.

Cement, Lime, and the like; Kilns for ——. H. Schmidt, Bonn, Germany. Eng. Pat. 12,803, July 16, 1900.

The slurry is fed into a rotating cylinder, in which it is dried by the heated gaseous products from a chamber, into which it subsequently enters, and in which the burning is completed.—J. W. H.

Cement and similar Materials; Manufacture of ——, *and Apparatus therefor.* G. Warren, Ilford, Essex. Eng. Pat. 6409, May 7, 1900.

The liquid sludge, from which the cement is made, is run into moulds to form cakes, small coal being added if desirable; these cakes are burnt in a special kiln.
—J. W. H.

Cement Substance for Ship Building Purposes. W. P. Thompson, Liverpool. From A. Johannsen, Flensburg, Germany. Eng. Pat. 8987, May 15, 1900.

The composition claimed consists of a mixture of Portland cement and powdered coke, with the addition of slaked lime and sawdust to lighten the mass.—J. W. H.

Cement out of Cement Slush; Manufacture of Solid Blocks of ——. S. S. Bromhead, London. From G. Möller and P. Pfeiffer, Berlin. Eng. Pat. 3282, Feb. 15, 1901.

Small blocks of dried cement are made by any suitable process, and these by repeated dipping in the slush and drying increase in size by layers until large enough for use.—J. W. H.

Cement; Apparatus for Calcining ——. B. B. Lathbury and H. S. Spackman, Philadelphia, U.S.A. Eng. Pat. 5346, March 13, 1901.

The calcination of the cement is carried out with powdered fuel, which is blown into the kiln by a current of air previously heated by employing it to cool hot clinker.
—J. W. H.

Stone, Artificial; Manufacture of ——, *and Apparatus therefor.* W. Dünkelberg. Eng. Pat. 20,639, 1900.

See under I., *page* 562.

X.—METALLURGY.

Calcium Carbide; Reducing Power of ——. F. v. Kügelgen. Zeits. für Elektrochem. 1901, **7**, [41], 541—550; [42], 557—568; and [43], 573—580.

Neumann (this Journal, 1901, 46) has stated that the reduction of metallic oxides by calcium carbide takes place according to the general equation—

$$3M'_2O + CaC_2 = CaO + 3M'_2 + 2CO,$$

where M' is a monovalent metal, the general equation for mixed oxides and chlorides being—

$$2M'_2O + 2M'Cl + CaC_2 = 3M'_2 + CaCl_2 + 2CO.$$

According to the author (this Journal, 1901, 126) the gas evolved during the reaction is carbon dioxide and not carbon monoxide. The general equations representing the reduction of metals by calcium carbide should thus be—

$$5M'_2O + CaC_2 = CaO + 5M'_2 + 2CO_2 \text{ and}$$
$$2M'Cl + 4M'_2O + CaC_2 = 5M'_2 + CaCl_2 + 2CO_2.$$

Further detailed experiments on the reduction of lead and copper confirm these equations and have also led the author to the conclusion that they take place in two stages. The general equation representing the reduction of PbO takes place in two stages, as follows :—

$$\text{(i) } PbO + CaC_2 = Pb + CaO + 2C.$$
$$\text{(ii) } 4PbO + 2C = 4Pb + 2CO_2,$$

whilst the reduction of mixed PbO and $PbCl_2$ is represented thus—

$$\text{(i) } PbCl_2 + CaC_2 = Pb + CaCl_2 + 2C$$
$$\text{(ii) } 4PbO + 2C = 4Pb + 2CO_2.$$

Details of experiments on the reduction of other metals are also given.

The author has arrived at the following conclusions :—Calcium carbide is a very powerful reducing agent, and is even capable of decomposing compounds of the alkali metals. Chlorides are much more easily reduced than oxides. With most chlorides, when once the reaction is started at a point, it usually proceeds throughout the entire mass, and very often with explosive violence. For the reduction of oxides, on the other hand, external heat must generally be applied. Bismuth oxide is an exception to this rule, as is also copper oxide when a large excess of carbide is employed. The easy inflammability of the mixture is characteristic of the reduction of chlorides, the application of an ignited match being often sufficient. Even when no very great amount of heat is liberated, the reaction can be easily started. Specially satisfactory results are obtained by the simultaneous reduction of mixed chloride and oxide.

Moissan has shown that molten carbide acts on the oxides of carbide-forming metals yielding metallic carbides. When, however, the reduction takes place at a lower temperature, as above, the metal obtained is free or almost free from carbide. At intermediate temperatures the amount of carbon in the reduced metal would increase until the temperature is sufficiently high, as in Moissan's experiments, for the formation of carbide, when carbide alone would be produced. As a rule, however, reduction by means of calcium carbide yields purer metals than if the reduction was effected by carbon alone, since the reaction takes place at a lower temperature.

When the carbide is employed in the proper proportion, only traces of calcium are present in the reduced metal. By using a large excess of carbide however, calcium alloys can be obtained. A copper alloy contained over 1 per cent. of calcium.

Calcium carbide can be utilised in the laboratory for various reducing purposes, but its application on a technical scale would be conditional upon the yield of metal. In the case of oxides this yield depends on whether the oxide is easily reduced by carbon or not. Both the calcium and the carbon of the carbide act as reducing agents, the former being the more powerful, as is indicated by the differentiation of the general equation into two stages. The greater the difficulty of the reduction of the oxide by carbon, the less will the carbon assist in the reduction, that is to say, more carbide will be required for the reduction, which will then take place chiefly at the expense of the calcium. This being the case, free carbon is liberated and the larger the quantity of carbon thus set free the greater is the difficulty of fluxing or of obtaining a regulus.

According to Neumann's equation, 342 kilos. of carbide are required per ton of copper reduced, whilst, according to the author, only 202 kilos. are necessary. This more nearly agrees with Frölich's estimation of one-tenth to one-quarter of a ton of carbide per ton of copper. In any case, the reduction of copper by carbide is scarcely likely to become a technical process. Carbide may, however, become useful in the preparation of other metals in the pure condition, as *e.g.*, for the reduction of nickel oxide or bismuth oxychloride.

Improvements might be effected in the case of oxides, easily reduced by carbon, by adding a certain amount of free carbon to the mixture. Again, if the oxides are difficult to reduce by carbon, *i.e.*, if the reduction takes place chiefly at the expense of the calcium, a saving might be effected by the addition of aluminium to increase the reducing power of the carbide, and to decrease the quantity of carbon.

The application of carbide for the production of alloys appears to offer better prospects. By proper choice of chloride and oxide, it is possible to simultaneously reduce metals, the separate reduction of which offers great difficulties. Alloys which are not easily prepared by fusing the constituents, owing to differences in the melting points or to oxidation, can thus be obtained.

Carbide may also become useful in refining metals. The process of reduction by means of carbide is not without hope if it be applied in its proper place. This consists not in attempting to replace existing processes of reduction, but rather in applying it in cases where existing methods are either useless or only give unsatisfactory results.—J. S.

Steel Production in Basic Siemens-Martin Furnaces. K. Poech. (From T. Turner.) Chem. Zeit. Rep., 1901, **25**, [36], 143.

Turner points out that, even in the West of Scotland, where the best "acid" steel is produced, the basic process is of importance in consequence of the increasing difficulty in procuring pure ores. To produce a steel of crucible steel quality, white or half hæmatite pig-iron is treated in the basic Siemens-Martin furnace, and then recarburised by spiegel or by means of the Darby process ; it is then finally run through the acid furnace or converter. In English practice, the basic Siemens-Martin furnace carries a charge of 30—40 tons ; it has a belt of (neutral) chrome ore between the acid walls and the basic hearth, which is of dolomite mixed with tar. About 70—80 per cent. of pig-iron and 30—20 per cent. of scrap iron are employed. To produce 1 ton of ingot, there would be used 0·675 ton of basic and 0·075 ton of gray pig-iron, and 0·338 ton scrap (making 1·088 ton in all) with 0·175 ton of ore, 0·2 ton of limestone, 0·025 ton of lime, 0·0022 ton of spiegeleisen, and 0·0022 ton of ferromanganese. The phosphorus in the finished product averages less than 0·05—0·06 per cent.—W. G. M.

Steel ; State of Combination of Iron with the Rare Elements in ——. Carnot and Goutal. Oesterr. Zeits. Berg-u. Hüttenw. **49**, 162. Chem. Zeit. Rep. **25**, [14], 127.

In blast-furnace ferrochrome, chromium occurs as the compound, $3Cr_3C_2.Fe_3C$. Tungsten occurs in tungsten steel as Fe_3W, or as $Fe_3C.WC$., according as the steel is poor or rich in carbon. Similarly, molybdenum occurs as Fe_2Mo_3, or as $Fe_3C.Mo_2C$. The electric conductivity indicates that these compounds are not in solution in the steel. Copper and nickel, on the other hand, seem to exist in the state of solution in or mixture with the steel, not as compounds with iron.—J. T. D.

Steel or Iron ; Influence of Tin on the Quality of ——. Stahl u. Eisen, **21**, 330. Chem. Zeit. Rep. **25**, [14] 128.

Samples of iron containing from 0·1 to 0·63 per cent. of tin were prepared and tested. All the samples forged well, though somewhat hard ; on rolling, those containing more tin split more readily at the edges ; they welded well, with the exception of that with 0·63 per cent. of tin ; they bent satisfactorily, both hot and cold. The tin was irregularly distributed in the ingots.

Crucible steel was also prepared containing 0·23, 0·50, 0·68, and 1·62 per cent. of tin. All samples forged well, the last being somewhat red-short ; none of them would weld. The breaking stress was 72·3—73·9 kilos. per sq. mm.—J. T. D.

Tin ; Influence of ——, *on the Quality of Iron and Steel.* A. Zugger. Chem. Zeit. Rep. 1901, **25**, [16], 143.

Observations upon the product of a basic Siemens-Martin furnace, in which tin was accidentally present, showed that a metal containing 0·55 per cent. Sn, 0·015 per cent. Sb, 0·03 per cent. As, and 0·182 per cent. Cu, rolled quite satisfactorily to plates ; on further rolling to sheet, however, cracks began to develop at the edges of the sheet, but they could not, as it was proved, be attributed to red-shortness. Bars

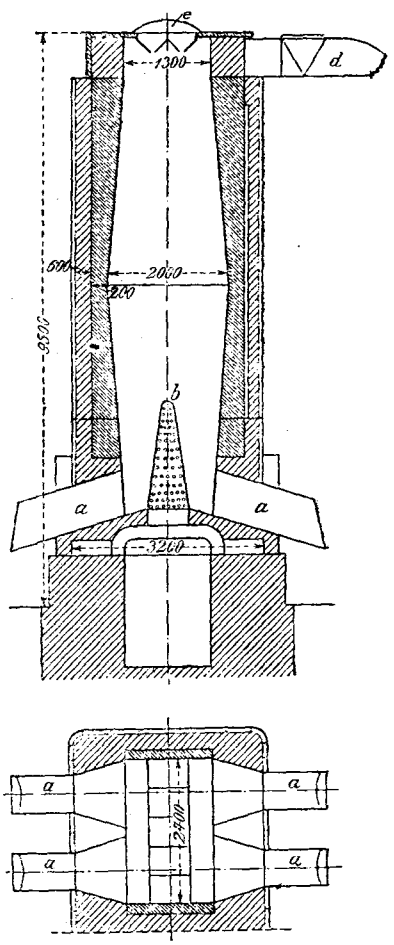
rolled from the ingot-iron folded completely over under the cold bending test, and showed a tensile strength of 40 kilos. with an extension of 31—34 per cent. Hence it appears to be proved that the presence of 0·55 per cent. of tin does not affect malleability, tenacity, or extensibility; the welding qualities at most are impaired.—W. G. M.

Mercury and its Production. A. Weiskopf. Zeits. angew. Chem. **14**, [18], 429—437, and [19], 465—469. (See also Trade Report, page 640.)

THE extraction of mercury is carried out in three stages: (1) Roasting the ore. (2) Condensation of the vapours. (3) Working up of the condensed products. These are prefaced by a sorting of the ore, according to its richness in mercury and the size of the pieces.

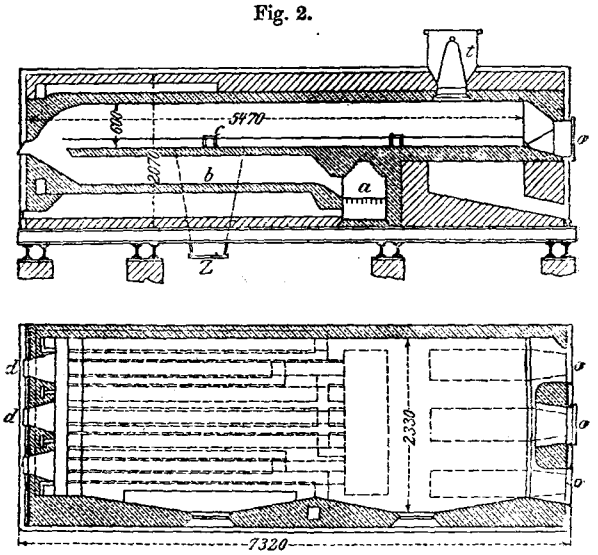
Roasting.—Modern furnaces have entirely superseded the well-known "Aludels." In some of these furnaces the ore and fuel are in direct contact; in others the furnace gases play on the ore, and the evolved vapours pass off with them. The former yield a less diluted vapour, and hence there is better condensation. Fig. 1 shows the construction of the Nowak furnace. In this, alternate

Fig. 1.



layers of fuel (charcoal) and coarse ore, each piece measuring from 40—90 mm. in diameter, are worked. In 24 hours, in three shifts of eight hours each, 12—14 tons are roasted, 1·5 cb. m. of fuel being required. Ten ovens employ, on an eight hours' shift, 14 men—eight to feed the furnace and six to carry the ore. The second type of furnace includes two kinds. (*a.*) That shown in the figure (Fig. 2), is a reverberatory furnace, on the hearth of which the ore is spread. It is cased in steel, and stands on a tray of steel plate, so that any accidental loss of mercury is avoided. Finely divided ore can be worked in it. In 24 hours 7·2 tons can be roasted, 3 cb. m. of beechwood branches or billets being used. For an eight hours' shift three men are needed—one at the furnace, two for the carriage of the ore. The work is very heavy and disagreeable, and this form of furnace is being superseded by another kind. This (*b*) is somewhat similar to the Gerstenhöfer pyrites burner, in which the ore, fed in at the top, gradually slides down over a series of stages, and reaches the bottom freed from mercury. These furnaces are made in two sizes, the larger dealing in 24 hours with 30—40 tons of ore, consuming 9 cb. m. of wood, and needing three shifts of nine men each; the corresponding figures for the smaller are seven tons, 8 cb. m., and two men. All these furnaces are worked from outside, and a fan beyond the condensing apparatus produces sufficient diminution of internal pressure to prevent any escape of fumes. The yield of the furnaces and the labour needed vary with the nature of the ore; the cinnabar ores are more easily dealt with than fahlerz. While the former yield a valueless residue, the latter yield one from which copper, silver, and iron are extracted (*e.g.*, 11·72 per cent. copper, 0·04 silver, 42·92 iron).

Fig. 2.



Condensation.—Curves expressing the density of saturated mercury vapour and the rate of loss by evaporation at different temperatures show that nothing is to be gained by cooling the gaseous products much below 20° C. The modern condensing arrangements are shown in Fig. 3. The U-shaped condensers are made of stoneware, and their open lower ends dip into water about 5 cm. deep, into which the condensed products fall; a water-spray over the condensers produces the cooling effect required. The uncondensed gases pass on into the large wooden settling-chamber (15,000 cb. m.), during their long and slow progress through which a further amount of deposition takes place; then they pass through the fan *f* into the chimney-shaft *g*. Even here a small amount of mercury is deposited.

Working-up of the "Stupp."—The deposited mud, or "Stupp," contains from 70 to 80 per cent. of mercury, chiefly as metal, partly as chloride or sulphide, with oxides and sulphates of calcium, magnesium, and iron, and (in the case of fahlerz) oxides of arsenic and antimony. It is mixed with quicklime, partly to absorb superfluous moisture, partly to saponify resinous and tarry matters, and to neutralise acids. Then it goes into a "Stupp mill," shown in Fig. 4, where it is squeezed between the knives carried on the rotating iron cross and those carried by the uprights fixed to the bottom of the vessel. The mercury sinks in drops to the bottom, falls through the holes (10 mm. diameter), which must be kept clear, and collects in the vessel as indicated. The residue in the mill still contains mercury, and is usually added to the ores to go through the process

Fig. 3.

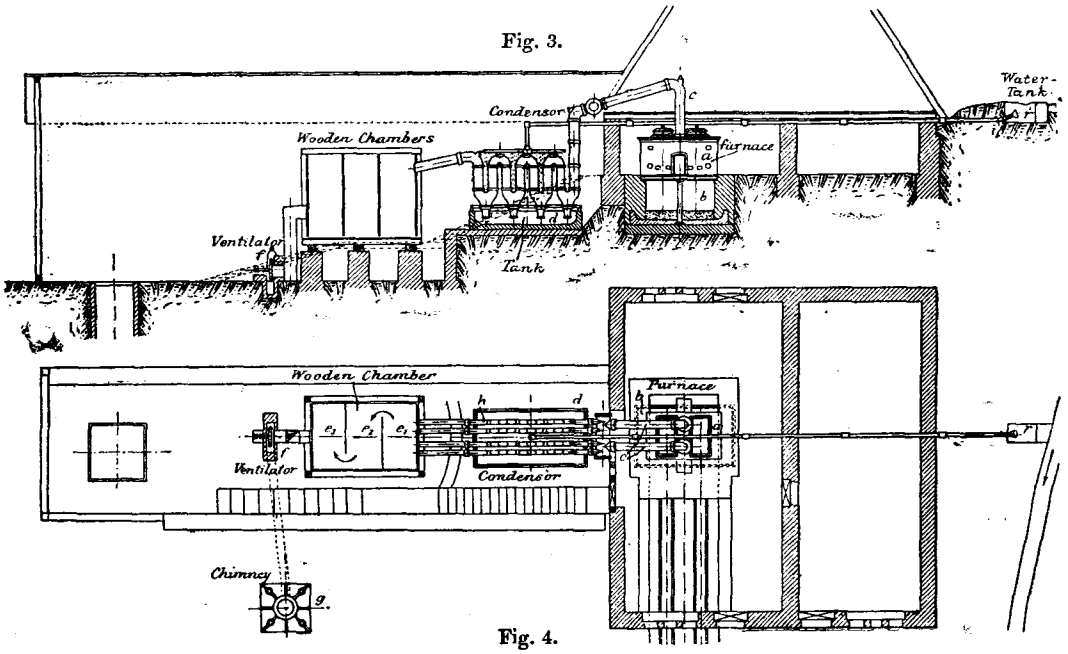
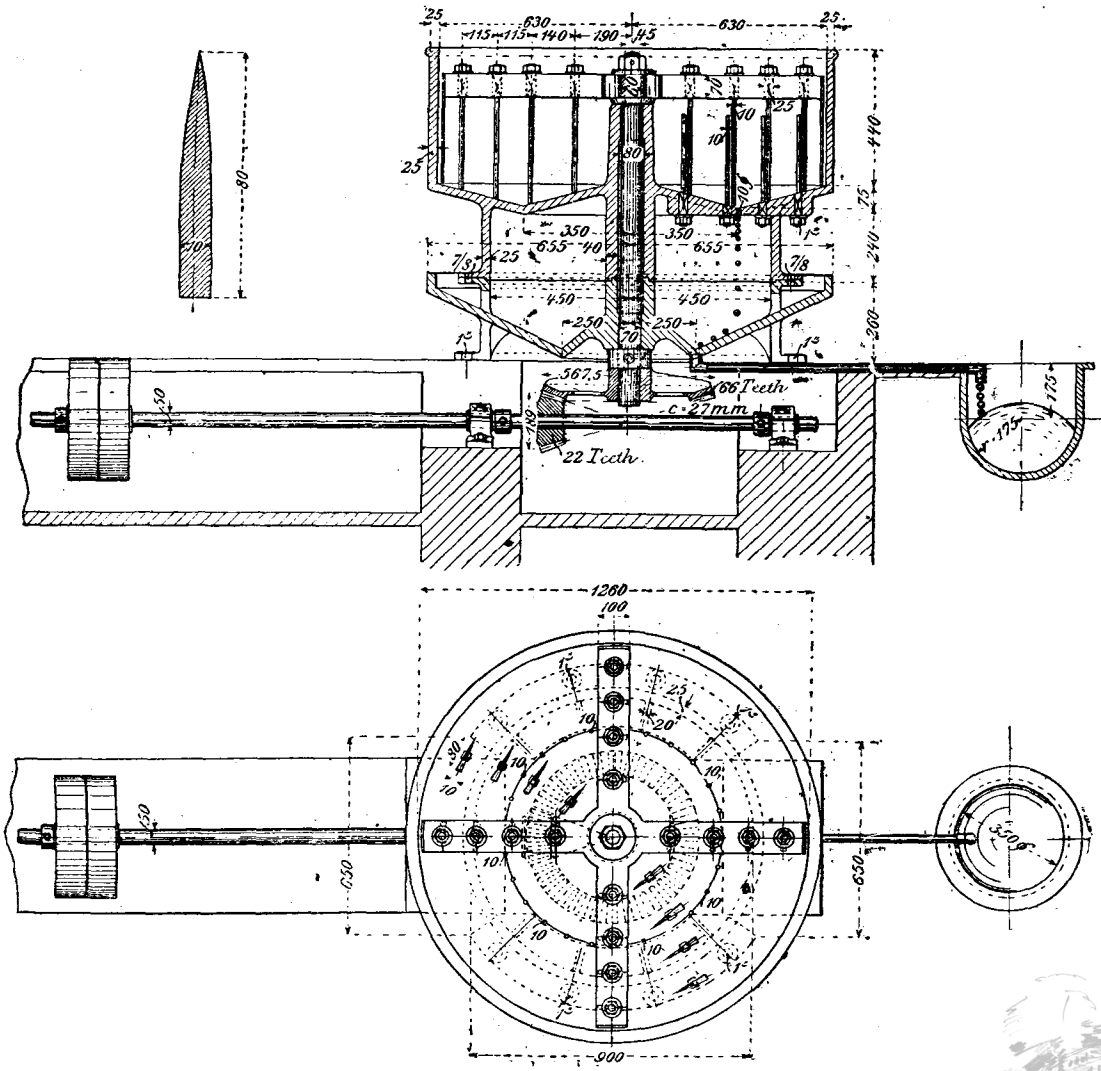

Water-Tank
Condensor
Wooden Chambers
Furnace
Ventilator
Tank
Wooden Chamber
Furnace
Ventilator
Condensor
Chimney

Fig. 4.

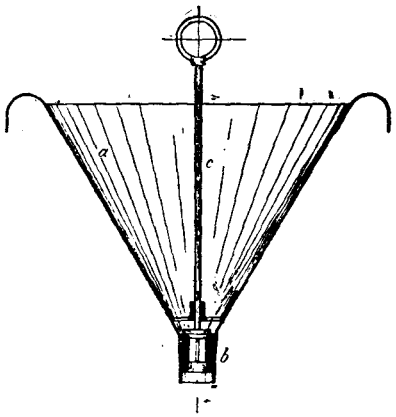

1260
900
630
650
567.5
66 Teeth
22 Teeth
c 27mm

again. The collected mercury is commercially pure; it only needs mechanical cleaning, which is effected by dipping into it the funnel *a*, Fig. 5, keeping the valve *b*, closed by pressure on the rod.

On letting go the rod *c*, the mercury-pressure opens the valve *b*, and the funnel is filled with clean mercury from the interior of the mass. When the funnel is lifted out the valve automatically closes, and is opened by pressure on the rod after the funnel has been placed in the mouth of one of the iron bottles in which the mercury is sold and transported.

The protection of the workers is an important factor in the conduct of the mercury industry. All is done that can be done by insisting on change of clothing, by the provision of baths, lard, mouth-washes of potassium chlorate, &c., to

Fig. 5.



prevent absorption of the metal; but as the air near the furnaces and stupp mill contains from 0·004 grm. to 0·008 grm. per litre of mercury vapour, and as a workman may thus inhale 0·4 grm. of mercury in a working day, these precautions are not always sufficient. When symptoms of mercurial poisoning show themselves, the workmen should be at once removed, and after recovery should be employed in some other industry, as they become more liable to a recurrence of the symptoms if they return to the mercurial atmosphere and surroundings.

The losses of mercury in the process occur from: (1) that remaining in the roasted ore; (2) that carried off in soluble form in the condensed water; (3) that carried off mechanically in the condensed water as finely-divided metal; (4) that escaping uncondensed into the atmosphere; (5) that lost during the various manipulations of the stupp and the metal. These losses were in former times enormous; amounted often to 80 per cent. of the total metal; now, with improved methods and appliances, they may be put at 10 per cent. in the aggregate.

Analytical Determination.—The percentage of mercury in the ores is determined by Eschka's method, which consists in mixing a sample (1—10 grms. according to the expected richness) intimately with iron filings in a porcelain crucible, covering the mixture with a layer of iron filings, and bending over the crucible a gold cover cooled on the outside. The crucible is heated for five minutes in a Bunsen flame, allowed to cool, the cover washed with alcohol and ether, dried at 40° C., and its increase of weight determined. In fluids, mercury is determined by precipitating with hydrogen sulphide, washing and drying the precipitate, and then determining the mercury in it by Eschka's method as above.

The author mentions two other processes of mercury-extraction. The first has only historical interest. The second method is possibly one for the future. Attempts to electrolyse a solution of cinnabar in alkali sulphide are being made, and may possibly be developed into a method applicable to the ordinary ores.—J. T. D.

Copper and its Alloys; Refining ——. Chem. Zeit. Rep. 1901, **25**, [15], 136.

A means, frequently employed for the removal of absorbed gases and cuprous oxide from copper, is the addition of the readily oxidisable metal magnesium. At a large foundry in Germany this is introduced into the melted copper before pouring, by binding it on the end of a copper rod with wire and stirring the melt The pieces of magnesium may be wrapped in thin copper sheet to prevent loss by wasteful oxidation. The violent boiling which occurs on the introduction of the magnesium greatly assists the elimination of the impurities. The manipulation, however, requires considerable experience and is attended with some danger, owing to the liability of the magnesium to catch fire at the surface or be thrown out of the crucible. The aluminium and magnesium factory at Hemelingen has recently introduced a copper-magnesium alloy, the use of which surmounts all the difficulties experienced in the older process. The alloy consists of equal proportions of copper and magnesium and is prepared from pure metals; it is easily powdered and has a specific gravity of 2·97 and melts at 450° C. There is not the slightest danger of waste, and the alloy acts more rapidly on the impurities of the copper than magnesium alone. About 50 grms. of alloy per 100 kilos. of copper are usually sufficient, but an addition of 100 grms. and upwards has an extremely favourable effect upon the density and strength of the copper. The mixture is also applicable for the refining of copper alloys of all kinds and the presence of up to 5 per cent. of magnesium in the castings endows them with valuable properties.—J. F. B.

Alloys containing Copper; Gradual Alteration of ——, *when left in Contact with the Air and with Alkali Chlorides.* Berthelot. Ann. Chim. Phys. **22** [7], 457—460. Chem. Centr. 1901, **1**, [18], 994.

In an earlier article (Ann. Chim. Phys. **4**, [9], 552), the author has shown how antique objects containing copper, undergo gradual alteration in the earth, and especially how the copper, in presence of small quantities of sodium chloride, is slowly converted into cuprous oxide. But it has been observed that many antique utensils consist completely of cuprous oxide, whilst still maintaining their forms or shapes. The author discusses the question as to whether such objects were manufactured of cuprous oxide, or whether, as he believes, they originally consisted of bronzes, of which the other constituents (Sn, Zn or Pb) disappeared in consequence of oxidation processes and formation of basic salts, whilst the copper remained behind in the form of cuprous oxide. For the confirmation of this hypothesis, the author studied the behaviour of copper alloys, when submitted to the simultaneous action of air and sodium chloride solution. Strips of brass were kept for two years in a dilute solution of sodium chloride in a loosely closed flask. After this time, the brass was partly converted into red cuprous oxide. At the same time, atacamite was formed, whilst the other metals of the alloy were found in solution. —A. S.

Tellurium; Preparation of Large Quantities of ——. E. Matthey. Proc. Roy. Soc. 1901, **68**, [444], 161—163.

In the course of several years' work on the extraction of bismuth from its ores, and the refining of the crude bismuth (see this Journal, 1891, 369; and 1893, 159), the author obtained large quantities of alkaline residues containing tellurium. He has now been able to treat these residues and extract from them a substantial amount, 26 kilos., of metallic tellurium. The alkaline residues obtained in refining the crude telluric bismuth were steeped in hot water, the solution acidified with hydrochloric acid, and the tellurium precipitated with sodium sulphite. The precipitate consisted of a mixture of bismuth and tellurium, the latter forming about 47·5 per cent. of the whole. The crude metal was dissolved in nitric acid, and again treated in the same way. The metallic tellurium obtained by this second treatment had the composition:—Tellurium, 97·00; bismuth, 2·15; copper, 0·65; iron, 0·10 per cent.

The metal, when broken, exhibits a crystalline fracture of needle-like structure and of bright metallic lustre. It does

not readily tarnish in the air at the ordinary temperature. If slowly cooled, it shows a crystalline form very much resembling that of bismuth. Its specific gravity is 6·27, as against 6·23, the density of uncompressed tellurium found by Spring. The melting point, as determined with the Le Chatelier pyrometer, was 450° C., or 5° lower than that given by Carnelly and Williams. A sample of chemically pure tellurium prepared by the author also melted at 450° C. The electrical resistance of tellurium is about 800 times that of copper, but is largely dependent on the crystalline condition, a rod cast and cooled quickly having a lower resistance than one that has been cooled slowly. In casting small rods of tellurium, of, say, $\frac{3}{8}$ in. diameter, there is much contraction, and partial separation takes place even after some hours. The thermo-electric power of tellurium appears to be great. The 57½ lb. (26 kilos.) of tellurium prepared by the author were derived from 187,019 lb. of crude bismuth, which resulted from the treatment of 831,168 lb. of mineral.—A. S.

Ore Sampler; The Calkins Umpire ——. Eng. and Mining J. 1901, **71**, [17], 534.

See under XXIII., *page* 617.

Sulphur; Determination of ——, *in Wrought Iron and Steel.* G. Auchy.

See under XXIII., *page* 620.

Vanadium in [*Iron*] *Slags and Cinders; Determination of* ——. C. H. Joüet.

See under XXIII., *page* 620.

Bismuth in Ores; Determination of ——. A. W. Warwick and T. D. Kyle.

See under XXIII., *page* 620.

Sodium; Use of ——, *in Blowpipe Analysis.* C. L. Parsons.

See under XXIII., *page* 618.

PATENTS.

Ores; Improved Method of Treating ——. M. Seligsohn, Denver, U.S.A. Eng. Pat. 23,660, Dec. 27, 1900.

A SOMEWHAT similar procedure to that patented by B. F. House (Amer. Pat. 590,739 of 1897) is protected. Air charged with "chemicals" is injected into the wet pulp, in or outside the mortar of a stamp battery, on an inclined amalgamated plate, in a Huntington mill, or otherwise, with the object of altering the physical conditions of the particles under treatment, and of thus facilitating amalgamation whilst preventing sickening or flouring. The "chemicals" may be chlorine or bromine (or both) with or without ammoniated air. The air charged with these gases is forced beneath the level of the "wet ore pulp."—W. G. M.

Ores and Metallurgical Products; Crushing and Lixiviating ——. E. C. H. Pape and W. S. Henneberg, Hamburg, Germany. Eng. Pat. 3044, Feb. 12, 1901.

BY this process the minimum of lixiviating solution for an ore can be employed. The stamps or other crushing machinery are covered with a certain head of water, and instead of the fine particles being carried through screens by a rush (and consequently an excess) of water, the whole of the material in the crushing mortar is kept agitated, as in jigging, and the finely crushed ore is allowed to overflow. By this means the milling water leaving the mortar with the ore may amount to as little as one-third of the weight of the ore. The crushing arrangement may be stamps or balls or other device, the movement of these being sufficient to give the necessary jigging motion. Centrifugal action in an open pan may also give the required overflow of fine ore around the upper edge. The lixiviating liquid may be used in crushing, or it may be mixed with the overflow pulp in rotating barrels outside.—W. G. M.

Furnaces for Smelting and Dephosphorising Iron and other Ores. E. T. Zohrab, Thurso, Caithness. Eng. Pat. 11,085, June 19, 1900.

To obviate the use of hot blast, two belts of tuyères are used in a blast-furnace, one in the usual position, just above the hearth, the other about the melting zone (a little above the boshes). Into the upper belt, peat gas produced from charring-ovens (such as those specified in Eng. Pat. 26,191 of 1896) is introduced, and into the lower belt, peat-gas, blown through an injector in such a way that it carries air with it. The upper tuyères may also be so arranged that they can be served with peat-gas and air when necessary. The fuel used in the furnace is preferably condensed peat or peat charcoal.—W. G. M.

Crucibles, more particularly Designed for Use in Connection with the Treatment of Ores. A. A. Crosby, Chicago, U.S.A. Eng. Pat. 2677, Feb. 7, 1901.

AN iron crucible is used, with means for introducing a supply of hydrocarbon fuel into the midst of the contents of the crucible, with the aid of a reservoir of liquid hydrocarbon and a system of coils of piping.—W. G. M.

Steel; Manufacture of Open Hearth ——. T. N. Muller, Saltburn-by-the-Sea, Yorkshire. Eng. Pat. 21,416, Nov. 27, 1900.

THIS patent covers the use of magnetic iron, sand, or finely-crushed magnetic iron ore, or a mixture of the two, alone or mixed with other oxides and oxidised compounds, or basic material, in the Talbot or Bertrand-Thiel, or other open-hearth furnaces into which fluid pig-iron may be poured or admitted for the purpose of making steel or ingot-iron.—W. G. M.

Metals and their Compounds; Converter Treatment of ——, *and Apparatus for that Purpose.* A. Reynolds, Sheffield. Eng. Pat. 5877, March 20, 1901.

A CONVERTER with two hollow trunnions is used, through one of which air may be passed and through the other reducing gas, each supply being controlled by a check-valve. The pipes conveying the gases from the trunnions may deliver separately into a pocket at one side of the converter, or may conduct them to tuyères placed at opposite sides of a converter of ordinary (side-blowing) construction. In blowing a charge of copper matte, the sulphide is melted in a cupola and charged as usual into the converter, where it is blown with air for a time. The converter is then turned down and the slag run off, after which reducing gas is blown through to effect deoxidation and to cool the highly heated charge. The gas is then stopped and air is again blown through, the oxidation and reduction being alternated until purification is complete. In treating iron, the metal is first decarburised by air and then deoxidised and recarburised to any desired extent by a blast of gas or vapour containing carbon.—W. G. M.

Coating or Overlaying with Gold, Silver, and other Metals. F. W. Howorth, London. From C. O. Harz and H. von Miller, München, Germany. Eng. Pat. 8363, May 5, 1900.

ANIMAL membranes, freed from grease, are painted with a solution of gelatin containing salts of chromic acid (chrome gelatin) and the desired metal, in leaf or foil, is applied thereon. The chrome gelatin is then hardened by the action of light on the untreated side of the membrane.

—R. L. J.

Aluminium; Uniting ——, *to Aluminium or other Metals* J. Y. Johnson, London. Eng. Pat. 6735, April 10, 1900.

THE aluminium surfaces to be united are cleansed well, and are then heated by means of a blast until they reach the exact temperature at which the metal softens sufficiently to enable a weld to be made with the hammer, and before oxide forms on the surfaces sufficiently to prevent welding. The critical moment is ascertained by slight hammering, and the temperature (which varies with the thicknesses of the pieces under treatment) must not be greatly overstepped, or the aluminium will become brittle and may fall to a

powder. At the right moment the weld is effected by working under the hammer.—W. G. M.

Titanic Oxide; Process and Method for Producing Concentrates containing High Percentages of ——. A. J. Rossi, New York; J. MacNaughton, Tahawus; New York State; and W. D. Edmonds, New York, U.S.A. Eng. Pat. 3582, Feb. 19, 1901.

In order to obtain from such material as titaniferous iron ore, a pure titanium compound, practically free from silica and other acids, suitable for the manufacture of ferro-titanium and the like, the iron ore is mixed with sufficient lime, or other suitable earthy base, to form titanate, and with sufficient carbon to reduce the oxide of iron and silica present; the mixture is then heated (preferably electrically) to 1,700° C. In this way a ferro-silicon is produced, and a slag of calcium titanate almost, or quite, free from silicon, sulphur, and phosphorus. The temperature must not greatly exceed 1,700° C., or titanic oxide will also be reduced.—W. G. M.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(*A.*)—ELECTRO-CHEMISTRY.

Chlorine; Production of ——, *from Ferrous Chloride by Electrolysis.* Roubertie and Pepin. Rev. Prod. Chim. **4**, [4], 51.

The operation is conducted in a wooden vessel containing a carbon anode and an iron cathode, the ferrous chloride solution being maintained in a highly concentrated state by an excess of the solid salt placed in a special compartment and kept in contact with the solution. A 4 to 5 volt current is employed, the ampèrage amounting to 600 ampères (maximum) per sq. m. of cathode surface. The chlorine liberated at the cathode is collected in a closed chamber enclosing this part of the apparatus, and is removed, through a conduit, for storage or further treatment. No secondary reaction occurs. With a consumption of 110 h.p. the apparatus will extract the chlorine from 1 ton of ferrous chloride in 24 hours.—C. S.

Anodes for Electrolytic Alkali Cells. A. T. Weightman. Eng. and Mining J., Aug. 4, 1900. (See also this Journal, 1899, 583.)

The author states that the anodes now employed in most electrolytic alkali works are "graphitized carbons" manufactured according to the process patented by Acheson (see this Journal, 1900, 450). He considers that the influence of current density and of temperature upon the life of carbon anodes is not sufficiently recognised. Samples of various carbons may be compared, as to durability, by submitting them to the action of 10 per cent. sulphuric acid for 10 hours, and determining the loss in weight.

A new form of platinum anode which is claimed to be highly efficient, and to cost only about 75 cents, has been devised by Le Sueur, and is in use at Rumford Falls. Short lengths of platinum wire are bunched together in glass tubes, and electrical connection is obtained by pouring in a small quantity of mercury.—A. S.

Pyrogenetic Reactions with the Aid of the Electric Current. W. Löb. Ber. **34**, [6], 915—918.

The electric arc (110 volts, about 10 ampères) was passed between carbon poles in a liquid or its vapour, the containing vessel being furnished with a reflux condenser and a gas-collecting apparatus. The experiments (still in a preliminary stage) have shown that the results are practically the same whether with continuous or alternating current, and whether the liquid or its vapour be used. *Water* yielded a gas containing hydrogen, 50; carbon monoxide, 40; carbon dioxide, 7; unsaturated hydrocarbons, 0·8; saturated hydrocarbons, 2 per cent. *Methyl alcohol* yielded formic acid, and a gas containing methane, 39; hydrogen, 45 per cent.; and small proportions of both oxides of carbon and of acetylene; no formaldehyde. *Glacial acetic acid* yielded carbon monoxide, 35; dioxide, 15·5; saturated and unsaturated hydrocarbons, 12 and 7 per cent. respectively. *Benzene.*—Chars, turns brown, evolves gas containing 86—90 per cent. of hydrogen, and traces of hydrocarbons. The behaviour of naphthalene is similar.

Instead of the electric arc a spiral of electrically heated iron wire was used, at a cherry-red heat; the yields here depended on the duration of the heating. *Methyl alcohol* gave formic acid, a little trioxymethylene, and gas—hydrogen, 72; carbon monoxide, 20; methane, 6·5 (as against 39 with the arc), and traces of carbon dioxide. *Benzene* evolved very little gas; in the liquid were found diphenyl, and another hydrocarbon, apparently diphenylbenzene. *Aniline* becomes dark, giving off ammonia and a little of some other gas; diphenylamine and carbazole were found in the liquid. *Nitrobenzene* darkened very much, gave off much nitric oxide, and a solid substance was formed, not yet identified.—J. T. D.

Cadmium; New Use for ——. Eng. and Mining J. 1901, **71**, [18], 554.

The Edison storage battery (see this Journal, 1901, 258), which comprises cadmium-copper couples in an electrolyte consisting of a 10 per cent. solution of caustic soda, is claimed to be less expensive, lighter, and more compact than an ordinary lead storage battery. The new battery, it is stated, can be discharged to zero voltage, whilst it only deteriorates very slowly, if at all.—A. S.

Monopersulphuric Acid (Caro's Acid). A. Baeyer and V. Villiger.

See under VII., *page* 578.

PATENTS.

Electric Furnaces of Great Power. [*Cooling by Water Circulation.*] O. Imray, London. From F. Morani, Rome, Italy. Eng. Pat. 10,580, June 9, 1900.

The electrode is supported by one or more yokes of suitable form, firmly held in a cross-head by bolts and heads, to which the surface of contact with the electrode is fixed by a metal tube, through which and an inner tube a complete circulation of water cools the supports of the electrodes, the contact of the conductors to the electrodes being effected by bolts and nuts. Various arrangements are described for leading the current to the walls of the furnace by metal conductors or yokes, or walls of a casing, or by groups of tubes, all cooled by water circulation. The liquid contents of the furnace are discharged through one or more slits formed between the walls of a metal casing cooled by water circulation.—G. H. R.

Accumulators; Electric [*High Voltage*] *and* [*Bipolar*] *Electrodes therefor.* A. Tribelhorn, Olten, Switzerland. Eng. Pat. 11,260, June 21, 1900.

In a high-voltage accumulator with double electrodes without rigid or solid core-plates, the neutral layer of the electrodes may be either (*a*) "impregnated by means of a substance rendering it impervious and not participating in the electro-chemical process, and opposing the passage of the ions from the positive to the negative side, for example, a solution of caoutchouc, solution compound, asphalt, collodion, amber," &c.; or (*b*) it may be provided with a diaphragm, or membrane of caoutchouc, which forms an elastic partition, which opposes the passage of the ions from the positive to the negative side, and does not impair the compensating action of the two halves of the active material during its expansion and contraction. The latter is contained in frames of which the opposite sides are provided in a longitudinal direction with recesses becoming larger as they extend from the edges to the middle of the side, and assisting to secure during the expansion of the positive half the retention of the negative active material in the frame, and prevent absolutely the escape of the active material, the faces being covered with bands or strips which electrically connect the two halves of the electrodes, and increase the solidity of the frame. The double electrodes, preferably conical in form, are surrounded by a ring of caoutchouc, and mounted in a trough contracted towards the bottom, or electrodes of type (*a*) may be arranged

vertically without special supports in receptacles where they form partitions. The double electrodes comprise "layers of fabric or material formed, for example, by threads of glass, which do not participate in the electrolysis, and extend horizontally and transversely through the electrodes at suitable intervals" to prevent their sinking. The substance which renders the neutral layer impervious also acts as an agglutinant between the parts of the double electrode, and agglomerates all the parts by means of horizontal layers of fabric, "a covering of bands or strips of lead, for example, being applied between the double electrodes and the walls of the receptacle, and serving to connect electrically the two halves of the electrodes."

—G. H. R.

Storage [*Alkaline-Zincate*] *Batteries.* T. A. Edison, Orange, U.S.A. Eng. Pat. 2490, Feb. 5, 1901.

THE closed receptacle for containing the active elements under pressure is formed of sheet metal, iron nickel-plated, so as to be unaffected by electrolytic action in an alkaline bath, and has numerous small openings in it made by displacing the metal so that the burr surrounding each opening will project inwardly, thereby increasing the area of contact of the active material which is held in position by a separate closing device. Oxides of nickel or cobalt mixed with graphite form one of the active elements in an alkaline electrolyte, and the oxidisable pole is composed of finely divided metallic iron mixed with graphite, an oxide of iron being employed of such hydration as will allow of its being reduced electrolytically to the metallic state.—G. H. R.

(*B.*)—ELECTRO-METALLURGY.

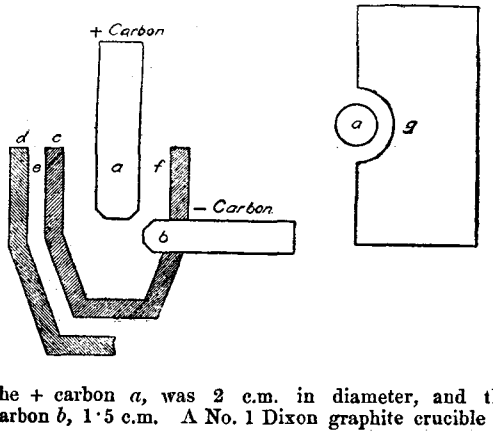
Antimony; Electrolytic Deposition of ——. A. Hollard. Eclair. électr. 1901, **26**, 165—166. Through Zeits. für Elektrochem. 1901, **7**, [41], 551—552.

THE author considers the disposition of the Classen cathode as the cause of the redissolving of antimony by polysulphide, since the dense polysulphide settles down on it. This can be prevented by placing the cathode in another position. A further disadvantage of the sodium hydrosulphide method lies in the fact that the solution dissolves copper (to the extent of 3 or 4 mgrm. per 100 c.c.) which is then deposited along with the antimony. The author recommends a solution of 13 grm. of potassium cyanide and 20 c.c. of concentrated sodium hydrosulphide in 220 c.c. of water. From this solution copper is not deposited owing to its small ionic concentration and to the fact that it exists chiefly in the form of a complex ion.

In order to determine antimony in commercial tin he employs a solution of 3 volumes of NaHS (sp. gr. 1·22) and 1 volume of 20 per cent. potassium cyanide, the current used being 0·05 ampères.—J. S.

Alloys made in the Electric Furnace ——. L. P. Hamilton and E. F. Smith. J. Amer. Chem. Soc. 1901, **23**, [3], 151—155.

THE furnace used by the authors is shown in the accompanying figure:—



The + carbon *a*, was 2 c.m. in diameter, and the — carbon *b*, 1·5 c.m. A No. 1 Dixon graphite crucible *c*, with an interior diameter of 5 c.m. and a depth of 7 c.m., was placed in a second graphite crucible *d*, with a diameter of 10 c.m. The space between the crucibles was filled with magnesia, whilst two carbon plates, 1·5 c.m. thick, of which *g* represents one, served as a cover. The carbon was inserted at about 3 c.m. from the bottom of the crucible which was lined with magnesia, so that the products should be free from carbon.

The material was introduced into *c* to about the level of the carbon *b*, and the arc between the poles started by means of a thin carbon pencil. The crucible was then partially covered by *g*, and the remainder of the substance introduced little by little, until 150 to 200 grms. in all had been added. The current was continued for some time, the total duration amounting to 10 to 15 minutes. The strength of the current was 145 to 160 A., and from 25 to 70 V.

Alloy of Copper, Tungsten, Iron, and Gangue.—This was obtained by fusing together commercial copper and a tungsten matte. It was lighter in colour but harder than copper, and was found to be the hardest of the copper alloys. Its specific gravity was 7·98, and its chemical composition:—copper, 66·68; tungsten, 23·03; iron, 5·74; and gangue, 5·04 per cent.

Titanium, Copper, Silicon, and Carbon.—This was not so hard as the preceding alloy. Its specific gravity was 7·616, and its composition:—copper, 90·98; titanium, 3·12; silicon, 3·51; and carbon, 2·08 per cent. It resembled brass in appearance. It was prepared by reducing rutile with carbon, and fusing the product with copper.

Columbite reduced with carbon and fused with copper, gave an alloy resembling copper. Its specific gravity was 8·38.

Copper and Molybdenum Matte.—The alloy was greyish-red in colour, and much harder than copper. Its specific gravity was 7·934, and its composition:—copper, 78·53; molybdenum, 8·53; iron, 2·71; carbon, 2·42; and gangue, 8·02 per cent.

Copper and Chromium.—On fusing chromium oxide and metallic copper in a carbon crucible, a greyish-red alloy with a specific gravity of 8·3146 was obtained. In hardness this came next to the tungsten and molybdenum alloys. Its composition was:—copper, 88·18; chromium, 3·22; iron, 1·35; carbon, 2·38; and gangue 4·13 per cent.

Copper, Aluminium, and Tungsten Matte.—A tungsten matte fused with equal parts of copper and aluminium, gave a yellow alloy with the following composition:—copper 34·11; aluminium, 24·89; tungsten, 32·67; iron, 2·12; and gangue, 6·56 per cent.

Iron, Titanium, and Tungsten.—10 grms. of iron were fused with the same quantity of titanium matte and of tungsten, 6 to 8 grms. of ferric oxide being added during the fusion. The alloy was steel-grey in colour, and had a specific gravity of 6·707, and the following composition:—iron, 82·15; titanium, 7·28; tungsten, 1·66; gangue, 6·63; and carbon, 2·30 per cent.

Iron, Chromium, and Titanium.—This alloy was made in the same way as the last one. It was steel-grey in colour, and had a distinct fracture and a specific gravity of 6·464. Its composition was:—iron, 76·41; chromium, 16·29; titanium, 2·47; silicon, 2·39; and carbon, 3·14 per cent. By largely increasing the proportion of chromium, an alloy with the following composition was obtained:—iron, 53·03; chromium, 40·37; titanium, 2·65; and gangue, 4·33 per cent.

Columbium, Tantalum, and Iron.—A brittle steel-grey alloy was obtained by reducing columbite with carbon, and fusing the product with iron in excess. Its composition was:—columbium, 59·76; tantalum, 18·77; iron, 15·73; gangue, 5·12; and tungsten, 0·63 per cent.

Columbium, Tantalum, Titanium, and Iron.—This was a hard grey alloy with the following composition:—iron, 80·03; columbium, 10·15; tantalum, 2·91; titanium, 3·18; carbon, 1·99; and silicon, 2·14 per cent.

—C. A. M.

PATENT.

Electrolytical Apparatus [*Stripping Tin from Scrap Metal*]. J. Matthews, Kings Heath; and W. Davies, Selly Park. Eng. Pat. 21,533, Nov. 28, 1900.

An outer tank or vat contains an inner one formed of a number of perforated blocks or slabs of insulating material supported by curved pieces of angle iron and metal uprights of girder T or other section. On this is mounted a revolving drum, having a number of radiating arms or rods passing through and supporting an outer circular face of insulating material. Instead of using the tank as a medium for the returned current, cathodes may be employed fitting into cells or holders provided with a well or pocket at the base to catch any granulated tin that may fall from the cathodes.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Lard Oil. M. Duyk. Bull. Assoc. Belge des Chim. **15**, (1), 18—19.

The author examined a sample of lard oil and obtained the following values:—Incipient solidification point, about 10° C.; sp. gr. at 14° C., 0·916; at 100° C., 0·8626; butyro-refractometer index (Zeiss), 52° at 40° C.; critical temperature in open tube, 75° C.; sulphuric acid thermal reaction, 47°; iodine value, 73; saponification value, 193; percentage of non volatile fatty acids, 97·4 per cent.; volatile acids, 0. The fatty acids are solid and hard at the ordinary temperature, melting at 35°, and solidifying at 31°; the specific gravity is 0·885 and the butyro-refractometer index 41° (at 40° C.). Under the action of nitrous vapours the oil solidifies in a short time, forming a faintly-greenish cake.—C. S.

Oils; Determination of the Heat of Combustion of ——, *as an Analytical Factor.* H. C. Sherman and J. F. Snell. J. Amer. Chem. Soc. 1901, **23**, [3], 164—172.

Apparatus and Method.—A bomb-calorimeter of the Atwater Blakeslee type was used by the authors for their determinations, and its hydrothermal equivalent found (1) by calculation from the weights and specific heats of the component materials; (2) from five combustions of cane sugar assumed to liberate 3,959 calories per grm.; and (3) by combustions with benzoic acid. To reduce the results obtained to constant pressure it is necessary to add $(\frac{1}{2}p-q)\frac{T}{M}$ calories per grm. where p represents the number of atoms of hydrogen, q the number of atoms of oxygen in the molecule, M the molecular weight of the substance, and T the absolute temperature of the calorimeter.

The following corrections were thus found to be required:—For American petroleums, 22 cals.; sperm oil, 18 cals.; castor oil and rosin oil, 14 cals.; and all oils consisting essentially of non-hydroxylated glycerides, 15 cals.

Ignition of the Oil.—A small quantity of fibrous asbestos was used as an absorbent, and this was placed in the platinum crucible and ignited directly by the electrically-fused iron wire.

The chief results obtained are given in the following table:—

Oil.	Specific Gravity at 15°·5 C.	Iodine Value.	Free Acid as Oleic Acid.	Heat of Combustion, per Grm.	
				Constant Volume.	Constant Pressure.
			Per Cent.	Calories.	Calories.
Raw linseed I. (1900), fresh	0·934	182·4	4·30	9,364	9,379
„ II. (1898)	0·938	175·9	1·22	9,379	9,394
„ III. old	0·947	156·7	5·30	9,215	9,230
Boiled linseed	0·953	150·7	7·40	8,810	8,824
Poppy seed	0·926	129·6	2·66	9,382	9,397
Maize I. (1900)	0·924	120·3	3·32	9,413	9,428
„ II. (1898)	0·926	120·7	2·56	9,436	9,451
„ III. crude	0·926	122·4	1·68	9,419	9,434
Cotton seed I. yellow	0·920	102·5	0·20	9,396	9,411
„ II. „	0·921	106·4	0·32	9,401	9,416
„ III. white	0·923	105·5	0·08	9,390	9,405
„ IV. crude	0·927	103·2	2·28	9,397	9,412
„ VII. old	0·941	93·7	2·03	9,168	9,183
Sesamé	0·924	105·3	1·65	9,395	9,410
Rapeseed	0·922	107·4	0·82	9,489	9,504
Castor	0·967	84·1	0·26	8,863	8,877
Earth nut	0·917	105·9	0·16	9,412	9,427
Almond	0·919	98·1	5·13	9,454	9,469
Olive	0·917	85·1	2·51	9,457	9,472
Menhaden (refined)	0·935	..	0·36	9,360	9,375
Codliver, fresh	0·927	165·6	0·56	9,437	9,452
„ old	0·933	137·3	1·50	9,277	9,292
Whale	0·924	126·6	0·60	9,473	9,488
Lard I. (1900)	0·917	74·3	0·74	9,451	9,466
Sperm	0·883	78·7	0·78	9,946	9,964
Rosin	0·989	76·9	14·40	10,145	10,159
Lubricating petroleum I.	0·881	..	..	10,797	10,819
„ „ II.	0·897	..	..	10,753	10,775
„ „ III.	0·905	..	..	10,682	10,704

From these results it appears that sperm, rosin, and mineral oils yield higher calorific results than fatty oils, and that the results given by non-drying glycerides are slightly greater than those from drying oils. Castor oil and boiled linseed oil show very low heats of combustion, as is also the case with old oils. The variations in the heats of combustion stand in closer relationship to the specific gravity than to the iodine absorption or acidity. For oils of the same kind the product of the heat of combustion multiplied by the specific gravity is practically a constant unaffected by age or exposure. This product is slightly higher for drying oils than for non-drying oils. In the case of the ordinary oils examined, it was found to range from 8·80 for the raw linseed oil II., to 8·63 for the earth-nut oil. The values for castor and boiled linseed oils were somewhat lower, whilst in the case of sperm oil and mineral and rosin oils they were higher.

The heat of combustion divided by the specific gravity yielded a result ranging from 10·0 to 10·3 for fresh unoxidised fatty oils, 11·2 for sperm oil, and 11·8 to 12·3 for the mineral oils.—C. A. M.

Oils; Bromine and Iodine Values of ——. H. T. Vulté and Lily Logan. J. Amer. Chem. Soc. 1901, **23**, [3], 156—159.

The authors have made a comparative determination of the bromine and iodine values of various oils. The bromine value was determined by McIlhiney's method (this Journal, 1900, 176), with the exception that 30 minutes were allowed before titration. In determining the iodine value, Hübl's method was used, the flasks being left in the dark for 24 hours before the titration. In each case the amount of substitution was determined, as in McIlhiney's bromine method.

The results thus obtained are shown in the subjoined table, in which the oils are grouped according to the increasing divergence between the bromine and iodine figures. As regards the oils in the first class, it makes but little difference whichever value be determined. In the case of rape oil there appears to be more substitution with iodine than with bromine, possibly owing to the longer time of action of the former.

Oil.	Iodine Value (Hübl).	Iodine Value Calculated from Bromine.	Difference of Averages.	Difference of Nearest Figures.
I. Olive	79·70— 80·40	80·31	0·25	0·09
Cotton-seed	97·50	97·41—97·00	0·295	0·09
Poppy seed	127·98—128·55	128·37	0·55	0·28
Linseed	155·12—155·52	154·80	0·52	0·32
Sweet almond	90·53— 89·64	90·20— 89·64	1·08	0·33
Earthnut	100·71—100·76	101·26	0·523	0·50
Whale	128·00	127·45—127·38	0·585	0·50
Lard	76·99— 77·36	76·03— 75·88	1·22	0·96
II. Sperm	79·95— 79·76	82·39— 82·08	2·422	2·13
III. Rape	103·69—103·37	99·03— 98·56	4·60	4·17
Castor	86·32— 87·15	78·74— 78·71	8·01	7·58
IV. Seal	93·31— 93·99	103·09—103·74	9·765	9·10
Codliver	122·09—122·79	132·86—132·18	10·08	9·39
Menhaden	176·05—175·65	186·94—186·86	10·825	10·36
V. Rosin	59·67— 58·95	10·60— 10·26	48·845	48·35

The authors suggest that the ratio between the two values might serve as an indication of, *e.g.*, the presence of menhaden oil in linseed oil.

Of the 15 oils only six gave any marked degree of substitution. The values obtained with these are given in the following table :—

Oil.	Total Bromine Absorption.	Addition Figure.	Substitution Figure.
Codliver	85·16— 84·67	84·52— 84·09	0·63— 0·58
Average	84·91	84·305	0·605
Menhaden	120·10—120·30	118·88—118·93	1·22— 1·27
Average	120·15	118·905	1·245
Sweet almond	59·54— 59·15	57·38— 57·02	2·16— 2·13
Average	59·345	57·20	2·145
Sperm	54·61— 54·56	52·41— 52·21	2·20— 2·34
Average	54·585	52·31	2·27
Castor	52·62— 52·80	50·09— 50·07	2·53— 2·73
Average	52·71	50·08	2·63
Rosin	103·67—109·23	6·74— 6·47	101·93—102·76
Average	108·90	6·605	102·345

(See this Journal, 1900, 213.)—C. A. M.

Fat Extraction Apparatus; New ——. W. Jerwitz.

See under XXIII., *page* 618.

PATENTS.

Fatty Matters from Wool, Sheep-Skins, and other Textile Fibres; Process and Apparatus for Extracting. C. D. Abel, London. From Délainage Verviétois et Cie., Renoupré-Verviers, Belgium. Eng. Pat. 2360, Feb. 6, 1900.

The material to be extracted is treated with carbon tetrachloride under water, or in closed chambers, the solution collected, and the solvent evaporated. Claim is also made for different modifications of apparatus in which the material is either made to circulate through stationary carbon tetrachloride, or the solvent through the material, or a combination of both methods is employed. In all three cases the solvent is protected from evaporation by being covered with water.—C. A. M.

Rape-Seed Oil and other Sweet Oils; Method of Purifying ——. F. Linde, Dortmund, Germany. Eng. Pat. 13,201, July 21, 1900.

The oil is mixed with milk (10 to 15 per cent.), heated to a temperature exceeding 100° C., and filtered. The residue can be used for fodder.—C. A. M.

Soap; Manufacture of ——. T. Parziale, Alexandria, Egypt. Eng. Pat. 10,912, June 15, 1900.

Cold cotton-seed oil is treated with flour and caustic soda solution in specified proportions, and the resulting soap incorporated with any other substances desired.—C. A. M.

Rosin Soap; Process of and Apparatus for Producing ——. C. L. Culmann, Hamburg, Germany. Eng. Pat. 16,989, Sept. 24, 1900.

Claim is made for the manufacture of a rosin soap having an acid reaction and containing free carbonic acid. The soap mixture is heated in an open vessel at a relatively low temperature (say 75°—80° C.), whilst made to circulate through the carbonic acid, which it thus absorbs.

The apparatus claimed for this process consists of a steam-jacketted boiler, containing a pair of concentric bells communicating with one another at their closed ends. The contents of the boiler are forced up into the inner bell by means of a vertical screw or conveyor, and passing laterally into the exterior bell fall back into the boiler. Separate claims are made for different parts and modifications of this apparatus.—C. A. M.

Liquids, Frothing ——*; Processes for Boiling, and Apparatus therefor.* M. Etfurt, Straupitz, near Hirschberg, Silesia, Germany. Eng. Pat. 5817, March 19, 1901.

The process claimed, which is specially intended for use in the manufacture of rosin soap, consists in conducting the froth through a "scumming chamber," where it loses the gas it contains whilst the liquid falls back into the boiler. Separate claims are made for cooling the scumming chamber, and for the condensation of the escaping steam or vapour.—C. A. M.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*A.*)—PIGMENTS, PAINTS.

PATENTS.

White Lead; New Process and Method for Producing ——. E. R. Blundstone, London. Eng. Pat. 8820, May 12, 1900.

Litharge is dissolved in lactic acid or in a solution of lead lactate until the liquid is strongly alkaline to litmus. Carbon dioxide is then led through the solution until the reaction is nearly neutral. The precipitated white lead is separated and washed; the filtrate is treated with litharge again. A denser product can be obtained by precipitating under pressure. The process may be worked in the cold.

—F. H. L.

Pigments or Paints; Manufacture of ——. F. A. Hyndman and W. B. Banyard, London. Eng. Pat. 9282, May 19, 1900.

The inventors claim that by mixing 85 parts of pigment or paint with 15 of methylated spirit they produce paints of greater brilliancy and permanence than heretofore possible.

—F. H. L.

Zinc Sulphide Suitable for Pigments, Manufacture of ——; and of Sulphocyanides as a By-product. E. Beringer, Charlottenburg, Berlin. Eng. Pat. 11,108, June 19 1900.

Into an enamelled digestor an ammoniacal solution of zinc is introduced, compounded from zinc sulphate, 230 lb.; water, about 90 galls.; ammonia (sp. gr. 0·91), about 230 lb. About 60 lb. of carbon bisulphide are added, the vessel is closed, the temperature rapidly raised to 100° C. with constant agitation, until the pressure rises to 60 lb. After four hours the pressure begins to fall; in 10 hours it will have sunk to about 22 lb., and the operation is finished. The zinc sulphide is collected, washed, dried, and ground; it is in a very finely divided state, and is suitable to be used as a pigment. The ammonia is converted into ammonium thiocyanate; any excess is recovered by treatment with quicklime.

The inventor refers to Ger. Pat. 97,820, in which, however, the zinc sulphide only appeared as a by-product in the manufacture of guanidine salts, and "of no commercial importance."—F. H. L.

(*C.*)—INDIA-RUBBER, &c.

Caoutchouc; Columbian ——. G. Springer. Gummi-Zeit. 1901, **15**, [20], 479.

The term "Columbian Caoutchouc" comprises several kinds of rubber obtained from the juice of the *Hevea* and *Castilloa*, but which vary considerably as to geographical origin and market value. The following may be distinguished:—"Virgin Columbia" (loss on washing, 15—20 per cent.), of good quality, rather like Para, and sold as such; "Carthagena" (Essequibo) (loss on washing, 25—60 per cent.), of medium value, consisting of black-coloured strings squeezed together into balls, of unpleasant odour, brownish-black when freshly cut, and often loaded with sand; "Guyaquil," recognised as black laminated pieces, very impure and of low value; "Colon" and "Panama," both sold in long strings and of medium value.

Twenty years ago "Columbian scraps" were for some time sold in London in considerable quantity, and were preferred to the genuine virgin Columbia. This rubber was derived from the *Sapium biglandulosum*, a forest-tree common at that time in Central and South America, the juice of which easily coagulates on the incisions and only requires to be scraped off. Subsequently this tree has been almost entirely destroyed, and its product no longer appears in the market.—R. L. J.

India-rubber; The Colouring of——. O. Markfeldt, Gummi-Zeit. **15**, 224.

The dyeing of india-rubber throughout its mass must be effected before vulcanisation. The dyes, or pigments employed for this purpose, must therefore be such as are capable of withstanding a temperature of 140° C., and not chemically acted upon by sulphur at that temperature. For the latter reason lead pigments cannot be used, and the same may be said of pigments containing iron, tin, mercury, and copper.

The dyeing of india-rubber with inorganic pigments is carried out by mixing them, together with the sulphur, with the india-rubber until a sufficiently homogeneous mixture has been obtained. Attempts have not been wanting to dye india-rubber by methods similar to those employed for the dyeing of textile materials.

The dyeing of india-rubber with aqueous solutions of the aniline dyestuffs has not proved successful, besides, such colours would considerably suffer in the subsequent operations. Hence only pigments are employed at present for the dyeing of india-rubber in the mass, zinc white, lithophone, antimony and vermillion being the most important.

The dyeing of india-rubber with solutions of the aniline dyestuffs, for which quite a number of patents have been taken, is altogether unsatisfactory, the shades being dull and very unstable. The results obtained with solutions of aniline dyestuffs in alcohol were perhaps better, but only moderately satisfactory. Much better effects have been recently obtained with solutions of certain aniline dye preparations in benzene, or carbon bisulphide, especially for the dyeing of thin, transparent articles, such as toy balloons. Cerasin Red, Red G, Orange, Dark Yellow and Soudan II., give particularly good results as regards permanency and brilliancy, but the shades assume brightness only when these articles are fully distended, the undistended article appearing dull.—C. O. W.

Para Rubber Mixings. G. Springer. Gummi-Zeit. **15**, [20], 327.

Para mixings are, strictly speaking, mixings consisting of pure Para rubber and sulphur. At present, however, all mixings of any kind of india-rubber and sulphur are described by the above term. All these float upon water and are further characterised by their peculiar grey colour. All high and medium grades of rubber may be used for Para-mixings provided the percentage of ash they yield on incineration does not exceed 0·6 per cent. and providing further that they can be vulcanised with a moderate proportion of sulphur. Also the colour of the india-rubber after vulcanisation is a guide as to suitability of any particular brand of india-rubber for the above purpose. No rubber exhibiting after vulcanisation a dark brown, or black colour should be used. Particularly some of the soft African brands are objectionable in this respect. Great care should be employed in the washing of the rubber for Para mixings and it is advisable to continue the process until the water is quite free from turbidity. In mixing the dry india-rubber with the sulphur only small batches at a time should be taken into operation as the time required for the mixing of large batches is apt to injure the quality of the mixing. Nor should mixings, especially those containing substitutes, be allowed to lie unvulcanised for any considerable length of time. Best qualities contain rarely more than 20 per cent. of Para, about the same quantity of Negro head, and about 30 per cent. of one or two of the better African qualities. Reduction of the percentage of Para above given is not advisable, the preferable alternative being to reduce the quality of the other rubbers used. Besides india-rubber probably all Para mixings contain at present substitute to the amount of about 10 per cent. Second grade Para mixings contain only 10 per cent. of Para or less, the amount of Negro head is reduced to 5 per cent, the amount of substitute raised to 12 per cent., the rest consisting of sulphur and lower-class rubbers. For still lower qualities no Para is used at all, nor as a rule any Negro head, only rubbers of third quality being employed with up to 15 per cent. of substitute and "floating" qualities of recovered rubber. The percentage of sulphur used in these mixings varies considerably, from 4·5 to 8·5 per cent., according to the vulcanising conditions observed. The quality of the sulphur employed, as also its uniform distribution in the mixing, are points of great importance. Flowers of sulphur are very unreliable, precipitated sulphur, in spite of its high price, is much to be preferred. The vulcanisation temperature should not exceed 146° C., and must be controlled by means of suitably placed thermometers. Revulcanisation of undervulcanised goods is generally unsatisfactory, and has invariably a deteriorating influence upon their quality. It is customary to subject these goods to a treatment with a dilute solution of caustic soda in order, as it is stated, to remove the free sulphur. Analysis of the goods before and after this treatment shows, however, that no such action takes place. The action of the alkaline bath is strictly confined to the surface, and is merely that of a soap solution. Indeed, soap solutions are at present used with good results for this final treatment.—C. O. W.

India-Rubber and Sulphur. G. Springer. Gummi-Zeit. **15**, [18], 294.

Vulcanised india-rubber, when examined under the microscope, is found to contain throughout its mass well developed crystals of free sulphur. No sulphur crystals occurring in the "flowers of sulphur", these crystals, in the writer's opinion, can only have been formed by the sulphur passing into solution in the india-rubber. The writer contends that in the process of masticating the india-rubber between hot rollers liquid hydrocarbons are formed, which are capable of dissolving the amorphous sulphur, depositing it on cooling in the crystalline form.

On heating a mixture of para-rubber and sulphur under the microscope the sulphur melts and gradually disappears. Subsequently small bubbles of a clear yellow colour form, which become opaque after a few hours and from which a mass of fine sulphur crystals penetrates into the india-rubber. On continued heating, the colour of the india-rubber darkens considerably, but remains transparent, and if the heating be continued long enough all free sulphur will permanently disappear.—C. O. W.

India-Rubber Waste. G. Springer. Gummi-Zeit. **15**, [21], 346.

THE employment of old india-rubber scrap, or waste, has assumed considerable dimensions since more or less satisfactory processes have been devised for converting this waste into a form in which it readily lends itself to re-incorporation with rubber mixings. The india-rubber waste is now collected and sorted according to its quality, in a systematic manner. The best waste consists of articles in the manufacture of which only rubber and sulphur have been used; this quality is therefore known as "best floating waste." The lower qualities consist, of course, of more or less heavily compounded goods. In one class are grouped the cuttings of waterproof cloth, bed sheeting, and the like, the rubber of which can bere covered by special processes now extensively carried out. "Hard rubber waste" is at present much sought after. When reduced to the state of an impalpable powder it can be added to hard rubber mixings.

In converting soft waste into a product suitable for re-introduction into rubber mixings, the first operation consists in freeing it from adhering dirt. It is then ground, and for about 20 hours boiled with caustic soda at a pressure of 60 lb. This removes the free sulphur. In spite of innumerable attempts, no process is known for the removal of the combined sulphur. After treatment with caustic soda, the waste is washed and dried in vacuo. The dry mass is then mixed with from 5 to 10 per cent. of benzene, or mineral oil, and heated for a few hours under hydraulic pressure, by means of 60 lb. steam. The product thus obtained is rolled into slabs, and is then ready for use in rubber mixings.—C. O. W.

Magnesia; The Employment of ——, in India-Rubber. G. Springer. Gummi-Zeit. **15**, [17], 281.

THE employment of magnesia in conjunction with india-rubber is not very extensive, its basic character making it suitable rather as a substitute for lime than for general use as a filling material. Lime is very generally considered a desirable addition to rubber mixings as a means of neutralising any free acids present, and as an adjunct to vulcanisation. But various investigations have shown that the addition of lime is conducive to the evolution of sulphuretted hydrogen, and there is besides the drawback of its being a heavy and more or less gritty powder, difficult to distribute evenly throughout the mixing. In all these respects magnesia, which forms an extremely light and impalpable powder, is much to be preferred. More important perhaps is the employment of magnesia in place of French chalk to counteract the stickiness of unvulcanised india-rubber. French chalk acts perfectly satisfactorily for this purpose in the cold, but is not capable of preventing adhesion when the india-rubber comes into contact with hot surfaces. In this case dusting the surfaces with magnesia is thoroughly effective, and this substance is therefore much used in the manufacture of rubber shoes and goloshes. Magnesium carbonate is capable of much more general application. It is employed not so much as a pigment as a filling material of special value. It has been found that the substitution of magnesium carbonate for chalk ("whiting") in "steam goods" tends to prolong the life of these articles.—C. O. W.

PATENT.

Electric Cables, Conductors, or Wires; Method of and Material for Covering [with India-Rubber] —— with Insulating Material. G. E. Heyl-Dia, Manchester. Eng. Pat. 11,217, June 20, 1900.

THE object of this invention is to cover cables with pure and with vulcanised rubber in one operation. Pure (*i.e.* non-vulcanised) and vulcanised rubber are each calendered separately into sheets; then one of each, or several of each arranged alternately, is passed together between warm rollers so as to make them adhere, yielding a composite sheet with pure rubber on one side and vulcanised rubber on the other. The whole is then cut into strips and applied to the cable by means of grooved pressure rollers, or in any other fashion, the pure rubber being next the conductor. Any rubber substitute capable of being vulcanised may be employed instead of the vulcanised rubber. The final heating to complete vulcanisation is preferably not carried out until the material has been applied to the conductor.

Eng. Pats. 8289 and 8290, 1900, are referred to.—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Sheep Skins with Wool on; Tanning ——. Leather Manufacturer, 1901, **12**, [3], 49—50.

THE skins after being thoroughly soaked in cold water and fleshed, are stretched on a table flesh side down, and the wool thoroughly washed with a strong solution of soap, to which has been added some sodium carbonate ("soda"), until the wool is perfectly free from grease, when it is thoroughly rinsed with warm water. The goods are now tawed by immersion in a solution of alum and salt for two or three days, the strength of the liquor being increased each day. A stock solution of alum and salt is made by dissolving 15 kilos. of alum and 9 kilos. of salt in 250 litres of water; for the bath, 25 kilos. of this solution are taken for each 375 litres of water used, and 5 kilos. of solution are added for each lot of skins treated.

After tawing, the goods are laid over a beam and allowed to drip until sufficiently dry, when they are brushed over lightly on the flesh side with the fat liquor made of three parts of dissolved soap and 15 parts of bone grease; if the skins are to be dyed, this operation must follow the dyeing.

The goods may be dyed with aniline dyestuffs, from 6 to 12 skins being operated upon at one time in a tub, keeping the goods in constant motion; when sufficiently dyed, the goods are rinsed in water containing a few drops of sulphuric acid. A seal brown may be dyed by alternately passing the skins through a Gambier solution and a solution of potassium bichromate "of 7° strength"; if desired white, the skins are bleached with sulphur dioxide gas.

—M. C. L.

Glacé Leather from Intestines. Leather Manufacturer 1901, **12**, [3], 53.

THIS is a process of producing glacé leather from the outer membrane of the intestines of animals, especially from the coecum or blind gut of cattle. The membrane may be tawed in a mixture of 500 grms. of wheat flour, 125 grms. of alum, 65 grms. of common salt, six egg yolks, and 2½ litres of water, for 2½ hours; the quantity being sufficient for 100 membranes. After removal from the tawing mixture, the goods are hung up to dry and when only slightly moist are worked over on the flesh side with a semi-circular blunt iron and are then dried, moistened, and worked for a second time with the iron tool.

A second method consists in treating the washed membranes with a 1 per cent. solution of hydrochloric acid until they are well plumped, when they are rinsed in a solution of soap and transferred to a solution of 40 grms. of alum and 40 grms. of salt, dissolved in 2 litres of water. The goods are transferred after a few hours soaking to a 3 per cent. solution of chromic acid which is gradually increased to 6 per cent. strength; the above quantitie being sufficient for 1,000 grms. of membranes. After absorbing sufficient chromic acid, they are placed in a mixture of ½ litre of egg yolk and 2 litres of water per 100 membranes, and left until the egg is well taken up; they are now stretched, dried, and glazed. If desired, zinc sulphate may be substituted for the chromic acid.

The membranes may also be tanned with ordinary vegetable tanning materials after being well cleansed in a sodium carbonate solution. They are afterwards subjected

to the action of one part of hydrogen peroxide solution dissolved in 10 parts of water and then washed. After tanning, the goods may be "nourished" with yolk of egg, and finished.—M. C. L.

Beam-House [Tanning] Experiment. Hide and Leather. 1901, **21**, [15], 28—29.

THIS experiment was made with the view of ascertaining what relation the amount of hide which enters the tanning liquor bears to that of the hide as bought. In carrying out the experiment no attempt was made at economic or improved methods, the hides being simply treated in the usual manner. Nine hides as nearly as possible alike in weight, spread, and texture, were divided into three packs and marked; No. 1 weighing 205 lb., No. 2 weighing 210 lb., and No. 3 weighing 197 lb. The total weight, 612 lb., of the wet hides consisted of 391·68 lb. of dry hide and 220·32 lb. of water. The soaking extended over a period of 72 hours, fresh water being used each 24 hours, the hides being drawn, put on a grating and drained, until all dripping had ceased, the drainage being conducted back to the soaking vat, the water well stirred and measured, and a large sample taken for the determination of the residue which it contained. This process was repeated on all of the three days during the soaking, and the results obtained were, 1st day, dry residue 118·324 lb. or 30·21 per cent.; 2nd day, dry residue 25·690 lb. or 6·56 per cent.; 3rd day, dry residue 5·280 lb. or 1·35 per cent.; thus showing a total loss of 149·294 lb. or 38·12 per cent.

On examination, it was found that the residue obtained on the first day was principally composed of manure, salt, and earthy matter; on the second day the insoluble and soluble matter were equally divided, whilst on the third day the residue was almost entirely soluble. After two days' soaking the hides were worked over on the beam and the fluid, dirt, &c. drained back into the vat and included in the residue from the second day's soaking; the shreds of flesh and fatty matter were carefully collected and dried, and on weighing gave 6·23 lb. or 1·59 per cent. A bacteriological examination of the soak water showed that seven different kinds of bacteria were present during all three days; two of these varieties were found capable of liquefying gelatin and exerted a destructive action on hide substance.

Liming.—Pure fresh lime was used, the amount varying for each pack; the goods were limed for five days, being drawn every 24 hours and allowed to drain into their respective vats; the quantity of water used in each case was 350 gallons. Pack No. 1 was placed in vat No. 1 which contained 51 lb. of lime, pack No. 2 into vat No. 2 containing 71 lb. of lime, and pack No. 3 into vat No. 3 containing 134 lb. of lime; a fresh quantity of lime was added each day so that when the liming was completed, the quantity equalled 50 per cent. of the original amount used; vat No. 1 containing 76·5 lb. of lime, vat No. 2, 105·5 lb. of lime, and vat No. 3, 201 lb. of lime. After completing the liming, the hides were drawn and drained thoroughly, the drainings being conducted back into the vats. An examination of the limes showed that in vat No. 1, pack No. 1, the loss in weight was 5·79 lb. or 4·4 per cent.; vat No. 2, pack No. 2, the loss was 5·16 lb. or 3·84 per cent.; vat No. 3, pack No. 3, 16·78 lb. or 13·31 per cent.

It will be seen that the minimum loss is in the lime of medium strength; however, when the hides came to be unhaired, it was found that pack No. 1 had not been sufficiently limed. Pack No. 2 was considered satisfactory, the hair being easily removed. Putrefactive germs were found in great numbers in vat. No. 1, whilst in vat No. 2, the number was somewhat diminished, especially after the third day; in vat No. 3 they were at no time so plentiful as in the others.

After liming, the hides were placed in a vat of water at a temperature of 85° F., and allowed to remain over night; the loss in hide substance during this operation being found to be 1·44 lb., or 0·37 per cent. They were then unhaired by hand, and the amount of hair removed was found after washing and drying, to weigh 28·04 lb., or 7·16 per cent. When unhaired, the hides were fleshed by hand, using the German spring flesher, and the fleshings washed and dried, when they were found to weigh 15·7 lb. or 4·01 per cent. The goods were now left over night in water at 60° F.; they were then stoned and worked out, and left for five hours in running water, when they were drained for 45 minutes, weighed, and samples taken for analysis. The net weight of the nine hides ready to tan was 654·84 lb., being 7 per cent. in excess of the original green salted weight; they contained 73·84 per cent. of water, and 1·8 per cent. of lime, which, on deduction, leaves 164·27 lb., or 24·36 per cent. of the weight of hide purchased, for tanning.

——	Per Cent.	Pounds.
Total green salted hide used for experiment..	..	612·00
Water in green salted hide	36·00	220·23
Total dry green salted hide.....	..	391·68
Loss in soaking	59·71	155·54
„ liming pack No. 1	4·4	5·79
„ „ No. 2	3·84	5·16
„ „ No 3	13·31	16·78
„ water before unhairing.....	0·37	1·44
„ unhairing	7·16	28·04
„ fleshing.....	4·01	15·70
Total Loss	..	228·45
Total hide ready to tan as shown by experiment.	..	163·23
Total hide ready to tan by actual weight before going to liquors.	..	164·27
Difference unaccounted for, 0·26 per cent. of dry hide.	..	1·04

The following will illustrate the true value of the weights and percentages in the foregoing:—

	Per Cent. Water.
Total dry green salted hide	36·00
After soaking.....	39·71
„ liming.....	43·55
„ unhairing.....	51·08
„ fleshing	55·09

—M. C. L.

Leather; "Spueing" of ——. Eitner. Der Gerber, through Leather Trades' Rev. **34**, [786], 275.

"SPUEING," or gumming, is the term used to denote a resinous exudation in which an oily substance collects in patches upon the surface of the leather, and when the process is prolonged, finally covers it with a thick viscous coat, which, as time goes on, becomes thicker and more sticky. This gummy or resinous exudation consists of the hydroxyfatty and polyhydroxyfatty acids of the fish oil, formed by the oxidation of the fatty acids of that oil. The oxidation is not always effected by the oxygen of the air, but appears to depend upon other causes, because the spueing in its worst form generally occurs in the warehouse, in the interior of bundles of skins, to which only a very small quantity of air can gain access.

It has been found by experience that all fish oils have not an equal power of becoming oxidised. Some varieties of fish oil are known to be characterised by this property in a high degree, such as shark oil, herring oil, and Japanese fish oil, whilst in others it would appear to depend upon the mode of manufacture, and upon the age of the oil. Oils of a low specific gravity may be said to oxidise more easily than those of a high specific gravity. Several cases of spueing have been investigated, and their cause attributed to the practice of converting lighter kinds of fish oil into more viscous and heavier kinds by the addition of a small percentage of wool fat or paraffin wax; also to the use of a substance sold as oxidised fish oil. Oxidised oils are more likely to spue than those which are not oxidised, which may perhaps be explained by the fact that more hydroxyfatty acids are formed, which under favourable circumstances are converted into polyhydroxyfatty acids, of which the exudation in great part consists. As many kinds of dégras sold in commerce consist to a greater or less extent of oxidised fish oil, it is very evident that leather stuffed with dégras, the artificial substitute for pure moëllon, may also be subject to the evil of spueing.—J. G. P.

Leather; Marbled ——. Hide and Leather, March 28, 1901.

A PROCESS recently patented by George Collin, of Berlin, for marbling or decorating leather by means of aniline dyestuffs, is carried out by moistening the surface of the tanned leather with water, the excess moisture being removed by rubbing with a dry cloth and those parts of the leather which are to appear bright are dotted, sprinkled or brushed with a solution made by dissolving 80 grms. of potash alum or 40 grms. of stannous chloride in 1 litre of water; the surface of the leather is allowed to dry, when it is covered for a few minutes with a dilute alkaline solution of soda or potash (about 10° Tw.), the surplus alkaline solution being removed by rubbing; the alum or stannous chloride solution is now again applied on the parts desired bright and the leather dried. The leather is now stained with a solution of the selected aniline colour when it will be found that the places mordanted with the alum or tin solution will not take the dye and therefore remain bright, by which means it is claimed, an effective marbling is produced. When using the acid dyestuffs, only the stannous chloride mordant must be employed in order to obtain good results.—M. C. L.

Leather; Manufacture of Patent Chrome Goat and Calf ——. Shoe and Leather Reporter. Feb. 28, 1901, 39.

Soaking Goat Skins.—The dry goat skins are placed in clean water for from four to five days, adding, if necessary, 5 lb. of borax to each 1,000 galls. of water; after which they are drummed with warm borax solution for about 20 minutes, afterwards being drummed in two changes of clean water for periods of 20 minutes.

Soaking Calf Skins.—Calf skins are soaked in clean water, two days being sufficient for fresh skins; salted skins require from three to four days.

Liming.—After soaking, the goods are weighed before liming; 1,200 goat skins or 500 calf skins are usually limed together in a vat. Five to seven days liming is generally given, experience having shown that skins intended for chrome leather should not be limed too long; the lime is slaked in the vat with an old lime liquor, together with $1\frac{1}{4}$ per cent. of "red arsenic," calculated on the weight of the dry skins; after slaking, the vat is filled with water, "plunged" well, and the goods thrown in; the limes are strengthened, and the goods hauled daily; when sufficiently limed the goods are ready to be unhaired. After unhairing and fleshing, the former operation being performed either by machine or hand as desired, the skins are well washed in a wash mill with cold water for 10 minutes, when they are allowed to drain and are ready to be bated.

Bateing.—The skins are bated in paddle wheels at a temperature of 75° F. for about $2\frac{1}{2}$ hours, using new manure. When sufficiently bated the skins are placed in a tub of warm water in order to open the grain and to permit of the removal of any remaining dirt in the slating operation, which follows next. After slating the skins either by machine or hand, they are paddled in a warm borax solution, using 4 lb. of borax to the pack of goods, in order to remove whatever lime may be left in the skin, after which they are drained and weighed.

Tanning.—The goods are now pickled by drumming in a solution made by dissolving 10 lb. of common salt in 15 galls. of water for every 100 lb. pelt weight of skins, for 20 minutes, when a quantity equal to 2 per cent. of hydrochloric acid, previously diluted with hot water, is added, and the drumming continued for another 15 minutes. The object of pickling the skins is to keep them open and plump, and to prevent them from drawing on the grain. The goods are now drummed for 30 minutes in a solution made by dissolving 2 lb. of potassium bichromate in 12 galls. of water for every 100 lb. of pelt, at a temperature of 70° F., when 4 lb. of potassium bichromate and $2\frac{1}{2}$ lb. of common salt dissolved in 15 galls. of water are added and the drum revolved for a further period of four hours or until the goods are coloured yellow through the thickest parts, when they are taken out of the drum, thrown over "horses," and left overnight covered with sacking, in order to keep off the light; next morning they are struck out by machine and are prepared for the reduction bath by dipping in a weak solution of sodium thiosulphate and acid, using $3\frac{1}{2}$ per cent. of sodium thiosulphate and 180 per cent. of the weight of the skins, of water. The skins are dipped well in this solution and thrown over a "horse," when they are ready for the reduction bath.

The reducing is done in the paddle, using 10 per cent. of thiosulphate of soda, and 5 per cent. of hydrochloric acid. The sodium thiosulphate after dissolving, is added together with sufficient water to the paddle, the acid is then added, the paddle set in motion and the goods thrown in as quickly as possible. The goods are paddled until no yellow colour is apparent, it being advisable to begin the reduction early in the morning and to paddle the skins all day; they are then left in the paddle overnight and run for one hour next day, when they are removed and paddled all day in running cold water, afterwards they are horsed up and left covered with sacks overnight, when they are ready for machine shaving. The goods are now drummed in a solution of borax for 20 minutes, using 10 lb. of borax dissolved in sufficient warm water, and are afterwards washed for 20 minutes in cold running water in the wash-wheel; three pints of sulphuric acid diluted with three pails of water is thrown into the wash wheel, with the plugs out, the goods are revolved two or three times, and are then washed for about 30 minutes in running water, when they are allowed to drain off.

The skins are now drummed for 30 minutes in a solution made by dissolving 3 oz. of Methyl Violet and 2 oz. of Nigrosine, in 30 galls. of warm logwood infusion at a temperature of 100° F. when a fat liquor, made by dissolving 10 lb. of a suitable soap in 15 galls. of boiling water to which 2 galls. of best fish oil, emulsified by boiling with a solution of 2 oz. of borax in water is added; sufficient cold water is now added to make up the solution to 45 galls., 10 lb. of egg yolk are added and the whole thoroughly mixed. The fat liquor is added to the drum at a temperature of 95° F. and the drumming continued 20 minutes, when part of the solution is run off and the following mixture added:—A mixture of 8 lb. of ferrous sulphate dissolved in sufficient water and 25 galls. of logwood infusion in which has been dissolved 5 lb. of gum arabic, is diluted to $2\frac{1}{2}$° Tw. $2\frac{1}{2}$ pailsful of this mixture are entered in the drum, which is revolved for two minutes, when the mixture is run off, the skins washed for five minutes in cold running water and then allowed to drain over horses for four hours, after which they are set out on the grain by machine, being afterwards given a light coat of a solution composed of two parts of glycerin and one part of water, on the grain side, and a slight coat of oil, preferably sperm or cod oil.

The goods are now tacked upon frames or boards, and dried, and then laid in piles till they are wanted for finishing. When required, the goods are staked, after damping in wet sawdust, for four or five hours, then fluffed on the flesh side, and the grain cleaned by brushing by hand, any grease being removed by rubbing a little "benzine" on the grain.

The goods are now fastened to frames, by means of string attached at distances of about 1 ft. all round the skin, and the first coat of varnish is applied; this varnish, or "daub" consists of 15 galls. of linseed oil heated up to 570° F. for eight or ten hours, allowed to cool to 400° F. The mixture is afterwards allowed to stand overnight, when it is again heated up to 600° F., and should then have acquired the consistency of a jelly; $\frac{1}{4}$ lb. of burnt umber, 4 lb. of litharge, and 1 oz. of sulphuric acid are now added, the mixture cooled down to a moderately warm temperature, 15 galls. of "benzine" added and the whole stirred. 10 galls. of benzine are now further added and the mixture again stirred; the quicker the benzine is added—the composition being stirred, the better. Two coats of this mixture are given to the grain surface of the skins, using a wooden slicker and working the mixture thoroughly into the leather, and drying thoroughly before applying the second coat. The skins are then dried in an oven for about 12 hours at a temperature of 120° F., rubbed over on the grain with a piece of pumice stone, in order to obtain a smooth surface and are given a coat of varnish. This varnish is made by boiling 20 galls. of raw linseed oil for eight to ten hours at a temperature of 550° F. to 580° F., adding 5 lb. of Chinese

blue after the oil has been boiling for two hours. The varnish is, after sufficient boiling, cooled, and 3 lb. to 4 lb. of lampblack, previously mixed with a little turpentine, are added, and the mixture diluted with benzine until it flows freely. This mixture is placed on the leather with a brush, and the skins dried for 12 hours in the oven at a temperature of 160° F., or until thoroughly dry, when the goods are again pumiced and a finishing varnish is applied; this is made by boiling 20 galls. of linseed oil until it is of the same consistency as the varnish just described; 5 lb. of Chinese blue are added after two hours' boiling, the composition being kept for 10 hours at a temperature of 650° F., 2 oz. of sulphuric acid being added during the operation; the varnish is cooled down overnight, and is ready to use when cool enough to bear the hand in it; the skins receive two coats of this varnish, applied with a brush, the goods being dried between the coats, and pumiced with a very fine pumice stone; the finishing coat is allowed to dry for two days in the oven; when dry, the goods are exposed to the sunshine for one day, and are then ready for market.

—M. C. L.

Picric Acid; Tanning with ——. F. Watenburger. Leather Manufacturer, 1901, **12**, [3], 54.

After tanning with a picric acid solution, the goods are allowed to drain, and are then transferred to a tanning extract solution of about 1° strength for about 12 hours, when they are removed, washed, partially dried, and fat-liquored, and are then dried and finished in the usual manner. It is claimed that the vegetable tanning material fixes the picric acid in the leather in an insoluble form which will not "rub off."—M. C. L.

Hide-Powder; Report on the Effect of Moisture and Time on the Quality of ——. A. Turnbull. Paper read before the Paris Conference of the I.A.L.T.C., 1900. Leather Trades' Rev. **34**, [786], 279.

In order to find out the effect of different amounts of moisture in hide-powder with regard to its keeping properties for purposes of analysis, a quantity of hide-powder was obtained and divided into four portions:—

Lot 1.—This portion was allowed to absorb moisture until it increased 2 per cent. in weight, and was then subdivided into five portions, which were placed in well-stoppered glass bottles, and the latter sealed with paraffin, and marked, respectively, "Lot 1*a*," "Lot 1*b*," "Lot 1*c*," "Lot 1*d*," "Lot 1*e*."

Lot 2.—This portion of the original hide-powder just as received was, as in the case of Lot 1, sealed up in five bottles, and labelled "Lot 2*a*, *b*, *c*, *d*, *e*."

Lot 3.—This portion was dried by means of a current of warm air until it had lost 2 per cent. in weight, and was then subdivided as before, and the separate bottles marked, respectively, "Lot 3*a*, *b*, *c*, *d*, *e*."

Lot 4.—The final portion was dried at 50° C. to constant weight, and was then apportioned in sealed bottles as before, and labelled "Lot 4*a*, *b*, *c*, *d*, *e*." The estimations of moisture gave the following results:—

——	(*a*.) January 23, 1900.	(*b*.) April 23, 1900.
	Per Cent.	Per Cent.
Lot 1	17·3	17·5
„ 2	15·5	15·3
„ 3	12·9	13·3
„ 4	9·8	9·5

For the object of testing these samples, an oakwood extract was used, each analysis being conducted on exactly the same lines. The following tables express the results obtained:—

Hide-powder: Oakwood Extract.

——	Lot 1.—17·3 moist.		Lot 2.—15·5 moist.		Lot 3.—12·9 moist.		Lot 4.—9·8 moist.	
	January.	April.	January.	April.	January.	April.	January.	April.
Tannins	26·4	26·5	26·4	26·4	26·4	26·7	26·3	26·
Non-tannins	13·6	13·7	13·6	13·8	13·6	13·5	13·7	13·
Insoluble	0·4	0·4	0·4	0·4	0·4	0·4	0·4	0·4
Water	59·6	59·4	39·6	59·4	59·6	59·4	59·6	59·4

Hide-powder: Solid Quebracho Extract.

——	Lot 1.—17·3 moist.		Lot 2.—15·5 moist.		Lot 3.—12·9 moist.		Lot 4.—9·8 moist.	
	March.	June.	March.	June.	March.	June.	March.	June.
Tannins	58·3	57·9	58·8	58·6	58·3	58·4	58·5	58·7
Non-tannins	11·2	11·6	10·7	10·9	11·8	11·1	11·0	11·8
Insoluble	12·1	12·0	12·1	12·0	12·1	12·0	12·1	12·0
Water	18·4	18·5	18·4	18·5	18·4	18·5	18·4	18·5

A blank test with water was made on the hide-powder, with the following results:—

Soluble in Water.

——	April.	June.	April.	June.	April.	June.	April.	June.
25 of first 30 c.c.	0·0113	0·0186	0·0154	0·0205	0·0196	0·0184	0·0209	0·02
25 of second 30 c.c. ...	0·0050	0·0030	0·0034	0·0022	0·0015	..	0·0010	0·00
25 of third 30 c.c.	0·0027	0·0028	0·0016	..	0·0016	0·0008	0·0008	0·00

It will be observed that the amount soluble in the first 30 c.c. is greater in June than in April, whereas in the succeeding 60 c.c. which would affect an analysis, the water-soluble portion in the first 30 c.c. increases, and in the succeeding 60 c.c. slightly decreases.—J. G. P.

Lactic Acid in the Tanning Industry. Leather Trades' Rev. **34**, [786], 278.

Lactic acid is used successfully for de-liming hides and skins. Half a kilo. of lactic acid is mixed with 100 litres of water. The skins are thrown into the bath, and allowed

to soak about a quarter of an hour, and then stirred for 15 to 20 minutes, and the process repeated. If used simply as a de-liming agent, the bath should be kept at a temperature of from 33° to 38° C., but if for swelling stock, the temperature must not be above 16° C.—J. G. P.

Tannin Estimation; Comparison of Volumetric Methods with the Results of the "Hide Powder" Method. A. Turnbull.

See under XXIII., *page* 623.

PATENTS.

Leather, Artificial; Manufacture of ——. C. Bauer, Imrie, and Co., London. From L. Gevaert-Naert, Bevere Audenarde, Belgium. Eng. Pat. 17,253, Sept. 28, 1900.

A leather-like substance is obtained by impregnating dressed, undressed, or felted fabrics with cellulose xanthate in solution, and then fixing the cellulose by means of steam or other recognised agents for decomposing this compound. Cellulose, dissolved in zinc chloride or in cuprammonium chloride may also be used. A second treatment with solutions of india-rubber or gutta-percha, dammar or other gums follows, and is effected either *in vacuo* or at the ordinary pressure. Either process may be omitted or repeated as often as desired, the final product being rolled or finished in various ways.—R. L. J.

Caseïn Glue; Composition of ——. R. W. James, London. From W. A. Hall, New York. Eng. Pat. 6815, April 1, 1901.

HITHERTO, although caseïn has been frequently used in place of glue, it has been found more suitable for sizing purposes than for joiners' work, as its compositions did not penetrate properly into the materials to which they were applied. According to the present process it is possible to prepare a caseïn glue free from this defect; it is strong, practically water-resisting, and particularly adapted for fixing veneers or for boat building. The new caseïn glue is made by mixing dry caseïn (preferably rennet-precipitated), 50 to 60 parts; sodium phosphate, about 20 parts; sodium sulphite, about 10 parts; and dry lime, either slaked or unslaked, 20 to 30 parts. This material dissolves in hot "or cold" water as ordinary animal glue does.—F. H. L.

XV.—MANURES, Etc.

Nitrification; The Organisms of ——. A. Stutzer. Centralbl. Bakt. 1901, [2], **7**, 168.

THE organism which produces nitrates was obtained from garden soil, and cultivated in a solution containing sodium nitrite (2), potassium biphosphate ("superphosphate") KH_2PO_4,H_2O (1), magnesium sulphate (0·3), sodium chloride (0·5), and potassium carbonate (0·5 grm. per litre). The cultivation is kept for some weeks, further amounts of nitrite being added as required, after which the nitrate organism is isolated on plates of agar. In a few more weeks, when there is no longer any nitrite reaction, the smallest colonies are selected. The organism is not coloured by aniline dyestuffs in aqueous solutions; Carbol-magenta is very suitable. The organism does not oxidise ammonia, but just manages to live when nitrogen is supplied in the form of nitrate. The usual organic nutrients are, as a rule, either poisonous, or hinder the oxidation of nitrites; some, as for instance mannitol, are indifferent. The reaction of the nutritive matter, the carbon dioxide, and the temperature are of importance. Only one variety of the nitro-microbe seems to exist.

In cultivating the nitrous organism, ammonium magnesium phosphate was employed instead of a soluble ammonium salt. The best dye for the nitrous organism is Carbol-magenta. The organism behaves towards organic nutrients similarly to the nitric organism. Several varieties seem to exist, differing both morphologically and physiologically.—N. H. J. M.

"Alinit." Biedermann's Centralbl. 1901, **30**, [4], 240—243.

ACCORDING to Stutzer and Hartleb, Stoklasa, and Lauck, Caron's "alinit" bacillus belongs to the group of hay bacilli; Stoklasa maintains that the microbe is *Bac. megatherium*, whilst Lauck believes it to be *Bac. subtilis*. Stutzer and Hartleb state, that it closely resembles *Bac. megatherium* and *B. mycoïdes*. Gerlach and Lüders have recently examined pure cultivations from alinit, and find that the bacillus behaves similarly to *Bac. subtilis*.

In 1898 and 1899, Gerlach made some vegetation experiments, in which alinit was employed both alone and in conjunction with glucose, straw, and glycerin. Inoculation was, in each case, without effect. Similar results have been obtained by Gyárfás (Wiener Landw. Zeit. 1900, **50**, 476); the simultaneous application of alinit and bone-meal also gave negative results.

—N. H. J. M.

XVI.—SUGAR, STARCH, GUM, Etc.

Glucose and Galactose; New Derivatives of ——. W. Koenigs and E. Knorr.

See under XXIV., *page* 626.

Sugars; Influence of Salts on the Rotatory Power of ——. J. de Kowalski and P. Tomartschenks.

See under XXIII., *page* 623.

Gum Arabic; Valuation of ——. O. Fromm.

See under XXIII., *page* 624.

Griess' γ-Diamidobenzoic Acid: A Reagent with its Compounds for Identifying Sugars, &c. B. Schilling.

See under XXIII., *page* 621.

Starch in Potatoes; Baumart and Bode's Method of Determining ——. P. Behrend and H. Wolfs.

See under XXIII., *page* 623.

PATENTS.

Steam or Vapour of any desired Pressure from Liquids Heated under Pressure but not Boiling; Process and Apparatus for Producing ——, *and the Application of the Process for Concentration of the Liquids.* C. Steffen. Eng. Pat. 12,805, 1900.

See under I., *page* 562.

Molasses Fodder; Apparatus for Making ——. R. Schrader. Eng. Pat. 3590, 1901.

See under XVIII. A., *page* 601.

XVII.—BREWING, WINES, SPIRITS, Etc.

Yeast; Antiseptic and Aseptic Action of Various Substances upon ——. Wehmer. Zeits. f. Spiritusind. 1901, **24**, [14], 137—138; [15], 147; and [16], 158—159.

THE effective dose of an antiseptic or germicidal agent varies within wide limits, even with one and the same micro-organism, depending, apart from concentration of the poison, on other factors which are often not sufficiently taken into account, such as vitality of the organism; conditions of nutrition and reproduction in respect to nature and reaction of the culture medium; temperature, age of the cultivation, time during which the reagent is allowed to act, number of cells employed in seeding, &c. Especially in the case of yeast is the available information vague and discordant; and in the present communication the author has collected and reviews some of the published investigations concerning the action of antiseptics on yeast, with the object of obtaining a comprehensive and comparative view of the effect of a series of substances, from a quantitative standpoint. The values in the following table may be

regarded as *approximately* accurate under *average* conditions. By "inhibitive value" is meant the maximum dilution at which a given reagent completely suspends the functions (reproductive and fermentative) of yeast, without, of necessity, killing the cells.

Substance.	Inhibitive Value		Lethal Dose.
	(*a*) for Reproduction.	(*b*) for Fermentation.	
Alcohol.....	10—20	5—10	.. ..
Citric acid.....	under 100	under 7	.. ..
Malic acid.....	„	„	.. ..
Lactic acid.....	50 (?)	12—15	.. ..
Sodium chloride.	10 (?)	10 (?)	.. ..
Succinic acid....	under 100	11	.. ..
Tartaric acid....	„	12	.. ..
Arsenious acid (alkaline salt).	over 130	10	1 per cent. within 20 days.
Acetic acid.....	..	70—140	..
Propionic acid...	..	„	..
Butyric acid.....	over 200	„	½ to 1 per cent. within 50 days.
Formic acid.....	..	200	..
Chloroform.....	..	100	1 per cent. = 0 (?).
Salicylic acid....	1,000	400	0·2 per cent. within 30 days.
Benzoic acid.....	„	400	0·2 per cent. within 30 days.
Oxalic acid.....	„	300—400	..
Formaldehyde...	over 1,000	400 and over.	¼ per cent. within 3—6 days.
Sulphurous acid.	over 1,000 (?)	1,000 (?)	0·1·5 per cent. within 15 minutes.
Mercuric chloride	„	„	0·1 per cent. within 3 days.

—H. T. P.

Yeast Cells and Yeast Enzymes; Comparative Resistance towards Injurious Agents of ——. Th. Bokorny. Chem. Zeit. 1901, **25**, [34], 365—366.

Oxalic Acid is very injurious to vegetable protoplasm. Yeast cells are killed by a 0·1 per cent. solution in four days; the yeast enzymes are fairly resistant, the zymase is not destroyed in five days by a 0·1 per cent. solution.

Acetic Acid.—The resistance of wine yeasts towards acetic acid was studied by Lafar. Bokorny finds that the zymase is destroyed by 0·2 per cent., but not by 0·1 per cent., in 24 hours. A 1 per cent. solution has no effect upon invertase, but is somewhat injurious to maltase.

Sulphuric Acid.—After 16 hours' exposure to 0·5 per cent. sulphuric acid, the yeast cells are killed, but not the mould yeasts, a 0·1 per cent. solution is not fatal; the yeast enzymes are more resistant to the mineral acids than the cells.

Caustic Soda.—The yeast cells are more sensitive to soda than the enzymes. Yeast is killed by 16 hours' exposure to a 0·5 per cent. solution, but survives treatment with a 0·1 per cent. solution. Invertase is not permanently destroyed by a 0·5 per cent. solution after four days, and maltase survives an exposure of 24 hours; both enzymes are destroyed by 1 per cent. caustic soda. Zymase is not destroyed by 0·5 per cent. soda in 24 hours.

Formaldehyde.—A 0·1 per cent. solution kills yeast in 16 hours, the zymase is also destroyed. The maltase is considerably injured by a 0·1 per cent. solution in 24 hours, and destroyed by 1 per cent. Invertase is not destroyed by a 5 per cent. solution in 24 hours; at this concentration yeast is killed in half an hour.

Mercuric Chloride in 0·02 per cent. solution is fatal to yeast in 24 hours, the zymase is also destroyed, as well as the maltase; the invertase is destroyed by a 0·5 per cent. solution.

Phenol.—Solutions of 1 per cent. and 0·1 per cent. kill the yeast cells in 24 hours, the former also destroys the zymase, but the latter does not; the same is also true for the maltase. Invertase will resist a 1 per cent. solution of phenol for 24 hours.

Thymol in saturated solution (0·1 per cent.) kills yeast in 16 hours, as also does a saturated solution of turpentine (0·001 per cent). Zymase seems to be just as sensitive as yeast cells to these antiseptics, maltase is not much more resistant, but the invertase is scarcely affected.

Alcohol.—Yeast will resist 10 per cent. alcohol for more than a month; it is killed after three months in 30 per cent. alcohol. Even absolute alcohol does not destroy zymase in eight days, exposure to 10 per cent. alcohol for a month weakens it. Maltase is more sensitive towards alcohol than zymase, 10 per cent. destroys it in a few days; invertase resists absolute alcohol for a long time.

For demonstrating the activity of the enzymes apart from the life of the cells, a 0·5 per cent. solution of caustic soda and a 0·1 per cent. oxalic acid are convenient for zymase (in addition to the antiseptics recommended by Buchner); for invertase, any of the above solutions are applicable, whilst for maltase, a 0·5 per cent. solution of oxalic acid would appear to be most suitable.—J. F. B.

Zymase. E. Buchner. Woch. für Brau. 1901, **18**, 197—201.

Continuing his reply to the paper on zymase by Macfadyen, Morris, and Rowland (this Journal, 1900, 1127), the author deals separately with each of the main contentions of these authors, and, in the light of the additional experimental evidence now to hand, he sees no reason to modify any of his previous conclusions, and adheres to his opinion as to the inconclusiveness of the English experiments (this Journal, 1901, 55). The author has worked throughout with Berlin and Munich bottom fermentation yeasts, but does not think it likely that the fundamental differences found are explained by any specific difference in the English top fermentation yeast. The present experiments prove that the quantity of gas evolved from the expressed juice increases with increase in the quantity of sugar added, and that lower concentrations of sugar do not yield better results than stronger solutions. Further, it is shown that the addition of toluene as an antiseptic up to 1 per cent. has practically no retarding action, whilst thymol causes considerable retardation at this concentration. With regard to the "auto-fermentation" of the expressed extract, the results of the author distinctly contradict those of the English chemists. The author generally allows that the gas evolved by auto-fermentation may be 10 per cent. of that obtained by the fermentation of sugar; the highest quantity observed was 23 per cent., and this was demonstrably due to the presence of glycogen. For the purpose of studying the effect of dilution, which was the most important evidence educed in support of the view of the English chemists as to the protoplasmic nature of the cell-juice, Buchner made three series of experiments. The cell-juice, containing sugar, was diluted with a 40 per cent. sugar solution in one series, with a 10 per cent. sugar solution in the second, and with water in the third; the first method being the best in Buchner's opinion, and the other two being comparable with the English experiments. At dilutions with equal volumes the first series showed no decrease in the quantity of gas, the second showed a distinct increase, owing to the fact that the undiluted extract could have fermented more than the 10 per cent. of sugar added, and the third series showed a slight decrease (20—25 per cent.) owing to the solubility of the gas. With regard to the proportion of carbon dioxide to alcohol formed, the author shows that this is very near the theoretical in the case of the active extracts with which he was dealing. He agrees that the sugar consumed is greater in quantity than the products of fermentation, and suggests the possibility of the presence of a synthesising enzyme.

The author is inclined to doubt the desirability of the important modifications which the English chemists have introduced in the method of preparing the juice.—J. F. B.

Catalase [*Tobacco Fermentation, Curing, &c.*]; *New Enzyme in Cured Tobacco and of General Occurrence.* O. Loew. U.S. Dept. of Agric., Report No. 68, 1901.

In the course of his studies on tobacco fermentation, the author encountered two kinds of oxidising enzymes, oxydase proper and peroxydase (see this Journal, 1899, 1161; compare also Grüss, this Journal, 1899, 1042). He has now discovered the universal occurrence of another enzyme

which gives none of the guaiacum reactions of the oxydases and to which he gives the name of "catalase" from its specific property of liberating oxygen from hydrogen peroxide. It was shown (*loc. cit.*) that the processes of curing and sweating of tobacco are accompanied by a destruction of the enzymes of the leaves; the oxydase and peroxydase generally survive the curing process, the former of these finally disappears in the sweating, leaving the more resistant peroxydase; this latter may persist for two years longer, and then only the catalase survives.

In spite of difficulties arising from its universal occurrence in living tissues, the author has been able to prove that catalase is distinct from all the known enzymes, none of which, when pure, have the power of "catalysing" hydrogen peroxide. Catalase occurs in two forms, α-catalase, which is insoluble in water, and which appears to be a compound of the soluble catalase with a nucleo-proteid, and β-catalase, which is soluble, and is of an albumose nature. α-Catalase is converted into the soluble β-catalase under the influence of very dilute alkaline media at moderately high temperatures, as for instance in the sweating process of tobacco manufacture, α-catalase is soluble in dilute alkalis (0·2 per cent. NaOH), and is re-precipitated by acetic acid. α-Catalase is best prepared from cured or sweated tobacco by washing out the soluble enzymes with water, and subsequently extracting with dilute alkali; the extraction, however, is not very efficient. It is difficult to remove all the peroxydase if green leaves be employed. β-Catalase is prepared by exhausting sweated tobacco, free from peroxydase, with water and salting out the enzyme with ammonium sulphate. Since the total catalytic power of tobacco increases during the sweating process, the existence of a zymogen may be inferred. Hydrogen peroxide has a destructive action upon catalase and the volume of gas liberated decreases somewhat rapidly. The catalases resist the effects of time for several years, they are destroyed by heating at a temperature of 71°—75° C. in water, but withstand higher temperatures in the dry state. The quantity of oxygen developed from hydrogen peroxide by catalase is increased by agitation. The presence of neutral salts as a rule has no effect upon the activity, but the presence of nitrates retards it. The presence of dilute acids retards the action, whilst dilute alkalis favour it; more than 0·5 per cent. of acid rapidly destroys the activity of catalase. Mercuric chloride (0·1 per cent. solution) is most injurious. β-Catalase is soluble to a considerable extent in 50 per cent. alcohol; absolute alcohol does not injure catalase in the cold, and boiling alcohol does not destroy it immediately. Baryta water and dilute hydrocyanic acid attack β-catalase, but have comparatively little effect upon α-catalase. Formaldehyde and free nitrous acid are most injurious.

No living plant or vegetable organ is free from catalase, although the relative proportions of the α and β forms vary considerably. In an elaborate series of tables the author shows the quantity of oxygen evolved from hydrogen peroxide by various leaves, seeds, and fruits under comparative conditions. β-Catalase is generally absent from green leaves, but is often predominant in seeds.

The flesh of fruit of an acid nature is poor in catalase, though slightly better results are obtained after neutralisation. Fatty seeds generally contain more catalase than starchy seeds. In the case of barley the total catalase is considerably increased after germination. The amount of catalase secreted by fungi is very considerable; there is also a large quantity in yeast and bacteria; the α-catalase is in greater proportion than the β. Catalase is also of general occurrence in the animal kingdom. The author discusses the question whether catalase is to be regarded as an oxidising enzyme and concludes that it may be classed as such, although several of the oxidising reactions, notably the guaiacum reaction, are absent. A characteristic oxidation brought about by catalase is the formation of quinone from hydroquinone. He then deals with the physiological importance of catalase. Its function must be to destroy hydrogen peroxide, but the formation of this body in the respiratory processes of living cells has not yet been proved; any explanation must account for the universal distribution of the enzyme. The recent researches of Bamberger and of Engler on the rendering active of oxygen in processes of autoxidation, make it very probable that hydrogen peroxide may be produced during respiration, and, since this body is a powerful protoplasmic poison, the presence of catalase would protect the tissues from destruction. The utility of catalase in the case of anaërobes like yeast, is still less clear, but it may have the effect of facilitating the breaking up of the sugar.—J. F. B.

Raisin Wines; Composition and Examination of ——. Aug. Schneegans. Archiv. der Pharm. **239**, 91—95.

The author has examined a number of raisin wines, made from different fruits, such as currants and sultana raisins. The wines were clear, and varied in tint from light yellow to pink. They had a pleasant taste but no bouquet. This is due to the previous drying of the fruit, which upon fermentation will not then yield the higher alcohols and acids which produce the bouquet by esterification. These wines are of much more value from a medicinal point of view than has hitherto been supposed, and, according to the author, should be recommended in preference to the heavier wines. In the following table, numbers 1 to 5, are wines from different varieties of currants, and 6 and 7 are from mixed currants and sultana raisins, while 8 is from sultana raisins alone.

100 *c.c. of Wine at* 15° *C. contain, in Grms.*

No.	Alcohol. By Weight. Per Cent.	Alcohol. By Volume. Per Cent.	Extractive.	Inorganic Matter.	Free Acids.	Volatile Acids.	Non-Volatile Acids.	Free Tartaric Acid.	Tartaric Acid.	Glycerin.	Phosphoric Acid.	Sulphuric Acid.	Prepared from
1	5·89	7·42	2·21	0·247	0·64	0·12	0·49	..	0·42	0·70	0·019	0·020	Currants.
2	6·73	8·48	2·35	0·216	0·78	0·09	0·67	..	0·49	0·73	0·021	0·021	„
3	7·60	9·57	2·52	0·272	0·71	0 12	0·56	..	0·39	0·74	0·023	0·027	„
4	8·84	11·14	2·96	0·242	0·64	0·10	0·51	..	0·17	0·78	0·027	0·024	„
5	11·42	14·39	3·18	0·234	0·71	0·14	0·53	0·03	0·22	0·86	0·044	0·022	„
6	6·59	8·31	1·91	0·218	0·52	0·11	0·38	..	0·22	0·50	0·023	0·013	Currants and sultanas.
7	6·79	8·56	2·30	0·274	0·63	0·15	0·44	0·01	0·22	0·65	0·036	0·017	Currants and sultanas.
8	6·59	8·31	2·55	0·304	0·58	0·12	0·43	..	0·07	0·57	0·033	0·019	Sultanas.

No. 2 contained: Chlorine, 0·005; lime, 0·014; and Fe_2O_3, 0·002 per cent.

The proportion of alcohol to glycerin varied between 100:7·53 and 100:11·88, and averaged 100:9·33. The proportion of the extract to ash varied between 100:7·35 and 100:12·92, and averaged 100:10·21. Free tartaric acid was found in small quantities in two of the wines.

—J. O. B.

Sparkling Wines; Secondary Fermentation of ——. E. Manceau. Comptes Rend. **132**, [16], 1003—1006.

The sparkling wines of Champagne are made by bottling a mixture of old and new wines from the vats, containing so much sugar as will yield, by fermentation in the bottles, carbon dioxide in amount producing a pressure of 4 or 5 atmospheres at 0° C. For this purpose, different wines need different amounts of sugar, and the explanation of

this, hitherto received, rests on the supposition that different wines dissolve very different amounts of carbon dioxide. The author's experiments, still in progress, have shown that this is not the case, but that many factors determine the result. 1. The amount of yeast at the time of bottling has but little influence, but its nature and origin influence the extent of the fermentation very considerably. 2. The fermentation as a rule goes farther at 20° than at 10° C. 3. The residue of unfermented sugar is greater in a more alcoholic wine; while in wines containing about 6 grms. of glycerin per litre, the addition of more glycerin diminishes the residual sugar.

The nature and amount of the gases dissolved in the wine at the time of bottling, and the proportions of phosphoric acid, nitrogenous substances, and other constituents, all appear to influence the resulting fermentation. The author's further experiments will be directed towards elucidating their separate effects.—J. T. D.

Yeast; Nutrition of ——. Part III. A. L. Stern. Proc. Chem. Soc. 1901, **17**, [238], 126.

THE author has determined the effect of varying the concentration of the sugar, the temperature of fermentation, the amount of seed-yeast, and the time, on the nutrition of yeast.

The experiments were made in exactly the same manner as those previously described (Trans. 1899, **75**, 202); this Journal, 1899, 933; the sugar used was dextrose, the nitrogenous nutriment, asparagine; and the inorganic nutriment, potassium phosphate, magnesium sulphate and calcium sulphate; and the yeast a pure culture from a Burton pitching yeast.

The conclusions drawn are:—

(1.) Any increase of nitrogenous or inorganic nutriment beyond a definite limit will not increase either the amount of nitrogen assimilated by the yeast, or the weight of the yeast. This limit is but little greater than the largest amount which the yeast is able to assimilate under the conditions of the experiment.

(2.) Any increase of the sugar is accompanied by an increase of the weight of nitrogen assimilated and of the weight of the yeast. This increase goes on up to the strongest concentrations which can be completely fermented. The rate of increase is greatest at the lowest concentrations, and falls off gradually as the concentration rises.

(3.) Temperatures between 12° and 25° C. have but little influence on the weight of nitrogen assimilated and the weight of the yeast crop. At higher temperatures reproduction is weakened.

(4.) The weight of the nitrogen assimilated and of the yeast crop is composed of two quantities: the weight of the seeding *plus* a quantity dependent on the composition of the solution.

(5.) The growth of the yeast is, during a portion of the fermentation, proportional to the amount of sugar fermented, and proceeds as long as any sugar remains unfermented.

From a consideration of these deductions, and of the work of others, the author concludes that there is an essential difference between the functions of the inorganic and nitrogenous nutriment on the one hand, and of the sugar on the other; that the former supply only material to the yeast, whilst the latter supplies both material and energy. Whether fermentation is caused by an enzyme or not, the author does not yet consider definitely determined; whilst there is certain amount of evidence to support this supposition, there is none to negative it.

PATENTS.

Yeast and Alcohol from Molasses or Sugar Juices without Addition of Cereals and without Lactic Acidity; Process for Obtaining German ——. F. R. Bramsch. Teplitz, Bohemia. Eng. Pat. 8306, May 4, 1900.

MATERIAL rich in nitrogen, such as linseed meal, is first heated with an acid (*e.g.*, hydrochloric acid) for about three hours, and then subjected to a suitable pressure, and boiled at that for about two hours longer. The liquid is drawn off, neutralised, and filtered, and the filtrate is decolorised by treatment with bone black. The preliminary open boiling causes complete softening and better extraction of the nitrogenous matter. The decolorised extract, with about 20 per cent. dissolved solids, is added to the purified molasses or saccharine juices before fermentation.

—J. F. B.

Fermenting; Apparatus for ——; Collecting the Gas given off during the Process of Fermentation and afterwards Using the Same to Re-Carbonate Ales, Sugar, Beer and other Beverages. J. House, Cardiff, and E. W. Lancaster, London. Eng. Pat. 8125, May 2, 1900.

THE inventors claim "a closed fermenting vat, divided into two chambers by means of a horizontal partition, the upper and lower chambers communicating with each other by means of a vertical yeast pipe and a vertical wort pipe, the vat being also fitted with a suitable attemperator coil and a hop extractor, consisting of a close coil of D-shaped pipes or of two concentric cones having a spiral water way between them, an opening at the bottom of the vat to admit sterilised air, and outlets for the air, yeast, and gas given off during fermentation." A cooler or refrigerator is claimed, consisting of a vertical cylinder having a stack of tubes therein, through which the liquid to be cooled passes, the cooling agent circulating outside the tubes. A carbonator and filters for fermented liquids are also specified.

—J. F. B.

Fermented Beverages and the like; Process for Dealcoholising ——, and Apparatus therefor. A. Müller, Stuttgart, Germany. Eng. Pat. 22,592, Dec. 11, 1900.

A de-alcoholised beverage, charged with carbon dioxide, is made from beer or other fermented liquor, without deterioration of taste or colour, by conveying the beverage into a vacuum pan, and, after the air has been expelled by a neutral gas, such as carbon dioxide, boiling it at a low temperature, the gas being conducted through it until it is freed from alcohol, after which the de-alcoholised extracted solution is charged with carbon dioxide gas, air being excluded.

—J. F. B.

Crude Spirit; Purification of ——. W. P. Thompson, London. From J. H. Lavollay and G. Bourgoin, Paris. Eng. Pat. 9210, May 18, 1900.

THE process claimed consists in treating the crude spirits with an insoluble manganate of an alkaline earth, *e.g.*, calcium manganate, in the presence of an electric current. The impurities are oxidised by the manganate under the influence of the current, whilst the aldehydes are reduced to alcohol by the electrolytic hydrogen. The process is carried out in any ordinary electrolyser without porous partitions; the electrodes may be of zinc, and the current is passed for 10—20 minutes with constant agitation.

—J. F. B.

XVIII.—FOODS; SANITATION; WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Honey; Artificially Coloured ——. A. Bömer. Zeits. Untersuch. Nahr. Genussm. 1901, **4**, [8], 364—366.

ATTENTION is drawn, in this paper, to the fact that honey, and, more especially, adulterated honey, is sometimes artificially dyed. The added colouring matter may be detected by the usual tests, and by dyeing wool fibres with the aqueous extract of the honey.—W. P. S.

PATENTS.

Flour of Cereals, Pulse, and their Derivatives; Desiccation and Sterilisation of ——. E. C. A. Fleurent, Paris. Eng. Pat. 11,502, June 25, 1900.

THE process claimed consists in first partially or wholly desiccating the flour by evaporation under reduced pressure at a temperature of about 45° C. The dry flour is then sterilised by heating to 80°—120° C. The object of the desiccation is to prevent the coagulation of the gluten and the discoloration of the flour during sterilisation.

—W. P. S.

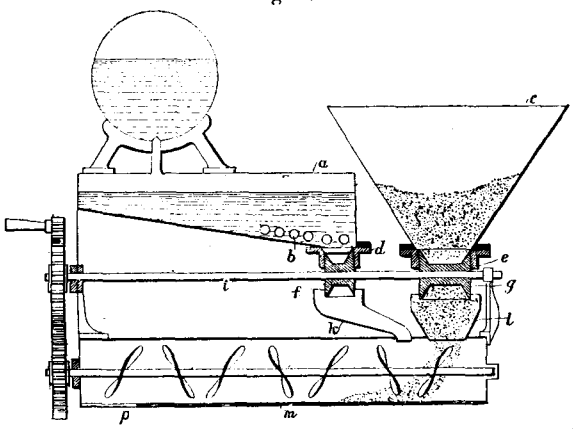
Milk; Process for Rendering Digestible Cow's and Goat's Milk. O. Imray, London. From Farbwerke vormals Meister, Lucius und Brüning, Hoechst a/Main, Germany. Eng. Pat. 12,231, July 6, 1900.

THE casein in the milk is first coagulated by the addition of rennet, and then broken up into fine flakes by shaking, beating, or otherwise mechanically agitating, the object being to prevent the formation of large clots of casein.—W. P. S.

Molasses Fodder; Apparatus for Making ——. R. Schrader, Hamburg. Eng. Pat. 3590, Feb. 19, 1901.

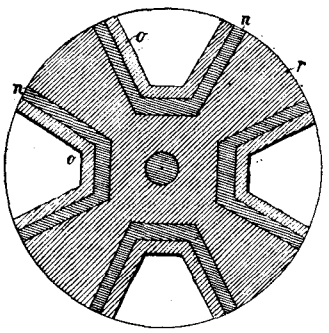
IN this apparatus, which is depicted in Fig. 1, the feeding of the molasses and the solid materials into the mixing

Fig. 1.



chamber is effected by means of rotating drums *f* and *g* (Fig. 1), fitted with chambers separated by wedge-shaped partitions. The drums are arranged above the mixing chamber, and are enclosed in casings attached to the storage vessels *a* and *c* in such a way that the latter are kept separated from the feed vessels until the contents of the previous chamber of the rotating drum have been discharged. The proportions between the molasses and the absorbent substances may be varied by the insertion of boxes *n* and *o* (Fig. 2), of a suitable number and size, into the chambers of either of the feed drums. The molasses pan is provided with a coil *b* for heating the molasses if necessary. The general arrangement of the apparatus will be seen from the figures.

Fig. 2.



—T. H. P.

Mother's Milk; A Substitute for ——. J. Meyenberg, Kent, Washington, U.S.A. Eng. Pat. 3750, Feb. 21, 1901.

THE process described in this specification is as follows:—Fresh milk is aërated to remove disagreeable flavours, and filtered through clean gravel. From one-half of this aërated and filtered milk the cream is removed, and from the skim-milk the casein is removed. The whey and cream thus obtained are added to the other half of the original milk together with the requisite amount of sugar. The product is then heated to boiling point in a closed vessel, evaporated under reduced pressure to the required consistency, and canned.—W. P. S.

Preserving Organic Bodies and Substances; Method and Apparatus for ——. A. Mészáros, Székesfejérvár, Hungary. Eng. Pat. 6371, March 26, 1901.

THE materials, such as meat, fruit, eggs, vegetables, corpses, &c., to be preserved are submitted, in closed receptacles, to the influence of the dry products of distillation of garlic or onions, which are previously passed through salt water and alcohol. The apparatus for carrying out the process is also claimed.—W. P. S.

(*B.*)—SANITATION; WATER PURIFICATION.

Drinking Water; Sterilisation of ——. A. Bergé. Mém. de la Soc. des Ing. Civ. de France, 1900, 601. Proc. Inst. Civil Eng. **143**, [1], 37—38.

THE author recommends the use of perchloric acid for the sterilisation of drinking water. A concentrated solution of perchloric acid is prepared by acting on potassium perchlorate with sulphuric acid (58° B.), and passing the gas evolved into water, by means of a current of air at constant pressure. One mgrm. of perchloric acid is sufficient to sterilise 1 litre of water, and the operation can be performed on any scale with equal facility. The French Board of Public Health has adopted the conclusions of various scientific authorities in support of the efficiency and innocuous character of the method. The process is being used, at the present time, at Lectoure (Gers, France) and at Ostend, whilst at Brussels, sewage water containing 1,000,000 bacteria per c.c. (16,000,000 per cub. in.) has been wholly cleared of germs by treatment with perchloric acid. The cost of the reagent should not exceed 0·26 centime per cub. metre (10*s.* per 1,000,000 galls.) of the water to be sterilised, even with bad water, whilst the charges for maintenance and redemption of plant would be extremely low.—A. S.

Waters; Determination of Nitrates in ——, *by Stannous Chloride.* H. Henriot.

See under XXIII., *page* 619.

PATENTS.

Filtering Media. C. Sharples, Stockport, Cheshire. Eng. Pat. 7383, April 21, 1900.

FIBROUS material, made up in the form of brushes, is used for the filtration of sewage effluent.—L. A.

Sewage and Sewage Effluents; Method of Treating —— R. H. Reeves, London. Eng. Pat. 9819, May 29, 1900.

THE sewage, before entering the precipitation tank, is mixed only with the chemicals required for deodorisation, the precipitant is sprayed on to the surface of the sewage in the tank, the effluent is then drawn off by a pump or other device and forced through a jet or nozzle, which converts it into fine spray or mist, and in this condition it falls on to a filter bed, or into a plain tank, according to the degree of purity required.—L. A.

Greasy Water; Purification of ——. E. G. Weddell, Tipton Green, Staffordshire. Eng. Pat. 7240, April 19, 1900.

THE greasy water first passes into a tank or chamber in which it is mixed with lime-water, and filtered to separate the precipitated calcium soaps. It then passes through a series of chambers, in which the excess of lime is removed, either by injecting fuel gas and filtering, or by mixing with hard water containing bicarbonates and filtering.—L. A.

Water; Apparatus for Softening and Purifying ——. W. Pemberton, Newcastle-on-Tyne. Eng. Pat. 7397, April 21, 1900.

THE apparatus comprises one or more tanks supplied with a suitable reagent, such as lime, a filtering chamber, preferably arranged under the tanks, and a valve mechanism controlling the supply to the filtering chamber of water from the main pipe mixed with a suitable proportion of water containing the reagent. The tanks are supplied through

branches from the main pipe, and the water containing the reagent is delivered from them through controlling cocks. This water and the water direct from the main pipe pass through separate valves into a mixing funnel, from which the mixed liquids pass through a pipe to the bottom of the filtering chamber. The two supply valves are operated automatically by a single lever connected to a float in the filtering chamber. The mixing funnel is provided with a perforated removable plate, which has depending tubular projections surrounding the perforations, for the purpose of better mixing the liquids.—R. A.

Water; Apparatus for Purifying and Softening ——. E. Froitzheim and M. M. Schumacher, both of Cologne, Germany. Eng. Pat. 1830, Jan. 26, 1901.

The water to be treated is fed into a pivoted two-chambered bucket, which as one chamber becomes filled, tilts and empties that chamber and brings the other chamber into position to be filled. The movement of the bucket acts automatically, through a pawl and ratchet mechanism, on a bucket conveyor, which raises proportionate quantities of a chemical solution (such as soda) from a reservoir and discharges it into the settling and filtering tank. The water, before entering this tank, may be heated by exhaust steam, &c. The tank is divided by a forked partition into three compartments, forming respectively the mixing, settling, and filtering chambers. Live steam, preferably mixed with air, may be supplied to the mixing chamber, to facilitate the mixing of the chemical solution with, and its action on, the water. From the mixing chamber the liquid descends to the settling chamber, and from the latter it rises to the filtering chamber, the settling chamber being provided with a cock for drawing off the deposited impurities. In the filtering chamber, a series of detachable filters are employed, each consisting of a perforated cylinder with a removable cover at one end, a perforated pipe, and means for connecting it to a common outlet pipe, the space between the cylinder and the perforated pipe being filled with a filtering material.—R. A.

Water and Aqueous Solutions; Processes of Eliminating Iron from ——. B. Teufer, Leipsic, Germany. Eng. Pat. 6121, March 23, 1901.

Hydrated ferric oxide, preferably freshly precipitated, is agitated with ferruginous water to remove the iron. The liquid may be filtered, or separated from the precipitate by decantation. The process is stated to be also applicable in removing iron from solutions of salts, such as "ferruginous solution of potash."—E. S.

XIX.—PAPER, PASTEBOARD, Etc.

Paper Sizing; Chemistry of ——. P. Friedlaender and H. Seidel. Mitth. tech. Gewerbe-Museums in Wien. 1901, **11**, 65—73.

The object of paper sizing is the diminution of the capillarity of the paper by cementing together the single fibres. After remarking that gelatin sizing is only used now for the best qualities of papers, the authors proceed to deal with the principles of the ordinary rosin sizing, which is almost universally employed. The raw materials are rosin, caustic soda or carbonate, aluminium sulphate, and starch. Rosin is a more or less variable mixture, containing abietic, sylvic, dextro- and lævo-pimaric acids; it becomes semi-fluid under boiling water and melts at about 135° C. The only factor which concerns the paper-maker is the saponification number (mgrms. of KHO required per 1 grm.) This varies with the different samples and is determined by boiling for half-an-hour with semi-normal alcoholic potash. The saponification number being known, it is an easy matter to prepare rosin soaps containing any desired percentage of free rosin. Neutral rosin soaps, fully saponified, dissolve to a clear solution in water, whilst, on the other hand, free rosin cannot be emulsified with pure water; the rosin size or "milk" is therefore a uniform and finely-divided emulsion of free rosin in a solution of neutral rosin soap. The proportion of free rosin employed varies greatly, according to requirements, but less than 25 per cent. of free rosin is never employed. The author gives the following example of the preparation of a rosin size:—300 kilos. of rosin are melted at 150°—160° C., 30 kilos. of caustic soda dissolved in 15—20 times the weight of water are then added and stirred up. The mixture is allowed to remain for 14 days, and a dark liquid which separates at the surface is drawn off. The soap is dissolved in 50 times its weight of hot water with the addition of 33 per cent. of starch. The size is added to the beater and a 5 per cent. solution of calcined sulphate of alumina is added to acid reaction. There has been considerable discussion as to the nature of the precipitate produced. Wurster argues that it consists of a mixture of free rosin and alumina, because it is largely soluble in ether. But, on the other hand, it has been shown that the resinates of copper, silver, and zinc are also soluble in alcohol and ether, and that the precipitate thrown down by alum is more finely divided and melts at a higher temperature than the free rosin precipitated by acid. The authors have made comparative precipitations with alum and acid and corroborate this latter point; they also show that the precipitate is almost completely soluble in ether-alcohol and that the filtered solution leaves an ash consisting of alumina, hence there is no doubt that the cementing precipitate is a resinate of aluminium.

In the second part of the paper the authors describe in detail the standard methods for the analysis of rosin soaps and sizes, and for the determination of free and combined rosin. The examination of glue sizes and of Mitscherlich's "Gerbleim," which is a mixture of caustic soda, glue, rosin, and sulphite wood liquors, is also dealt with.—J. F. B.

Cellulith. G. Springer. Gummi-Zeit. **15**, [20], 329.

Brunswig's cellulith is produced by grinding wood pulp in a paper "beater" until an apparently homogeneous pulp, free from every trace of wood fibre, is obtained. This pulp is then drained of the bulk of its moisture by allowing it to run into a vat provided with a bottom of metallic gauze, and is subsequently dried, either in the air or in rooms at a temperature of 40° C. The product thereby contracts very greatly, and forms, finally, solid masses of the hardness of horn, which are sold under the name of cellulith. Its specific gravity is about 1·5, it is not inflammable, can be worked with tools like wood or horn, and is very resistant to oils, fats, alcohol, and petroleum. It is used as a substitute for horn, ebonite, buffing and polishing wheels.—C. O. W.

"Celluvert." G. Springer. Gummi Zeit. **15**, [20], 329.

Celluvert consists of hydrocellulose charged with zinc chloride. In 1878 it was first used in America for the manufacture of waterproof cardboard. It is obtained by parchmentising paper by means of zinc chloride and uniting a number of the sheets so treated by pressing. More recently it has been displaced in the market by "vulcanised fibre." It used to be sold in hard, but somewhat pliable, slabs, either red or black in colour. It resisted the action of hot and cold water, also of fats and oils. It used to be employed in place of leather, india-rubber, ebonite, metal, horn, and celluloid.—C. O. W.

PATENTS.

Peat Fibre for Use in the Manufacture of Paper or Paper Pulp; Process for the Production of Purified and Bleached ——. Jensen and Son, London. From M. Krause, Berlin. Eng. Pat. 6831, April 11, 1900.

Peat is sludged in a hopper with water that has been aërated and rendered alkaline (*e.g.*, by passage over layers of coke and lime) and allowed to stand for two hours or longer, when the following changes are stated to occur. Humic and other acids are neutralised, organic compounds of iron, &c. are rapidly oxidised, and putrefactive bacteria promote an alkaline bleach. After draining, the mass is cut up finely and evenly in a mincer and made into paper stock, with or without additional treatment. Designs for a suitable plant are attached to the specification.—R. L. J.

Pulp Fibres from the Waste Waters of Paper Manufactories; Apparatus for Separating ——. W. P. Thompson, London. From E. Füllner, Warmbrunn, Silesia, Germany. Eng. Pat. 7842, April 27, 1900.

This device consists of a collector in the form of a hopper, having radially-arranged flanges or wings on the outside which strengthen the hopper and at the same time, by means of an outer ring of closing partitions or walls, made preferably of removable boards, form a number of deposition cells round the foot of the hopper, into which the fibres deposited from the waste liquor on the floor of the hopper are directed at will, according to their quality or colour. The waste liquor, with fibre in suspension, is led into the hopper from a bell-shaped mouthpiece situate half-way down the hopper, and with a coarse sieve plate in its neck; the fibres sink to the floor and pass, by wide pipes, to the cells, whilst the waste liquor wells up and overflows at the mouth of the hopper.—R. L. J.

Paper; Manufacture of ——. Johnsons and Willcox, London. From G. J. Wildridge, Airdrie, N.B. Eng. Pat. 9517, May 24, 1900.

Two layers of pulp, one of the composition for blotting paper and the other for sized paper, are caused to adhere to each other in the moist condition by pressure only, no gum or adhesive being used. One side of the composite web so obtained is perfectly absorbent, the other may be printed or written upon, and does not fray or break up as ordinary blotting paper does by the friction of the hand.
—R. L. J.

Paper temporarily Transparent; Process for and means of Rendering Non-transparent or Opaque ——. Cheesbrough Royston, Liverpool. From L. H. A. von Giese, Diedenhofen, Germany. Eng. Pat. 630, Jan. 10, 1901.

To make ordinary paper transparent for a time, so that tracings, &c., may be made in any desired portion, the part is painted with a volatile liquid, such as benzene, ether, methylated spirit, chloroform, or a solution of camphor (3 parts) in benzene (7 parts), all of which evaporate without injury to the surface.

A device suitable for the purpose consists of a small metallic drum with a flat tongue-like extension. A cotton wick is rolled up inside and just emerges from the chisel-edged aperture of the tongue. The drum is filled with the liquid through an air-tight cap at one end, and the saturated wick-end then stroked over the paper where desired. An improved form of the apparatus has a spring slide which closes over the end of the tongue to prevent evaporation of the liquid when not in use. The process and the device are claimed.—R. L. J.

Fibrous Compositions; Manufacture of ——. H. H. Lake, London. From The National Package Co., Glen Falls, New York State, U.S.A. Eng. Pat. 4125, Feb. 26, 1901.

This composition is prepared by mixing together in equal parts a fibrous material such as waste paper, corn stalks, bagasse, &c., or, if a more or less fireproof substance is required, asbestos or mica, plaster of Paris, and rosin. The product is submitted to hydraulic pressure at a temperature sufficient to melt the rosin, whereby a hard and tough material is formed, which can be used for constructing barrels or kegs that have to be exposed to hot water or to oil. Pigments may be incorporated into the composition, or may be dusted on to the dies of the press; and the finished article can be made at one operation by suitable arrangement of the hydraulic press.—F. H. L.

Celluloid-like Substance; Method of Producing ——. E. Zühl, Berlin. Eng. Pat. 4326, Feb. 28, 1901.

Phthalic acid and phthalonic acid ($HO_2C.C_6H_4.CO.CO_2H$), also their anhydrides and esters, easily dissolve nitro-cellulose, forming a celluloid-like substance. For instance, 100 kilos. of nitro-cellulose are mixed with a solution of 50 kilos. of phthalic acid or anhydride, and treated in the usual manner for the manufacture of celluloid. The claim suggests that the camphor usually employed may also be one of the constituents.—T. A. L.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

Acids; Action of ——, *upon the Carbonates of the Alkaline Earths in the Presence of Alcohol.* C. Vallée. Bull. Soc. Chim. 1901, **25**, [8], 393—395.

Pelouze found that acetic acid does not act upon carbonates in the presence of absolute alcohol. The author has now found that (1) acetone and methyl alcohol also prevent the action of acetic acid upon calcium carbonate. (2) The action of sulphuric acid in the presence of alcohol (10 c.c. of normal acid diluted to 100 c.c. with absolute alcohol) on excess of calcium carbonate is very slow, but is complete (in four months). The velocity of reaction increases with decrease in the strength of the alcohol, and the velocity decreases regularly as the reaction proceeds. Barium and strontium carbonates behave similarly. An increase in the quantity of acid, the strength of the alcohol remaining the same, accelerates the reaction. (3) Acetic acid (10 c.c. of normal acid diluted with absolute alcohol to 100 c.c.) does act upon calcium carbonate; the reaction is extremely slow (half complete in $3\frac{1}{2}$ months). The velocity decreases regularly as the reaction proceeds, and increases with decrease in the alcoholic strength. (4) Nitric acid, similarly diluted with alcohol, in the presence of strontium nitrate, is at once neutralised by strontium carbonate. With barium nitrate and carbonate several hours are required.—A. C. W.

Basic Bismuth Salicylate. C. Martinotti and L. Cornelio. Boll. Chim. Farm. **40**, 141—148; Chem. Centr. 1901, **1**, [17], 966.

Commercial preparations of bismuth salicylate differ considerably in composition, the proportion of salicylic acid varying between 5 and 67 per cent. and that of bismuth oxide between 37 and 79 per cent., and it has, consequently, not been deemed possible to attribute a definite chemical composition to these preparations. From the results of an investigation as to the constitution of normal and basic salicylates, and the behaviour of bismuth salicylates towards water, acids, &c., the authors conclude that commercial bismuth salicylates contain, mostly, only bismuth salicylate of the formula—

$$C_6H_4(OH)(COOBiO) \text{ or } C_6H_4(OH)[COOBi(OH)_2].$$

For the preparation of bismuth salicylate, the authors use a method based on the following reaction:—

$$3C_6H_4(OH)(COONa) + 2Bi(NO_3)_3.5H_2O = 2C_6H_4(OH)(COOBiO) + 3NaNO_3 + 3HNO_3 + 8H_2O + C_6H_4(OH)(COOH).$$

A solution of 1 kilo. of sodium salicylate in 10 kilos. of water at 50° C., is treated with 2 kilos. of normal bismuth nitrate moistened with about one-tenth of its weight of water, the mixture well shaken, diluted with about 25 litres of water at 50° C., agitated for 10 minutes, filtered, and the precipitate washed with 50—60 litres of water at 50° C., centrifugalised, and dried at 60° C. Bismuth salicylate prepared in this manner forms a light, amorphous, white, faintly acid powder, which, when freed from nitric acid, other salts, and as far as possible from free salicylic acid, should yield, on analysis, at least 60 per cent. of Bi_2O_3. Like other basic bismuth salts, the salicylate readily undergoes alteration by washing with water.—A. S.

Bismuth; Basic Nitrates of ——. F. B. Allan. Amer. Chem. J. **25**, [4], 307—315.

When poured into water at ordinary temperature, nitric acid solutions of bismuth furnish only $BiONO_3 + H_2O$, if the precipitate be quickly filtered off, the concentration of the acid being high at the point of contact, whilst the reaction on subsequent contact with the weaker acid is too slow to effect any change in a short time. At 21° C. the salt $Bi_{12}O_{13}(NO_3)_{10} + 9H_2O$ is in equilibrium with nitric acid solutions from 0·03 to 0·32 normal, and the salt $BiONO_3$ with solutions from 0·425 to 0·72 normal. At 50° C. the salt $Bi_4O_5(NO_3)_2 + H_2O$ is in equilibrium with the acid

solutions from 0.057 to 0·285 normal, and the salt $Bi_{12}O_{13}(NO_3)_{10} + 9H_2O$ with solutions from 0·285 to 0·466 normal. At 75° C. the salt $Bi_4O_5(NO_3)_2 + H_2O$ is in equilibrium with the acid solutions from 0·109 to 0·314 normal. No basic nitrate between $Bi_4O_5(NO_3)_2 + H_2O$ and $Bi_{12}O_{13}(NO_3)_{10} + 9H_2O$ exists at 50°.

The many basic nitrates that have been described are accounted for by the slowness of the reaction, or a concentration of the mother liquor at which a monovariant system was possible. It is also found that some time elapses before the commencement of a reaction between the higher basic nitrates and water containing nitric acid.—C. S.

Mercury Salicylate; Determination of Mercury in ——. E. Rupp. Archiv der Pharm. **239**, 114—118.

MERCURY salicylate is one of the recent additions included in the new *Pharmacopœia Germanica* (Editio IV.). The author criticises the method given for determining the mercury, and points out that varying results are obtained according to the length of time and temperature at which the H_2S is passed in. He advises instead a method which he claims to give consistent results and to be at the same time very simple. 0·3 grm. of the salicylate is rubbed down with a little water and 25 c.c. of N/10 iodine solution is run in. After standing for an hour in a well-stoppered flask, the excess of iodine is determined by titration with thiosulphate. Several determinations required 16·5—16·8 c.c. of N/10 iodine; practically no variation being noticed whether the action was allowed to continue for one hour or fifteen. Theoretically 0·3 grm. of the salicylate should require 17·85 c.c. of N/10 iodine solution; the low results obtained are due to the fact that mercuric salicylate reacts with haloid salts to form a double compound, but as the error due to this is a constant, the results are quite reliable. —J. O. B.

Methyl Alcohol; Pure ——. — Rotten. Rev. Prod. Chim. **3**, [23], 357.

METHYL alcohol may be entirely freed from acetone by exposing the nearly boiling liquid to the action of a current of chlorine gas, which is inert towards the alcohol but converts acetone into mono- or di-chloro compounds, which are afterwards separated by fractional distillation, the alcohol being finally freed from chlorine by the aid of lime.—C. S.

Formaldehyde. C. Harries. Ber. 1901, **34**, [4], 635—637.

KEKULÉ found (Ber. **25**, 2435) that when the vapour evolved on heating solid paraformaldehyde was condensed by means of solid carbon dioxide and ether, a liquid mono-molecular formaldehyde was obtained. The same substance is produced in almost quantitative yield if liquid air be used as the refrigerant. The paraformaldehyde is heated in a hard-glass test tube (a combustion tube in a furnace for larger quantities), from which the vapours are conducted by means of a wide tube into a U-tube, blown out at the bend, the lower half of which is placed in a dish containing about 300 grms. of liquid air. The paraformaldedyde is heated from the front backwards; the vapours condense to a white crystalline mass, which melts, on warming, to a turbid liquid. The turbidity is easily removed by filtering through a small folded filter. In attempts to dry the formaldehyde vapours by means of calcium chloride, the gas was partly absorbed and partly polymerised. In the receiver a little liquid collected, which soon polymerised; the vapours of the polymer detonated when it was heated in a test tube. The same explosive property was observed in a preparation of paraformaldehyde obtained from Kahlbaum several years previously; obviously it could not be used in the preparation of the liquid formaldehyde. Phosphorus pentoxide acts on the gas, causing the formation of carbon, perhaps according to the equation, $CH_2O + P_2O_5 = C + 2HPO_3$. The melting point of the solid mono-molecular formaldehyde was determined, by means of a toluene thermometer, to be about 92° C. The mono-molecular aldehyde in ethereal solution unites with aceto-acetic ester at —50° C., forming an oily compound, without separation of water. This substance cannot be distilled undecomposed; it is violently decomposed by caustic soda; on heating, it yields formaldehyde, water and high-boiling compounds.—A. C. W.

Heptane from Coniferous Trees. W. C. Blasdale. J. Amer. Chem. Soc. 1901, **23**, [3], 162—164.

T. E. THORPE (J. Chem. Soc. **35**, 297) found an insecticide sold as "Abietine" to consist of nearly pure normal heptane. This substance had previously been stated by Wenzell (Amer. J. Pharm. 4th Ser. **2**, 97) to be obtained by distilling an exudation of *Pinus Sabiniana*. These facts have led the author to examine the exudations from different species of conifers.

1. *Pinus Jeffreyi.*—The fresh material from recently cut trees was a clear white fluid, which rapidly became thick on standing, and lost its pleasant orange-like odour. On distillation it yielded about 3 per cent. of a mobile colourless liquid, which on purification distilled mainly between 96° and 98° C.

2. and 3. *Pinus Sabiniana.*—The exudations closely resembled that of *P. Jeffreyi* but were more viscous. They yielded about 16 per cent. of a mobile distillate.

4. *P. Murryana.*—The material was a light yellow semi-solid substance with an odour of turpentine. On distillation it yielded a liquid with a terpene odour, most of which boiled at 153°—160° C., though part boiled as high as 180° C. The fraction boiling at 158°—160° C. was employed for a series of determinations.

5. *Abies concolor var. Lowiana.*—This exudation closely resembled Canada balsam. It yielded 20 per cent. of a terpene-like liquid distilling, in the main, between 155° and 160° C.

6. *Pseudotsuga taxifolia.* — The hardened exudation yielded about 9 per cent. of a terpene-like liquid, most of which distilled between 157° and 160° C., and the remainder between 160° and 165° C.

From the characteristics of these different distillates, the author concludes that *P. Jeffreyi* and *P. Sabiniana* yield normal heptane, whilst the other species mentioned yield terpenes resembling those obtained from other coniferous trees.—C. A. M.

Methylheptenone; New Synthesis of ——. W. Ipatiew. Ber. 1901, **34**, [4], 594—596.

THE constitution of methylheptenone—

$$(CH_3)_2{:}C{:}CH.CH_2.CH_2.CO.CH_3,$$

has been ascertained by Barbier and Bouveault and by Tiemann and Semmler (Ber. **28**, 2128). Barbier and Bouveault obtained an unsaturated ketone by the action of sodium acetylacetone on dimethyltrimethylene bromide, $(CH_3)_2.CBr.CH_2.CH_2Br$, from which caustic soda separated methylheptenone in small quantity (Comptes Rend. **112**, 1243). The author has studied the action of sodium aceto-acetic ester upon the dibromides $C_nH_{2n}Br_2$; acids with closed chains are formed only when the bromine atoms are united to primary carbon atoms or to a primary and a secondary. By the action of sodium aceto-acetic ester upon the above dimethyl-trimethylene bromide, dimethyl-allylaceto-acetic ester is formed; it boils at 120°—122° C. under 11—12 mm. pressure. A second unexamined product boiled at 145°—150° C. When heated with baryta or weak alcoholic potash, this ester produces a ketone identical in all respects with natural methylheptenone.—A. C. W.

Camphor; New Method of Manufacturing ——. The Ampère Electrochemical Co. Rev. Prod. Chim. **4**, [3], 36.

THE process is based on the reaction of oil of turpentine on oxalic acid, or some other body capable of yielding up COOH, so as to form esters, which are then oxidised to camphor.

For this purpose five parts by weight of oil of turpentine, free from water, are heated to a temperature below the boiling point, *e.g.*, 120°—130° C., along with one part of anhydrous oxalic acid. The reaction produces a mixture of borneol, its oxalic and formic esters, camphor, and polymerisation products, in which the borneol esters are decomposed by the action of caustic alkali (lime), leaving a mixture of borneol, camphor, and impurities. The latter are then eliminated by distillation, and the borneol is oxidised to camphor by a mixture of potassium bichromate and sulphuric acid.—C. S.

Citraptene or Lemon Camphor. E. Theulier. Bull. Soc. Chim. 1901, **25**, [8], 465—468.

A QUANTITY of 2,500 grms. of lemon oil, when concentrated *in vacuo* until the terpenes were completely removed, gave 200 grms. of a very deep yellow substance, which, on cooling, formed a semi-fluid mass containing crystals. The crystalline portion was separated by filtering; it then formed a waxy mass. The portion insoluble in hot alcohol was composed of salts of potassium and iron, probably citrates. The alcoholic solution of the citraptene contained two substances, melting at about 76° and 145° C., the former is present only in traces, and always separates from alcoholic solution in the amorphous form, the latter crystallises in small prismatic needles.—A. C. W.

Mandragora Root; Bases of ——. H. Thoms and M. Wentzel. Ber. 1901, **34**, [6], 1023—1026.

HYOSCYAMINE has previously been found amongst the bases contained in the mandragora root (this Journal, 1898, 1071). The aurichloride of another alkaloid melted at 205° C. This base was now isolated in the following manner:—The roots were exhausted with alcohol containing tartaric acid and water, the extract concentrated *in vacuo*, and extracted with petroleum spirit and ether. It was then exactly neutralised with hydrochloric acid, sodium bicarbonate added, and the solution extracted with ether. After two repetitions of this process, a base was obtained, the hydrobromide of which was identical with the salt of Schmidt's scopolamine in composition and optical rotation. A third base, belonging to the piperidine series, is also present.—A. C. W.

Alkaloids; Micro-chemical Investigation of ——. E. Pozzi-Escot. Comptes Rend. **132**, [15], 920—921.

THE author has examined the crystalline structure, micro-chemically, of the picrates of strychnine, cocaine, brucine, atropine, morphine, codeine, and quinine. Of these, strychnine is the only one, the picrate of which is in the least characteristic.—J. T. D.

Cinchona Alkaloids; Perbromides of ——. A. Christensen. J. prakt. Chem. 1901, **63**, [6, 7, 8], 313—351.

QUININE, cinchonine, and cinchonidine, when dissolved in hydrobromic and acetic acids, and treated with bromine, produce crystalline perbromides of the formula, $AlkBr_2.2HBr.Br_2$. The bases contained in the perbromides are dibrom-addition compounds of the alkaloids; they have the composition, $C_{20}H_{24}Br_2N_2O_2$ (quinine), and $C_{19}H_{22}Br_2N_2O$ (cinchonine and cinchonidine).

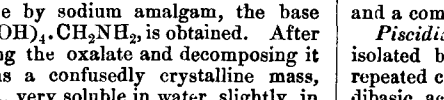
A dibromo-derivative of cinchonidine is unknown; Skalweit's base (Annalen, **172**, 103) is a perbromide of the formula, $C_{19}H_{22}Br_2N_2O.Br_2$.

The salts of the bases contained in the perbromides are formed from 1 mol. of base and 2 equivalents of acid. One-half of the acid is more loosely combined than the other, so that a solution of the salt in 50 per cent. alcohol liberates iodine from a solution of potassium iodide and iodate.—A. C. W.

Glucamine; New Base derived from Glucose. L. Maquenne and E. Roux. Comptes Rend. **132**, [16], 980—983.

BY reducing glucosoxime by sodium amalgam, the base *glucamine*, $CH_2OH.(CHOH)_4.CH_2NH_2$, is obtained. After forming and recrystallising the oxalate and decomposing it by lime, the base forms a confusedly crystalline mass, melting at 127°—128° C., very soluble in water, slightly in alcohol, lævo-rotatory, not reducing Fehling's solution. Its taste is at once sugary and caustic, it absorbs carbon dioxide, and exhibits all the characters of a powerful base.

The *oxalate* crystallises readily from alcohol in hexagons, melts at 180° C., at a somewhat higher temperature gives off water, and forms a slightly yellow mass, of the composition $C_2O_2(NC_6H_{14}O_5)_2$. Other salts have not yet been isolated in a well-defined condition. The free base precipitates metallic salts like ammonia; it redissolves in excess the precipitates formed with ferric, cupric, and mercuric salts. It yields iodoform with tincture of iodine. Its action on nitrous acid is yet obscure; but it behaves towards ethyl oxalate, benzaldehyde, acetylacetone, and potassium cyanate, as a primary amine. Its constitution is, according to the author's notation, amino- 1 hexanepentol $\frac{2.4.5}{3}6$.



It is perhaps identical with the compound found by Fischer among the products of reduction of glucose-hydrazone.—J. T. D.

Theobromine; Properties of ——. Th. Paul. Archiv der Pharm. **239**, 81—90.

THEOBROMINE crystallised from aqueous solutions may be dried without loss at a temperature of 50° C. The base is not hygroscopic. The solubility in water at 18° C. is 1 : 3232. It is slightly more soluble in dilute hydrochloric acid, but not so much so as is required by theory for an undissociated hydrochloride. It is more soluble in caustic soda solution, showing that its characters are more of an acid than of a basic nature. Theobromine possesses much weaker basic properties than betaine, but on the other hand its acid characters are more marked.—J. O. B.

Anæsthetic [Ethyl-o-Anisidine Formate]; New ——. C. Goldschmidt. Chem. Zeit. 1901, **25**, [31], 329.

THE author had previously announced the anæsthetic properties of methenyl-*o*-anisidine, obtained by the action of orthoformic ester on *o*-anisidine (Chem. Zeit. 1898, **22**, 1033). Methenyl-*o*-anisidine appears, however, to change into ethyl-*o*-anisidine formate, which is a strong local anæsthetic; it was obtained by Claisen's method (Annalen, **287**, 360). By long boiling of *o*-anisidine with orthoformic ester, ethyl-*o*-anisidine formate is obtained; the methenyl-*o*-anisidine, when once isolated, is stable, and distils unchanged at 253° C. The guaiacol sulphonic acid salt of methenyl-*o*-anisidine is obtained by dissolving its constituents in alcohol and precipitating by ether; it is very soluble in water and has anæsthetic action.—A. C. W.

Jamaica Dogwood; Constituents of ——. P. C. Freer and A. M. Clover. Amer. Chem. J. 1901, **25**, [5], 390—413.

THE active principle of the bark of the root of Jamaica dogwood (*Piscidia erythrina*) has been stated by Hart (Amer. Chem. J. **5**, 9) to be a resinoid called *piscidine*, crystallising from alcohol in colourless prisms, melting at 192° C. This so-called piscidine really consists of a mixture of two distinct compounds, melting at 216° C. and 201° C. respectively, and it is stated that a pharmacological investigation by Cushny has shown that there is no foundation for the assumption that this mixture constitutes the active principle of the bark.

The authors have made a detailed investigation as to the constituents of Jamaica dogwood, and, by means of a series of extracts of the powdered bark of the root, have isolated the following substances:—*Piscidic acid*, $C_{11}H_{12}O_7$, m. pt. 185° C.; a compound, $C_{23}H_{20}O_7$, m. pt. 201° C.; a compound $C_{22}H_{18}O_6$, m. pt. 216° C.; a substance melting at 50°—80° C.; a compound, $C_{25}H_{22}O_7$ (?), m. pt. 159° C.; and a compound, $C_{20}H_{22}O_7$, m. pt. 150°—155° C.

Piscidic acid, obtained from the aqueous extract, was isolated by means of its lead salt, and was purified by repeated crystallisation from methylpropylketone. It is a dibasic acid, $C_9H_8O(OH)_2(CO_2H)_2$, extremely soluble in water, slightly so in ether, and insoluble in chloroform, benzene, and petroleum spirit (ligroin). It resembles, in its properties, mucic and saccharic acid, is decomposed on heating, giving an odour of caramel; and is instantly oxidised by permanganate of potash, forming potassium carbonate, oxalate, and, apparently, formate. The ethyl ester, $C_9H_{10}O_3.CO_2H:CO_2C_2H_5$, m. pt. 207° C.; anilide, $C_9H_{10}O_3(CO.NH.C_6H_5)_2$, m. pt. 196° C.; acetyl derivative of ethyl ester, $C_9H_8O(C_2H_3O_2)_2.CO_2H.CO_2C_2H_5$, m. pt. 149° C.; and bromine derivative, $C_{11}H_{12}O_2Br_2$ (?), m. pt. 234° C., were prepared.

The compound, $C_{23}H_{20}O_7$, crystallises from alcohol in highly refractive rectangular prisms, is very soluble in chloroform, moderately soluble in benzene and acetic acid, sparingly soluble in alcohol, and insoluble in ether, petroleum spirit (ligroin), and alkalis. It contains two methoxy groups, and by the action of methyl alcoholic potash, yields two compounds, one, $C_{22}H_{20}O_8$, melting at 159° C., and the other, $C_{22}H_{20}O_7$, melting at 136° C.

The compound, $C_{22}H_{18}O_6$, crystallises from alcohol in fine yellow needles, soluble in benzene and chloroform, sparingly soluble in ether and alcohol, and insoluble in ligroin, water, and alkalis. It contains two methoxy groups.

The substance melting at 50°—80° C., could not be obtained in a pure state, and the quantity was too small to admit of analysis. It is probably a glucoside; when heated with slightly acidified alcohol, it deposits crystals of the compound $C_{22}H_{18}O_6$, melting at 216° C., whilst the mother liquor readily reduces Fehling's solution.

The compound ($C_{25}H_{22}O_7$?), melting at 159° C., could not be obtained in a perfectly pure state; it yielded colourless crystals from alcohol, soluble in chloroform and benzene, sparingly soluble in ether and ligroin, and insoluble in aqueous acid or alkaline solution.

The compound, $C_{20}H_{22}O_7$, separated from 70 per cent. alcohol in small, needle-shaped, colourless crystals, melting with decomposition at 150°—155° C., very soluble in ether and methyl alcohol, moderately soluble in acetone, sparingly soluble in ether, chloroform, benzene, and hot water, and insoluble in ligroin. It dissolves in hot alkalis with a red colour. The compound reduces Fehling's solution and ammoniacal silver nitrate, and gives a green coloration with ferric chloride. A diacetyl derivative, $C_{24}H_{26}O_9$, melting at 183° C., was prepared. When heated with slightly acidified alcohol, it yielded a compound, $C_{18}H_{16}O_6$, melting at 275° C.; and by heating with caustic potash and methyl iodide, a compound, $C_{20}H_{20}O_6$ (?), melting at 141° C.—A. S.

Balsam of Peru. Chem. and Druggist, 1901, **58**, [1108], 638—642.

P. PREUSS describes the method of cultivating the "balsam-tree" and the preparation of the balsam (see this Journal, 1901, 150). H. Thoms and C. Mannich give the results of the analysis of four samples:—(1) Peru balsam as exported from San Julian; (2) "rag balsam" (balsamo de trapo); (3) "bark balsam" (balsamo de cascara); and (4) balsam made from bark brought from Central America by P. Preuss and extracted with ether (yield, 18·5 per cent.).

——	1.	2.	3.	4.
Specific gravity at 15° C.	1·404	1·408	1·612	..
Cinnamein, per cent.....	64·6	65·8	51·4	37·4
„ ester number	263·6	260·6	250·0	..
Resin, per cent.	18·2	17·3	28·7	26·5

—A. S.

Oil of Rue. Thoms. Pharm. Zeit. **46**, 110. Pharm. J. 1901, **66**, [1608], 485.

THE author finds that the oil of rue from *Ruta graveolens*, L., is free from terpenes, but contains about 5 per cent. of normal methylheptylketone; its chief constituent is normal methylnonylketone. The author also detected free fatty acids and a phenol, but could not confirm the presence of laurinaldehyde and the compound, $C_{12}H_{14}O$, reported by Williams.—A. S.

Calamus Oil; The Constituents of ——. H. Thoms and R. Beckstroem. Ber. 1901, **34**, [6], 1021—1023.

A FRACTION of calamus oil boiling between 272° and 340° C. was saponified and fractionated *in vacuo*. From the highest fraction a crystalline substance, $C_{15}H_{26}O_2$, melting at 166°—167·5° C., separated. From the mother liquor of this compound, asarone, $C_{12}H_{16}O_3$, was isolated. The original oil contained free fatty acids and eugenol.

A colourless oil, of very pleasant odour, was obtained by shaking the calamus oil fraction with sodium bisulphite solution, and decomposing the compound with sodium carbonate. On standing, the odour was lost and crystals separated, which were found to be asaryl aldehyde; the oxime, $C_{10}H_{13}O_4N$, melted at 137° C.—A. C. W.

Saffron; Estimation of Red Sanders Wood in ——. A. Beythien. Zeits. Untersuch. Nahr. Genussm. 1901, **4**, [8], 368—369.

THE author finds that a determination of the amount of crude fibre in samples of saffron affords a good indication of the amount of adulteration should the same be mixed with sanders wood. The latter contains from 62·12 to 62·93 per cent. of crude fibre, whilst the quantity in saffron varies from 4·54 to 5·47 per cent. "Bastard" saffron contains from 11·87 to 12·53 per cent. of crude fibre. A sample of saffron, which contained 20·33 per cent. of crude fibre, was, from the above figures, calculated to be adulterated with 26·6 per cent. of sanders wood, and this conclusion agreed favourably with a microscopic examination of the sample.

In estimating the crude fibre in saffron, the latter should be first boiled with water until freed from colouring matter, as boiling with acid precipitates a large quantity of crocetin, which blocks the filter.—W. P. S.

Licari canali (Bois de rose femelle); Essential Oil of ——. E. Theulier. Bull. Soc. Chim. 1901, **25**, [8], 468—475.

THE essential oil of the *bois de rose femelle* is sometimes confounded with oil of linaloë (this Journal, 1896, 919); it is therefore called oil of linaloë of Guiana. The essential constituent of both oils is linalool; the odour of the former is, however, much finer than that of oil of linaloë. The density of the essential oil of *bois de rose femelle* is 0·8735 at 14·5° C., and its optical rotation −15° 20′ to −13°; the corresponding values for Mexican oil of linaloë are 0·898 and −7° 33′.

The wood, which is also known as *licari canali*, is imported into France from Guiana in the form of logs, 1·20—1·30 m. in length, and varying in diameter from that of small branches up to 1 m. or even more. The rugged greyish-maroon-coloured bark is devoid of oil. The wood is of a bright golden-yellow colour, with reddish-yellow veins. The density of the wood at 15° C. is 0·6789; it is much less hard than sandal wood. Unhealthy trees produce inferior oil. A tree of medium age produces the largest yield of oil, the upper portion of the trunk and the large branches producing more than the other portions. The heart of a log gives a larger yield than the outer portion.

The ground or chipped wood is distilled with steam; the residue is used as fuel or packing. The yield of essential oil varies from 1 to 1·6 per cent., the mean being 1·4—1·5 per cent. After rectification the oil is colourless; its odour is soft and agreeable, with a characteristic piquancy, and is very persistent.

On fractionation the principal portion of the oil distils at 194°—200° C.; this fraction was identified with *l*-linalool. By fractionating 10 kilos. of oil it was proved to be free from methylheptenone and other substances boiling below 192° C. Geraniol and other compounds boiling above 210° C. are also absent. The quantity of saponifiable matter was about 0·3 per cent. The oil is thus almost entirely composed of *l*-linalool. It is soluble in 2 vols. of alcohol of 70°. It is principally employed for perfuming soaps, for which its neutral character is an advantage.

—A. C. W.

Anthranilic Acid; Derivatives of ——. H. Mehner. J. prakt. Chem. 1901, **63**, [6, 7, 8], 241—312.

THE author summarises his conclusions as follows:—

Methyl anthranilate (2 mols.) condenses with formic or acetic aldehyde (1 mol.), without the presence of acids, to form methylene or ethylidene diamine derivatives, the imide hydrogen of which cannot be replaced by acid groups. The methylene diamine is transformed by acids into a derivative of diaminodiphenylmethane, whilst the ethylidene diamine is decomposed. Different products are obtained from methyl anthranilate and aldehydes in acid solution; acetaldehyde is polymerised to aldol, which then unites with 2 mols. of the ester.

Diazotised methyl anthranilate unites with primary amines to form diazo-amino compounds, which are fairly stable and crystallise well. Methyl anthranilate does not unite with diazo compounds in dilute acetic acid solution.

Anthranilic acid readily unites with diazo salts to form unstable diazo-amino compounds, the stability of which is increased by the presence of negative groups in the diazotised amine and decreased by alkyl groups.

After treatment with nitrous acid, anthranilic acid does not unite in dilute acetic acid solution with primary amines to form diazo-amino compounds; the condensation, however, takes place in pyridine solution.

o-Toluene diazochloride and *m*-xylene diazochloride do not unite with anthranilic acid, apparently because in one ring a methyl and in the other a carboxyl group would come into the *o*-position with regard to the anticipated diazo-amino group.

Diazo-aminobenzene-*o*-carboxylic ester, its homologues and substitution products, on boiling with dilute alcohol, yield methyl alcohol and derivatives of phen-β-triazone. The ring formation is facilitated by the presence of negative groups in the nucleus of the amine united to the diazobenzene-*o*-carboxylic ester, but is hindered by the presence of alkyl groups. The same triazones are obtained by diazotising *o*-aminobenzanilide and its homologues.

On heating with alcoholic alkalis, the triazones are decomposed with formation of the corresponding diazo-aminobenzene-*o*-carboxylic acids. The latter are also obtained by the saponification of their esters. The compounds obtained are identical with those produced by diazotising the amine, united in the above case with the diazotised methyl anthranilate, and combining with anthranilic acid. This is another case of the identity of the diazo-amino compounds obtained from $XN_2Cl + YNH_2$ and $YN_2Cl + XNH_2$.

A simple apparatus (see page 623) was employed for the estimation of the diazo-nitrogen in both crude and purified diazo-amino compounds.—A. C. W.

Geranium; Essence of Cannes ——. Jeancard and Satie. Bull. Soc. Chim. 1901, **25**, [9], 516—519.

THE authors have previously shown (this Journal, 1900, 272,) that the determination of physical and chemical constants serves in a measure to distinguish between the different geranium essences. In the present paper they present in tabular form a series of experiments on six samples taken weekly, during the season, showing the weekly minimum, medium, and maximum specific gravity, rotation, solubility in 70 per cent. alcohol, surface tension, specific viscosity, alcohols, saponification numbers hot and cold, and true ethers. During these experiments the residues from the determination of the saponification numbers and those from the alcohol determinations were preserved and examined. That which came from the determination of the saponification number in the cold is "neutral" "geranium"; that from the saponification number hot is "saponified" "geranium"; that from the determination of alcohol geranium "acetyl"; the characteristics of these are tabulated.

Assuming that neither in saponification hot or cold, or in the acetylation are there found resins or polymerides, the following conclusions are drawn from the tabulated averages:—

(1) Neutralisation diminishes specific gravity, rotation, and surface tension, whilst increasing the solubility.

(2) Acetylation increases specific gravity and surface tension, whilst diminishing specific viscosity and solubility.

(3) Saponification increases viscosity and solubility. Neutralisation increases the solubility in 70 per cent. alcohol.

This explains why Cannes geranium is a little more soluble than other essences. Cannes geranium contains less free acid. The authors find that the free acids are present in the wood 3 per cent., leaves 54 per cent., and stalks 43 per cent.—C. T. T.

Essential Oils; Surface Tension and Viscosity of —— Jeancard and Satie. Bull. Soc. Chim. 1901, **25**, [9], 519—523.

DUCLAUX'S method for determining surface tension of liquids is as follows:—A 5 c.c. pipette provided with a capillary orifice, is calibrated to 100 drops of distilled water at 15° C. If this pipette be filled with a liquor of surface tension F, specific gravity D, and gives n drops at 15° C., then $\frac{F}{f} = \frac{100}{n}D$, f being the surface tension of distilled water at 15° C. If the value n is known to be between 140° and 300°, a correction is made—

$$N = n - [0{\cdot}002\ (n - 100) + 0{\cdot}00036\ (n - 100)^2].$$

The authors have determined the surface tensions and viscosities of a number of essential oils, and give the results in a series of tables, in which the oils are grouped, according to the classification of Charabot, Dupont and Pillet, as:—Alcohols and alcoholic esters (*e.g.*, benzyl acetate), Aldehydes (*e.g.*, benzaldehyde, Phenols (*e.g.*, eugenol), Ketones, Hydrocarbons, &c. From their average results they draw the following conclusions:—

Alcohols.—First, essences of which the principal constituent is geraniol, free or combined; average surface tension is higher than that of essences of which the principal constituent is linalool. *Secondly*, specific viscosity of an ester is less than that of the corresponding alcohol. *Thirdly*, for essences, diminution of specific viscosity corresponds generally to increase of saponification number.

Phenols. — First, for essences containing eugenol; decrease of surface tension corresponds to decrease of phenols. *Secondly*, for essences containing thymol and carvacrol; the non-phenolic portions have a surface tension and viscosity less than that of the phenolic portion.

Aldehydes.—First, the surface tension appears to diminish with content of aldehydes. *Secondly*, the viscosity of aldehydes is generally less than that of the corresponding alcohols.—C. T. T.

Myrcenol and its Constitution. P. Barbier. Comptes Rend. **132**, [17], 1048—1050.

THE alcohol ($C_{10}H_{18}O$) was prepared from myrcene by Power and Kleber's method. It is a colourless, oily, fragrant liquid, slowly polymerising and becoming viscous. It boils at 99°—101° C. at 10 mm. pressure; density, 0·9012 at 14·5° C.; refractive index for sodium light 1·47787, hence molecular refraction, 48·35. It must therefore contain two double linkings. It gives with acetic anhydride a monacetyl derivative; and it must be, from its mode of formation, a tertiary alcohol. It is certainly different from licareol. On oxidation with chromic acid it yields acetone, an aldehyde $C_{10}H_{16}O$, similar to limonal, but not identical with it, and levulinic acid. No methylheptenone is formed, but some of the original myrcene is regenerated. On oxidation with permanganate, followed by chromic acid, it gave acetone, and a mixture of levulinic and succinic acids. From this evidence the author deduces the formula $C(CH_3)_2:CH.CH_2.CH_2.C(CH_3)OH.CH:CH_2$, and hence that of myrcene is—

$$C(CH_3)_2:CH.CH_2.CH:C(CH_3).CH:CH_2.$$

That a tertiary alcohol should form an aldehyde on oxidation is unusual, but the author explains it by supposing that myrcene is first formed by dehydration of the alcohol, and is then directly oxidised.—J. T. D.

Perfumes; New Method of Improving ——. Lavollay and Dourgoin. Rev. Prod. Chim. **3**, [23], 358.

THE method consists in treating essential oils, either singly or mixed, with 20 to 25 grms. per hectolitre of calcium manganate or other manganate, in presence of an electric current of suitable intensity, for example, 3 to 10 ampères per square metre of electrode surface, the operation being in no case prolonged beyond 20 minutes. Under these conditions the manganates are said to act more energetically than ozone, the action being also more easily controlled and cheaper.—C. S.

Piperine Colloid, Form of ——, *with Especial Reference to its Optical Refraction and Dispersion.* H. G. Madan. Proc. Chem. Soc. 1901, **17**, [238], 127.

THE author has examined the conditions under which crystalloid piperine is converted into the colloidal allotropic

form of the substance. He finds that while crystallised piperine when heated to its melting point, 135° C., solidifies on cooling into a transparent resin-like substance, the latter, when thus prepared, is not permanent, but reverts spontaneously in the course of a few months into the crystalloid form. The same change takes place quickly and completely when the substance is heated to 100° C. If, however, piperine is kept for an hour at a temperature of 180°, the resulting product is much more stable, if not absolutely permanent as a colloid. It does not become crystalline when heated to 100° or above, nor has the lapse of 2½ years had any effect in changing it back into the crystalloid condition.

The results of a determination of the refractive indices of colloid piperine for some of the principal spectrum lines were given, from which it appears that, while its refraction is very high ($\mu_D = 1{\cdot}684$), its dispersion is quite extraordinary, the visual spectrum being nearly four times as long as that given by a prism of dense flint glass having the same refracting angle. The coefficient of dispersion ($\mu_{H\gamma} - \mu_{H\alpha}$) is 0·142, while that of carbon bisulphide is only 0·057.

Bismuth by Electrolysis; Quantitative Determination of ——. K. Wimmenauer.

See under XXIII., *page* 620.

"Saccharin"; New Reaction for ——. A. Leys.

See under XXIII., *page* 622.

Catalase [*Tobacco Fermentation, Curing, &c.*]; *New Enzyme of General Occurrence.* O. Loew.

See under XVII., *page* 598.

Morphine; Determination of ——, *by means of Potassium Iodate and Arsenious Acid in Alkaline Solution.* C. Reichard. Chem. Zeit. 1901, **25**, 31, 328.

See under XXIII., *page* 624.

Alkaloids; Methods of Exhausting Drugs for Determination of ——. O. Linde.

See under XXIII., *page* 624.

Cloves; Notes on the Approximate Analysis of ——. A. McGill.

See under XXIII., *page* 625.

PATENTS.

Iodine and other Products from Sea Weed; Extraction of ——. J. Thesen, Christiania, Norway. Eng. Pat. 15,233, Jan. 30, 1900. (Under Internat. Convent.)

Sea weed, more or less dried, is heated with dilute (5—20 per cent.) sulphuric acid in open or closed vessels. The iodine present goes into solution, and may be recovered by adding an oxidising agent such as sodium nitrite, and finally separated by distillation or extracted with petroleum, petroleum spirit or other mineral oil. The solution also contains potassium sulphate, which may be crystallised out after removing the iodine. The sea weed residue, after drying forms a pulverulent manure, containing almost all the original nitrogenous constituents of the sea weed.

—T. A. L.

Stannic Acid; Manufacture of —— *by Combustion of Tin Vapour.* C. P. Bary, Paris. Eng. Pat. 9231, May 18, 1900.

Metallic tin is heated, electrically or otherwise, to a temperature of 1,700° C. out of contact with air. The resulting tin vapour is then burnt in a current of air or oxygen. The stannic oxide thus produced is more homogeneous and in a better state of sub-division for enamelling, &c., than is the ordinary oxide, whilst owing to the absence of metallic tin, it has not the grey tint often observable in the older samples.—W. G. M.

Antiseptic Compounds; Manufacture of ——. A. Zimmermann, London. From The Chemische Fabrik auf Actien vorm. E. Schering, Berlin. Eng. Pat. 10,845, June 14, 1900.

In Eng. Pat. 8429 of 1893 (this Journal, 1894, 414) a process is described for the production of alkaline antiseptic liquids from silver salts and non-toxic organic bases, such as ethylenediamine, and piperazine, &c., such solutions having the property of not coagulating albumin. According to the present specification, these compounds can be obtained in a solid form. For example, the compound from silver nitrate and ethylene diamine is obtained by stirring 6 grms. of powdered silver nitrate into 5 grms. of 90 per cent. ethylene diamine at a temperature below 40°—50° C. The clear solution is mixed with 18 grms. of absolute alcohol, and on cooling, the mass solidifies to a crystalline jelly. After filtering, the product is washed with alcohol and ether, and dried in the dark over lime. The product has the formula $2(H_2N.CH_2.CH_2.NH_2).AgNO_3$, is readily soluble in water with a strong alkaline reaction, and melts at 65° C. Piperazine silver nitrate is obtained by quickly mixing 2·5 grms. of silver nitrate in 2·5 c.c. of water and 5·1 grms. of piperazinum in 10·1 grms. of absolute alcohol, with constant agitation, when the product separates in a crystalline form. The specification gives the physical properties and melting points of a number of these new substances.—T. A. L.

Saponine from Horse-Chestnuts; Obtaining ——. L. Weil, Strassburg, Alsace, Germany. Eng. Pat. 3217, Feb. 14, 1901.

Ripe horse-chestnuts, after peeling, are ground and dried at 40°—50° C. The oil and resin are removed by extraction with benzine or petroleum spirit, and a subsequent extraction with 93—96 per cent. alcohol dissolves the saponine, which separates on concentration and cooling. The crude product is dissolved in alcohol, treated with freshly precipitated hydroxide of lead (from lead nitrate and ammonia), and finally precipitated by pouring the alcoholic solution into ether. If necessary this process is repeated. After drying, the saponine forms a white powder, readily soluble in water, giving a strongly frothing solution. The yield of saponine is about 10 per cent. of the weight of the nuts.—T. A. L.

XXI.—PHOTOGRAPHY.

Metol-Developer; Limit of Dilution of ——. E. Kastner. Phot. Rundschau, **15**, 91. Chem. Zeit. Rep. **25**, [14], 128.

Metol can be used in very dilute solution. A solution of 0·5 grm. of metol and 5 grms. each of sodium thiosulphate and sodium carbonate, in 2 litres of water, forms a developer suitable both for instantaneous and for longer exposed plates. The time required for the development varies from two to three hours.—J. T. D.

Potassium Percarbonate; Medium for Destroying Thiosulphate in Photographic Films. E. Valenta. Phot. Corr. **38**, 235. Chem. Zeit. Rep. **25**, [14], 128.

"Antihypo," a new preparation, consisting essentially of potassium percarbonate, for destroying excess of thiosulphate after fixing and washing, was found by the author to effect its object perfectly without injuring the film.

—J. T. D.

PATENTS.

Photographic Prints; Process for Obtaining ——. C. D. Abel, London. From The Actien-Ges. für Anilin Fabr., Berlin. Eng. Pat. 12,313, July 7, 1900.

The processes hitherto employed for producing photographic images by means of chromates depend either on treating the exposed print with metallic solutions or on using the mordanting properties of the chromium dioxide which is formed by light to deposit dyes on the paper. According to the present invention, the oxidising property of the chromium

dioxide is made use of to convert colourless aromatic compounds into colouring matters of any desired shade. Thus the process yields positive pictures. A method has been described by Lumière in which manganese salts and oxidisable bodies are used somewhat similarly, but here the images were produced by the action of the unchanged chromates, and the pictures were negative. The new process is as follows:—Ordinary unsensitised photographic paper is coated with a solution of 16 to 20 grms. of ammonium bichromate in 100 c.c. of water, to which, in order to avoid sinking in of the image, 6 grms. of gelatin or gum may be added. The paper is dried in the dark, and exposed under a negative for half the time required in the gum process. The prints are washed quickly with plenty of fresh water, finally rinsed in a bath of 1 c.c. of sulphuric acid in 1 litre of water, and then developed with 1 grm. of *p*-phenylenediamine, 1 or 2 grms. of sodium bisulphite, and 600 c.c. of water in the cold. The prints are washed first in acidulated, afterwards in pure water, and possess a pure dark brown colour. Other organic compounds which give characteristic colours are dimethyl-*p*-phenylenediamine, tolylene-*p*-diamine, 1 : 5 - naphthylenediamine, *p* - aminophenol, methyl-*p*-aminophenol, *o*-*p*-diaminophenol, *β*-triaminophenol, pyrogallol, 1 : 5 - dihydroxynaphthalene, *p*-aminodiphenylamine, *p*-diaminophenylamine, aniline, and dimethylaniline.—F. H. L.

Photographic Reducer; Manufacture of a ——. C. D. Abel, London. From The Actien-Ges. für Anilin Fabr., Berlin. Eng. Pat. 12,375, July 9, 1900.

The well-known photographic reducers, potassium ferricyanide or potassium ferric oxalate, mixed with sodium thiosulphate, cannot be kept ready made, but must be prepared afresh each time as required. The inventors overcome this inconvenience by compounding a mixture of 5 kilos. of powdered potassium ferric oxalate and 50 kilos. of powdered anhydrous sodium thiosulphate. This is permanent in the dry state, and for use only has to be dissolved in water. Potassium ferricyanide cannot be employed in a similar manner.—F. H. L.

Bromide Photographic Prints; Treatment of ——, *to Render them more Permanent, with Range of Colours.* T. J. Smith, London. Eng. Pat. 22,201, Dec. 6, 1900.

The prints are treated in a bath of potassium bichromate, 1 oz.; hydrochloric acid, ½ oz.; water, 20 oz., till they acquire a creamy colour. They are then well washed and redeveloped with sodium sulphantimoniate, 1 oz.; water, 30 oz. They are next rinsed, and toned with ammonium sulphide, 1 oz.; water, 20 oz. Finally they are washed and fixed in an alum bath, as is done with "carbons." By this process an image of silver sulphide in bichromated gelatin is obtained, which is said to be permanent; a variety of tones ranging from red to dark brown may be produced, and the whole method of working is cheap.—F. H. L.

XXII.—EXPLOSIVES, MATCHES, Etc.

Explosion Catastrophe at the Griesheim-Elektron Chemical Works. Zeits. angew. Chem. **14**, 459—461.

The works lie to the west of Griesheim, between the railway and the Main. The chief products are mineral acids, soda (caustic and carbonate), potash, chromates, bleach, permanganate, aniline derivatives, and carbon tetrachloride. There are four main divisions: the old acid and soda works, on the river bank, adjoining the village of Griesheim; the aniline works, next the soda works; the recent electrolytic works, further from the river; and the "Mainthal" works, further inland still. Between the last and the aniline works are the works of Marx and Müller and of Nötzel Istel and Co., both of which suffered in the explosion.

About three in the afternoon of April 25th a small fire occurred, through the heating of a shaft or bearing, in the picric acid division of the aniline works. Through insufficient water-pressure at the time, it was not promptly extinguished; alarm was given, and the buildings were emptied of workpeople. In spite of the efforts of the works brigade and that of Griesheim, the fire spread, and about half-past four there was a severe explosion. Dr. Jacobi and his men after this beat a retreat, when a second much more powerful explosion occurred, which wrecked all around, and broke windows in all the neighbouring villages. Heavy wreckage, some of it burning, was hurled in all directions, so that fires occurred in the other divisions of the works, and in those of neighbouring firms.

Fire brigades from all around were soon on the spot, but the smoky atmosphere, the heaps of *débris*, and the fear of further explosions, greatly hindered their work. Several small explosions did occur, but no further damage was done. None of the boilers exploded, the fires having been all drawn and the steam blown off in time.

Griesheim was emptied, from a fear that the great store of over 100,000 litres of benzene might fire or explode; the wind was unfavourable for this, however, and though the roof had been damaged by the explosion, nothing further happened here.

For 600 metres around, roofs were lifted and walls displaced; a child playing on the river bank was hurled into the river and drowned; people in the neighbourhood were thrown down and often injured; the wall of the building in which Dr. Jacobi and his men had taken refuge was blown down and buried them in its ruins. In all, 24 persons were killed, some are missing, 20 were severely and many others slightly wounded.

Two days after the explosion, work was partially resumed in the "Elektron" and the acid works; but not till the 28th was all danger over in the aniline works and in those of Marx and Müller and of Nötzel Istel and Co.

Picric acid manufacture, it is stated, will not be begun again, even if no Government regulation should be made to prevent it.

It was generally believed, as the result of experiments, that moist picric acid would not explode, but would only burn. The catastrophe at Griesheim shows only too clearly that there are circumstances under which this is not the case. Prompt investigation of these circumstances is demanded.—J. T. D.

Explosives; Manufacture of —— *with Solidified Oil.* Ger. Pat. 119,593, Jan. 7, 1900. G. Girard, Paris. Zeits. angew. Chem. 1901, **14**, [18], 449.

A potash or soda soap with a vegetable oil or resin oil is dried at 120°—130° C., added to the oil to be solidified, to the extent of 10—15 per cent., the whole well stirred, heated to 150°—170° C. for two or three hours, and filtered if necessary. At the ordinary temperature the product is a plastic solid, but at 80° C. it is liquid, and can be mixed with chlorate, perchlorate, or nitrate, as desired. The oil itself may form the combustible constituent of the explosive, or there may be added, at will, nitro or azo compounds, nitro ethers, or any mixture of them.—J. T. D.

Nitrocellulose; Researches on the Stability of ——. W. Will. Mitth. a. d. Centralstelle f. Wissensch. Techn. Untersuchungen Neu-Babelsberg bei Berlin, 1900, [2], 5—24.

After enumerating and criticising the methods at present employed to determine the stability of nitrocelluloses, the author describes a new method, based on the quantitative estimation of the nitrogen evolved (as oxides) when the guncotton is heated at a definite temperature (135° C.).

The results so obtained are plotted in curves to illustrate the effects produced by various conditions of manufacture, and to show the relationship between this method of examination and the results given by other well-known test methods.

The figure, page 611 (Fig. 1) diagrammatically represents the apparatus employed. The sample of guncotton (2½ grms.) packed in a glass tube, *a*, 15 mm. wide and 10 cm. high, is heated by the oil-bath, *b*, at a constant temperature, the nitrogen oxides so produced are swept over ignited copper, *d*, where they are reduced, and the nitrogen is retained in the measuring tubes, *g*. Care must be taken that the acid decomposition products do not condense in any portion of

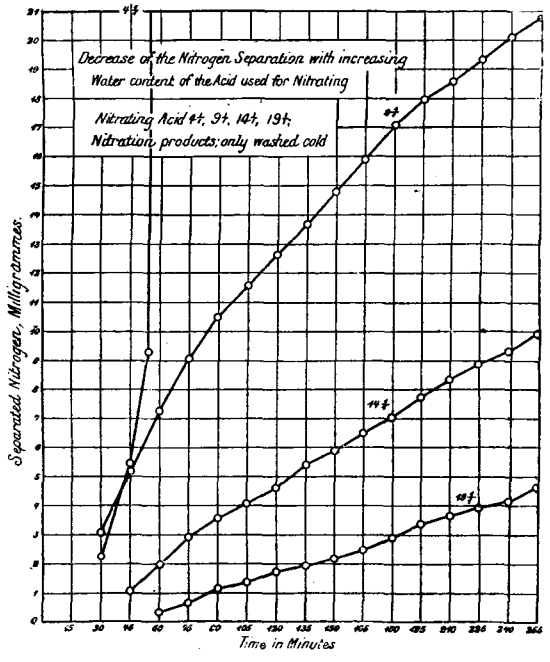
the apparatus. The air in the whole apparatus is first displaced by a stream of carbon dioxide issuing from the generator, *f*, through the scrubbers, *e*, and this stream is maintained throughout the experiment, the gas being absorbed at the end of the system by strong soda solution.

To guard against the danger of explosions, which occur repeatedly, the tube *a* and oil-bath *b* are surrounded by a large casing with walls composed of iron p'ate and strong glass.

As raw materials, an untreated cotton, similar to that commonly employed in the gun-cotton industry, was obtained, the effect of special chemical treatment being not yet sufficiently well known to risk its possible influence in the course of the experiments. The composition of the nitrating acid is indicated in the paper by a compound fraction (see curves at end), in which the unit represents the amount of water present per 100 parts, and the fraction denotes the ratio of H_2SO_4 (numerator) to HNO_3 (denominator) in the remaining portion; thus $4\frac{3}{1}$ is an acid mixture containing 4 per cent. of water, 72 per cent. of sulphuric acid and 24 per cent. of nitric acid, *i.e.*, the ratio of H_2SO_4 to HNO_3, after subtracting the water, is as 3 is to 1.

To nitrate the cotton it was placed in a centrifugal machine at 15° to 20° C. with 50 volumes of the nitrating acid, and stirred after 10 minutes to prevent local heating. After one hour the acid was drained off, the remainder removed by whirling, and the cotton then washed with at least 100 volumes of cold water, which washing was renewed over a period of eight days. It was then cut into lengths about 2 mm. long, washed again for eight days, and finally centrifugalised. As thus prepared, it was used for most of the experimental steps of purification. For example, if boiled nitrocellulose were required, the centrifugalised

PLATE 1.



product would be taken and boiled in an enamelled iron vessel together with distilled water and 10 per cent. of precipitated calcium carbonate, the water-level being kept up by further additions of distilled water, as required. The brownish liquor was drained off, the cotton washed with distilled water for 48 hours, drained, and dried on filter paper at a temperature not exceeding 40° C.

The results plotted in curves are here given in full, in order, and to show (1) the influence of the composition of the nitrating acid on the speed of nitrogen evolution, *i.e.*, decomposition of the nitro-cellulose, *vide* Plates 1 and 2; (2) the effect of incomplete purification on speed of decomposition, Plates 3 and 4; (3) the relation between quantitative decomposition and the results obtained by the usual tests, Plates 5, 6, 7*a*, 7*b*; (4) the effect on the rate

PLATE 2.

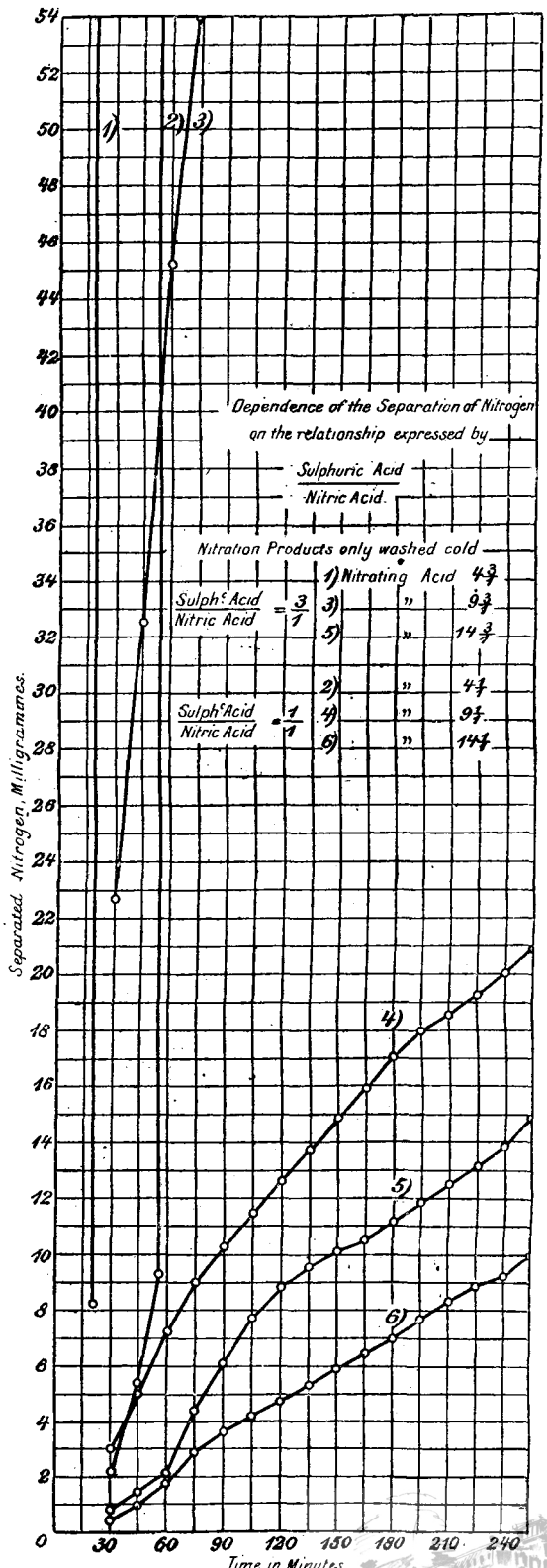


Fig. 1.

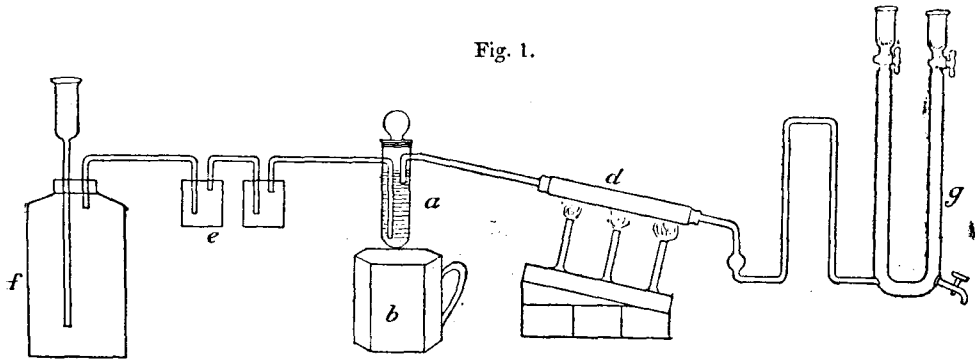
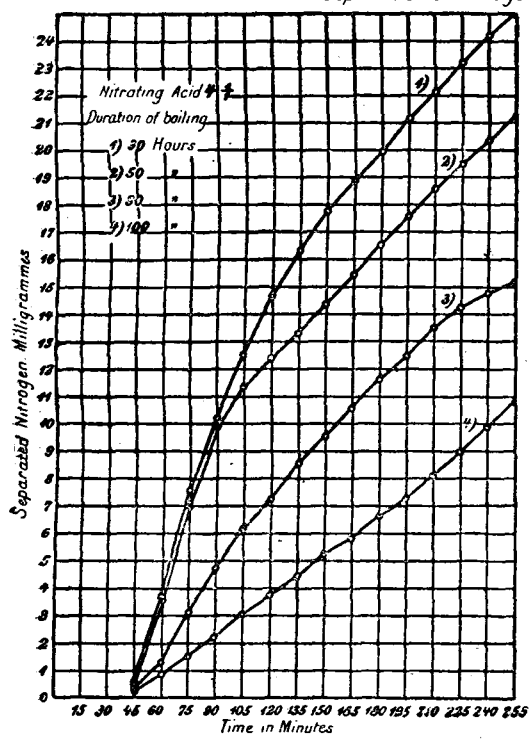
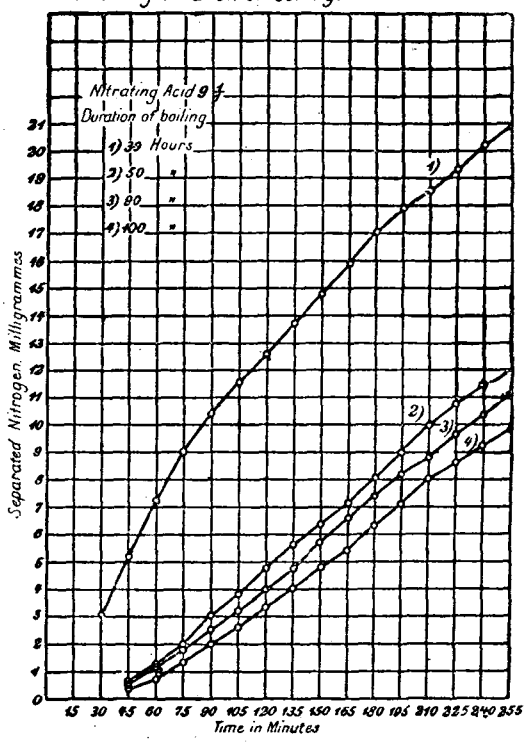
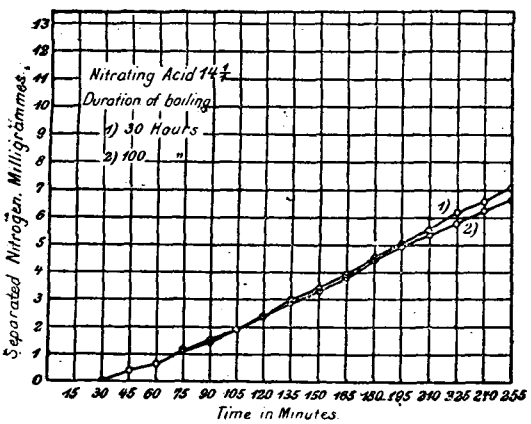


PLATE 3.

Decrease of Separation of Nitrogen with increasing duration of boiling.







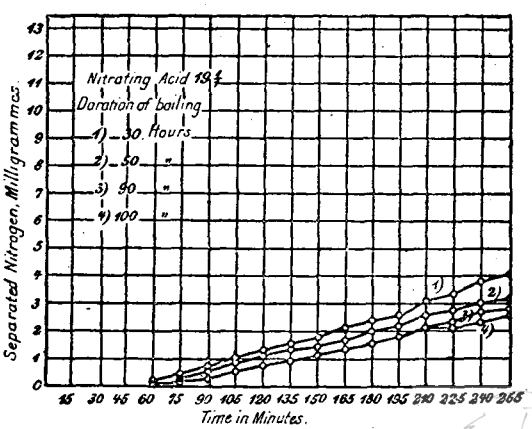


PLATE 4. (For Plate 5, see page 615.)

Decrease of Separation of Nitrogen with increasing duration of boiling

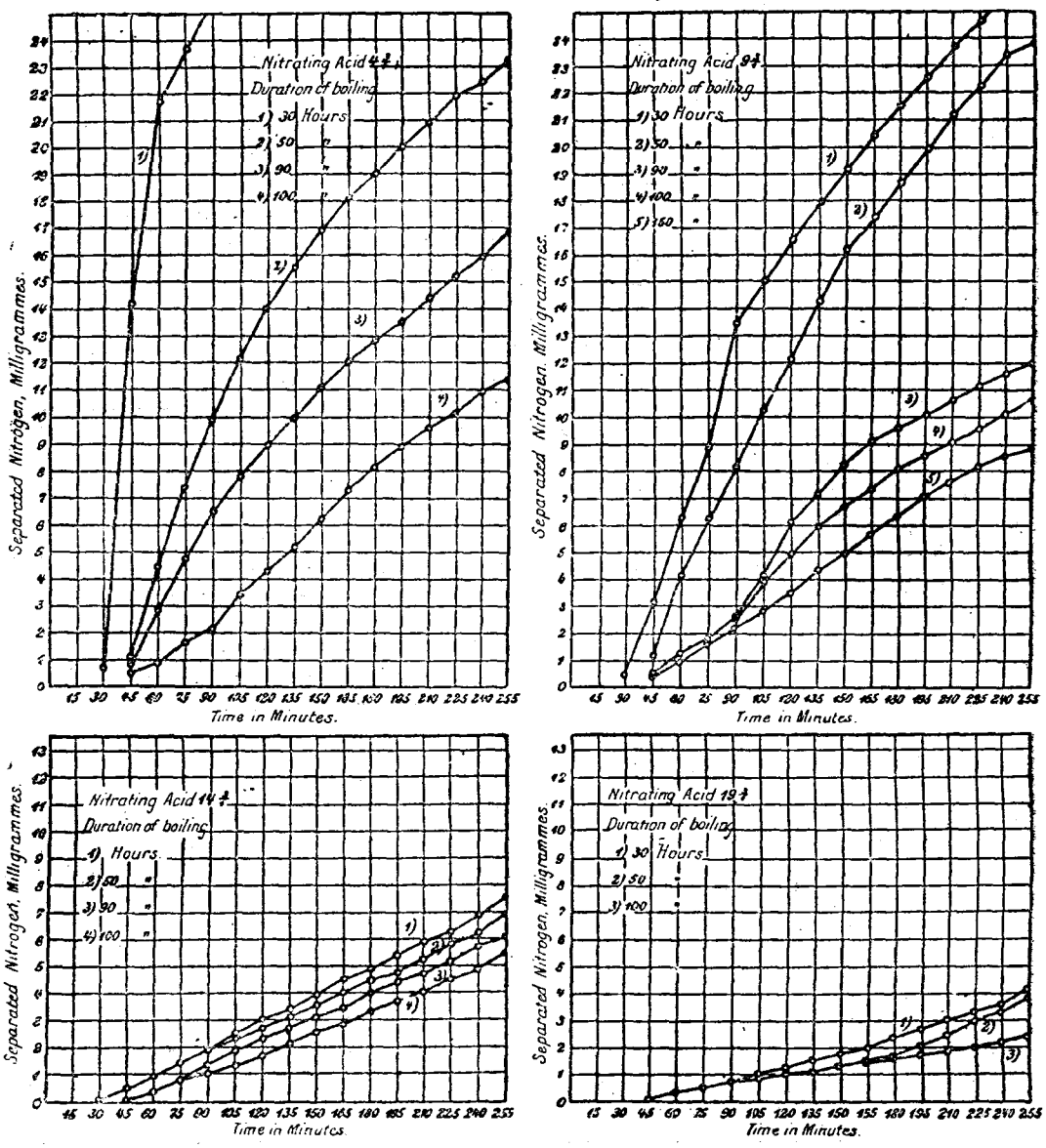
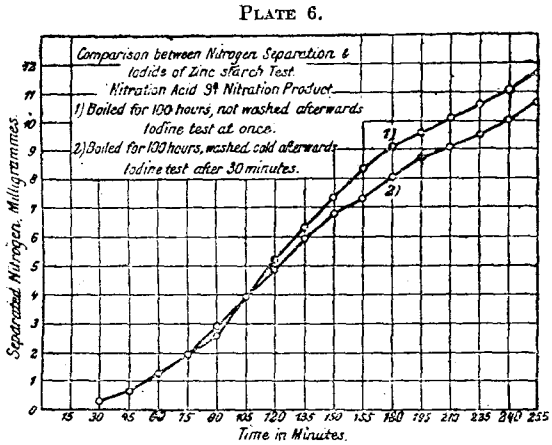


PLATE 6.



of decomposition caused by the use of different temperatures during testing, Plates 8, 9, 10; and (5) the regular decomposition of nitrocellulose purified to a certain limiting stage, the existence of which became evident in the course of this work, Plates 11, 12*a*, and 12*b*. The chief deductions are summarised under the following heads:—

Relation of Stability to Nature of Raw Materials used.—Brunswig has established by other methods, that the time of appearance of the first traces of separation products is a function of the nitrating acid. It is also clear that under similar conditions and with similar acid ratios, the speed of decomposition falls according as the amount of water used, *i.e.*, as the dilution of the acid is greater. In Plate 1, for example, the nitrating acid 4¼ containing 4 per cent. of water, produces a gun-cotton that easily explodes, the product of 19¼ acid, however, is highly stable.

Again, under similar conditions of dilution, the speed of decomposition depends on the relation between the acids.

PLATE 7a.

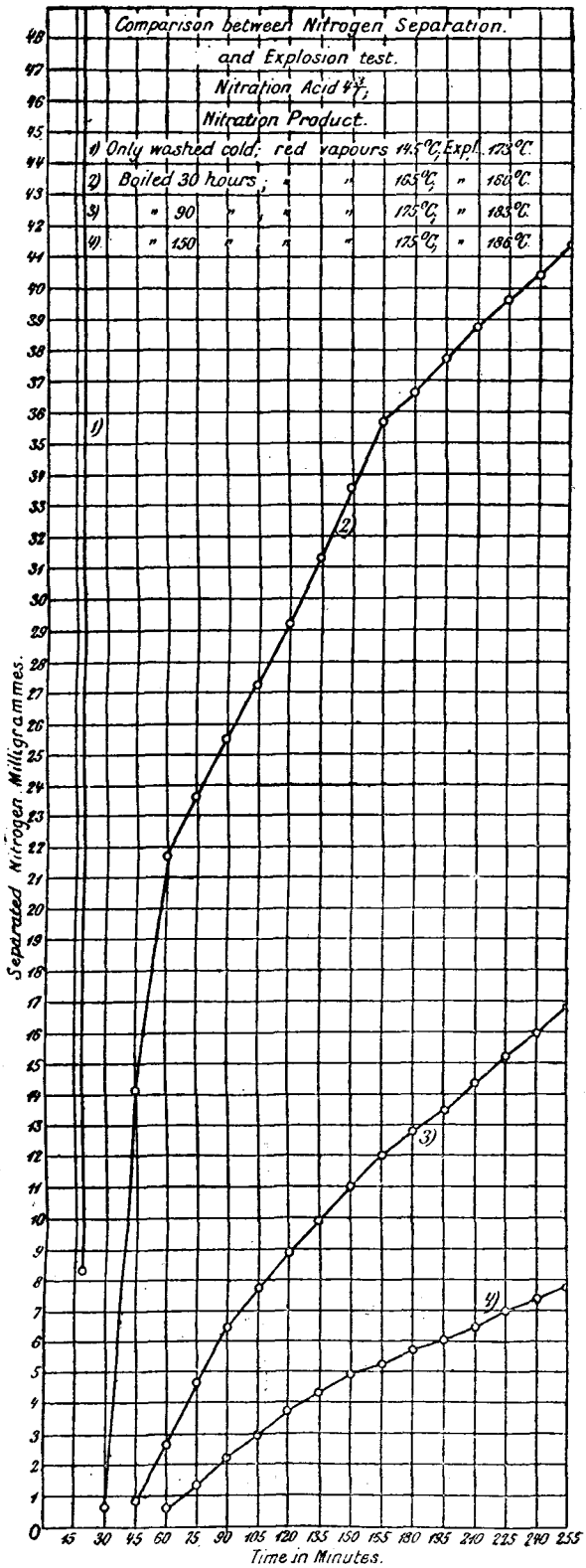


PLATE 7b.

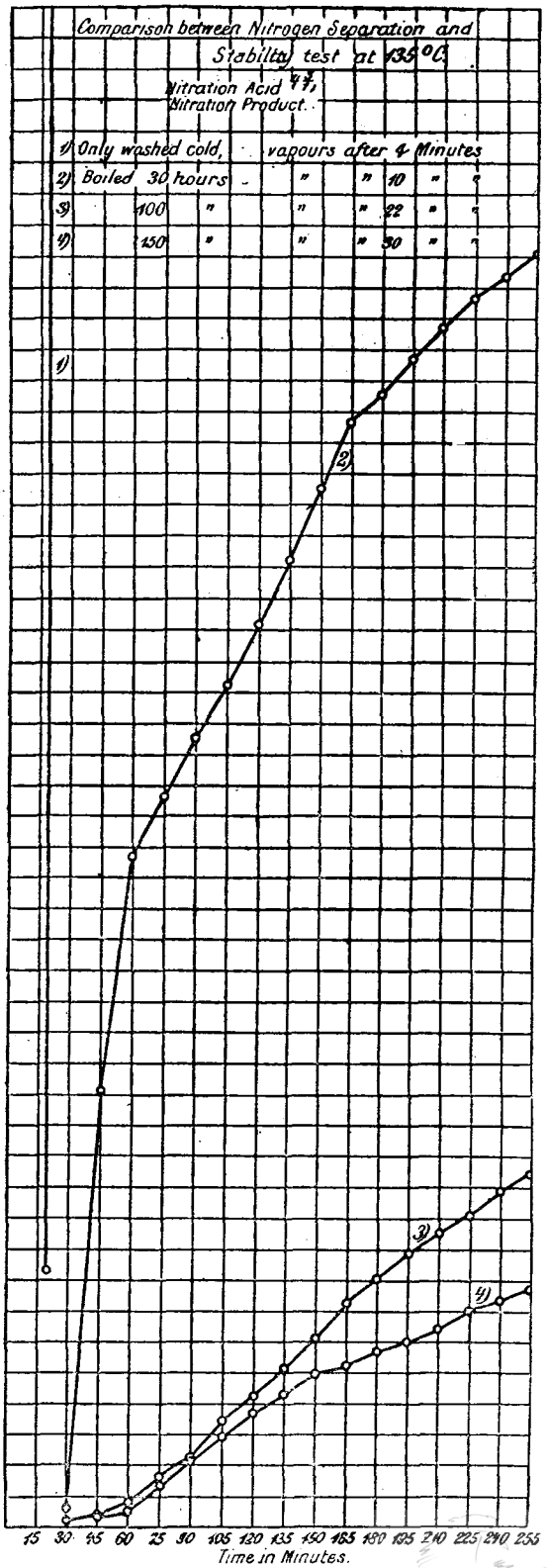


PLATE 8.

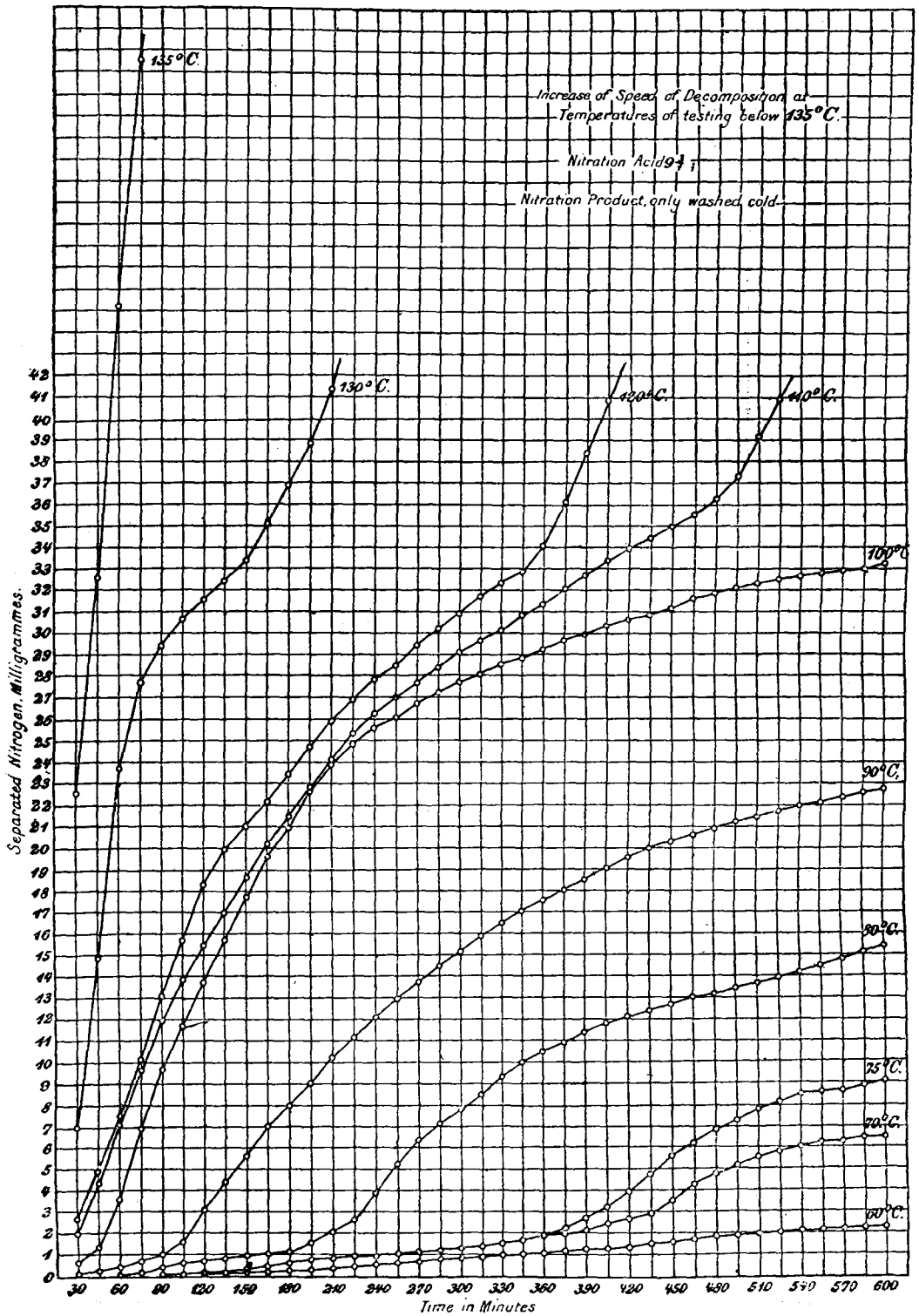
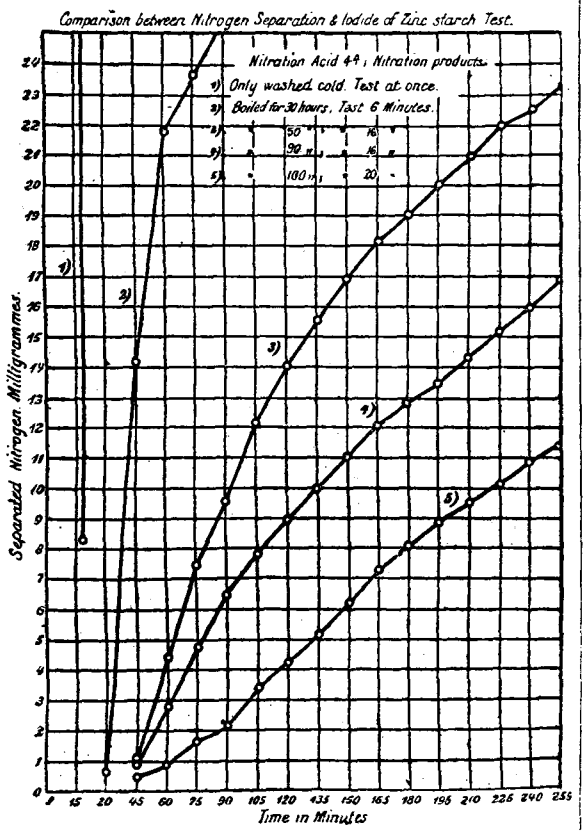

Increase of Speed of Decomposition at Temperatures of testing below 135° C.
Nitration Acid 9-1
Nitration Product, only washed cold
135° C.
130° C.
120° C.
110° C.
100° C.
90° C.
80° C.
75° C.
70° C.
60° C.
Separated Nitrogen. Milligrammes.
Time in Minutes

PLATE 5.



An increase of H_2SO_4 increases the speed, as is evident from Plate 2, where the steep curves have the acid ratio $\frac{3}{1}$.

Relation between Stability and Mode of Preparation.—Plates 3 and 4 indicate that insufficient washing lowers the stability. These experiments were conducted with boiled gun-cottons, and there appears to be a limit, beyond which boiling does not further increase the stability.

The relation of stability to the degree of comminution of the cotton was also investigated and found to be very slight, contrary to the general view that only finely divided cotton can be efficiently stabilised. Similarly the effect of adding small quantities of mercury or sublimate was found to be slight.

Relation between Stability as judged by the Quantitative Method and other Methods.—No definite relations were established between the speed of decomposition and (1) the actual nitrogen contents; (2) the solubility of the nitrocellulose in ether-alcohol (2 : 1); or (3) its viscosity.

Plate 5 shows that a nitrocellulose, which gives a slow reaction to the zinc-iodide-starch test also decomposes slowly. The iodide test, however, is largely influenced by the presence of impurities, and it may be markedly affected by washing the gun-cotton, whilst the speed of nitrogen evolution, under similar conditions, is not correspondingly altered (compare Plate 6). There is greater harmony between the quantitative method and the ordinary decomposition tests, as Plates 7*a* and 7*b* show clearly.

Results of the Quantitative Method at various Temperatures.—In Plates 8—12 are exhibited the results of varying the temperature of decomposition, in which experiments larger quantities of gun-cotton were employed.

In conclusion, the author considers that the best definition and test of a stable nitrocellulose, is that it should give off at a high temperature equal quantities of nitrogen in equal times. For the purposes of manufacture, it is specially important that the material should be purified to its limit, *i.e.*, the point at which further washing produces no further change in its speed of decomposition measured in the manner described.

PLATE 9.

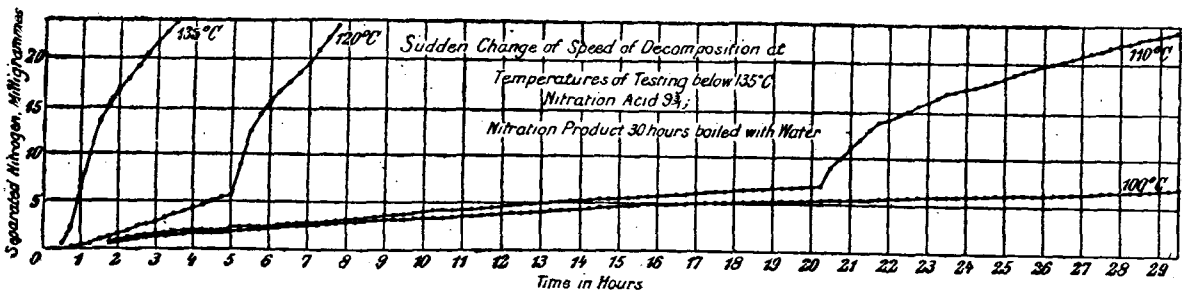


PLATE 10.

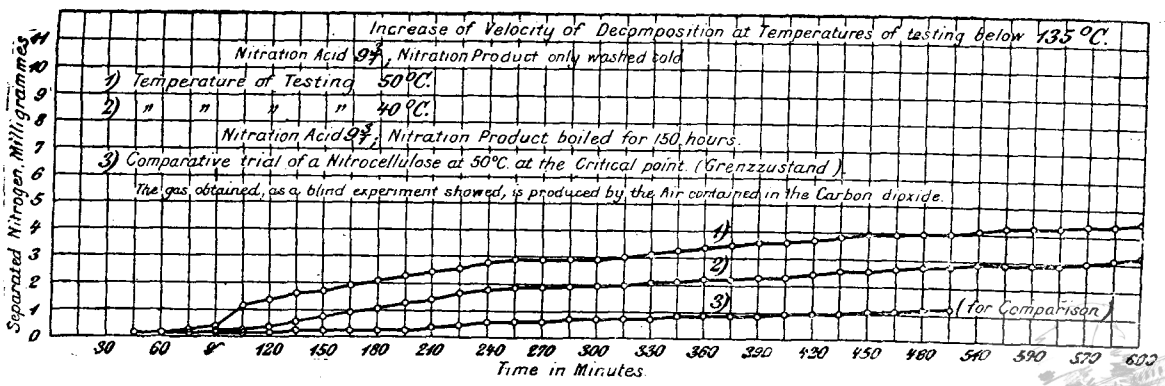


PLATE 12a.

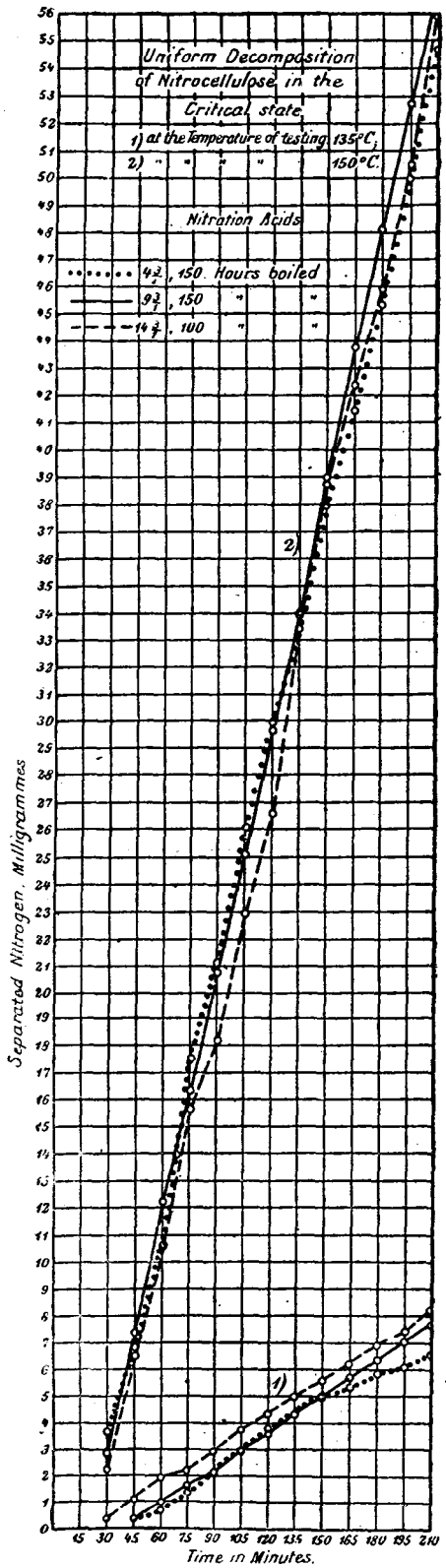


PLATE 12b.

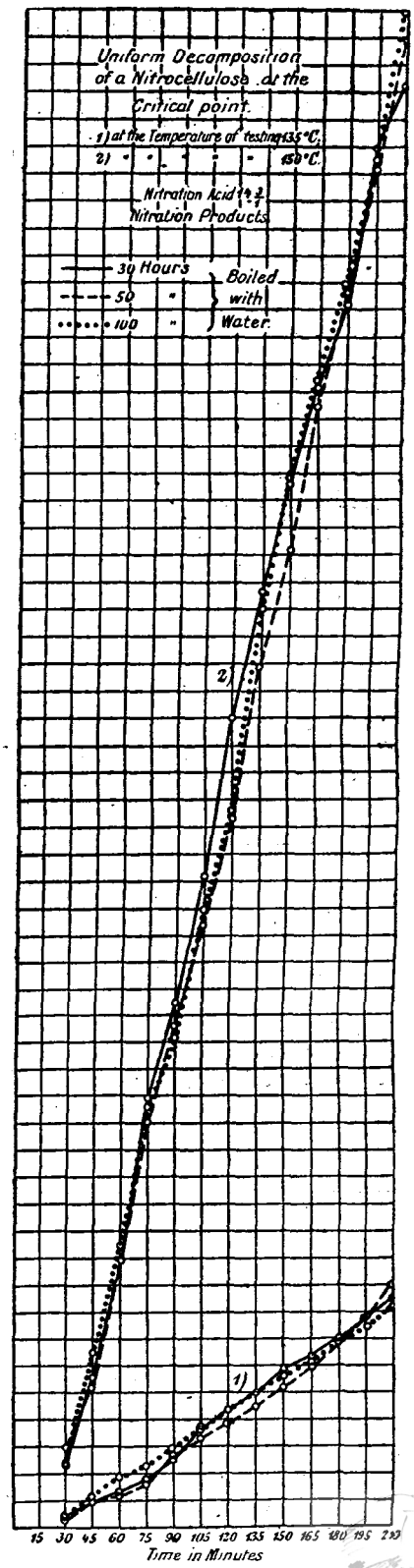
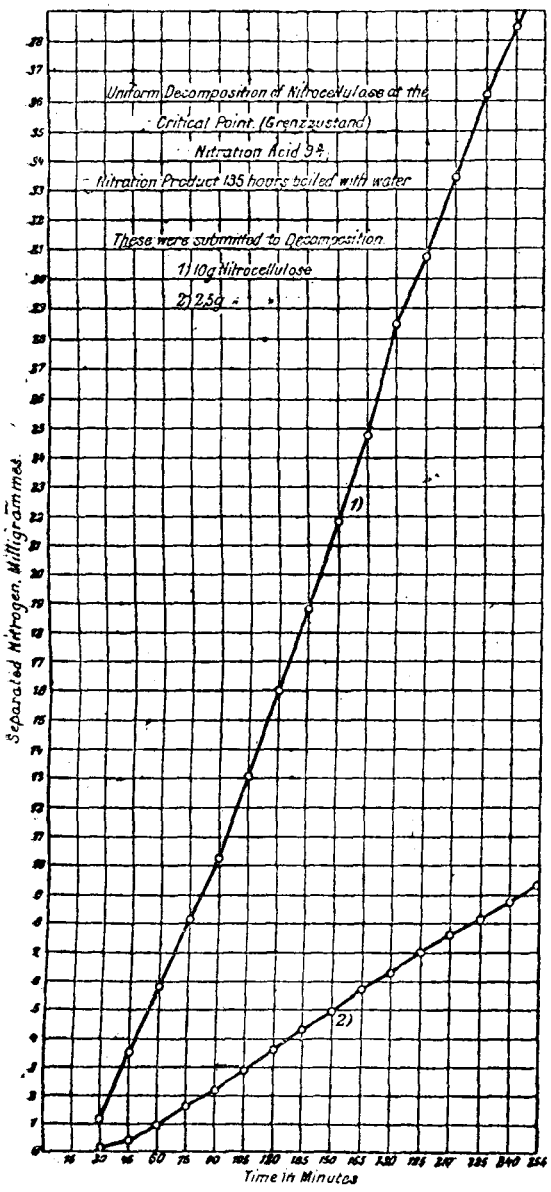


PLATE 11.



The influence of other manufacturing conditions which may impair or increase the stability will be considered later.—R. L. J.

Phosphorus; Ignition Temperature of ——. F. Eydmann, jun. Rev. trav. chim. Pays-Bas, 1900, **19**, 401, through Chem. Zeit. Rep. 1901, **25** [15], 129.

THE substance is placed under water in a test-tube of 30 mm. diameter; the test-tube is closed with a cork with three holes, carrying a thermometer and two tubes, one of which reaches nearly to the bottom of the tube. The test-tube stands in a larger vessel, filled with water, which can be heated gradually. During the experiment the water or melted phosphorus is gently agitated by passing a current of air or oxygen through the tubes. As soon as the phosphorus melts, the thermometer dips into it. According to several experiments, the ignition point of phosphorus is 45° C., whether the atmosphere of gas be air, pure oxygen, or oxygen diluted with carbon dioxide. The concentration of the oxygen has no effect upon the ignition temperature of phosphorus.—J. F. B.

Picric Acid; Process for Manufacturing ——. Wenghöffer.

See under IV., *page* 570.

PATENTS.

New Explosives. J. de Macar, Embourg, Belgium. Eng. Pat. 10,456, June 8, 1900.

A SERIES of new explosives is made by mixing lead nitrate with (1) derivatives of phthalic acid; (2) salts of derivatives of phthalic acid; (3) nitro-hydrocarbons such as dinitrobenzene or dinitronaphthalene; (4) azo-derivatives, such as azobenzene, amidoazobenzene, or diamidoazobenzene; (5) the combinations formed between the nitrous derivatives of the hydrocarbons and the azoderivatives; or (6) the nitrocelluloses. Mixtures containing lead nitrate and 17 per cent. of dinitroxylene, 10 of amidoazobenzene and 45 of dinitrocellulose respectively are quoted as examples.—E. W. W.

Smokeless Explosives or Colloids; Making ——. J. B. Bernadou, Philadelphia, U.S.A. Eng. Pat. 2253, Feb. 1, 1901. Compare Eng. Pat. 11,567, June 26, 1900 (this Journal, 1900, 851).

A COLLOID powder consisting of nitrocellulose insoluble in ether-alcohol and containing a higher percentage of oxygen than that of the pentanitrate, is claimed together with a process for manufacturing it by cooling a mixture of insoluble nitrocellulose and ether to a temperature below that required to freeze a mixture of 95 per cent. (by volume) absolute alcohol and water. At such a temperature the nitrocellulose is dissolved or colloided by the ether, and the residue, after drying, may be used as a smokeless powder or as a cementing agent for binding together other ingredients of such powders. Any ordinary form of closed mechanical mixer fitted with a cooling jacket can be used for gelatinisation and kneading.—E. W. W.

Explosives; Manufacture of ——. J. V. Skoglund, Brooklyn, U.S.A. Eng. Pat. 5231, March 12, 1901.

A METHOD of manufacturing an explosive mixture of salts containing a high percentage of ammonium nitrate, from sodium nitrate and ammonium sulphate. About equal weights of the commercial salts are dissolved in water, the solution cooled, and the crystals of sodium sulphate removed. To the mother liquor a compound such as calcium nitrate which forms an insoluble sulphate is added. After removing the precipitated sulphate, the liquid is evaporated and a residue containing a large proportion of ammonium nitrate obtained.—E. W. W.

Explosive Charges for Guns. R. W. Scott, Philadelphia, U.S.A. Eng. Pat. 5264, March 12, 1901.

THIS invention relates to a method of retarding the combustion of a gun charge by temporarily protecting a portion of the charge from ignition, by inserting a barrier of combustible material. The latter may be made of a plastic explosive substance moulded into an envelope or sheath, wholly or partly inclosing the charge, or may be used to coat the grains of the charge, or in a variety of other ways, according to the object to be attained. By this means, it is stated, the chamber pressure is greatly reduced, owing to the slowness of ignition, and the strains are distributed throughout the bore of the gun, since the projectile has traversed a portion of the barrel before the rapid combustion of the charge occurs.—E. W. W.

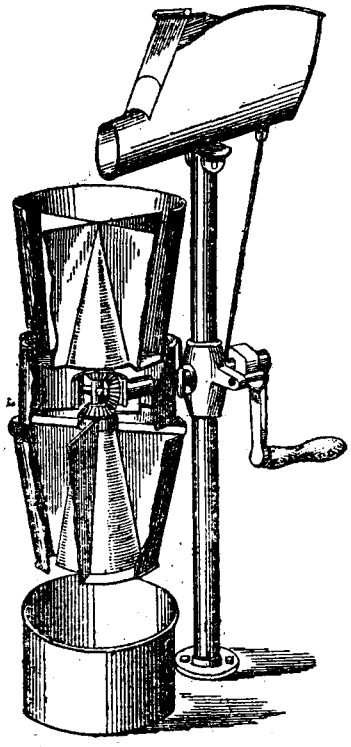
XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

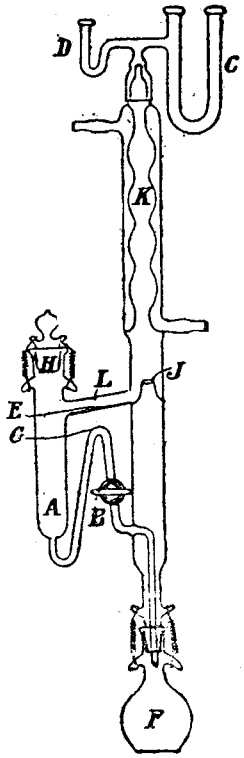
Ore Sampler; The Calkins Umpire ——. Eng. and Mining J. 1901, **71**, [17], 534.

IN the accompanying figure is shown a sectional view of the "Calkins umpire ore sampler," by means of which, it is claimed that 100 lb. of crushed ore can be reduced to an assay sample in 10 minutes, and smaller quantities of from 1 to 10 lb. can be accurately sampled to 4 oz. in 1 or 2

minutes. The machine is 3 feet 7 ins. high, and weighs 55 lb.; it can be easily and thoroughly cleaned. The upper and lower buckets are revolved in opposite directions by



means of gear wheels, and the hopper is continually agitated. Each bucket is divided into four divisions, two being closed and two open; therefore, if 16 lb. of ore be fed into the machine from the hopper, 8 lb. will remain in the upper bucket, 4 lb. in the lower bucket, and 4 lb. or one quarter of the original amount will pass into the receptacle at the bottom. By repeating the operation, the original sample may be reduced to $\frac{1}{16}$, $\frac{1}{64}$, $\frac{1}{256}$, &c., as desired.—A. S.



Fat-Extraction Apparatus. W. Jerwitz. Chem. News, 1901, **83**, [2164], 229.

The author refers to the difficulty, when using ether in a Soxhlet fat-extraction apparatus, of obtaining a tight joint between the condenser and extractor, and he claims that this disadvantage is overcome in the apparatus which he has devised and which is shown in the annexed figure. The two clamps of special construction which are provided for use with the apparatus, give a solid, safe, but not absolutely rigid grip. The stopper H and the connection between the condenser and the flask, F, are secured by means of spiral springs as shown. When using the apparatus, the cartridge containing the material to be extracted is placed in A, the stopcock B is closed, and ether poured into A, till it reaches to about E, after which the stopper H is inserted and secured by the springs. The siphon is set working by opening B, and the ethereal solution of the fat flows into F. The flask, F, is heated in a water-bath, in which it is allowed to hang free; it is advisable to govern the temperature of the bath by means of a thermo-regulator. The ether distils off through J, is condensed in K, and flows back through L into A. When A becomes again filled with ether up to G, the siphon acts and the ether flows into F. The process is repeated until extraction is complete, when the stopcock B, is closed, and the whole of the ether is distilled off into A. C is a calcium chloride tube to prevent entrance of moisture, whilst D which contains mercury acts as a safety valve, in the event of the calcium chloride tube becoming choked.—A. S.

INORGANIC CHEMISTRY.—QUALITATIVE.

Sodium; Use of ——, in Blowpipe Analysis. C. L. Parsons. J. Amer. Chem. Soc. 1901, **23**, [3], 159—161.

W. Hempel (Zeits. anorg. Chem. **16**, 22), first made use of metallic sodium as a reagent in qualitative analysis, but was unable to obtain satisfactory results on charcoal. By the following method, however, the author readily obtains a reduction. A small piece of sodium, 3 or 4 mm. in diameter, is hammered out flat, and the powdered substance to be reduced kneaded into it with a knife. The mass is placed on charcoal and ignited, the reduction being immediately complete. The residue is heated in the blowpipe flame, and as the sodium oxide and hydroxide sink into the charcoal, any fusible metallic particles collect into a button.

The reduction takes place with silicates, chlorides, carbonates, borates, sulphates, &c., as readily as with the oxides themselves, and in the case of minerals, the method gives results nearly as certain as with the pure salts of the metals.—C. A. M.

Cobalt; Some Reactions of ——. E. Donath. Zeits. anal. Chem. 1901, **40**, [3], 137—141.

If a few drops of a solution of a cobalt salt be added to concentrated potash lye (30 per cent. and over), there results, especially on heating, a more or less intense blue solution, although a portion of the cobalt hydrate always remains undissolved. The same reaction can be better observed by adding a piece of solid caustic alkali to a hot solution of the cobalt salt. The blue solution is also obtained on adding any of the cobalt compounds which are insoluble in water to the concentrated alkali. Being a solution of cobaltous hydrate, this blue solution absorbs oxygen from the air, and is decolorised with the formation of a brownish-black deposit on the walls of the test-tube; the change may also be effected by oxidation with hydrogen peroxide. The only other metallic oxide yielding similar solutions with the caustic alkalis is cupric oxide, but these solutions are permanent in the air. The blue solutions of the two metals can be differentiated by the addition of powdered Rochelle salt. The blue colour due to cupric hydrate is not affected, or, if anything, rather intensified, but the blue solutions of cobaltous hydrate are either decolorised, or, if concentrated, are changed to a reddish tint. Further, the addition of potassium cyanide decolorises the blue solutions of copper instantaneously, whilst the cobalt solutions are changed to a yellow, yielding an intense brown where they come in contact with the air. If complete solution of the cobaltous hydrate be desired, it may be obtained by adding glycerin to the caustic alkali employed; the blue solutions so obtained turn to a fine green on oxidation by the air or hydrogen peroxide. The cobalt in the glycerin-alkali solution is therefore in a different condition from that in the tartrate-alkali solution. In the author's opinion the solubility of the metallic hydrate in concentrated alkali and the characteristic behaviour with tartrates are due to differences in the equilibrium of the ions.—J. F. B.

Metallic Sulphides; Precipitation of ——, by Thiosulphate. E. Donath. Zeits. anal. Chem. 1901, **40**, [3], 141—143.

The author draws attention to the great utility of sodium thiosulphate in presence of mineral acid for the separation of copper as sulphide from cadmium and zinc. It may also be employed in place of sodium carbonate and sulphur for the decomposition of minerals containing arsenic and antimony in addition to metals of the copper group. If a solution of a cadmium salt be treated with an excess of ammonia and then strongly acidified with acetic acid, the addition of powdered thiosulphate to the boiling solution causes the precipitation of the whole of the cadmium mixed with sulphur after half an hour's boiling. With a zinc salt, on the other hand, under the same conditions, only a minimum quantity of the zinc is precipitated as sulphide. The separation of cadmium and zinc by this method is not quantitative, since cadmium sulphide carries down with it variable quantities of zinc sulphide. Similar relationships exist between nickel and cobalt; under the same treatment, with long continued boiling, nickel is entirely precipitated, but cobalt salts in the pure state yield no sulphide. But when nickel salts are mixed with cobalt, manganese, or iron salts, variable quantities of these metals are co-precipitated with the nickel sulphide. The method answers admirably for the rapid and easy detection of small quantities of nickel in presence of large quantities of cobalt.—J. F. B.

Selenium; Detection of ——, in Sulphuric Acid. Ad. Jouve. Bull. Soc. Chim. 1901, **25**, [3], 489—491.

Of the known methods for the detection of selenium, two only are sensitive. Firstly, the codeine test (morphine is equally applicable), which gives an intense blue coloration in a sulphuric acid solution of selenious acid, sensitiveness 1 in 200. Secondly, the method by which sulphur dioxide is passed into the sulphuric acid, diluted with four volumes of water; warm solutions should be used. The selenious acid is reduced, selenium being precipitated as the red modification; maximum sensitiveness 1 in 10,000. Selenic acid is only influenced with difficulty by sulphurous acid, and not at all in the codeine reaction. The author recommends as a test (sensibility 1 to 100,000), that crude acetylene should be passed through the solution under examination, when, if selenium be present, a more or less intense red coloration results, due to selenium in suspension. As a check there are two means:—Firstly, sulphuric acid containing free selenium in suspension, dissolves the latter on warming, with a green coloration. Secondly, the red liquid after the passage of C_2H_2, is diluted, and oxidised with permanganate, until there is a permanent rose coloration, excess of $KMnO_4$ is destroyed by sodium sulphite in excess, and the liquid is warmed. The permanganate changes the selenium to selenious acid, then the sodium sulphite brings it back to the state of selenium, and, on warming, very light red flakes of selenium are obtained, which can be collected and identified by the odour evolved on ignition, or by their solubility in alkalis, and by suspension in sulphuric acid and warming, producing the green colour as above. The author adds that the sensitiveness as regards rapidity of production of red colour is increased if the acetylene contain vapour of hydrochloric acid.—C. T. T.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Sulphuric Acid; Determination of Water or of Sulphur Trioxide in Concentrated or Fuming ——. H. Rabe. Chem. Zeit. **25**, [32], 345—346.

If ordinary concentrated sulphuric acid be added to fuming acid, the latter ceases to fume when the water in the concentrated acid added is exactly equivalent to the sulphur trioxide in the fuming acid.

To assay concentrated sulphuric acid, a sample of known composition, approximating to that of the sample to be tested, (say containing 5 per cent. of H_2O) is added from a burette to 25 c.c. of fuming acid, carefully shaking and blowing out the fumes after each addition, till the point is just reached at which no more fuming occurs. The experiment is repeated with the sample of acid to be tested. The percentages of water are inversely as the amounts of the acids used, so that if 24·8 c.c. of the known acid and 30·5 c.c. of the unknown acid were required, the latter contained $24{\cdot}8 \times 5 \div 30\ 5 = 4{\cdot}07$ per cent. of water.

By a precisely similar process, running concentrated acid from a burette successively into equal quantities of a known and an unknown sample of fuming acid, the percentage of SO_3 in the unknown sample can be determined.

The process is easy and rapid, and is not affected by coloration or turbidity of the samples. For works purposes, it is sufficient to compare the volumes run out of the burette, or equal volumes of the fuming acids, as the differences in density are usually small; but the densities can be determined and calculations by weight made where necessary. Great care must of course be taken to preserve the known standard acids from possible alterations of composition.

—J. T. D.

Nitric Acid in Alkali Nitrates: Detection and Determination of ——. E. P. Perman. Chem. News, 1901, **83**, [2161], 193.

If an alkali nitrate be heated gently with certain sulphates, for instance, with lead sulphate or ordinary alum (anhydrous), the sulphate of the alkali metal is formed, and red fumes of nitrogen peroxide are evolved. In applying the method quantitatively, the alkali nitrate is heated with anhydrous potash alum, at first very gently, and finally to a low red heat for one or two minutes. The residue consists of alkali sulphate, together with alumina and excess of alum, nitrogen peroxide and oxygen having been expelled. The loss in weight may be calculated as N_2O_5.

The author has obtained the following results:—Percentage of NO_3 in KNO_3:—Found 61·5, 61·2, and 61·3; calculated, 61·4.—A. S.

Nitrates in Waters; Determination of ——, by Stannous Chloride. H. Henriot. Comptes Rend. **132**, [16], 966—968.

The author confirms quantitatively Divers and Haga's statement that stannous chloride in strongly acid solution converts nitric acid into hydroxylamine. He applies this reaction to the determination of nitrates in waters, as follows:—Prepare (1) a standard solution of stannous chloride by dissolving 14 grms. of tin in hydrochloric acid, making up with strong hydrochloric acid to a litre. Preserve this in a flask fitted with a two-holed cork, one tube through which communicates with a carbon dioxide generator, and the other, a siphon tube, communicates, through a rubber tube and pinchcock, with a 10 c.c. pipette or burette, the upper portion of which also communicates with the carbon dioxide generator. Measured quantities of this solution can thus be delivered without access of air. The vessel should be shaken before use, and the first 10 c.c. should be rejected. Prepare (2) a standard iodine solution by dissolving 8 or 9 grms. of iodine and 20 grms. of potassium iodide in a litre of water. Standardise, either by thiosulphate or by solution of potassium nitrate, as below. For analysis, place 50 c.c. of the water in a 125 c.c. flask, and evaporate to dryness on the sand-bath at 110° C. Cool, add 10 c.c. of hydrochloric acid and 10 c.c. of the stannous chloride solution, close with a cork through which passes a glass tube about 10 cm. long, having an equal length of rubber tube on its outer end; boil vigorously for 10 minutes under a hood. Meanwhile proceed in the same way with a blank experiment. Remove the flask, pinch the rubber tube, connect it with a carbon dioxide generator, and allow to cool; then add 10 c.c. of water, a few drops of starch paste, and titrate with the iodine. The difference between the sample and the blank represents the stannous chloride oxidised by the nitrates: 14 parts, by weight, of nitrogen are equivalent to 6 × 127 parts of iodine. Experiments with known amounts show always a slight loss of nitrogen, which is, however, negligible for waters containing not more than 1·5 mgrm. of nitric nitrogen per litre. Organic matter does not affect the results, but any iron should be removed by ammonia before evaporating.

—J. T. D.

Sulphur; Determination of ——, *in Wrought Iron and Steel.* G. Auchy. J. Amer. Chem. Soc. 1901, **23**, [3], 147—151.

A SOURCE of error in the analysis of iron and low carbon steel is that sulphur may escape during the solution of the sample in strong nitric acid. Thus, for example, an iron which, when dissolved in *aqua regia* in the ordinary way showed 0·006 per cent. of sulphur, gave 0·012 per cent., when the solution was made very slowly. In evolution methods there is a further source of error due to the escape of part of the sulphur in combination with the carbon. It is shown by a series of comparative results obtained by the *aqua regia* method of slow solution, and the cadmium chloride evolution method, that the latter is sometimes accurate and sometimes not, and that it is therefore not possible to find a universally true correction. The author considers it best to note the greatest error in a series of tests, and to apply one-half of this amount as a correction.

The presence of iron oxide in the barium sulphate is a difficulty in the *aqua regia* method when the precipitation is made from a solution not containing much free acid. Arnold's method of cold precipitation has given low results in the author's hands, whilst Blair's method of having only 5 c.c. of hydrochloric acid in the liquid also gave figures from 0·004 to 0·008 per cent. too low. By having this amount of acid (5 c.c.), boiling for 30 minutes, and allowing the liquid to stand over night, the author found that no iron was precipitated, and that the precipitation was as complete as in Blair's method. In all cases, however, he recommends the addition of at least 0·002 per cent. to the result.

Experiments are described to show that although *aqua regia* is necessary to dissolve high carbon steels, the hydrochloric acid must be sparingly used or the percentage of sulphur will be too low.—C. A. M.

Bismuth in Ores; Determination of ——. A. W. Warwick and T. D. Kyle. Eng. and Mining J. 1901, **71**, [15], 459.

THE authors state that the methods given in text-books for the determination of bismuth in ores, are either inaccurate or clumsy and laborious. They propose a modification of the method devised some years back by Pattison Muir, and claim that it is simple, accurate, and rapid (a determination can be completed in 40 minutes). 1 grm. of the finely crushed paste is treated with 5—10 c.c. of concentrated nitric acid, the solution evaporated to dryness, the residue broken up, heated for a short time with 5 c.c. of nitric acid, then 25 c.c. of water added, and the whole rinsed into a beaker, and made up to about 100 c.c. with hot water. 5 grms. of ammonium oxalate or oxalic acid are added, the solution boiled vigorously for a few minutes, and the precipitate washed twice by decantation with boiling water, the washings being passed through a filter. The final wash water should be neutral to litmus, indicating that the bismuth oxalate has been completely converted into basic oxalate. The precipitate on the filter is dissolved in 5 c.c. of hydrochloric acid (1 : 1), and the filter washed twice with water, the liquid in each case being run into the beaker containing the main portion of the precipitate. The precipitate is dissolved by gentle warming, the solution made up to 250 c.c. with hot water, then neutralised with ammonia, and the precipitate re-dissolved by the addition of a slight excess of sulphuric acid (1 : 4). The solution is finally titrated at 70°—100° C. with standard permanganate solution. Lead, iron, copper, zinc, arsenic, and tellurium have no influence on the results, which are stated to be accurate within 0·1 per cent.—A. S.

Bismuth by Electrolysis; Quantitative Determination of ——. K. Wimmenauer. Zeits. anorg. Chem. **27**, 1—21; Chem. Centr. 1901, **1**, [19], 1066.

THE author finds that the methods hitherto proposed for the direct electrolytic determination of bismuth do not give satisfactory results. He proposes the following method:—As anode, a bucket electrode is used with a total surface of about 70 sq. cm. and which is so arranged that it can be rotated by a small water turbine. Quantities of bismuth up to about 0·4 grm. can be separated with sufficient accuracy and certainty, if the solution contain 1—0·5 c.c. of concentrated nitric acid per 0·1 grm. of Bi_2O_3, and is electrolysed by a current of at least 0·05 ampère, the electrolyte being kept in motion. The time occupied is 3—4 hours; the temperature is raised to 50° C. and maintained at that point till the electrolysis is finished. The tension of the current is always 2 volts. The length of time taken for complete electrolysis may be reduced by using a higher current strength, say 0·1 ampère, at the beginning, but so soon as the precipitate begins to darken in colour, the current strength must be reduced to 0·05 ampère and maintained thereat till the finish. If the electrolyte is not kept in motion, superoxide is formed. Before cutting off the current, the deposited bismuth is washed with pure water, and rinsed with alcohol and ether.

If bismuth nitrate be the substance under examination, glycerin may be used with advantage as a solvent; 0·1—0·3 grm. of bismuth nitrate is dissolved in 2—4 c.c. of a liquid consisting of 2 parts of water and 1 part of glycerin, the solution is diluted to 150 c.c. and electrolysed in the manner described above.—A. S.

Vanadium in Slags and Cinders; Determination of ——. C. H. Joüet. School of Mines Quarterly, 1901, **22**, [2], 143.

THE author gives a description of Pope's method (Trans. Amer. Inst. Mining Eng. **1899**, 372). A mixture of 10 grms. of the finely powdered slag, 50 grms. of sodium carbonate, and 6 grms. of sodium nitrate is fused, a small portion at a time, in a platinum crucible, and the fused mass heated for 1 hour to a very high temperature, by two blast lamps. The melt is digested in boiling water, and the residue again fused, &c., in order to effect the complete separation of the vanadium. The united aqueous extracts are treated with alcohol to reduce the manganese, then almost neutralised with nitric acid, and boiled to expel carbon dioxide. The precipitate of alumina and silica may retain some chromium and vanadium. It is filtered off, evaporated with sulphuric and hydrofluoric acids, the residue fused with sodium carbonate and the aqueous extract of the melt nearly neutralised with nitric acid, boiled for a short time, and filtered. The filtrate is added to the main one, and the whole made slightly acid with nitric acid, then alkaline with a few drops of sodium carbonate, boiled for a few minutes and filtered, and the filtrate treated with barium nitrate. The precipitate formed consists of barium vanadate and carbonate, together with chromate and phosphate, if the corresponding elements are present. It is collected on a filter paper and the paper, with its contents, digested for some time with dilute sulphuric acid. The barium sulphate is filtered off, the filtrate concentrated to about 100 c.c., then made alkaline with ammonia and heated to induce the formation of ammonium metavanadate. The solution is then nearly saturated with solid ammonium chloride, and 200 c.c. of a mixture of equal volumes of alcohol and ether added, and the whole allowed to stand for several hours in a cool place. The ammonium metavanadate which crystallises out, is filtered off, and washed, first with an ammoniacal solution of ammonium chloride containing some alcohol, and finally with alcohol-ether. The precipitate is dried, ignited, treated with two or three drops of nitric acid, again ignited and weighed. In order to obviate any error due to contamination of the precipitate with small amounts of phosphorus, chromium, or tungstic acid, the vanadic oxide, V_2O_5, is dissolved in sulphuric acid (1 : 1), the solution diluted and reduced with sulphurous acid. The excess of sulphur dioxide is expelled by boiling in a current of carbon dioxide, and the V_2O_4 re-oxidised by titration at 70°—80° C. with N/100 permanganate.—A. S.

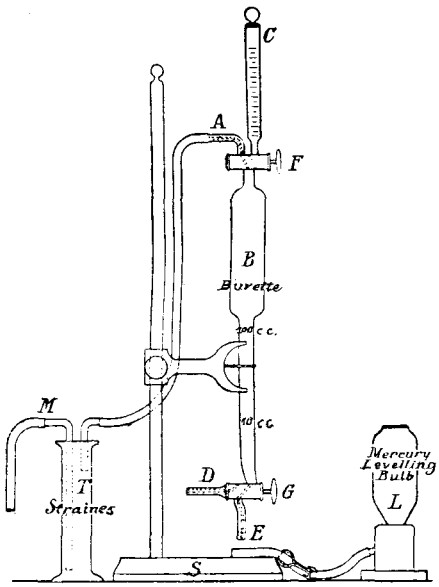
Gold Chloride; New Method of Titrating ——. H. Reeb. Chem. Zeit. Rep. 1901, **25**, [16], 144. From Phot. Mittheil. 1901, **38**, 128.

A 1 PER CENT. solution of gold chloride and a 0·1 per cent. aqueous solution of sodium thiosulphate are prepared. About 3—6 drops of saturated potassium iodide solution

are then added to 1 c.c. of the gold chloride, producing a deep brown colour, indicating the liberation of iodine. The brown colour is then discharged by means of the standard thiosulphate solution, added drop by drop, the volume of the latter required for the purpose being accurately read off. The number of c.c. of thiosulphate used multiplied by four gives the titre of the gold chloride in percentages. In order, for example, to decolorise 1 c.c. of the one per cent. gold chloride solution, 10 c.c. of the 0·1 per cent. solution of thiosulphate are used; the titre of the chloride will then be :—10 c.c. × 4 = 40 per cent., *i.e.*, 100 grms. of the chloride contain 40 grms. of pure gold.—W. G. M.

Hydrogen Sulphide; Determination of ——, in Illuminating Gas. C. C. Tutwiler. J. Amer. Chem. Soc. 1901, **23**, [3], 173—177.

THE apparatus shown in the figure was devised by the author for testing the progress of purification in gasworks. The method employed is based on the well-known reaction, $H_2S + I_2 = 2HI + S$.



The apparatus consists of a burette, in communication with a 10 c.c. stoppered cylinder, graduated in tenths of a c.c. On the burette itself there are only two marks, one at 100 c.c. and the other 50 mm. from the bottom. A wash bottle, containing cotton, is placed between the gas supply and the burette to arrest tar.

In making a determination, the taps are turned so that A and E communicate with the burette. The gas supply is connected with A, or, if it be crude gas, with M. After a few minutes the bottom tap G is turned so as to close the burette. The levelling bulb L is attached to E, with the usual precautions, the tap G opened, and the mercury brought to the 100 c.c. mark. The lower tap is now closed and the top one (B to A) momentarily opened to bring the gas to atmospheric pressure. The mercury is next withdrawn to G, the burette disconnected, and about 5 c.c. of starch solution drawn in through A. A standard solution of iodine is then placed in C, and allowed to enter the burette drop by drop, with a shake after each three or four drops. This is continued until the starch becomes permanently blue. The difference between the original quantity of iodine solution in the cylinder and the amount used corresponds to the hydrogen sulphide in the gas. The strength of the iodine solution should be about 0·0017076 grm. of iodine in 1 c.c. One c.c. of this will then be equivalent to 100 grains of hydrogen sulphide in 100 cb. ft. of gas.

Three grains of H_2S in 100 cb. ft. of gas can be detected by means of the apparatus.—C. A. M.

ORGANIC CHEMISTRY.—QUALITATIVE.

Dyestuffs; Detection of ——, Application to Indophenols. C. Camichel and P. Bayrac. Comptes Rend. **132**, [14], 1901, 882—885.

THE method employed depends upon the absorption of light by solutions of the indophenols in alcohol, ether, carbon bisulphide, and chloroform. Taking wave lengths as abscissæ and the transmission coefficients as ordinates, the authors obtain parabolic curves convex towards the axis of the abscissæ. The branch of the curve corresponding to the red rays rises more rapidly than that corresponding to the green and blue. The minimum of transparency is given by the lowest point of this curve and has been determined for a number of substances. This value is independent of the concentration for all substances of which the absorption coefficient is proportional to the concentration according to Beer's law, but it varies with the solvent according to a law which is, however, different from that enunciated by Kundt. Two series of indophenols have been investigated, one containing two tertiary nitrogens, as for example $O:C_6H_4:N.C_6H_4.N(CH_3)_2$, the other in which the second tertiary nitrogen is replaced by a primary, $O:C_6H_4:N.C_6H_4.NH_2$. The authors have determined the effect of substituting a primary for a tertiary nitrogen and of substituting certain radicles (CH_3, C_2H_5, $(CH_3)_2:CH$, $CH_3.CH_2.CH_2$) in the *o*- or *m*-position in the original phenol. They arrive at the following conclusions :—1. The replacement of a tertiary by a primary nitrogen whatever the solvent, causes a displacement of the minimum of transparency towards the most refrangible end of the spectrum. 2. An ortho substituent in the phenol from which the indophenol is derived, whatever the solvent, causes a displacement which may exceed the preceding. 3. A meta substituent causes a very slight displacement. 4. The method proposed by the authors is applicable for the detection of dyestuffs of which the absorption coefficients follow Beer's law. In the case of blood it is possible to determine the position of the first black band, the error not exceeding 1/10th of the distance between the two sodium lines. With the help of the first two laws it is possible to determine the formula of a phenol, only a very small quantity being required for the test. It is only necessary to convert the product into an indophenol with a primary or tertiary nitrogen and to examine its alcoholic solution.

—T. A. L.

Griess' γ-Diaminobenzoic Acid; a Reagent, with its Compounds, for Identifying Sugars, &c. B. Schilling Ber. 1901, **34**, [6], 902—907.

ALTHOUGH useful as a reagent for characterising sugars phenylhydrazine does not offer a good means of identifying maltose and isomaltose in the products of the hydrolysis of starch. For this purpose, γ-diaminobenzoic acid [$NH_2:NH_2:CO_2H = 1:2:3$] was suggested by Griess who, in conjunction with Harrow, described condensation products formed by this acid with various sugars. These compounds, which were stated to be well crystallisable, and hence readily purified, have both an acid and a basic character, are optically active in both acid and alkaline solution, and do not reduce Fehling's solution. The author gives a method for preparing the acid, which is, however, attended with considerable difficulty, and has examined the action of it on glucose, maltose, isomaltose, and lactose. With glucose and maltose the results obtained agree with those of Griess and Harrow, but the author regards the condensation products as benzimidazol derivatives, since by the action of potassium permanganate, the sugar residues are split off and benzimidazol carboxylic acids are formed. With isomaltose, no solid product was obtained but the existence in solution of a condensation compound was shown by the action of permanganate. Lactose and γ-diaminobenzoic acid yield a compound forming small pyramidal crystals melting at 206° C.

Against the general applicability of this acid as a reagent for sugars are its difficult preparation as well as the bad yields of the condensation products, which the author finds are not readily purified. It may, however, in particular cases, be made use of, as the melting points of the products

are in general more constant than those of the phenyl osazones.—T. H. P.

"Saccharin" (Benzoylsulphonic Imide); New Reaction of ——. A. Leys. Comptes Rend. **132**, [17], 1056—1058.

EVEN very dilute solutions of "saccharin" give, when treated with dilute copper sulphate or ferric chloride and hydrogen peroxide, a violet coloration. To 5 c.c. of a solution of saccharin (say 0·01 per cent.) there are added two drops of ferric chloride solution made by diluting 2 c.c. of solution of density 1·075 to 100 c.c. with water, and 2 c.c. of hydrogen peroxide solution made by diluting 1 c.c. of the 10-volume solution to 200 c.c. with water; the colour developes in 30 or 35 minutes.

Saccharin in Milk.—To 100 c.c. of 10 per cent. solution of potassium bisulphate add 10 c.c. of absolute alcohol, and into 100 c.c. of this liquid pour 50 c.c. of the milk; filter from the precipitated casein and fats, and shake the clear liquid with ether. Separate, evaporate the ethereal solution, and dry at 90° C. Take up the residue with 5 c.c. of boiling water, allow to cool, and taste; if it tastes sweet, examine it for saccharin as above.

Saccharin in Butter.—Dissolve the butter in a mixture of equal volumes of chloroform and absolute alcohol; add twice the volume of water, shake, allow to settle, and separate. The separated chloroform retains the fats; the aqueous-alcoholic solution contains the saccharin. Evaporate the latter, and test as above.—J. T. D.

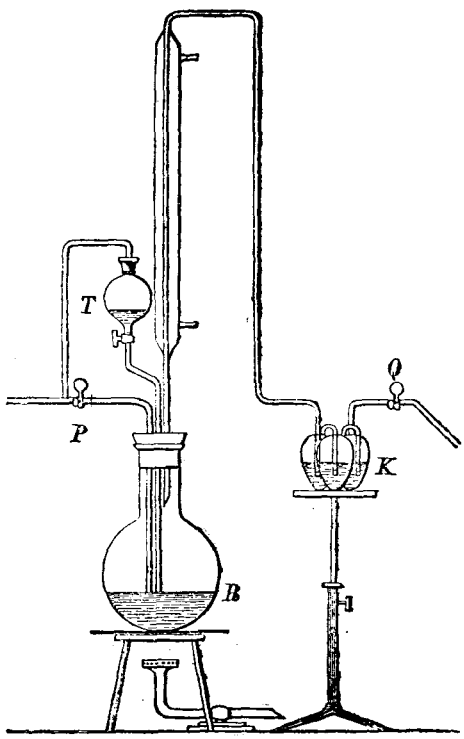
ORGANIC CHEMISTRY.—QUANTITATIVE.

Nitroso Group in Organic Compounds; Determination of ——. R. Clauser. Ber. 1901, **34**, [6], 889—895.

THE method consists in warming a solution of the nitroso-compound and phenylhydrazine in glacial acetic acid when benzene is formed and nitrogen is given off, two atoms of the latter for each nitroso group, according to the following typical equation,

$$R.NO + C_6H_5NHNH_2 = R.N: + C_6H_6 + H_2O + N_2.$$

The estimation is carried out in the apparatus shown in the figure.



The 300 c.c. flask R is provided with a three-hole cork carrying a condenser, tap funnel T, and a tube provided with a stop-cock P, and is connected with a carbon dioxide gas supply. The tube connected with the top of the tap funnel is for the purpose of preserving uniformity of pressure. The upper end of the condenser is connected to a Liebig's bulb apparatus K, containing water, and by means of a three-way tap Q, to a nitrometer tube. In order to make use of the apparatus, 0·1 to 0·2 grm. of the nitroso compound is dissolved in 20—30 c.c. of glacial acetic acid in the flask. After the whole apparatus is filled with carbon dioxide gas by passing a current through for several hours, a quantity of phenylhydrazine (4—5 times the theoretical quantity) dissolved in 30—40 c.c. of concentrated acetic acid is run into the flask by means of the tap funnel. The current of carbon dioxide gas is stopped and the flask is gently warmed, when a vigorous evolution of gas takes place, and the colour of the solution turns red. The reaction, except in the case of substances sparingly soluble in acetic acid, is complete in about 10 minutes. The nitrogen remaining in the apparatus is expelled by passing carbon dioxide gas through for five minutes, until no further increase in the volume of the nitrogen takes place. After standing 1—2 hours, the gas is transferred by means of the three-way tap Q to a eudiometer. A few drops of benzene are added and the nitrogen then is completely saturated with water and benzene vapour, when its volume is read off in the usual way. If P be the percentage of nitroso groups in the substance, V the volume of nitrogen in c.c., w the sum of the tensions of water and benzene at $t°$ in mm., g, the weight of the substance in grammes, b, the barometric height, s, the weight of 1 c.c. of nitrogen at 0° C., and 760 mm, then—

$$P = \frac{3000\, s\, V\, (b - w)}{760 \times 28\, (1 + \alpha t)\, g}$$

For substances soluble in glacial acetic acid, the method is accurate within 0·5 per cent, but in the case of insoluble substances in which the reaction proceeds very slowly, a 2 per cent. error has been observed.—T. A. L.

Nitro Group; Applicability of a Volumetric Method for the Determination of the ——. P. Altmann. J. prakt. Chem. 1901, **63**, [6, 7, 8], 370—380.

THE method is based on the ready and complete reduction of the NO_2 group by acid stannous chloride solution. The following solutions are used:—(1) Stannous chloride: 150 grms. of tin are dissolved in strong hydrochloric acid, the solution decanted off, mixed with 50 c.c. of hydrochloric acid, and made up to 1,000 c.c. The solution of the tin should not be accelerated by platinum chloride; variable results would then be obtained. (2) Rochelle salt: 90 grms. of anhydrous sodium carbonate and 120 grms. of Rochelle salt dissolved to 1 litre. Also filtered starch solution, decinormal iodine solution, and standardised permanganate.

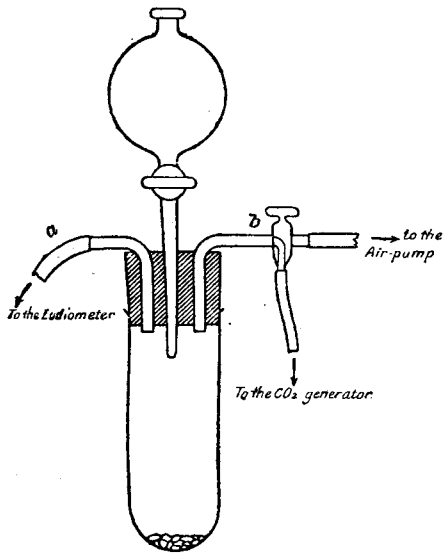
0·2 grm. of the material, chemically pure and carefully dried, is weighed into a 100 c.c. flask and warmed with 10 c.c. of the stannous chloride solution. The solution is then quickly cooled and made up to the mark; then 10 c.c. are withdrawn, and the Rochelle salt solution added until the precipitate just redissolves, water and starch are added, and the liquid titrated with iodine solution. Good results were obtained with nitrobenzene sulphonates, *m*-nitrobromobenzoic acid, barium dinitrobenzene sulphonate. An end-reaction cannot be recognised in the case of nitrophenols and nitronaphthalene; good results were, however, obtained by titrating with permanganate instead of iodine, the stannous chloride being standardised against the permanganate; *o*-nitrophenol, *m*-dinitrobenzene, potassium nitrotribromobenzene sulphonate, ammonium nitrodibromobenzene sulphonate, *m*-nitrobenzene sulphonamide, &c. gave favourable results by this method. In the case of nitraniline, the reduction must be effected by heating for several hours in a closed tube in the water-bath. The method is not applicable to picric acid, nitronaphthalene, and similar compounds, the amines obtained from which are oxidised by the permanganate.

In practice, iodine solution is used first; if this does not give a sharp end-reaction, permanganate is tried, employing Rieth's modification with ferric chloride (Volumetric,

p. 290). If no end-reaction is then obtained, ordinary methods must be employed.—A. C. W.

Diazo-Nitrogen in Diazo-Amino Compounds; Apparatus for the Determination of ——. H. Mehner. J. prakt. Chem. 1901, **63**, [6, 7, 8], 304—307.

GOLDSCHMIDT and Reinders decomposed diazo-amino-benzene by boiling with dilute sulphuric acid in a small flask, from which the air had been expelled by carbon dioxide, and collected the nitrogen over caustic soda in a eudiometer (Ber. **29**, 1369). During the long time required to replace the air completely by carbon dioxide, diazo-amino compounds are transformed to some extent into amido-azo compounds. The author has devised a simple apparatus in order to obviate this small error; the substance is not brought into contact with the acid until the evolution of nitrogen may commence, thus strong hydrochloric acid may be used, and a more rapid reaction obtained. Under these conditions amido azo compounds are not produced.



A thick-walled test tube, 10—12 cm. long, and 3 cm. in diameter, is closed by a rubber stopper, through which pass two glass tubes, and the stem of a tap funnel. The former are cut off close to the stopper, the latter is drawn out to a point. The tube *b* connects by means of a 3-way cock either with a filter pump or with a Kipp's carbonic acid apparatus.

The substance is placed at the bottom of the test-tube, the tube of the tap funnel filled with water to just above the tap, the tube *a* closed by a clip and the air pumped out of the apparatus. Carbon dioxide is allowed to enter, and the apparatus again evacuated. After repeating this process several times, the air in the rubber tube connecting to the eudiometer is driven out by a rapid current of carbon dioxide. The gas passing through the apparatus should now be completely soluble in caustic soda. The 3-way tap is closed, and strong hydrochloric acid admitted from the funnel until the test-tube is filled to the extent of about one-fifth. The liquid is now heated to boiling; the evolution of nitrogen is soon finished, boiled water is then run in from the funnel until the apparatus is nearly full. The last traces of gas are expelled in a few moments by a current of carbon dioxide. It is stated that, with careful working, the accuracy of a Dumas' nitrogen determination is attained, or even exceeded. The time required is very short.—A. C. W.

Tannin Estimation; Comparison of Volumetric Methods with the Results of the "Hide-Powder" Method. A. Turnbull. Paper read before the Paris Conference of the I.A.L.T.C. 1900. Leather Trades' Rev. **34**, [786], 279.

THE following experiments were undertaken with the object of testing F. Jean's method for the estimation of tannin, to see what relation the results might have with those obtained by the Loewenthal method and the hide-powder method as now used by the I.A.L.T.C. The following table gives the results obtained by analysing a known mixture of gallotannin and gallic acid:—

——	Tannic Acid.	Gallic Acid.
Actual amounts in milligrammes, per c.c.	0·456	0·452
Jean's method, using Jean's separation	0·424	0·485
Loewenthal method, using separation method of—		
Jean.....	0·527	0·390
Procter.....	0·457	0·445
Hunt.....	0·426	0·470

In the subsequent experiments, Jean's method of separation was used with the Jean method of analysis, but with the Loewenthal method, Procter's method of separation was used. The following table gives the results by the three methods:—

——	Jean Method.	Lowenthal Method.	Procter's Hide-powder Method.
Oakwood:—			
A. Gallotannic acid.	13·3	16·6	27·0 tannins.
Gallic acid.....	4·7	2·8	13·0 non-tannins.
B. Gallotannic acid.	10·6	25·5	26·6 tannins.
Gallic acid.....	4·8	2·7	16·0 non-tannins.
C. Gallotannic acid.	15·5	17·3	26·1 tannins.
Gallic acid.....	4·2	2·5	17·2 non-tannins.
Mixed oakwood:—			
D. Gallotannic acid.	17·4	21·2	33·8 tannins.
Gallic acid.....	5·3	3·3	6·7 non-tannins.
Hemlock:—			
Gallotannic acid.	13·6	15·2	28·8 tannins.
Gallic acid.....	1·9	1·2	13·0 non-tannins.
Solid quebracho:—			
Gallotannic acid.	39·3	45·8	58·8 tannins.
Gallic acid.....	2·2	1·8	10·7 non-tannins.

(See also this Journal, 1901, 159).—J. G. P.

Sugars; Influence of Salts on the Rotatory Power of ——. J. de Kowalski and P. Tomartschenko. Arch. Sc. phys. nat. Genève, **11**, [4], 294—299. Chem. Centr. 1901, **1**, [18], 984.

THE authors examined the influence of the chlorides, bromides, and iodides of sodium, potassium, and ammonium, at different concentrations, on the rotatory power of cane sugar, invert sugar, and glucose, in the latter case after the multi-rotation had ceased. The rotation of the cane sugar is lowered, and at equal concentrations the effect is less, the stronger the individual salt becomes dissociated. Also, for each salt the influence is smaller, the stronger it is dissociated at the corresponding dilution. Similar results are obtained with invert sugar and glucose. The lævo-rotation of the invert sugar is increased; the dextro-rotation of the glucose is lowered.—A. S.

Starch in Potatoes; Baumert and Bode's Method of Determining ——. P. Behrend and H. Wolfs. Zeits. angew. Chem. **14**, 461—465.

THE authors' method of determining the starch-content of potatoes, by the specific gravity of the potato, cannot claim scientific accuracy, though it affords good comparative results and is very useful in the rapid valuation of potatoes for the purposes of the distillery. The conversion of the starch into dextrose and determination of the amount of the latter, too, only gives the "starch-value," not the actual starch content, of the tuber.

Baumert and Bode's method (this Journal, 1900, 1152) consists shortly in completely drying the finely divided potato substance, bringing the starch into solution in water under high pressure, filtering, rendering alkaline with soda, and finally precipitating the starch from an aliquot portion, washing and drying the precipitate, and determining its quantity from the loss of weight on burning it off. The authors have subjected the method to a searching investigation, and are completely satisfied with it. They suggest

that the asbestos-tubes for the filtration should be not less than 20—22 mm. in internal diameter. The starch should be burnt off in a stream of oxygen; this is much quicker and more convenient than to use air. The author's figures (duplicate analyses of 12 potatoes), show a maximum difference between the duplicates of 0·10 per cent., an average difference of 0·06 per cent. (the figures running from 18·84 to 24·62 per cent.). Moreover, by analysing known mixtures made to imitate the composition of a potato, the authors have shown clearly, in confirmation of Baumert and Bode's statement, that the whole of the starch is precipitated and weighed, and that it is precipitated free from any of the associated substances.

The method is exact, though not rapid. It will be of the greatest value for physiological research and for the purposes of potato culture, as also in the starch industry. It will not probably be very generally used in distillery work, though here the authors have used it for determining starch in the "insoluble residue" of sweet mash, to discover the amount of unaltered starch; for this it is more convenient and more exact than the (yet very good) Maercker diastase method.

—J. T. D.

Gum Arabic; Valuation of ——. O. Fromm. Zeits. anal. Chem. 1901, **40**, [3], 143—168.

THE first portion of the paper is devoted to the methods employed in the examination of gum. These methods are necessarily entirely empirical, and the results are only comparable among themselves. The observations were carried out on filtered solutions of 1·035 density at 15° C., it being proved that these solutions always contained about 8·5 per cent. of dry substance. The observations include those of colour, tendency to froth, and behaviour towards basic lead acetate and alkaline copper solution. The volume occupied by the swollen insoluble matter when allowed to settle in water was always noted. The viscosity of the solutions was determined by Engler's viscosimeter and referred to water at 20° C. The free acidity was tested by titration with decinormal soda in 50 c.c. of the solution. The acidity of the solutions increases and the viscosity decreases on keeping; standard conditions must therefore be observed. The optical activity, α_D in the 1 dm. tube, is generally between $-2°$ and $-3°$, but abnormal variations between the limits of $-4°\ 52'$ and $+9°\ 8'$ are recorded. The variations in optical activity are not due to the presence of sugar. Nearly all the samples were precipitated by basic lead acetate, and very nearly all had a certain variable cupric reducing action. The most important factor in the value of a gum is its adhesive strength. This was determined relatively by soaking sheets of standard unsized paper in the solution, drying off, and determining the increase of weight and increase of tensile strength of the paper. From these a "breaking length" was calculated, indicating the cohesion of the dried gum and its adhesion to the fibres of the paper. The process is somewhat tedious, and a special chamber is required to ensure constant hygrometric conditions in making the tests of tensile strength. The increased elasticity imparted to the paper was also observed and expressed as per cent.

The author draws his conclusions from the results of the examination of several hundred samples. It is never quite safe to substitute the various physical and chemical tests for the more tedious direct test of adhesive power. But in most cases it is found that if the other properties, notably viscosity, acidity, and lævo-rotation are up to the normal average, the adhesive power will also be good. The author gives a definition on his arbitrary scale of the normal values of these factors; variations, whether above or below the normal, generally indicate deficiency in strength. Certainly no safe conclusion can be drawn from viscosity alone, as is sometimes attempted.

The prices of the samples on the whole tended to vary with the adhesive strength, though some were found to be too dear and others cheap; the external appearance, however, also has an influence on the price, which often varies in much wider limits than the relative strength of the gum. The author states that no differences could be detected in the samples of different origin. The author's methods are sufficiently exact to control bulk deliveries against samples. The tests show that gums gradually alter on keeping—a fact which is recognised in the trade by the term "ripeness." This alteration on storage is marked by a deterioration as regards strength; the viscosity and acidity also decrease; the elasticity is not affected. This deterioration is accompanied by an increase in the quantity of gelatinised insoluble matter.—J. F. B.

Alkaloids; Methods of Exhausting Drugs for Determination of ——. O. Linde. J. Pharm. Chim. 1901, **13**, [8], 374—377.

THE author criticises and reviews the methods for alkaloidal extraction, and divides them into two classes.

A.—Operations requiring no special apparatus:—

1. Extraction by neutral solvents, utilizing cold or hot water as the solvents. To this class may be added methods where a solution of sodium salicylate is used (see method for opium extraction, German Pharmacopœia, 4th edition).

2. Extraction by acid solvents. To this class is added methods in which very concentrated solutions of chloral hydrate are employed (J. Pharm. Chim. 1900. 6, **11**, 180, see also this Journal, 1900, 569).

3. Methods of treatment with alkalis and subsequent treatment by a neutral solvent. These methods are very numerous; as bases there are used, lime, potash, soda, magnesia, ammonia, oxide of lead, alkali carbonates; as solvents, ether-chloroform mixtures, alcohol, petroleum spirit, benzine, carbon tetrachloride, amyl alcohol, ether-alcohol.

B.—Operations in which special apparatus is required, divided into two classes:—

1. Methods in which a percolater is required, the solvent varying according to the method (acidulated water, alcohol, ether, &c.) These methods generally exhaust the drugs perfectly, but they are open to objection on account of the comparatively large amount of solvent required necessitating subsequent evaporation, and thus introducing risk of injury or loss in the case of certain alkaloids.

2. Methods in which extraction apparatus is required (as Soxhlet, Barthel, Tollens). In such methods the powdered drug is usually mixed with lime or magnesia and submitted to the solvent action of ether-chloroform, &c.; or by neutral solvents to which ammonia has been added.

The author concludes that methods based on the employment of extraction apparatus give perfect results; their only inconvenience being that the apparatus is usually very fragile, but there are certain very simple types (Tollens, Barthel); and these do not even require the use of water as a condensing agent, they can therefore be installed in any laboratory. The quantity of drug to be taken should be equivalent to 0·1 grm. to 0·5 grm. of active principle. As a solvent, ether is recommended, as it dissolves almost all alkaloids, but in certain cases (*e.g.*, nux vomica) chloroform is preferable.—C. T. T.

Morphine; Determination of ——, *by Means of Potassium Iodate and Arsenious Acid in Alkaline Solution.* C. Reichard. Chem. Zeit. 1901, **25**, [31], 328—329.

IN the presence of acids, morphine separates iodine from potassium iodate. The iodine is, partially at least, in some peculiar state of solution, since the brown coloration obtained in dilute solutions is made deeper by the addition of ammonia. Aqueous iodine solutions are at once decolorised by morphine salts; on standing, the colorless solution becomes brown, but the coloration is not removed by the addition of excess of morphine. The iodine separated on the addition of potassium iodate to morphine solutions can be completely removed by chloroform and other solvents, the residual solution then gives no coloration with ammonia. The dark coloration produced by ammonia is not removed by an alkaline arsenite, it is therefore necessary to extract the iodine by a solvent and act on the solution in the solvent with the arsenite. Chloroform is the most suitable solvent, it is used until the last portion is colourless; the iodine solution is shaken with an excess of arsenite solution and starch paste, and the excess of arsenite titrated by $\frac{1}{100}$ N-iodine solution. Three molecules of morphine = 1 atom of iodine. The results of the control analyses quoted are quite satisfactory.

In regard to the estimation of morphine by means of silver nitrate (this Journal, 1901, 160), it has now been found that an ammoniacal solution of silver chloride is much more suitable, especially in the case of morphine hydrochloride. Very dilute solutions of morphine separate in the cold a black powder of silver. The reaction appears to be adapted to the estimation of morphine in opium; greyish-black silver is separated in a few minutes from very dilute, almost clear, filtrates of opium solutions.

—A. C. W.

Cloves; Notes on the Approximate Analysis of ——. A. McGill. Analyst, 1901, **26**, [302], 123—126.

The author records analyses of genuine cloves (*Caryophyllus aromaticus*), tabulating his results. Previously recorded analyses give water from 2·90 to 16·39 per cent. (König, "Zusammensetzung," &c., 744); the author finds a smaller range, the extremes being 4·1 and 7·5 per cent. On exposing samples of powdered cloves *in vacuo* over sulphuric acid it was found that loss of weight continued to occur for many days, but that the rate decreased very rapidly after the first period of 24 hours, while the acid continued to grow deeper in colour till it became quite black from separated carbon; the explanation being that water only is given off during the first period, and that loss of weight after this period is essentially due to volatile oil. A sample was found to suffer 11·45 per cent. total loss in 16 days. The same sample lost 3·65 per cent. in 24 hours over sulphuric acid at ordinary pressure, and 4·72 per cent. in 54 hours with only slight discoloration of acid. The author recommends that moisture be determined by exposure for 24 hours over sulphuric acid under the reduced pressure of 60 mm. mercury; total volatile matter by exposure in a current of dry air at 98° C. for 24 hours. A slight increase of volatile matter is observed after previous treatment with petroleum spirit. 2 grms. of the sample are allowed to stand at the ordinary temperature, after addition of 25 c.c. of ether, until this ether is evaporated, then dried in a water oven at 98° C. as described; total extractive matter is obtained by Soxhlet extraction to constant weight. The lowest value for the percentage of volatile oil found in genuine whole cloves is 12·1 per cent. The author concludes that no sample of cloves of good quality should yield less than 14 per cent. of volatile oil when assayed as above. If the total volatile matter be determined by treatment with petroleum spirit before drying at 100° C., this treatment being repeated, the minimum amount of volatile oil should be 16 per cent.—C. T. T.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Radium; Action on Selenium of the Radiation of ——. E. Bloch. Comptes Rend. **132**, [15], 914—915.

Selenium cells, of resistances 30,000 and 654,000 ohms respectively, were exposed in the dark to the radiation from a sample of radio-active barium carbonate of activity about 1,000 times as great as that of uranium. The active salt was covered by black paper, and was distant everywhere about 1 mm. from the selenium cells. The resistances of the cells were reduced after ten minutes to 29,000 and 64,000 ohms respectively: an effect equal to that of feeble diffused light.—J. T. D.

Radio-active Lead. K. A. Hofmann and E. Strauss. Ber. 1901, **34**, [6], 907—913

In continuation of their investigations on radio-active material separated from pitch-blende, bröggerite, &c. (this Journal, 1901, 290), the authors have obtained from the more soluble portions of the lead chloride prepared from these minerals, a compound, which, when converted into sulphate and precipitated as barium sulphate, gives an equivalent of 85·98. The similarity existing between these compounds and those of lead indicates that they correspond with a divalent metal which would hence have the atomic weight 171·96, and would occupy in the periodic system a gap between tin and lead. This "radio-lead" sulphate exerts a strong action on a photographic plate in the dark and when acted on by cathode rays, it retains this activity for some weeks. On heating to redness for a quarter of an hour the activity suffers only slight diminution. When the sulphate was converted into sulphide by means of ammonium sulphide solution it was found to be quite inactive; a strongly active sulphate converted into sulphide in a Rose crucible by hydrogen sulphide, and then re-converted into sulphate, was found to be active, although to a less marked degree than the original material. By digesting the sulphate with aqueous potassium iodide, a yellow iodide is obtained, which, under the action of the cathode rays, exhibits a green fluorescence and superficial decomposition, but does not affect a photographic plate. The property of being rendered active is hence peculiar to the sulphate, although the sulphide seems to possess it in a latent form. That an intimate connection exists between phosphorescence and radio-activity is shown by heating the sulphate with sulphur, by which means a mixture of sulphate and sulphide is obtained; this mixture is caused to fluoresce and phosphoresce under the action of cathode rays, but it has no action on a photographic plate, whilst on converting the whole mass into sulphate again, a material is obtained which is rendered intensely active by cathode rays. The action of radio-lead sulphate on a photographic plate is exerted through sheets of paper, glass, aluminium, and caoutchouc, the last-named causing a spreading out of the action. The action on the electroscope coincides with that of Becquerel or ultra-violet rays, the sulphate causing the gold leaves to collapse much more quickly than if left to themselves.—T. H. P.

Stannous Chloride; Oxidation of Solutions of ——, *by means of Free Oxygen.* S. W. Young. J. Amer. Chem. Soc. 1901, **23**, [3], 119—147.

In a former communication (J. Amer. Chem. Soc. **23**, 21,) it was shown that there must be a strict definition of the reagents taking part, before it is possible to determine the constant of velocity or the order of a reaction.

In the reaction between stannous chloride and free oxygen an acceleration occurs, and the author suggests that that this may be due to the hydrochloric acid formed during the reaction retarding the hydrolysis, or possibly to a catalytic action of the products of the reaction. Since the velocity constants for different series of measurements show great variations, he is inclined to attribute the phenomenon to catalytic influences, and in the second part of his paper discusses the detection and qualitative investigation of some of these agents.—C. A. M.

Silver Oxide; Action of Hydrogen Peroxide on ——. Berthelot. Comptes Rend. **132**, [15], 897—904.

If equivalent quantities of well-cooled dilute solutions of silver nitrate, sodium hydroxide, and hydrogen peroxide be taken, any two of them be mixed, and the third be then rapidly added, the first result is the formation of the black peroxide Ag_2O_2. This almost immediately begins to decompose, and a rapid evolution of oxygen occurs, which practically ceases when an amount equal to the available oxygen of the original hydrogen peroxide has been given off. Part of this arises from the complete dissociation of some of the silver peroxide, according to the equation $Ag_2O_2 = Ag_2 + O_2$; but another portion of the silver peroxide gives off only half this proportion of oxygen, while the resulting monoxide combines with a third portion of the peroxide ($Ag_2O_2 = Ag_2O + O$; $Ag_2O + Ag_2O_2 = Ag_4O_3$) to form an intermediate oxide. A third stage in the reaction consists in the very slow dissociation (hastened by rise of temperature, or by addition of dilute sulphuric acid) of this oxide, according to the equation $Ag_4O_3 = 2\,Ag_2O + O$. Direct experiments have shown that this last evolution of oxygen proceeds from the solid substance, not, as Baeyer has supposed, from a supersaturated solution of the gas in the suspending liquid. When strong solutions of the reacting substances are used, or the temperature is allowed to rise, the reaction approximates more and more closely to the complete dissociation, $Ag_2O_2 = Ag_2 + O_2$.

—J. T. D.

Borides; Production of some New Metallic ——. S. A. Tucker and H. R. Moody. Proc. Chem. Soc. 1901, **17**, [238], 129.

THE authors describe the formation and properties of four new borides, those of zirconium, chromium, tungsten, and molybdenum. These compounds were prepared by bringing an intimate mixture of the metal with boron under the influence of a temperature produced by a current of from 200 to 275 amperes and 60 to 75 volts in the electric furnace.

Combination takes place in a few minutes with the formation of compounds which are crystalline, hard, of a high specific gravity, not easily attacked by acids, and having very high melting points. The compounds thus prepared have the following formulæ Zr_3B_4, CrB, WB_2, and Mo_3B_4.

The authors were unable to produce the borides of copper and of bismuth in a like manner, and it seems therefore as if boron had no affinity for the members of the copper group, but that it combines readily with the members of the iron group.

Tobacco Smoke; Chemistry of ——. H. Thoms. Schweiz. Woch. **39**, 27; Pharm. J. 1901, **66**, [1607], 459.

TOBACCO smoke was drawn through absorption tubes containing caustic soda, sulphuric acid, &c., and the contents of the tubes subsequently examined. The smoke was found to contain the bases, nicotine, pyridine (from the decomposition of nicotine), and ammonia, carbonic, and butyric acids, (but no hydrocyanic acid), carbon monoxide, and two volatile oils, one of which could be steam-distilled from the tobacco. In 15,000 grms. of tobacco there are 6 grms. of this oil. It is dark coloured and similar to a balsam, the smell resembling that of camomile oil. It boils at 295°—315° C., and contains a phenol. The other oil, which is extremely toxic, is obtained by extracting with ether the sulphuric acid and soda through which the smoke had passed. 20,000 grms. of tobacco yield 75 grms. of the oil; it is dark coloured, has a narcotic odour, boils mainly at 220°—230° C., and contains nitrogen and sulphur, and a phenol boiling at 190°—200° C. About 75 per cent. of the nicotine present in the tobacco passes over with the smoke, part being decomposed into pyridine and other bodies.

The ash amounted to 20·09 per cent. of the tobacco burned, and 18·82 per cent. of this ash was carbonaceous. The mineral matter consisted chiefly of the carbonates of calcium and potassium, the phosphates of calcium and magnesium, potassium chloride, silicates and silica.—A. S.

Glucose and Galactose; New Derivatives of ——. W. Koenigs and E. Knorr. Ber. 1901, **34**, [6], 957—981.

BY the action of five molecular proportions of acetyl bromide upon glucose, the authors have obtained acetobromglucose, analogous to the acetochlorglucose of Colley. Unlike the chloro-compound, acetobromglucose is readily obtainable in the crystalline form, and its purity is thus ensured. It is dextro-rotatory, melts at 89° C., and reduces Fehling's solution. With silver acetate and glacial acetic acid it yields the pentacetylglucose, melting at 131° C. Solutions of the potassium compounds of phenol and β-naphthol in methyl alcohol yield the corresponding glucosides. The authors noticed the extreme ease with which the bromine is replaced by methoxyl or ethoxyl, by shaking acetobromglucose with the absolute alcohols in presence of silver or barium carbonates or pyridine. Tetracetyl derivatives of the β-alkyl glucosides were thus obtained; these are readily saponified by caustic alkalis in the cold to the β-glucosides. Acetobromglucose therefore belongs to the β-series, and the pentacetyl glucose, melting at 131° C., is the β-isomeride. In no case would any of the α-series of glucosides be detected. Colley's acetonitrose [acetonitroglucose] was prepared by treating acetobromglucose or β-pentacetylglucose with fuming nitric acid in chloroform solution. The nitric group of acetonitrose is just as readily replaceable as the bromine of acetobromglucose, and in exactly similar manner. Since the pentacetylglucose is very easily obtained, it forms the most convenient starting point for the preparation of the β-glucosides. The authors have not succeeded in obtaining the isomeric α acetonitroglucose. Further, starting from pentacetylgalactose, the authors have prepared acetonitrogalactose, tetracetyl β methylgalactoside, and finally β-methylgalactoside. Attempts to synthesise di-saccharides by means of acetobrom- and acetonitroglucose led to no successful results.—J. F. B.

PATENTS.

Oxidising or Raising Chemical Compounds to a Higher Oxide; Process for ——. U. Wedge, Bayonne, New Jersey, U.S.A. Eng. Pat. 2010, Jan. 29, 1901

THE patentee claims the use of ozone or ozonised air for various processes of oxidation.—T. A. L.

Extinguishing Fires in Vessels containing Inflammable Liquids. F. Shuman, Philadelphia, U.S.A. Eng. Pat. 5515, March 15, 1901.

A SOLUTION of a carbonate or sulphite—preferably bicarbonate of soda—is stored at the bottom of the vessels, or where it can be quickly discharged into it, and sulphuric or other acid is stored above in a vessel from which it can be discharged on the outbreak of fire within the vessel. The carbon dioxide evolved on the contact of the acid with the solution passes to the surface of the oil or other inflammable liquid, and forms a layer thereon which is stated to exclude air and thereby extinguish the fire. The discharge of the acid may be effected automatically by the burning of a cord or connection when fire breaks out on the surface of the liquid.—J. A. B.

Research.

RESEARCH AT THE SCIENTIFIC AND TECHNICAL DEPARTMENT OF THE IMPERIAL INSTITUTE.

Annual Rept. of Imp. Inst. for 1900.

During the last year a large number of natural products, derived from India and the Colonies, have been submitted to scientific investigation with the view of utilising them commercially.

Among the investigations which have been reported to the Government of India may be mentioned those on various new fibres, gums, tanning materials, medicinal plants, and rubbers.

Reports have also been made to the Indian Government on certain special questions connected with the production, in India, of tanning extracts from those plants which have proved to be suitable for this purpose, and also, through the Commercial Intelligence Branch of the Board of Trade, in connection with the question as to the competition between natural and artificial indigo and its bearing upon the indigo industry in India, and the possible improvements which may be effected in this industry with the view of increasing the production and decreasing the cost of this valuable dyestuff.

Reports to the respective Governments have been made on coal from Victoria (Australia), clay from British Columbia, fibres and minerals from British Central Africa, rubber from Zululand, the ash of the aloes plant of Natal, mica from Canada, and leather from New South Wales.

Investigations have also been conducted with certain medicinal and poisonous plants from Egypt. The Egyptian Government having had its attention frequently drawn to the poisonous effects on animals of a small vetch, *Lotus arabicus*, abundant on the banks of the Nile, an investigation was conducted in the Laboratories of the Department on the nature of the poison, with material collected in Egypt by Mr. E. A. Floyer, Member of the Egyptian Institute and Director of Egyptian Telegraphs. The mystery attending the poisonous action of this plant has been completely cleared up. In a preliminary communication to the Royal Society by Professor Dunstan and Mr. Henry (the Salters' Company's "Research Fellow" attached to the Institute), it has been shown that at certain stages of its growth, this plant, when intimately mixed with water, develops prussic acid to a considerable extent, which accounts for its poisonous effects. They have proved that

this poison originates in a glucoside contained in the plant, of which a full account will be given in a forthcoming paper.

The publication last year of papers in the *Journal of the Chemical Society*, describing the results of an investigation for the Government of India of Indian henbane, *Hyoscyamus muticus*, has led to the examination of the same plant which is abundant in Egypt. It has been proved that the Egyptian hyoscyamus is even of more value than the Indian plant as a source of the pure alkaloid, *hyoscyamine*, which is a valuable medicinal agent. As a consequence of these investigations a demand has already sprung up for this plant among chemical manufacturers.

With the increased facilities which are being afforded to this Department, it is hoped that its usefulness to the Governments of India and the Colonies will be still further extended. It is now in a position to supply scientific and technical information in reference to natural products of every description which are likely to be of importance either from a commercial or scientific standpoint and also to advise in reference to the establishment or improvement, in India or the Colonies, of those industries which involve applications of science.

Société Industrielle de Rouen.

It has been decided to organise, in connection with an Exhibition of Arts applied to the Adornment of Tissues and Textiles, a congress of Learned, Technical, and Industrial Societies at Rouen on Aug. 19th next.

Communications are invited, and further particulars may be obtained from the Secretary of the Société Industrielle de Rouen.

Victorian Gold Jubilee Exhibition, Bendigo, 1901—2.

May, 1901.
Exhibition Offices,
Market Square, Bendigo.

The Committee of the Victorian Gold Jubilee Exhibition have to announce to manufacturers of chemicals, chemical apparatus, and assaying goods, that they have made the following arrangements for bringing the manufactures of exhibitors in these lines as prominently before the public as possible:—

A model laboratory 25 ft. × 16 ft. is to be provided by the Bendigo School of Mines, equipped and stocked under the direction of the Lecturer in Chemistry and Assaying at that institution.

The laboratory will be provided with furnaces, apparatus, and fittings for the carrying on of assaying and chemical processes.

It will be situated near to the metallurgical and assaying exhibits, and will do much to make the court an interesting and attractive one.

Where exhibitors so desire it, exhibits such as balances, apparatus, crucibles, platinum ware, and other articles demanding closer examination than can be made in show cases, will be removed, at regular times, and in rotation (or at the request of visitors), to the laboratory, and there displayed so that they can be carefully examined by chemists and assayers who may be interested in them.

All care will be taken that the goods are not damaged by careless handling.

In the case of furnaces, &c., exhibitors can make arrangements to have these actually worked in the laboratory.

In all cases goods on exhibition in the laboratory will be prominently labelled with the name of the manufacturer or exhibitor. Such labels should be forwarded with the exhibits. A good supply of circulars and catalogues should also be forwarded, for distribution frem the laboratory, when the goods are on exhibition there.

The Committee feel sure that manufacturers and others will fully appreciate the advantages that such a method of exhibition afford from an advertising point of view.

A detailed description of all exhibits in the way of chemicals, assaying goods, and chemical apparatus, will be written by Mr. H. C. Boydell, B.Sc., A.O.U.S.M., (Lecturer in Chemistry, Assaying, and Metallurgy, at the Bendigo School of Mines). Mr. Boydell has been connected with mining, metallurgy, and assaying in many parts of Australasia, and will particularly emphasize such articles as are new to Australia.

These descriptions will be published in the Australian, and other mining papers, and also in the papers of mining districts. Sketches of the best and newest appliances will also be given. Exhibitors should forward notes and sketches of such points in their goods as they wish special attention drawn to.

Exhibits will be carried to and from the Exhibition free of charge, on the Government railways, and must reach Bendigo not later than the end of September.

Geo. V. Allen,
General Secretary.

New Books.

The Extra Pharmacopœia. By William Martindale, Late President and Examiner of the Pharmaceutical Society, and W. Wynn Westcott, M.B., &c., H.M's. Coroner for North-East London. 10th Edition. H. K. Lewis, 136, Gower Street, London, W.C. 1901. 10*s.* 6*d.*, by post, 10*s.* 9*d.*

This is the 10th edition of the Extra Pharmacopœia, which is of pocket-book size (med. 24 mo.), and contains preface and subject-matter filling 544 pages, also Appendices I., II., and III., 14 pages; and the Index and Posological Table, pages 559—664, and finally the Therapeutic Index of Diseases and Symptoms, pages 665—685.

Merck's Annual Report on the Year 1900. Vol. VIII. (Complete German Edition, Vol. XIII.). March 1901. F. Boehm, 16, Jewry Street, E.C.

A compilation of facts ascertained during 1900, and a description of substances which have, during this period, gained importance in the various branches of medical chemistry. The book (Report) contains 205 pages of subject matter, and ends with a Biographical Index, an Index of Authors, and the General Index of Subjects and Substances. Then follows an Index of Diseases, Symptoms and Indications for Treatment, and finally a Tabulated Index of Approximate Prices of the Principal Substances (remedies) specified in the Report.

Recent Parliamentary Publications. Patents, Designs, and Trade Marks. Eighteenth Report of the Comptroller-General. 163. Price 3*d.*

The report of the Comptroller-General of Patents, Designs, and Trade Marks for 1900 shows that the number of applications in respect of patents continues to show a falling off. In 1897, 30,952 were applied for, and these have decreased annually to 23,922 in 1900. The applications for designs amounted to 16,952 last year. The applications for trade marks numbered 7,937 in 1900, but in spite of this general decrease, the total receipts increased from 225,701*l.* to 226,091*l.* The number of applications accompanied by provisional specifications was 18,117, which is 1,912 less than in 1899, while those accompanied by complete specifications were the most numerous ever received in one year. The number of complete specifications filed after provisional ones had been sent in was 7,288 in 1900, and the total number of specifications was 31,210. The applications from England and Wales have decreased from 15,354 in 1899 to 13,775 in 1900, and those from Germany have decreased from 2,921 to 2,631, and for France from 1,031 to 946. The United States, on the other hand, account for 3,189 instead of 3,022 in 1899. The system of deposit accounts for Patent Office publications which was started in June 1900, has met with a favourable reception. Eighty-three accounts with a *minimum* of 2*l.* were opened in that year. Under this system it is no longer necessary to send the deposit of 8*d.* in ordering a given specification. Volumes of collected abridgments relating to certain selected portions of the subject-matter of a few abridgment classes have now been issued and placed in the Patent Office Library. Three further guides to the contents of the library have been published at 6*d.* a number, *viz.*: (1)

key to the classification of French patent specifications; (2) photography; (3) laws of industrial property and copyright.

PRACTICAL DICTIONARY OF ELECTRICAL ENGINEERING AND CHEMISTRY (German, English, and Spanish). Treating especially of Modern Machine Industry, The Foundry and Metallurgy. By PAUL HEYNE, assisted by Dr. E. SANCHEZ-ROSAL.

In 3 volumes:—I. German—English—Spanish. II. English—Spanish—German. III. Spanish—German—English. Gerhard Kühtmann, Dresden. H. Grevel and Co., London. 1899.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

COUNTERVAILING DUTIES ON DUTCH SUGARS—REVISED RATES IN BRITISH INDIA.

The Board of Trade have received a copy of a notification (Customs Circular 8, of 1901) issued by the Government of India on 7th May 1901, imposing, with effect from 1st June last, the following revised rates of additional duty on sugars produced in, or exported from, Holland:—

Kinds of Sugar.	Bounties Bestowed.	Additional Duties to be Levied.
		Per Cwt. Rs. a. p.
Raw sugar produced in Holland from beet-roots.	1·50 florins per 100 kilos. of hard refined.*	0 15 3
Sugar refined from beet-root raw sugar produced in Holland.	1·72 florins per 100 kilos.	1 1 5
Sugar refined from imported raw sugar.	0·22 florins per 100 kilos. in addition to bounty, if any, allowed on the raw sugar by the country of production.	0 2 3 in addition to the countervailing duty, if any, on the raw sugar.

The former rates were:—

Kinds of Sugar.	Bounties Bestowed.	Additional Duties to be Levied.
		Per Cwt. Rs. a. p.
Raw beet sugar of less than 98 per cent. polarisation.	2·2354 florins per 100 kilos. of hard refined (100 per cent.).*	1 7 0
Raw beet sugars of at least 98 per cent. polarisation.	¾ths of above bounty, or 1·7655 florins per 100 kilos. of hard refined.*	1 2 0
Refined beet-root sugars / Refined sugar from materials other than beet-root raw sugar.	0·2946 florins per 100 kilos. of hard refined, in addition to above rate.	0 3 0 in addition to above rate.

* The output of refined sugar from raw is computed by deducting from the polarisation of the raw sugar twice the glucose, four times the ashes, and 1½ per cent. for loss in refining.

EXEMPTION FROM DUTY OF VINEGAR AND LIGNEOUS ACID FOR PREPARATION OF AMYLIC ACID IN THE NETHERLANDS.

A translation has been received at the Board of Trade, through the Foreign Office, of a Dutch Royal Decree, dated the 15th ult., providing for the exemption from excise and import duty, subject to certain restrictions, of vinegar and ligneous acid destined for use in the preparation of amylic acid.

DUTY ON GLUCOSE AND MOLASSES.

In Committee of Ways and Means of the House of Commons on 11th June, the Chancellor of the Exchequer (Sir M. Hicks-Beach) moved a resolution effecting certain changes in the new Customs duties proposed to be levied on molasses and glucose. On molasses and on sugar and sugar extracts which cannot be tested by the polariscope, if containing 70 per cent. of sweetening matter the amended duty will be 2*s.* 9*d.* per cwt.; if containing less than 70 per cent. of sweetening matter and more than 50 per cent. the duty will be 2*s.*; and up to 50 per cent. the duty will only be 1*s.* The Chancellor's original proposal, as embodied in the Finance Bill, was that in these cases there should be a uniform duty of 2*s.* The duty upon glucose at present is only 1*s.* 8*d.*, a sum fixed on the assumption that glucose contains 40 per cent. of sweetening matter. But since that calculation was made it has been pointed out that solid glucose contains considerably more sweetening matter than 40 per cent., and the duty upon it will accordingly be raised to 2*s.* 9*d.* Liquid glucose also has a higher sweetening power than Sir Michael at first supposed, and the duty upon it will be increased to 2*s.* The duties will operate from 11th June, when corresponding Excise duties will also be imposed. The Chancellor explained that some of these changes are necessary in the interests of the revenue, while others are required in order to put manufacturers in this country on a footing of equality with foreign producers.

The resolution was agreed to after some discussion without a division.

The following is the text of the resolution:—

Resolved—That there shall be charged on and after the 11th day of June 1901, the following Customs duties:—

Molasses and all sugar and extracts from sugar which cannot be tested by the polariscope:—

	Per Cwt. *s.* *d.*
If containing 70 per cent. or more of sweetening matter	2 9
If containing less than 70 per cent. and more than 50 per cent. of sweetening matter	2 0
If containing not more than 50 per cent. of sweetening matter	1 0
Glucose:—	
Solid	2 9
Liquid	2 0

And there shall be charged on and after the same date on glucose Excise duties equivalent to the Customs duties charged on that article.

EXPORT OF SPIRITUOUS PREPARATIONS.

Chemist and Druggist, June 15, 1901.

The Custom House have issued an order, dated June 5, relating to amendments in the Code which governs the export of medical tinctures in bond. Paragraphs 212–3–4 and 256 have been cancelled and the following paragraphs substituted:—

Paragraph 212.—"Tinctures or medicinal spirits may be removed under bond to a ship's side, for exportation or use as ship's stores, in bottle or metal canisters holding any quantity, packed in cases each containing not less than 1 bulk-gallon, and the bottles or canisters may be of different sizes; but no consignment of tinctures or medicinal spirits may consist of less than 2 bulk-gallons. Medicinal spirits, flavouring-essences, and perfumed spirits, may be packed in the same case provided that they are packed in separate internal compartments or packages. Jars covered with wickerwork and iron drums, each containing not less than 2 and not more than 10 bulk-gallons, may be exported uncased. 'Free goods' may be packed in the same case as medicinal spirits, flavouring-essences, or perfumed spirits, provided that they are placed in an internal package, distinctly marked 'Free Goods,' and that the consignment consists of not less than the minimum quantity of tinctures or medicinal spirits, flavouring-essences, or perfumed spirits, allowed to be exported on drawback at one time, exclusive of the 'free goods.'"

Paragraph 213.—"Flavouring essences may be removed under bond to ship's side, for exportation, in bottles or metal canisters holding any quantity, packed in cases each containing not less than 1 bulk-gallon, and the bottles or cannisters may be of different sizes; but no consignment of flavouring-essences may consist of less than 2 bulk-gallons.

Jars covered with wickerwork, and iron drums, each containing not less than 2 and not more than 10 bulk-gallons, may be exported uncased."

Paragraph 214.—"Perfumed spirits may be removed under bond to a ship's side, for exportation, in bottles holding any quantity, packed in cases each containing not less than 1 bulk-gallon, and a single case may be exported. The bottles in each internal package in a case must be of uniform size and contain spirits of the same strength."

Paragraph 256.—"When tinctures or medicinal spirits, flavouring-essences, or perfumed spirits, are exported to the Isle of Man, and samples of them have been sent to the Government Laboratory for examination, the abstract of the notice to pack (Form No. 111–6) is to be sent by the first post direct to the Collector of Customs at Douglas. The Principal of the Laboratory will, without delay, send the result of the analysis of the samples to the Collector at Douglas on the notice to pack, to guide him in charging the insular duty. The Collector will certify on that document to the receipt of the goods and to the charging of the insular duty. He will then forward the notice and the abstract to the Collector of the Inland Revenue Collection from which the goods were removed, in order that drawback and allowances may be paid in the usual way."

United States.

(*From the Board of Trade Journal.*)

Customs Decisions.

Lentisco.—So-called "lentisco," being the ground leaves of the *pistacia lentiscus*, or mastic tree, is not sumac, but is used as an adulterant for sumac, and is a tanning-bearing substance of less value than sumac. It is dutiable at the rate of ¼ cent. per lb. and 10 per cent. *ad valorem* under paragraph 20 of the Tariff.

Mercurial Preparations.—Mercury sulphocyanate, mercury nitrate, mercurous nitrate cryst., mercury oxycyanide, and mercury bichloride are dutiable as chemical compounds under paragraph 3 of the Tariff at the rate of 25 per cent. *ad valorem*, and not as medicinal preparations.

Paraldehyde.—Paraldehyde is not dutiable under the provisions of paragraph 2 of the Tariff, which is limited in its application to perfumeries and toilet waters, nor can it properly be classed under the provisions of paragraph 3 as a chemical compound. It is a "medicinal preparation," and as such is dutiable under the provisions of paragraph 67 as an article in the preparation of which alcohol has been used.

Salol and Chloral Hydrate.—In accordance with a decision notified in August last, and published in the *Board of Trade Journal* for the 6th September, salol and chloral hydrate, in the preparation of which alcohol is used, have hitherto been admitted at the rate of 25 per cent. *ad valorem* under paragraph 68 of the Tariff. The question, however, has again been raised whether these articles should not be dutiable under paragraph 67 of the Tariff, and until this point shall have been re-determined it has been decided that salol and chloral hydrate, as above, are to be dutiable under paragraph 67 at the rate of "55 cents per pound, and in no case less than 25 per cent. *ad valorem*.

Drawback on Sugar, Glucose, Saccharin, &c. in the United Kingdom.

Chem. and Druggist, June 8, 1901.

The Board of Customs has issued a General Order relating to drawback and export regulations on sugar and cognate goods exported or deposited for use as ships' stores, or removed to the Isle of Man. The drawback is not allowed except upon sugar, molasses, glucose, or saccharin, which have gone through a process of manufacture in this country. The amount of drawback which will be paid on articles containing saccharin or glucose is equal to the duty in respect of the quantity of that article which has been used in the manufacture or preparation. Full details are given how to set about drawback claims generally, and the particulars required. As regards examinations and sampling, liquid glucose manufactured in the United Kingdom and contained in drums and other large packages, also glucose in a solid form, will be examined to the extent of 2 per cent. of each parcel; one sample of 8 ozs. is to be taken. Saccharin need not be sampled to the extent of more than 1 oz. In the case of liquids of British manufacture containing sugar or saccharin, such as malt extract, ginger-ale, or like preparations, and sweetened waters (whether aërated or not), the schedule should state the number of bottles of each size and variety, the number of gallons of each kind, and the percentage of dutiable article or articles contained in them. When sweetened condensed milk is presented for drawback, the schedule should specify the number of tins of each description in each case, the net weight per tin, the total net weight per case, and the percentage of refined cane-sugar added.

Monopoly Duties in Switzerland on Products Manufactured with Alcohol.

Bd. of Trade J., June 6, 1901.

With reference to the notice which appeared at p. 194 of the *Board of Trade Journal* for the 25th April last, respecting the Swiss Spirit Law of the 29th June 1900, the Board of Trade have now received a copy of the German official *Nachrichten für Handel und Industrie* for the 16th April last, containing a translation of a notice, issued by the Swiss Customs authorities on the 27th March to the effect that the following new monopoly duties are to be leviable under the Law in question on products manufactured with alcohol.

Tariff No.	Article.	Monopoly Rates per 100 Kilos., Gross Weight.
		Frs. Cts.
13	Iodoform (until further notice).....	Exempt.
14	Salol (until further notice)	"
15	Formic ether, chloroform, acetic ether, nitric acid (until further notice).	"
15	Fusel oil (amylic alcohol) (until further notice).	80 00
15	Fruit ethers.....	As spirits of superior quality, see below, Nos. 461–3.
20, 21	Pharmaceutical preparations, containing alcohol, so far as they are not intended for the manufacture of brandy and liqueurs, per degree and per 100 kilos., gross weight.	1 05
	Extracts and essences containing alcohol, for the toilet, or for the manufacture of distilled spirits, liqueurs, lemonade, &c., such as "*Alcool de menthe*," "*Bittergeist*," essence of cognac, essence of rum, "*Extrait de menthe*," &c.	As spirits of superior quality, see below, Nos. 461–3.
23, 24	Perfumery and cosmetics containing alcohol, per degree and per 100 kilos., gross weight.	1 05
64	Sulphuric ether (until further notice).	Exempt.
106	Spirit-varnishes and polishes manufactured with non-denatured alcohol, per degree and per 100 kilos., gross weight. N.B.—Spirit varnishes and polishes containing 6 per cent. of their weight and over of shellac or other resins will be considered as denatured and exempted from the monopoly tax.	1 05
375, 376	Vinegar (until further notice).....	Exempt.
377	Fruits preserved in alcohol.....	As spirits of superior quality, see below, Nos. 461–3.
379, 395	Juice of fruit or berries prepared with alcohol: (*a*) Containing at most 3·5 per cent., by volume, of alcohol:— (1) Consignments of 50 kilos., gross weight, and upwards, per degree and per 100 kilos., gross weight.	0 80
	(2) Consignments under 50 kilos., gross weight, per degree and per 100 kilos., gross weight.	1 00
	(*b*) Containing more than 3·5 per cent., by volume, of alcohol.	As spirits of superior quality, see below, Nos. 461–3.

MONOPOLY DUTIES IN SWITZERLAND, &c.—*cont.*

Tariff No.	Article.	Monopoly Rates per 100 Kilos., Gross Weight.
		Frs. Cts.
460	Raw spirit, spirit, spirits of wine, and alcohol. N.B.—The importation of raw spirit, spirit, spirits of wine, and alcohol is a monopoly of the State.	Exempt.
460	Anhydrous alcohol:—	
	(*a*) In quantities over 50 kilos., gross weight, and over.	100 00
	(*b*) In quantities less than 50 kilos..	125 00
475	Transparent glycerine soap (until further notice).	Exempt.

OFFICIAL NOTICES.

CARRIAGE OF COLLODION COTTON ON BOARD SHIP.

The Board of Trade are advised that for the purposes of the Explosive Substances Act, 1875, Collodion Cotton is defined as follows:—

Collodion Cotton consisting of thoroughly purified nitro-cotton (*a*) of which not less than 15 per cent. is soluble in ether and alcohol, and (*b*) which contains not more than 12·3 per cent. of nitrogen.

Where this substance is "used or manufactured with a view to produce a practical effect by explosion," it is by Section 3 of that Act an explosive in any form. But in cases where it is not intended to be used by itself as an explosive, it is not regarded as coming within the meaning of the Act when it is

(*a*) in solution in alcohol and ether,
(*b*) wet, or
(*c*) saturated in methylated spirit and contained in air-tight cases.

In such circumstances it will not be considered as "dangerous goods" within the meaning of Section 446 of the Merchant Shipping Act, 1894.

It is however regarded as an explosive when dry.

COURTENAY BOYLE,
Secretary,
WALTER J. HOWELL,
Assistant Secretary.

I.—GENERAL.

CHEMICAL INDUSTRIES OF PERNAMBUCO, BRAZIL.

Foreign Office Annual Series, No. 2591.

Match Factory.—A match factory has closed its doors owing to the stamp tax. This tax lays the obligation on the factory to buy and hold a stock of stamps greatly beyond its financial resources. The mere fact of obliging every box of matches to be sealed with this stamp entails greatly increased cost of the article, for there is no machine yet invented for sticking these stamps on the boxes in the way laid down by the Government; consequently it has to be done by hand. The result is that foreign matches can successfully compete with the home-made article.

Sugar Refinery.—The sugar refinery (the largest in Brazil), which was erected in 1894–95, has proved a failure, although the machinery is of the very best make. The system, however, of refining did not prove suitable for the native markets, or perhaps the venture was over-capitalised by the payment of a large sum for the patent, by disasters while building, and by the lack of sufficient working capital, entailing the borrowing of large sums at high interest, or perhaps technical men were not employed as elsewhere to manage and supervise the different processes of refining. The result is a loss of several thousand contos de reis.

Soap Works.—There are several soap works giving good results, turning out coarse yellow soap, but no toilet kinds.

Gunpowder Factory.—A successful result has been obtained by a Swede (Mr. Lundgren) in his gunpowder factory, which he erected himself in 1894.

Distilleries.—Nearly all the local distilleries have come to grief owing to excessive taxation and prohibitive imposts. Rum is now produced only in the sugar factories in the interior of the State, and at the time of writing is a drug on the market.

Cotton-seed Oil Factories.—Cotton-seed oil factories, which were flourishing five or six years ago, are now either closed or just barely making headway owing to the favourable tariff granted to the United States on imported oil.

CHEMICAL IMPORTS OF CANARY ISLANDS.

Foreign Office Annual Series, No. 2605.

Candles.—The United Kingdom still sends nearly all the candles consumed; those from Spain and France are used chiefly for ecclesiastical purposes.

Soap.—The United Kingdom keeps her hold on the monopoly of the soap business, and did a much bigger trade in 1900 than in the previous year.

Starch.—The United Kingdom has entirely ceased to ship starch, and the trade has passed into the hands of Belgian manufacturers, who are now appropriating much of the trade that has hitherto gone to Germany.

Drugs.—The Mother Country has beeen pushing the drug branch of her trade at the expense of the United Kingdom and Germany. The imports in 1900 were: Spain, 44 tons; the United Kingdom, 26 tons (76 tons in 1899); and Germany, 3 tons (23 tons in 1899).

Beer.—Germany's lighter beers are still preferred in this market, and she sends twice the quantity shipped from the United Kingdom.

Glassware.—Germany has taken the lead again in the glassware business, the cheapness of her wares giving her an advantage over her competitors, Spain, the United Kingdom, France, and Belgium. Belgium, which in 1899 beat Germany in the race for first place, lost more than half her trade in 1900, while German imports were more than double those of the previous year.

Paper.—There was a considerable improvement in this trade in 1900, and most of the new business fell to the United Kingdom. Germany still keeps the lead. France and Italy send a little, and Belgium has begun to compete.

Paint and Varnish.—Imports in 1900 were double those of 1899. The business is almost all in the hands of the United Kingdom.

Cement.—The United Kingdom has lost her hold on the cement branch of the island trade. French shippers, however, have been making strenuous efforts to recover lost ground, and are now doing nearly as much business as their Belgian competitors, who have been enabled by cheap freights to overcome all opposition from both the United Kingdom and France. There is always a steady demand for this article, and price is the only consideration with buyers.

Chemical Manures.—The chemical manure business was formerly a monopoly of the United Kingdom, and she still holds the greater part of this trade, but France, Spain, and Germany have all begun to send shipments to meet the increasing demand.

Mention should be made of a few specialities which are brought to the islands in important quantities, the chief being the following:—

Powdered Sulphur.—Powdered sulphur, which comes in bags from Sicily and Marseilles, and is used for the vines.

Chemicals.—Thousands of tons are sold annually of grey and white sulphate of ammonia, superphosphate of bones, superphosphate of lime, ground dried blood, nitrate of soda and raw nitrate of potash. These are used for the manufacture of artificial manures.

CHEMICAL TRADE OF SIVAS, TURKEY.

Foreign Office Annual Series, No. 2588.

The Vice-Consul expresses the belief that a good energetic agent at Sivas could secure a good deal of business in such articles as chemicals, drugs, stationery, and possibly candles and soap.

Chemical Imports of Tangier for 1899.

Foreign Office Annual Series, No. 2603.

Candles.—The trade in candles has remained stationary. The United Kingdom retains her hold on the market in this article, 3,390 cwts. of the total of 3,576 cwts. having been supplied by British manufacturers. Germany supplied a few cases, some of them having probably been stearine candles of Dutch make.

Cement.—There still appears to be no sale for British Portland cement in presence of the competition of the cheaper though inferior foreign cements; thus, out of 1,200 barrels imported, only 30 barrels came from the United Kingdom.

Chemical and Drugs.—The principal items included under the heading of chemicals and drugs are potash to a value of 1,182*l.* from Germany and Belgium; saltpetre, 532*l.*, also from Germany; and soda, 400*l.*, principally from the United Kingdom. Saltpetre, being a monopoly, can only be imported on Government account.

Matches.—British matches meet with little sale in this country, the cheaper French and Italian small wax matches being preferred.

Oil.—A good oil crop, and consequent fall in price of olive oil of local manufacture, accounts for the comparatively small amount of cotton-seed oil imported this year —2,788*l.* worth, as compared to 9,040*l.* in the preceding year. Linseed oil was imported to the value of 112*l.*, and 742*l.* worth of olive oil came from Spain.

Potash.—About 90 per cent. of the item "chemicals" was potash for soap manufacture. The other articles of imports do not call for any particular notice.

Dyes.—676*l.* worth of indigo and 324*l.* of cochineal are the principal items under the head of dyes.

Trade of Bordeaux in 1900.

Foreign Office Annual Series, No. 2612.

Exports.—The following remarks apply to the exports of the United Kingdom only, unless specially mentioned as otherwise:—

The pure oils exported were olive oil, 7,742 cwts. against 7,340 cwts. in the previous year; ground nut oil, 16,023 cwts. against 16,811 cwts. in 1899; practically, no castor or cotton-seed oil went to the United Kingdom last year.

There was a great increase in exports of resin and turpentine, the forest owners finding prices for these articles more remunerative than those for pit-wood.

Under chemical products the principal items exported were chestnut and other vegetable tannin extracts, 72,000 cwts. against 83,960 cwts. in 1899; oxide of zinc, 131 cwts. against 772 cwts.; crystallised acetate of copper, 536 cwts. against 496 cwts.; glycerine, 357 cwts. against 1,103 cwts.; raw tartar, 2,516 cwts. against 2,185 cwts.; cream of tartar, 35,587 cwts. against 37,832 cwts; and chemical manures, 1,558 cwts. against 146 cwts. in the previous year.

The principal item under perfumery is that of common soap, 234 cwts. against 433 cwts. in 1899.

The only item of importance under pottery and porcelain is that of white porcelain, 56,621 cwts. against 43,705 cwts. in the previous year. This article comes from the Havilland factories at Limoges.

9,584 cwts. of common paper formed the principal item under this heading against 10,955 cwts. in 1899.

Under the heading of prepared hides the only items of note were 4,150 cwts. of tanned goat and sheepskins against 5,010 cwts.; other, 1,073 cwts. against 132 cwts.; curried calfskins, natural colour, 441 cwts. against 274 cwts.; and tinted or dyed dressed calfskins, 842 cwts. against 2,112 cwts. in 1899.

The whole of the manganese ore went to the United Kingdom in addition to which was sent 3,451 tons of iron ore, 1,384 tons of copper ore, 776 tons of zinc ore, and 740 tons of lead.

Imports.—The following remarks apply exclusively to the trade with the United Kingdom, unless otherwise stated:—

The principal items under fish oils were, whale oil, 464 cwts. against 206 cwts.; cod liver oil, 627 cwts. against 639 cwts.; and other oil, 1,150 cwts. against 468 cwts. in the previous year.

Of oil seeds, 647,788 cwts. came from Senegal.

The amount of cotton oil imported was 9,948 cwt. against 9,802 in 1899.

No chemical wood pulp was imported from the United Kingdom in 1900.

There was again a falling-off in coal-tar, but there were 32,420 cwts. of bitumen imported, against 24,530 cwts. in 1899.

The principal items of importation under iron and steel were cast-iron, containing less than 25 per cent. of manganese, 122,192 cwts., against 87,129 cwts., and tinned-plates, 59,540 cwts., against 25,039 cwts. in the previous year.

The principal item imported under copper was under the heading of "pure or alloyed with zinc or tin; of first fusion, cast in lumps, bars, pigs, or slabs," 1,717 cwts., against 3,069 cwts. in 1899.

The lead imported was chiefly in crude lumps or pigs, 10,148 cwts., against 16,391 cwts. in the previous year.

There was a very great falling-off in the importation of chemicals, owing principally to the freedom from disease of the vines. The principal items were bicarbonate of soda, 72 cwts. against 921 cwts. in 1899 and 2,768 cwts. in 1898. Sal ammoniac, raw, 5,858 cwts., against 9,924 cwts.; refined, 3,993 cwts., against 1,511 cwts. in 1899; super-phosphate of lime, 2,687 cwts., against 6,304 cwts. in 1899, and 21,253 cwts. in 1898; chemical manure, 112,188 cwts., against 166,910 cwts. in 1899, and 77,867 cwts. in 1898; creosote, 124,079 cwts., against 367,570 cwts., and sulphate of copper, 217,839 cwts., against 229,192 cwts. in the previous year. The falling-off in the importation of creosote is due to the substitution by the railway companies of sulphate of copper as a preservative for railway sleepers and timber generally. Other items imported were caustic soda, 233 cwts., against 198 cwts.; oxide of lead, 177 cwts., against 345 cwts.; carbonate of magnesia, 34 cwts., against 412 cwts.; chloride of lime, 147 cwts., against 269 cwts.; glycerine, 1,170 cwts., against 1,167 cwts.; sulphate of lime, 746 cwts., against 787 cwts. in the previous year.

The quantity of ground oil paint imported fell off to 191 cwts. from 283 cwts. in 1899 and 751 cwts. in 1898. There does not, however, appear to have arisen a competitor outside France, as nearly all the paint imported came from the United Kingdom.

There was an increase in the importation of coloured washes for wall papers, &c., the amount imported being 445 cwts., against 70 cwts. in 1899. The amount of ink for writing or printing imported was 31 cwts., against 20 cwts. in 1899.

There was a falling-off in all toilet soaps, transparent soap figuring for only 12 cwts., against 123 cwts., and other kinds 21 cwts., against 49 cwts. in 1899.

Of condiments were imported 109 cwts. of mustard and 109 cwts. of others, including sauces. The blacking imported amounted to 87 cwts. out of a total of 98 cwts.

The principal articles of pottery imported were common earthenware, 93,855 cwts., against 71,834 cwts.; varnished or enamelled pottery, 503 cwts., against 169 cwts.; fine earthenware, 107 cwts., against 153 cwts.; and porcelain, 109 cwts., against 80 cwts. in the previous year.

The only item of importance under the heading of glass was 5,026 cwts. of empty bottles, against 6,477 cwts. in 1899.

63. The principal kinds of paper imported were fancy paper, 33 cwts.; other writing paper, 2,168 cwts., against 2,142 cwts.; paper-hangings, 351 cwts., against 228 cwts.; and cardboard, 49 cwts., against 212 cwts. in the previous year.

64. The chief items under prepared hides are tanned or dressed goat, sheep, or lamb skins, 173 cwts., against 315 cwts.; other tanned or dressed skins, 183 cwts., against 327 cwts. in the previous year.

Sulphate of Copper.—About 2,000 tons of sulphate of copper remained over from 1899, and 11,186 tons were imported in 1900, so that the consumption reached about 13,000 tons in all. Of these fully 10,000 tons were used against the cryptogamic diseases of the vine, and the remainder for railway sleepers, telegraph poles, &c.

The syndicate founded in 1899 among the most important British manufacturers of sulphate of copper, renewed their agreement for 1900, and on the strength of this, high prices were asked at the beginning; towards May, however, after finding that manufacturers outside the syndicate had been underselling them, they had to lower their prices, and thus rendered trade most unprofitable to dealers and re-sellers in France.

As the vines were healthy and very little troubled by mildew, proprietors applied only very small doses of sulphate of copper, and were thus saved what has been a very heavy annual tax on their resources. The stock remaining at the end of the year was, however, small.

Brimstone.—The importation of brimstone during 1900 amounted to about 3,700 tons, of which about 2,500 tons were transformed into sublimated and ground sulphur for the treatment of vines. A sudden outbreak of oidium in some vine districts towards May and June cleared off the existing stocks, and rendered trade pretty brisk, although prices, which were a little below those of the previous year, kept steady during the whole season.

Creosote.—The French railway companies did not buy any creosote in 1900, using instead sulphate of copper for the preservation of sleepers. Consequently, the speculators, who bought up all available stocks in anticipation of heavy orders from France, were deceived in their calculations, and a very heavy fall in prices was the result. Creosote continues to be used as an insecticide against the vine disease called "cochenille," and seems to give satisfactory results.

Tanning.—During 1900, tanning maintained its activity, and this industry, although in its infancy here, seems to be developing steadily. The continued rise in raw material has not made the task an easy one for the tanner, who, in the face of a very small stock and defective classing, experienced great difficulties in the conduct of his business. Thanks to good work and the continued rise in prices, the production sold well both at home and abroad.

Manure.—During 1900, the total output of manure from Mazamet was 126,676 cwts., valued at about 22,400*l.*

Turpentine.—In 1900 there was a greatly increased production of turpentine, and stocks are large and probably amount to from 24,000 to 30,000 cwts. Prices rose during the first quarter of 1900 to 98 fr. 45 c. (3*l.* 18*s.*) per 2 cwts. in May, and then fell steadily throughout the year, being 83 fr. 50 c. (3*l.* 6*s.* 9*d.*) in December 1900, and in March 1901, only 77 fr. 10 c. (3*l.* 1*s.* 9*d.*) per 2 cwts. f.o.b., subject to a discount of 3½ per cent. Thus the average price for the 12 months was 86 fr. (3*l.* 8*s.* 10*d.*).

Resin and Pitch.—As in the case of turpentine, there was also a great production of resin and pitch, owing to the rise in prices, but in this case the greater part of the stocks were sold, and of resin especially, hardly any remains on hand.

Pauillac.

Coke Ovens.—The works of "La Société des Hauts Fourneaux de Pauillac" includes two blast furnaces, which are estimated to consume 90,000 tons of coke per annum. The coke will be produced on the premises by means of 60 Coppée ovens, and it is proposed to employ for this purpose British coal only. The yearly quantity that will be required is estimated at 110,000 tons. It is calculated that each blast furnace will ensure a daily production of 90 tons of pig-iron and 130 tons of other castings. There is a subordinate factory for the extraction of chemical products from coal. This department is expected to yield 3,000 tons of coal tar and 100 tons of sulphate of ammonia per annum.

Chemical Products.—A manufactory of chemical products near Pauillac is among the projects which have so far taken no practical shape. It may here be mentioned that there is a large demand for sulphur and sulphate of copper in the Médoc, and on the opposite side of the river for the preservation of vines from cryptogamic disease. There appears to be some opening at Pauillac for British trade in this direction.

Bayonne.

Chemicals.—The chemical works of St. Gobain, although not yet completed, have now started working. During the past year they received from the United Kingdom, principally from Middlesborough, Birkenhead, and Liverpool, 3,310 tons of phosphates, and recently, 2,252 tons from Coosau River, United States of America.

A German firm is about to construct works at Nay, near Pau, for the extraction of dye from chestnut wood, which is very plentiful in this district, and which has up to the present always been sent to Nantes.

Manganese Ore.—Although small quantities of manganese ore are annually shipped to Belgium and the United Kingdom, the shipments have never reached anything like the proportions expected. The mines are at St. Gerons and at Aras; the former send their shipments from Pauillac, in the Gironde, where they have their depôt.

Italian Imports and Exports in 1900.

Foreign Office Annual Series, No. 2,616.

Division.—In the official returns the imports and exports are divided in four large categories in accordance with their nature:—(1) Raw materials for manufacture; (2) other materials for manufactures; (3) manufactured articles; (4) articles of food and drink. The Consul has adopted the same system in analysing the importation and exportation of the principal goods, distinguishing those which showed an advance from those that underwent a decrease.

Category 1.

Subjoined are the articles that had a larger importation into Italy in 1900:—

Articles.	Value.	
	1900.	Increase over 1899.
	£	£
Medicinal herbs	30,920	900
Gum and resin	154,825	12,390
Barks and roots for dyeing purposes.	426,635	9,693
Raw hides	1,734,558	88,669
Cement	570,374	77,985
Non-oleaginous seeds	194,371	68,324
Bones	48,640	24,778
Raw india-rubber	265,314	19,247

Over two-thirds of the gum and resin came from the United States. Cements came principally from France, but of late factories have been started in Italy to meet increasing demand. Non-oleaginous seeds come from India and Australia. The United Kingdom only exports the oleaginous kinds to Italy. The United Kingdom export of raw india-rubber was 6,983 cwts. out of a total of 13,470 cwts.

The following showed a falling off in import:—

Articles.	Value.	
	1900.	Decrease under 1899.
	£	£
Cinchona	8,832	4,156
Indigo	83,152	77,290

The greater part of cinchona bark now comes from Holland. Limited requirements for indigo depressed the trade.

The following showed increased export in 1900 :—

Articles.	Value. 1900.	Value. Increase over 1899.
	£	£
Cements	139,987	12,995
Mercury	62,064	12,894
Sulphur	1,897,388	158,912

The most important article which seems to be reviving after the serious depression recorded in 1896 and 1897 is sulphur. The United Kingdom purchased, in 1899, 472,893 cwt. The largest customers are, however, the United States, which bought, in 1900, 3,262,887 cwt.

The following showed decreased export in 1900 : —

Articles.	Value. 1900.	Value. Decrease under 1899.
	£	£
Tartaric materials	517,839	17,450
Medicinal herbs	111,272	15,560
Barks, &c., for dyeing	110,672	25,564
Raw hides	864,365	23,642
Mineral ores	6,090,806	2,005,125
Seeds	100,210	16,463
Manures	25,495	14,087

Almost one-third of the mineral ores was shipped to the United Kingdom (2,225,868 cwts. in 1900). Since 1896, the quantity to the United Kingdom has fallen by one-half. Holland, which bought of Italy, in 1896, 73,165 cwts., purchased in 1900, 1,098,314 cwts.

Category 2.

The following imports showed an increase over 1899.

Articles.	Value. 1900.	Value. Increase over 1899.
	£	£
Fixed and other oils (except olive oil).	602,582	202,990
Ammonia	136,833	26,151
Oxides	44,473	7,515
Carbonates	125,896	5,929
Chlorates	91,808	4,718
Nitrates of soda and potash	241,957	49,878
Sulphate of soda	943,929	163,390
Charcoal	36,173	12,795
Wood pulp	271,252	32,275
Tanned hides	599,551	2,174
Raw iron, copper, steel, bronze alloys, in ingots, &c.	1,876,444	397,478
Stearic acid	68,306	14,100
Grease of all kinds	567,464	51,630

British oil exports to Italy, such as cod and others, have slightly increased.

Our exportation of acids, potash, and caustic soda, oxides, and sulphates also increased, but there was a decrease in chlorates and nitrate of potash. The latter article, which used to be supplied in quantity by the United Kingdom, has fallen very rapidly since 1896, apparently to the advantage of Central and South America and Germany. The greater part of the sulphate is provided by the United Kingdom, but the United States of America have tried to compete since 1896, so that they have almost trebled their consignments of this article to Italy.

One good feature, as far as Italian interests are concerned, is the increased importation of the chemical products recorded above. The increase is very marked, and as these products are intimately connected with local industries and manufactories, the improvement is worth noting.

Charcoal and wood pulp are not supplied by us, and tanned hides only for a small value.

A good portion of the iron, copper, &c., returned in the preceding schedule are provided by us, as may be seen in the list of British imports.

Stearic acid was imported from Holland, Germany, Belgium, Austria-Hungary, and from sundry other countries. Italian orders in Holland and Germany for stearic acid increased in 1900 as well as in the United Kingdom.

The following imports under the second category showed a decrease under 1899.

Articles.	Value. 1900.	Value. Decrease under 1899.
	£	£
Nickel, unwrought	25,823	1,715
Lead	57,345	6,660
Gold	76,080	14,572

Nickel is almost exclusively supplied by Austria-Hungary and Germany, and lead in bars is chiefly provided by Spain. In 1900 the latter country sent 48,209 cwts. out of an aggregate total of 63,983 cwts. The quantity of lead from the United Kingdom has never been very great and in 1900 it was still less so. The trade of zinc in bars has for many years been in the hands of Germany. Our exportation amounted to 4,579 cwts. in 1896, and in 1900 it was reduced to only 957 cwts. Gold in any shape is not on the British import list.

In the second category straw and esparto exports increased 22,131*l.* to 392,601*l.*

The following exports showed a decrease :—

Articles.	Value. 1900.	Value. Decrease as compared with 1899.
	£	£
Olive oil mixed with sulphur	39,306	48,682
Volatile oils and extracts	356,880	6,417
Sundry acids not specified	147,040	28,198
,, juices	161,272	3,648
Barks and roots for dyeing purposes, ground.	201,122	31,905
Charcoal	69,128	20,898
Silver scraps	109,339	28,172

The limited oil production of 1900 explains the reduced exportation of the first two articles in the preceding list. No reason is given for the falling-off in acids and juices, unless it be due to increased absorption on the part of local manufactories. British markets seem to be providing themselves elsewhere with barks and roots, judging from the following figures of the exports to the United Kingdom :—

	Cwts.
1896	351,133
1897	319,652
1898	215,695
1899	285,190
1900	184,347

France, which used also to import from Italy large quantities, has, since 1896, reduced her orders to almost one-half.

Category 3.

In the third category (manufactured articles) the following imports increased :—

Articles.	Value.	
	1900.	Increase over 1899.
	£	£
Petroleum	643,181	43,498
Alkaloids	173,398	54,038
Soap and perfumery	63,466	5,562
Paper	75,809	7,029
Mirrors and glassware	42,939	8,880
India-rubber articles	124,802	11,694

The alkaloid trade is chiefly in the hand of Germany. Very small quantities come from United Kingdom and Switzerland.

Soap is chiefly imported from—

Country.	Quantity.	
	1899.	1900.
	Cwts.	Cwts.
France	8,928	9,197
Germany	4,247	5,330
Belgium	1,802	4,371
United Kingdom	3,796	3,918

Compared with former years the importation from the United Kingdom was somewhat larger (4,867 cwts. in 1896). The quantity imported from the three other countries mentioned above has been regularly increasing. British soap is considered here as quite an article of luxury, both on account of its excellent quality and high price, rendered still higher by a duty of 2*l.* sterling per quintal (220 lbs.) Most consumers have recourse to the cheaper brands, and hence the decrease in the British article.

There was a slight advance in the importation of paper. Up to a few years ago British paper was in great request in Italy, but the decrease is due probably to local production. The British system of manufacture has been adopted, and Italy produces close to 2,000,000 cwts. of paper yearly. The number of mills is now 424, employing 15,766 workmen. The mills are distributed all over Italy. In former years France used to supply the best class of hanging paper; but the demand has almost ceased, as the same article is produced here at a smaller cost. An attempt was made to introduce on the Italian markets lincrusta of British origin for wall decoration in lieu of paper, but its price and the duty proved too heavy for the purchasers.

Visitors, however, continue to ask for British note paper, and that is the reason why in the import returns for the United Kingdom in 1898, 1899, and 1900, a certain quantity of it passed the Italian customs.

The following imports showed a decrease :—

Articles.	Value.	
	1900.	Decrease under 1899.
	£	£
Colours and varnishes	563,110	54,375
Articles of nickel	46,127	980
Starch	54,622	12,203

Germany suffered most from the diminished importation of colours. The depression may continue, as attempts are being made to produce locally colours and varnishes. Fairly good imitations of British colours have been made, and their cost less than for the qualities brought from abroad, considering the heavy duty and taxes on drying substances containing alcohol.

The following exports in the third category show increase as follows :—

Articles.	Value.	
	1900.	Increase over 1899.
	£	£
Matches	120,052	15,264
Paper	268,378	42,334

The following showed decrease :—

Articles.	Value.	
	1900.	Decrease under 1899.
	£	£
Soap and perfumery	90,712	4,233
Colours, dyes, varnishes	44,709	7,204
Candles	21,151	4,969

Category 4.

In the fourth category the following imports showed an increase :—

Articles.	Value.	
	1900.	Increase over 1899.
	£	£
Mineral waters	32,444	3,274
Beer	95,898	2,482
Alcohol	60,026	1,042
Olive oil	719,312	112,252
Cocoa and chocolate	107,728	6,280
Meat extracts	64,022	7,310

Mineral waters chiefly originate from Austria-Hungary, Germany, and France, and bottled wines almost exclusively from the latter two countries. Wine in casks is imported from Austria, Spain, Turkish Dominions, &c.; beer from Austria-Hungary, Germany, and Switzerland. There is no importation of British beer. Alcohol comes from Austria-Hungary, Germany, and France.

Of late years the importation of olive oil has risen very rapidly, owing to industrial demands, and also to local bad crops :—

	Galls.
1896	697,004
1897	716,980
1898	3,573,702
1899	3,347,982
1900	3,882,670

There was an increase of 18,788*l.* in the export of alcohol to 113,961*l.*; and a decrease of 903,428*l.* in the export of olive oil to 1,138,120*l.*

II.—FUEL, Etc.

Peat as a Substitute for Coal in Sweden.

Foreign Office Annual Series, No. 2590.

H.M. Consul at Stockholm, in a recent report, says that it is interesting to note that at the Carpalund refinery peat is exclusively used as fuel instead of coal; it is not put into the furnaces direct, but first converted into gas in generators. Altogether peat is being extensively experimented with in Sweden as a substitute for coal.

Both refineries and factories maintain steady efforts to keep abreast with the times.

Calcium Carbide Industry of Sweden.

Foreign Office Annual Series, No. 2590.

In a recent report to the Foreign Office, H.M. Consul at Stockholm states that the manufacture of calcium carbide, which suits a country like Sweden, with its numerous waterfalls, is greatly increasing.

III.—TAR PRODUCTS, PETROLEUM, Etc.

The Beaumont Oil Fields (Texas, U.S.A.).

Bd. of Trade J., May 23 *and* 30, 1901.

The centre of the new Texas oil field is 84 miles east of Houston, the largest railroad centre in the State, and 18 miles from Port Arthur, one of the principal seaports on the Gulf coast. The first gusher was struck Jan. 10, 1901, and at once an enormous stream of oil, the full size of the 6-inch casing, spouted to a height of over 200 feet, being forced upward by a pressure of over 6,000 lb. to the square inch. The pressure was so great that it was impossible to check the flow, which continued for 10 days before being brought under control. The flow could not then be measured, but was conservatively estimated at 25,000 barrels a day.

From the information now obtainable, it is evident that the amount of illuminating oil which can be obtained from the Beaumont crude oil will not exceed 20 per cent. of somewhat inferior quality, so it never will enter into serious competition with the illuminating oil from the Eastern field. The quantity and quality of the lubricating oil that can be obtained has not been fully ascertained, but it is probable that it will produce a large amount of lubricating oil of excellent quality. As it is well known that lubricating oil is the most profitable part of refining, this will add very greatly to the value of the Beaumont oil. It is safe to say, however, that at least 75 per cent. of the production will be a fuel oil, and its importance to the South, and, in fact, the whole country, must be considered from that standpoint.

The Beaumont field is almost identical in relative location and geological conditions with the Russian oil fields. The Russian oil is a heavy fuel oil of about 26° B. gravity. The Beaumont oil is also a heavy fuel oil, and is 23° B. gravity. The Russian oil fields are located near the shore of the Caspian Sea, and the Beaumont oil fields only a few miles from the Gulf of Mexico. The most productive oil strata in the Russian field are found from 1,000 to 1,500 ft. below the surface and at practically the same depth below sea level, both of which facts are exactly the same in the Beaumont field. The Russian wells are entirely through loose soil and sand, which is the same in the Beaumont wells. The Russian and Beaumont oils both have an asphalt base and many other characteristics in common. The Russian field now produces over 185,000 barrels a day, and if the first wells can be taken as an indication, the Beaumont field will certainly become even more productive when sufficiently developed. . . . .

Fuel oil from the wells in the Beaumont field can be piped to the seaboard at Port Arthur and run into tank steamers at a total cost of production and transportation not exceeding 10 cents a barrel, and $3\frac{1}{2}$ barrels of oil are equal to one ton of coal for steam purposes.

The Oil Paint and Drug Reporter of New York says:—Of the oil itself little need be said, as accurate descriptions of its physical appearance, and also analyses, have already been published. It has been claimed that other grades have been discovered, but these claims have not been proved or confirmed. In fact, all sorts of claims are made for the Beaumont oil in the extravagant statements of the company prospectuses, but the plain fact is that the oil is fit only for use as fuel, and latterly some doubts have been entertained as to its value for this purpose, by reason of the large percentage of sulphur contained, which is believed will prove very destructive to boiler flues, and every metallic surface with which the combustion gases come in contact. Doubtless some valuable products can be made from this oil, but the expense of producing them would prove too great. So far as it is at present known, the oil is suitable only for the manufacture of asphaltum mixtures for paving and other purposes, for application to roads to prevent dust, and for fuel, though, as above noted, its use as fuel may be restricted by reason of its sulphur content. That uses will be found for the product need not be doubted, but until its proper use is established and a market found, the oil has no actual commercial value, and it is idle to speculate as to what the production of any well is really worth.

Mineral Oil Industry of U.S.A.

Bd. of Trade J., June 6, 1901.

The recent oil discoveries in Texas and on the Pacific Coast lend especial interest to some facts just presented by the Treasury Bureau of Statistics regarding the exports of mineral oils from the United States. These show that the exportations of the fiscal year about to end will probably be the largest in the history of the industry which has increased its exports from 204,000,000 galls. of illuminating oil in 1875 to 721,000,000 galls. in 1900. In the quarter century from 1876 to 1900 the total value of mineral oils exported from the United States was about 1,200,000,000 dols., an average of about 48,000,000 dolls. a year; and during recent years it has averaged about 60,000,000 dolls. per annum, or 5,000,000 dols. per month.

One especially interesting feature of the development of the oil industry is that there has been a remarkable decrease in the price to the consumer during the period in which the actual exportations and the net value of the exports have been increasing. The average value of the illuminating oil exported in 1876 was about 15 cents per gallon, and in 1877, an exceptional year, 20 cents per gallon. By 1891 the price had fallen to about 10 cents per gallon, the figures for that year being 332,000,000 galls., valued at 34,000,000 dols. By 1891 the average price was about 7 cents per gallon, the exports of that year having been 564,000,000 galls., valued at 41,000,000 dolls. By 1898 the average export price was about 5 cents per gallon, the quantity exported having been 824,000,000 gals., and the value reported to the Bureau of Statistics by exporters through the Customs collectors 42,922,682 dols. In the nine months of the present fiscal year (ending April 30 last), for which the figures are completed by the Treasury Bureau of Statistics, the total exports of illuminating oil amounted to 569,624,751 galls., valued at 37,939,514 dols., or $6\frac{2}{3}$ cents per gallon; while the total value of all mineral oils exported, including crude, lubricating, and illuminating oils, naphthas, and residuum was 52,745,096 dols., and for the full fiscal year seems likely to amount to 70,000,000 dolls.

VI.—DYEING, Etc.

Textile Dyeing in Philadelphia.

Foreign Office, Annual Series, No. 2,621.

Textile dyeing forms an important industry in Philadelphia, one-sixth of all the dyeing done in the United States being done there. 90 per cent. of this work is turned out in the mills of Kensington. In addition to the amount of dyeing done in the regular dye-houses, the majority of carpet and rug manufacturers dye their own yarn.

The bulk of the finer dyes are imported from Europe, East India, and South America. The principal dye business is in cotton yarns from the east and south; these yarns are sent to the dyer in hanks and are dyed without disturbing them in any way. Wool and cotton stock also constitute an import feature of this work. Wool stock is sent to be dyed before it is carded or spun. Cotton stock is the raw cotton, which is dyed in the same manner as wool before being spun. Practical dyers make from 10 dols. (2*l.*) to 12 dols. (2*l.* 8*s.*) per week, and assistants from 8 dols. (1*l.* 12*s.*) to 9 dols. (1*l.* 16*s.*). A considerable number of children are employed in the dye-works; the factory law of Pennsylvania prescribes 12 years as the lowest age for child workers, but as a rule there are very few at work under 13 years of age.

There are at present 64 plants in Kensington, employing 3,210 people, paying 2,002,877 dols. (400,575*l.* 8*s.*) in wages, and 2,744,574 dols. (548,914*l.* 16*s.*) for materials, and turning out 6,098,776 dols. (1,219,755*l.* 4*s.*) worth of work.

VIII.—GLASS, POTTERY, AND ENAMELS.

Earthenware at Porto Allegre, Brazil.

Foreign Office, Annual Series, No. 2602.

In the earthenware trade, the bulk of which is in the commoner qualities, such as granite, painted and common chinaware, the United Kingdom has the principal share, but painted and common chinaware from Holland is beginning to compete, and is generally better finished, the material being whiter and the glaze more brilliant. The colours in the painted ware are also brighter and the designs neater, while the shapes in both classes are newer and prettier than in similar British goods, which continue to come in the old designs and shapes, no endeavour being made, apparently, to get out of the beaten track, so that anything in the way of novelty offered by competitors immediately attracts attention. The differences are not great, but they are sufficient.

A kind of porcelain, or half porcelain as it is called, comes chiefly from Germany and Belgium; glassware from Bohemia and Germany. Common glassware—tumblers, lamp chimneys, medicine bottles, &c. — is now little imported, being made locally.

New Products in the Glass Industry.

Scientific American, May 1901.

M. Leon Appert has lately read an interesting paper before the Société des Ingénieurs Civils, relating to the progress of the glass industry as shown at the Paris Exhibition. After describing the different processes of manufacture, he mentions several new products which have lately been brought out. One of these is called glass stone by its inventor, M. Garchey. It has been found that when certain kinds of glass are cooled, and then slowly reheated, a kind of precipitation takes place in the mass. The inventor uses a glass rich in lime, such as bottle-glass, for this purpose. The glass, cooled to a point somewhat below fusion, is submitted to a temperature of 1,200° C., and the plastic mass then undergoes a strong pressure by means of powerful hydraulic presses. The piece, after it comes out of the press, is annealed in the usual way. This product is said to possess remarkable qualities of hardness, inalterability, and resistance to wear. It is more elastic than ordinary glass, and is thus much less fragile. Its properties render it well adapted for paving blocks or tiling, and it may be used to advantage on the outside of buildings. The author mentions also the "strengthened glass" which has come into use, this being a flat glass plate containing a metallic network in the centre which renders it far superior to ordinary glass as regards solidity and resistance. In case of fire it will stand the highest temperature without bending. This glass may be obtained in two different ways. The French process, due to M. Appert, differs from the American, in which the rigid metal network is forced into the glass sheet; in the French process, two separate sheets of glass are rolled, and the network is introduced between them, the whole being pressed together in the rolls. Another glass which may be considered as new, although known for some years past, is that known as "opaline." This glass, of a milk-white or greenish hue, has come into use of late for tile-work, and it may in a great many cases replace ordinary tiles at a less cost. The underground stations of the Paris Metropolitan are entirely lined with these "opaline" tiles, which produce an agreeable effect. The St. Gobain glass works had an important exhibit of opaline glass at the Exhibition. The author mentions also the perforated glass, which facilitates the ventilation of dwellings, and thus renders great service from a hygienic point of view. Plates of glass for buildings, roofs, &c., are now being made of very large size, up to 15 and 18 ft. in length, and glass tubes are made as large as 20 ins. in diameter.

X.—METALLURGY.

Mineral Production of Great Britain.

Description of Mineral.	1900.				1899.
	Coal Mines Act.	Metalliferous Mines Act.	Quarries.*	Total.	Total.
	Tons.	Tons.	Tons.	Tons.	Tons.
Alum shale	1,308	..	..	1,308	5,820
Arsenic	..	4,081	..	4,081	3,829
Arsenical pyrites	..	9,573	..	9,573	13,519
Barytes	..	27,456	2,000	29,456	24,664
Bauxite	..	5,779	..	5,779	8,009
Bog ore	..	..	4,153	4,153	4,321
Chalk	..	10,124	4,363,207	4,373,331	4,678,132
Chert and flint	..	3,721	73,972	77,693	68,834
Clays and shale	3,025,054	111,570	10,913,070	14,049,694	15,064,857
Coal	225,170,163	..	11,137	225,181,300	220,094,781
Copper ore and precipitate	..	9,488	..	9,488	8,319
Fluorspar	..	1,413	35	1,448	783
Gold ore	..	20,802	..	20,802	3,047
Gravel and sand	2,215	27,026	1,807,961	1,837,202	1,771,276
Gypsum	..	152,720	55,318	208,038	212,563
Igneous rocks	..	97,698	4,536,603	4,634,301	4,709,925
Iron ore	7,667,578	1,863,714	4,496,916	14,028,208	14,461,330
„ pyrites	9,078	3,201	..	12,279	12,230
Lead ore	..	31,985	25	32,010	30,999
Limestone (other than chalk)	28,064	589,042	11,287,930	11,905,036	12,302,890
Manganese ore	..	1,362	..	1,362	415
Mica	..	..	..	..	650
Ochre, umber, &c.	..	4,270	10,930	15,200	16,314
Oil shale	2,282,221	..	..	2,282,221	2,210,824
Petroleum	..	..	..	..	5
Phosphate of lime	..	..	620	620	1,446
Rock salt	..	159,860	..	159,860	179,010
Salt from brine	..	..	1,701,487	1,701,487	1,735,883
Sandstone	105,594	258,346	4,655,894	5,019,834	5,212,624
Slate and slate slabs	..	166,695	419,645	586,340	639,840
Sulphate of strontia	..	..	9,121	9,121	12,629
Tin ore (dressed)	..	6,000	800	6,800	6,392
Uranium ore	..	41	..	41	7
Wolfram	..	8	1	9	94
Zinc ore	..	24,675	..	24,675	23,135

* Exclusive of the produce of most of the quarries less than 20 ft. deep, but including the produce of all open workings for iron ore, bog ore, ochre, phosphate of lime, sulphate of strontia, and tin ore.

Mineral Production of Algeria.

Nachrichten für Handel und Industrie, Berlin, March 25, 1901.

From 1896 to 1900 the colony of Algeria exported 3,845,566 tons of minerals, of which 1,136,197 tons were phosphates, 2,553.649 tons iron ore, 3,503 tons copper ore, 16,391 tons lead, and 135,826 tons zinc. The net results of the mineral production in 1900 were 50,000 tons less than in the preceding year, as the following table shows :—

Description.	1899.	1900.
	Tons.	Tons.
Phosphates	281,113	273,500
Iron	633,304	604,053
Copper	1,596	24
Lead	6,217	2,084
Zinc	36,952	30,250
Total	959,182	909,911

The decline was important in all branches of the mining production, but the outlook for the year 1901 is far better than that of 1899. Work will be resumed in the Guerromna mines, which have been abandoned since 1893, and a number of projects will be initiated, as new companies with powerful capital have been organised. Many large and rich mineral deposits in the interior cannot be developed on account of their distance from the sea and the lack of transportation facilities. The completion of the recently projected railways will have a beneficial effect on the mineral industries, and the colony will become of importance through its mineral wealth.

Mineral Production of Spain.

Eng. and Mining J., May 18, 1901.

The figures for the mineral production of Spain, other than iron and steel and coal and coke, are given below, collected for the *Revista Minera* of Madrid. The figures are all in metric tons.

Silver.—The total production of silver in Spain in 1900 was 94,507 kilos., all of it being obtained by desilverizing lead except about 3,000 kilos. recovered from the dry ores of Hiendelaencina. In addition 89,355 kilos. were exported in argentiferous lead. This makes the silver output in 1900 a total of 183,862 kilos., against 169,451 kilos. in 1899 ; showing an increase of 14,411 kilos.

Quicksilver.—The production in 1900 was 32,237 flasks, compared with 39,364 in 1899, showing reduction of 7,127 flasks, or 18·2 per cent. Of last year's output 30,612 flasks were produced by the Almaden mines, 1,125 by the Sociedad el Porvenir, and 500 by other companies. The falling off was entirely in the Almaden product.

Copper.—The production of copper-bearing pyrites in the provinces of Huelva and Sevilla last year was 2,652,640 tons, which compares with 2,521,875 tons in 1899 ; showing an increase of 130,765 tons. Of last year's production 1,894,504 tons were from the Rio Tinto mines, and 449,226 tons from those of the Tharsis Company. Of copper ores other than Pyritic there were mined last year 4,500 tons, of which 3,184 tons were from Navarra. The exports of copper material were, in metric tons :—

——	1899.	1900.	Changes.
Cupriferous pyrites	948,852	1,029,141	I. 80,289
Copper precipitate	28,444	29,084	I. 640
„ matte	14,509	19,591	I. 5,082
Black copper	1,089	1,235	I. 146

The total production of copper from Spanish ores las year is estimated at 45,000 tons.

Lead.—The production of lead in Spain is given as follows in metric tons.

——	1899.	1900.	Changes.
Lead exported—			
In pigs, bars, &c.	162,131	153,937	D. 8,194
In pipe, &c.	664	544	D. 120
In ores	6,212	3,109	D. 3,103
Lead consumed in Spain	13,500	14,000	I. 500
Totals	182,507	171,590	D. 10,917

The production of white lead, made chiefly in Linares, was 3,000 tons last year. The lead used in making this is included above in the lead consumed.

The lead exported in bars and pigs was as follows, in metric tons :—

——	1899.	1900.	Changes.
Argentiferous lead	68,955	71,492	I. 2,537
Soft lead	93,176	82,445	D. 10,731
Totals	162,131	153,937	D. 8,194

Exports of lead ores in 1900 were 5,181 tons, of which 3,524 tons were argentiferous galena. The total showed a decrease of 5,173 tons from 1899.

Zinc Ore.—The zinc ore mined in 1900 was 87,000 tons, against 119,770 tons in 1900. The exports in 1900 were 41,776 tons blende and 19,423 tons calamine, a total of 61,199 tons. There is only one zinc reduction plant in Spain, the Arnao Works of the Real Compania Asturiana, where 5,784 tons of spelter were made last year. In 1899 the output was 6,328 tons ; the decrease in 1900 being 544 tons.

Iron Pyrites. — Exports of pyrites last year were 356,019 tons, against 321,278 tons in 1899, an increase of 32,741 tons. Of these exports, 54,756 tons were ordinary crude pyrites ; the balance was from the mines of the province of Huelva, and was the residue left after the copper had been extracted by the wet process.

Manganese Ore.—The manganese ores mined in 1900 reached a total of 131,450 tons, all of which was exported, going chiefly to Germany. The production in 1899 was 139,352 tons, showing a decrease of 7,902 tons last year.

Asphalt.—The output of asphalt in Aleva was 3,261 tons, and in Navarra 443 tons, a total of 3,704 tons, which compares with 2,537 tons in 1899.

Cement.—The production of hydraulic cement last year was 147,831 tons. Nearly all of this was made in Guipuzcoa, Gerona, and Barcelona. The production in 1899 was 169,966 tons, so that the decrease last year was 22,135 tons. There were two works in operation last year making Portland cement, and their output was 20,000 tons.

Salt.—The total production of salt in Spain during the year 1900 was 480,000 tons, against 598,000 tons in 1899. The exports were 205,562 tons, against 326,357 tons in 1899. The decrease of 120,795 tons was due largely to the loss of the colonial trade.

Mining Industry of Huelva, Spain.

Foreign Office Annual Series, No. 2592.

Mr. Vice-Consul Ricketts reports as follows :—

An incontestable proof of the actual prosperity of this district is to be found in the Treasury returns for 1900, for, although 1899 was a relatively good year, the tax on mining, the chief and almost the only industry in the province, yielded last year over 50,000*l.* more than in that period. The outlook for copper seems to be as favourable as possible for producers, and, whilst offering inducements for the discovery and opening up of new mines in this neighbourhood, gives increased confidence and encouragement to those who are interested in the older established concerns. A glance at the particulars which are here given with regard to the output of some of the latter, taking into consideration the sustained high price of copper and the favourable rate of exchange (which varied from 29 to 38 per cent. at different periods during the year), cannot be other than

satisfactory to all who are aware of the enormous amount of British capital invested in the best of these undertakings, and a perusal of the return of imports from the United Kingdom will show that this is not only an important but a growing market for British trade.

Annex A shows the principal articles of import from the United Kingdom to the port of Huelva during the year 1900. Compared with the previous year, the following show a satisfactory increase:—Cement, coal, coke, naphtha, and manufactured iron and steel. The only items in the list which show an important decrease, on comparison with 1899, are railway material and anchors and chains, but these, of course, are supplied to meet special requirements and are not the object of a continuous steady demand. Rather less tin was imported than in the previous year, but all that was used came from the United Kingdom.

It is worthy of note that 1,245 tons of chemical manures were imported from the United Kingdom last year, and there will probably be a still greater demand in 1901. Another new feature, which has attracted some attention, has been the importation of some cargoes of Jarrah wood railway sleepers from Australia, an experiment which has met with great success.

Annex B deals with the exports to the United Kingdom, but there is nothing special to report in connexion with the same. Cupreous iron pyrites was again the principal article exported to the United Kingdom, and the quantity sent forward amounted to over 500,000 tons, or 10,000 tons in excess of the quantity shipped in 1899.

There was also an increase in the export of sulphate of copper, cork (both in bales and manufactured), copper matte, chestnuts, &c., whilst precipitate of copper, iron pyrites, lead ore, mercury, manganese, oranges and dried figs were in less demand, but not to an appreciable extent, except precipitate of copper.

To account for the slight reduction in the shipments of iron pyrites, it may be mentioned that almost invariably the mines working iron pyrites work also copper pyrites, and with copper at its present price the inducements are much greater to work the latter than the former.

The total extraction of mineral at the Rio Tinto mines during 1900 was 1,985,760 tons, with an average copper content of 2·74 per cent., as compared with 1,701,730 tons (2·72 per cent. copper) during 1899. The overburden removed in 1900 was 1,572,216 cubic yards, bringing the total removed up to 17,500,000 cubic yards.

The following was the distribution of the mineral last year:—

	Tons.
For export	781,377
„ smelter	111,250
„ washing in heaps	1,092,133

The exportation of ore during that period was divided as follows:—

	Tons.
Shipped to Great Britain and the Continent	554,572
„ United States	226,194

Of the products exported, 20,391 tons were in the form of precipitate (or "cascara,") and 19,344 tons in the form of matte. The sulphate of copper manufactured, and which was divided in about equal parts between Spanish consumption and export, amounted to over 4,000 tons.

The total shipments from this company's pier at Huelva amounted to 820,800 tons in 383 steamers, or an average of 2,143 tons.

The number of hands employed during the year by the Rio Tinto Company was 10,243, and the number of fatal accidents 14, or 1·36 per 1,000. The total mileage of railway is 74 miles main line, 85 miles mine lines with 21 main line and 50 mine line locomotives, and 2,600 ore waggons, in addition to passenger rolling stock. New lines are being constructed to Nerva and Zalamea, chiefly for the use of the company's operatives, who will get very cheap rates.

The new smelting and Bessemerising works, which will have two of the largest-sized copper furnaces ever made, and two large copper converters, for making blister copper, are nearly completed. All the required plant for these new works was obtained from the United States.

The Tharsis mines are reported to have shipped during the past year some 8,000 tons of precipitate of copper, 73,163 tons of washed ore, and 227,486 tons of cupreous iron pyrites.

The Peña de Hierro mine shipped some 50,000 tons of copper pyrites ($2\frac{1}{2}$ to $3\frac{1}{2}$ per cent.) during 1900, and 397 tons of precipitate.

Tinto and Santa Rosa shipped 41,384 tons of cupreous pyrites, most of it high grade, but some very poor.

Sotiel Coronada produced 16,000 tons of cupreous pyrites, 285 tons of washed ore, and some precipitate.

El Castillo del Buition produced 14,560 tons of iron pyrites and a little precipitate.

The San Miguel mines shipped 669 tons of precipitate.

At the Las Herrerias Mine 52,000 tons of pyrites, (about $1\frac{1}{2}$ per cent. Cu.) were extracted. All this was treated at the mines and gave about 1,090 tons of precipitate (82 per cent.) all shipped to the Tyne.

The Campanario Mines produced 744 tons of cupreous pyrites.

The Societé Francaise des Pyrites de Huelva have completed the railway for the Perrunal Mines, and exportation will commence in June. The output, in iron pyrites especially free from copper, is estimated at 100,000 tons per annum. This company also exported last year 70,000 tons of mineral from their old mine "Confessionario," now exhausted.

Whilst referring to the **taxation on minerals**, special attention is due to the recent legislation on the subject. The decision at which the Director-General of Customs lately arrived with regard to the export tax on minerals is of vital importance to Huelva as the largest shipping port of copper ore in the Peninsula. The whole question is long and involved, but the chief features can be roughly sketched as follows:—An export tax of $1\frac{7}{17}d.$ per metric ton on iron ore, and of 1*s.* $2\frac{9}{17}d.$ on copper ore was imposed in March, 1900. This tax, although much resented by the mining interest, worked relatively smoothly at first, and produced an enormous revenue, it being always understood that mineral and washed ores, containing so small a percentage of copper, as to have no commercial copper value were free of the copper tax. Towards the end of 1900, however, the Director-General of Customs not only decided to the contrary, but ordered the Customs officials to collect full copper dues on all washed ores, and to make the order retrospective to all shipments since March, 1900. This produced a great outcry, as it was beyond the power of mine owners or merchants to recover the tax after delivery of the mineral to buyers abroad, and a committee of representatives of the principal mines proceeeded to Madrid, and protested with some measure of success against what was considered so arbitrary a proceeding.

It has been temporarily agreed, that this arrangement will probably soon be confirmed by Royal Decree, that:—

"All ore under 1 per centum of copper be classed as iron pyrites.

"Ore declared to the Custom-house to contain under 1 per cent. and found to contain more than 1 per cent. but less than 1·15 per cent. of copper will pay transport and export taxes as copper, but will not be subject to fine.

"Ore found to contain more than 1·15 per cent. of copper will be liable to the fines and penalties which obtain generally for false declaration, unless so declared."

This applies to crude and to washed ores, and to all shipments made since March, 1900, of which the Custom-house have samples in Madrid. In future, samples of all cargoes will be sent to Madrid and there assayed to prove the veracity of shippers' declarations.

Manganese.—The export of manganese from this province, which in 1899 amounted to no less than 148,419 tons, or nearly 25 per cent. of the world's consumption, went back in 1900 to 129,916 tons, or a reduction of 18,503 tons. Of these 129,916 tons, 1,213 tons only were shipped to the United Kingdom, 2,221 tons to France, and the remaining 126,482 tons to Antwerp. Fully three-fourths of this quantity may be classed as poor ore averaging 31 to 32 per cent. of manganese metal and 28 to 30 per cent. of silica;

the high contents of silica render these ores unfit for treatment of ferromanganese and for fair class iron ore, whilst they are found suitable for the "Minette" ores of Luxembourg, which require silica. This circumstance accounts for the large export to Belgium.

The Huelva production for 1901 is likely to again show a falling-off, as the manganese formation occurs in limited superficial lodes, which are being fast exhausted, and the poor quality and, in most cases, the heavy transport charges are a serious obstacle to the working of new mines at a greater distance from the port.

Exact particulars of the world's production of manganese during 1900 are not available at the time of writing, but the following table shows part of the European consumption for that period and the source of supply :—

	Tons.
Caucasus	325,221
Chile	12,662
India	89,845
Turkey	21,355
Japan	250
Portugal	1,375
France	8,555
Greece	9,064
Brazil	74,910
Australia	5
Java	2,203
Southern Russia	17,313
Asia Minor	1,187
	563,945
Huelva	129,916
Total	693,861

To the 693,861 tons must be added the United States importation, the United States home production, and the German, Belgian, Swedish, Austro Hungarian, and Russian home production and consumption; thus the world's consumption of manganese ore for 1900 can be safely computed at 900,000 tons, or an increase of nearly 300,000 tons over the consumption of the previous year.

Annex A.—Return of Chemical Articles of Import from United Kingdom to the Port of Huelva during 1899–1900.

Articles.	Quantity.		
	1899.	1900.	
	Kilos.	Kilos.	Tons.
Cement	271,891	479,281	471
Coke	12,612,564	13,434,625	13,223
Pitch and tar	831	14,403	14
Mineral oils	81,575	..	..
Naphtha	55,207	147,625	145
Copper	17,172	18,320	18
„ tubes	9,961	8,467	8
Tin ingots	13,117	10,510	10
Copper wire	..	4,234	4
Beer	8,573	4,780	4
Chemical manures	..	1,265,487	1,245

Annex B.—Return of Principal Articles of Export to Great Britain from the Port of Huelva during the Years 1899–1900.

Articles.	Quantity.		
	1899.	1900.	
	Kilos.	Kilos.	Tons.
Cupreous pyrites	521,261,156	533,525,425	523,155
Copper precipitate	30,082,662	27,587,125	27,105
„ matte	14,508,745	19,555,080	19,247
„ sulphate of	24,210	129,147	127
Iron pyrites	66,783,940	39,780,360	39,154
Lead ore	1,092,290	525,380	521
Mercury	820,000	205,000	201
Manganese	103,820	..	..

Iron Production of Bilbao, Spain.

Foreign Office Annual Series, No. 2598.

The Biscay ores are generally divided into four classes, the vena or campanil, campanil (inferior), rubio (limonite), and siderite or spathic.

The following analysis of Biscay ores has been made :—

	Spathic.		Vena.	Campanil.	Superior Rubio.	Inferior Rubio.
	Superior.	Inferior.				
Peroxide of iron	1·400	2·850	81·157	75·357	78·514	72·950
Protoxide of iron	52·070	47·300	..	..	..	..
Peroxide of manganese	1·480	1·100	1·340	2·110	0·900	0·780
„ copper	..	..	..	..	..	..
„ lead	..	..	..	..	..	..
Alumina	0·170	0·300	1·200	1·840	1·250	1·700
Lime	1·700	1·560	1·310	5·530	0·850	0·500
Magnesia	0·450	0·870	0·450	1·540	0·550	0·250
Sulphuric acid	0·351	0·675	0·042	0·035	0·058	0·095
Phosphoric acid	0·040	0·045	0·035	0·025	0·031	0·055
Arsenic acid	..	..	..	..	..	..
Carbonic acid	33·633	32·957	0·100	0·093	0·650	0·850
Titanic acid	..	..	..	..	..	..
Silica	6·590	8·990	6·210	5·300	7·120	9·750
Combined water	0·480	1·480	0·120	0·700	4·100	6·950
Moisture	1·620	1·950	8·150	7·470	6·100	6·100
Total	99·984	100·077	100·114	100·000	100·123	99·980
Metallic iron	41·474	38·780	56·809	52·746	54·959	51·065
Manganese	0·935	0·695	0·846	1·333	0·568	0·492
Phosphorus	0·017	0·019	0·015	0·010	0·013	0·024
Sulphur	0·140	0·270	0·016	0·014	0·025	0·040
Loss in calcination	32·270	32·480	..	..	..	..
Iron in the calcined ore	58·100	55·500	..	..	..	..

The vena is the purest of all the Biscay ores, and was the only one worked when production was limited to the demands of local work. The Bessemer process caused campanil to be largely used, but both the vena and campanil beds are now nearly worked out.

The spathic iron is found at times in small quantities mixed with masses of campanil or rubio, and in "pockets" bedded in other minerals, but more generally underneath them.

The vast proportion of ore now shipped from here consists of rubio.

It has been estimated that the mines in Biscay now being worked, and others which are constantly being opened, can put out 57,000,000 tons before they are exhausted, and those of Santander 10,000,000 tons.

The first attempts at calcination for the utilisation of siderite were made in 1881, but it was not until 1889 that it was resorted to on a large scale. At present

there are about 33 kilns which turned out 613,575 tons in 1899.

The treatment of spathic ore by calcination was fully dealt with by Mr. Consul Smith in his report for 1894. The same process is now made use of for the treatment of inferior rubio. It has been found to answer well.

The increasing demand for the rich hematites of Biscay and the exhaustion of the beds also led mine-owners to resort to mechanical means for washing this ore and the contents of their old rubbish heaps. Two systems are in use: 1. The cylinder of open metal work revolving horizontally in a tank, thereby allowing the clay, &c., to drop through; 2. The drum system ending in a cone in which the ore mounts and the water descends with the dirt. The mineral comes out clean at the top and falls on a table where it is further dealt with. The latter system has been almost generally adopted, and has given excellent results, especially as regards old and formerly valueless rubbish heaps which have turned to good account.

The exports for the last seven years have been as follows:—

To—	Quantity.						
	1894.	1895.	1896.	1897.	1898.	1899.	1900.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
United Kingdom	2,806,686	3,006,600	3,572,993	3,167,147	3,060,801	3,966,129	3,101,563
Holland	618,903	586,656	768,795	814,995	830,311	861,669	703,766
Germany	..	4,175	5,229	37,939	11,618	32,821	66,401
Belgium	83,859	121,605	129,037	280,108	154,526	205,953	207,925
France	280,411	299,527	312,370	316,971	285,561	282,109	200,322
Italy	..	964	1,810	..	976	..	..
United States of America	563	17,530	37,166	31,907	3,042	59,657	49,445
Austria-Hungary	..	..	..	..	1,382	..	..
Norway	..	..	..	..	..	4,425	..
Total	3,790,422	4,037,057	4,827,400	4,649,067	4,348,217	5,412,763	4,329,322

The largest shipment came from the Orconera Iron Ore Company, Limited, and amounted to 1,408,499 tons.

Mining Industry of North of Spain.

Bd. of Trade J., May 30, 1901.

H.M. Consul at Corunna, reporting on the mining industry in Spain (Foreign Office Annual Series, 2586), states that the arsenical gold pyrites of his district contain an average of about 20 per cent. arsenic, and from 10 to 16 dwts. of gold to the ton. The mispickel of Cornwall contains no gold, and only 8 per cent. of arsenic, and it is well known that these mines are the most paying and prosperous in Cornwall.

Considering the above figures, it certainly appears, therefore, that the Corunna mines are well worth attention, not only for the gold contained, but even more so for the arsenic. An influential group of Cornish capitalists, largely interested in the arsenic trade, has lately sent out an engineer and mining expert to report on these mines.

The Consul states that the demand from England for an ore as pure as possible from phosphorus has, perhaps, led exporters at Bilbao to cater too much for the British market. It is worthy of note that the German ironworkers make use of highly phosphoric ore, which British ironmasters will not even look at. A case has been quoted in which an engineer of the Government of India railways refused to accept a large supply of rails because the steel was found on testing to contain 0·005 of phosphorus above the usual British specification of 0·06 per cent. In Germany steel rails are made and passed with 0·08 and even 0·10 per cent. of phosphorus. It is contended by some that the British specification of 0·06 per cent. as a standard of purity is too high, and that a rail is perfectly sound and has as long a life with a percentage of phosphorus not exceeding 0·08 as one that has a much lower percentage, provided always that care is taken to regulate other ingredients. If, however, the percentage of 0·08 is exceeded the rail becomes crystalline in structure, and hence brittle and more liable to break. However this may be, the result is that Germans are able to obtain ore in large quantities and cheaper than British traders, and can utilise ore that a British ironmaster will not have at any price.

The Consul is of opinion that, coming as these statements do from a man who has had considerable experience in British foundries, they should not be dismissed without careful examination. Spathic ore and washed ore were both eagerly bought up by German houses and used for years before they were touched by British ironfounders, the simple reason being that British makers would not make the necessary changes in their plant.

It is a curious geological fact that the further west the mines are from Bilbao, the more phosphoric is the ore. The highly phosphoric ore of Galicia practically all goes to Germany. A considerable amount of Bilbao capital is now beginning to be invested in Galician mines.

The report from the British Vice-Consul at Gijon, states that the advantages to be gained by a close study of the mineral wealth of that district, have been commented on in previous reports, especially since the question of cheap minerals has become so acute, and the Vice-Consul wishes to impress the importance of becoming more acquainted with the undeveloped coalfields. iron, manganese, and other deposits of minerals in the province. The coal area is calculated to be 540 square kilos., of which only an insignificant part is being worked, owing to want of better facilities for transport and improved accommodation for shipment. The iron area is calculated at 145 sq. kilos., of which 14 kilos. alone are being worked.

Iron ore in Asturias gives 40 to 51 per cent. It contains a considerable proportion of silica and some sulphur, but this defect is fully compensated by the abundance of the mineral, as there are beds of 5 kilos. long, with seven layers, some of which present a depth of 10 metres.

Mineral Exports of Milo (Cyclades) in 1900.

Foreign Office Annual Series, No. 2599.

The principal exports of Milo were mineral produce amounting to 338,267 dr. (8,210*l.*), and consisted of 16,976 tons of manganese ore, of which 16,050 tons were shipped for the United Kingdom, and the remainder to Laurium; 847 tons of sulphur sent to different Greek ports; 13,447 pieces of mill-stone to Crete and Greece; 96 tons of kaolin, of which 80 tons went to the United Kingdon, and 1,485 okes (37½ cwts.) of gypsum sent to Greece. The total exports show a considerable decrease compared with the preceding year, but for no special reason beyond the ordinary fluctuation of trade. The minerals exported to the United Kingdom were carried in for British steamers representing an aggregate of 16,950 gross tonnage.

Mercury Production.

A. Weiskopf. Zeits. f. angew. Chem. **14**, 429, 432.

(See also p. .)

Mercury occurs in nature as a cinnabar HgS, in combination with sulphides of copper, silver, and iron. It is found

in sedimentary stone formations in Almaden, in Spain, similarly at Huancaorlia, in Peru, and in Idria; the same mineral occurs between the strata, and also mixed with bitumen and earthy deposits. Other important sources are the mines of the Napa Consolidated Quicksilver Mining Company, at New Almaden, New Idria, Altoona, and Ætna, all in California, where the cinnabar is mixed with serpentine, trachite, and basalt, and their adjacent chalk formations. It is also found at Kotterback and Dobschau, in Hungary, and at Monte Amiata, in Tuscany, as cinnabar; at Cornacchino and Montebuono as an ore, containing from 0·4 to 1·2 per cent. of mercury. In Russia, at Nikitowka, in the district of Bachmunt, a cinnabar is mined which is similar to that of Almaden, the ore yielding from 0·6 to 20 per cent. of mercury.

The United States of America produced in 1898 31,092 bottles of mercury, worth 1,188,627 dols., and in the year following, 638 bottles less, but owing to the market condition the value in the latter year was 264,118 dols. more. The prices in 1899 were the highest since 1890. In January 1899, mercury was selling in San Francisco at 42 dols. per bottle ($76\frac{1}{2}$ lb.) for home use and 37·50 dols. for export; but in December the prices had advanced to 51·50 dols. for home use and 47 dols. for export. During the past twenty years all the American quicksilver has come from California. except 65 bottles from Oregon in 1887, and Texas during the last year or two has supplied 1,000 bottles from the Terlingua quicksilver-mining district. In Hungary the Rima Muranyer Gewerkschaft, including Dobschau, produce about 1,000 kilos. per annum. The Russian output is calculated to be 200,000 pouds of mercury, 1 poud equalling 16·38 kilos. From statistics collected by the Metal and Metallurgical Society at Frankfort, it appears that the world's production in 1899 was as follows:—

	Tons.
Spain	1,357
United States of America	993
Austria-Hungary	500
Russia	360
Italy	206

The production for the ten years ending 1899 is shown by the following table, the figures as to quantities referring to metric tons:—

Year.	U.S.A.	Spain.	Austria-Hungary.	Russia.	Italy.	Total.
1890	796	1,819	542	292	449	3,898
1891	794	1,790	570	324	330	3,803
1892	971	1,657	542	343	325	3,838
1893	1,047	1,665	512	200	373	3,697
1894	1,056	1,609	519	196	258	3,638
1895	1,179	1,506	535	434	199	3,835
1896	1,036	1,524	564	492	186	3,802
1897	965	1,728	532	617	192	4,034
1898	1,059	1,691	491	362	173	3,775
1899	993	1,357	500	360	206	3,416

In addition to the sources above named, Mexico, China, Japan, Chile, and Peru also contribute, but the amount cannot be accurately ascertained.

Thus in Mexico there is a large number of small mines, which give no statistics; but apart from these, the following has been the production in metric tons:—

1893	286	1897	294
1894	309	1898	535
1895	213	1899	324
1896	218		

Mining in Laurium and Eubœa, Greece.

Foreign Office Annual Series, No. 2,614.

Laurium Mines.—The output from the mines was rather below the average, and there is a prospect of calamine zinc ore being shortly entirely exhausted in this district, although expensive works have been carried on to discover new veins. About 10 years ago the output of this ore amounted to 60,000 tons in the year, whereas in 1900 the output fell to 18,500.

The ancient deposits of pit refuse are also being gradually worked out, and the lead foundries of the Greek company have of late not been fully working in consequence. This company has, however, purchased a considerable number of lots in the mines recently discovered in the island of Myconos, and it is expected that the foundries of the company will be well supplied for some years to come by the Caramania lead mines recently purchased by them.

The following are the quantities of marketable ore yielded by the mines all over the district:—

	Tons.
Manganese iron ore	320,245
Hematite iron ore	171,377
Roasted calamine or zinc ore	18,505
Export rich galena	1,552
Arsenic lead smokes	2,260
Speiss	1,832
Total	515,771

In addition to the above, 264,614 tons of raw and dressed lead ore passed through the furnaces, out of which 16,719 tons of pig-lead were produced, containing an average of about 81 ozs. of silver to the ton of lead.

Mining in Eubœa.—Attention should be drawn to the mining operations now carried on in the island of Eubœa. The company called "Petrifite, Limited," which was floated three years ago with a share capital of 12,450,000 dr. (300,000*l.*), the majority of the shareholders being British, has concessions for the extraction of magnesite in the north of the island. During last year 7,500 tons of this mineral were exported by the company, who are augmenting their appliances so as to enable them to increase the output on a a very considerable scale.

The Greek company called the "Société des Travaux Publics" has also concessions and ship annually some 20,000 tons of magnesite.

The green Cippolino marble at the South of Eubœa is being brought out now in larger quantities, as also is that from Tinos, and each of these marbles, more especially the former, is establishing a sound demand.

XII.—OILS, Etc.

Cinghalese Oils.

Imp. Inst. J., June 1901.

Among the Cinghalese exhibits in the Paris Exhibition of last year were several oils obtained from trees and plants growing wild in that colony. Some of these have a considerable reputation among the natives of India and Ceylon as medicinal agents. With the view of making them better known, the *Tropical Agriculturist* of Ceylon, for March and April 1901, contains brief notes on some of the more important of them.

Duhudu Oil is obtained from *Celastrus paniculatus* belonging to the Natural Order *Celastraceæ*. The seeds of this shrub yield a dark-red pungent oil, which has a tendency to deposit a solid fat when kept. It is taken internally as a nerve stimulant, and is recommended for external application to sores.

Iriya Oil is the product of *Myristica irya* (*Myristicaceæ*), an evergreen shrub. The oil expressed from the bark is commonly applied to cutaneous diseases by the natives.

Wal-del Oil is extracted from the seeds of *Artocarpus nobilis*, of the Natural Order *Urticaceæ*. The outer shell of the seed when roasted is used as a food-stuff, while the juice obtained from the bark finds application as an insecticide.

Makulu Oil, which is obtained from the seeds of *Hydnocarpus venenata* (*Bixineæ*), a large tree growing generally in the riparian forests, is of the consistency of soft butter. It is known in South India as *Thertag oil*, and is recommended as a valuable substitute for *Chalmugra oil* in the treatment of leprosy.

Divikaduru Oil, expressed from the seeds of *Tabernæmontana dichotoma*, of the Natural Order *Apocynaceæ*, frequently known as the "forbidden fruit" and "Eve's apple." The oil is used in native embrocations.

Madol Oil, the product of *Garcinae echinocarpa* (*Guttiferæ*). The seeds yield a thick oil, which can be used in lamps for burning. It is also used medicinally as a vermifuge.

Dorana Oil is obtained from the wood of *Dipterocarpus glandulosus* (*Dipterocarpaceæ*). It is a dark-coloured resinous oil. Like the *Makulu oil*, it is used in the treatment of leprosy, and as a substitute for gurjun balsam, derived from an allied species of plant. It forms a good varnish when mixed with Hal resin, and a red lacquer with vermilion.

Me Oil is yielded on crushing the seeds of *Bassia longifolia*, belonging to the Natural Order *Sapotaceæ*. It solidifies on standing, and is applied externally in the treatment of skin diseases. The residual cake, known as "Arappo," is exported to the Indian coast.

Kekuna Oil, sometimes known as candle-nut oil, is obtained from the seeds of *Aleurites tribola* (*Euphorbiaceæ*). It is suitable for soap-making and cloth-dressing. It resembles and is an efficient substitute for linseed oil in mixing paints. The yield of oil from the seeds is about 50 per cent. The roasted nut forms an agreeable food.

Domba Oil is obtained from the nuts of *Calophyllum inophyllum* (*Guttiferæ*), sometimes known as the Alexandrian laurel, a moderate-sized tree, growing generally by the sea coast. The yield of oil from the nuts, which are sometimes exported to India, is considerable, averaging up to 60 per cent. The oil is chiefly used as an embrocation for rheumatism and for burning. Domba oil is sold in Burma at four times the Calcutta price of castor oil, which it resembles.

Mr. L. Field, in reporting on Me oil, Kekuna oil, and Domba oil, predicts a considerable demand for them, if they can be supplied cheaply.

Margosa or *Kohomba Oil*, the product of *Asadriachta indica* (*Meliaceæ*), is an antiseptic, and as such is used as a cattle medicine. It is also applied externally for rheumatism. Unfortunately, it has a very objectionable odour, which may prevent its more extended use.

Kon Oil.—This oil, which is clear and quite liquid, is used for illuminating purposes, and in culinary operations. It is the product of *Schleicheria trijuga*, belonging to the Natural Order *Sapindacaceæ*, and is reported to have been introduced into German commerce under the name of Macassar oil.

Market for Soap in Spain.

Chem. and Druggist, June 1, 1901.

The American Consul-General at Barcelona states that the manufacture of soap in Spain has greatly increased during late years, the large exports of Cuba having fostered this industry, and enabled it to acquire an importance that but for that outlet it would never have reached. Especially was this the case before the loss of the colonies, for in 1897 Spain exported 7,045 tons to her dependencies, and only 112 tons to other countries. During 1900 the exports amounted to 6,107 tons, while those for the first two months of this year reached 1,792 tons. In the province of Barcelona alone there are over 100 soap factories. Cocoanut oil forms one of the principal ingredients of the soap made in Barcelona, the cocoanuts or copra being brought from the Pacific Islands, and crushed at the oil mills. Vegetable oils were formerly imported, but, owing to the high protective duties now levied on them, the principal firm in the Spanish trade found it necessary to cross the frontier and erect a mill at Barcelona, in order to compete with the local oil-crushers. Caustic soda of 60° strength is imported from England; the present price is 23.65f. per 100 kilos. c.i.f. Barcelona. The price at which the common yellow soap is sold for export is equal to about 1½*d.* per lb. delivered f.o.b. at Barcelona.

Bean Oil and Cake at Swatow, China.

Foreign Office Annual Series, No. 2620.

To the Swatow farmer the supply of cheap beancake is of the first importance. With no system of rotation in the cultivation of his land, and raising crop after crop of sugar-cane on the same fields, the soil soon becomes exhausted and artificial manures necessary. In this connexion beancake has been found to meet the farmer's requirements and the nature of the soil; it is an excellent fertiliser, besides being both cheap and plentiful.

Locally, the import of beans furnishes a new industry to Swatow in extracting and manufacturing bean oil. In addition to ordinary hand-presses there are now steam mills in full operation, with the result that, after satisfying the heavy local demands, 43,250 cwts. were shipped to Hong-Kong, valued at 48,276*l.*, which in 1900 reached 59,868 cwts., valued at 73,569*l.*

XIV.—TANNING, LEATHER, GLUE, SIZE, Etc.

Quebracho Exports from Buenos Ayres.

Foreign Office Annual Series, No. 2615.

Quebracho Colorado (red quebracho) is a hard wood which has been used with success in the tanneries of Europe for some eight or ten years past and is now coming into use in the United States of North America.

The following figures show the export of quebracho wood from the Argentine Republic during the last nine years, viz.:—

	Tons.
1892	29,700
1895	155,000
1900	225,000

The quebracho tree, known to exporters as Gran Chaco quality, is found in a large belt of forest which commences at the Fives Lille Station in the province of Sante Fé, and runs due north to the 26th parallel of latitude. This belt of quebracho forest is about 15 to 20 miles in width and is situated on the west bank of the River Paraná at a distance of several miles from this river. The largest works are at Calchaqui, Guampita, Espin, Vera, Colmena, Garaboto, Golondrina, and other stations on the line of railway recently purchased by a French company from the Sante Fé Government.

Quebracho Colorado is also found in Corrientes and the interior provinces of the Argentine Republic, but it contains a smaller percentage of tannin than the Gran Chaco wood and is therefore not exported, but a ready sale is found for it in the Argentine Republic for railway sleepers, piles for wharves and bridges and for rafters of buildings.

The Gran Chaco wood, exported for tanning purposes, is shipped in the form of logs from which the bark and sap-wood have been pealed.

The best Gran Chaco wood gives the leather a clear light yellow tint and considerably increases its weight. Very good results are obtained by mixing quebracho with hemlock, oak, gambier, and other tanning materials, and the expense of tanning sole leather is thereby reduced.

Herr Eduard Muller in his pamphlet on the subject gives the following

Comparative Analysis.

	Tannic Acid. Per Cent.
Quebracho wood from the Gran Chaco	22—28
Empedrado	18—20
Santiago del Estero	10—17

The specific weight of the poorer kinds of quebracho wood is about 20 to 30 per cent. less than that of quebracho from the Gran Chaco.

For some years the quebracho forests have been worked in a primitive manner, but lately two factories for the manufacture of extract of quebracho have been erected in the Argentine Republic with German capital, and British,

French, and North American companies are now considering the advisability of putting up similar factories on a large scale.

The principal port for the shipment of Gran Chaco quebracho is that of Colastiné in the province of Santa Fé.

The value of quebracho sleepers exported in 1900 amounted to 47,000*l.*; in 1899, to 3,000*l.*; of quebracho extract in 1900, to 119,200*l.*; in 1899, to 63,400*l.*; of quebracho logs in 1900, to 479,600*l.*; in 1899, to 318,800*l.*

XV.—MANURES, Etc.

The Phosphates of Christmas Island.

Bd. of Trade J., May 30, 1901 (*see also this Journal*, 1901, 167 *and* 405).

The report for 1900 on Christmas Island, recently issued by the Colonial Office, says that two kinds of phosphate are found on the island, lump phosphate and granulated phosphate, and it is understood that the latter is slightly the more valuable of the two. Granulated phosphate has the appearance of coarse white sand, and so far none has been shipped, as it cannot be handled with the plant now in use.

A large number of narrow pits have been dug, and the quantity of phosphate discovered is enormous. In some cases at a depth of 40 feet the bottom of the deposits has not yet been reached.

To give some idea of the amount of phosphate, details are appended of seven pits sunk in a row across the summit of Phosphate Hill at intervals of 60 yards:—

Pit 1.—Lump phosphate from surface to 6 feet.
Granular phosphate from 6 feet to 11 feet.
„ 2.—Lump phosphate from surface to 2 feet.
Granular phosphate from 3 feet to 26 feet.
„ 3.—Lump phosphate from surface to 22 feet.
Granular phosphate from 23 feet to 36 feet, and limestone not yet reached.
„ 4.—Lump phosphate from surface to 13 feet.
Granular phosphate from 13 feet to 22 feet, and limestone not yet reached.
„ 5.—Lump phosphate from surface to 12 feet.
Granular phosphate from 12 feet to 24 feet.
„ 6.—Lump phosphate from surface to 7 feet.
Granular phosphate from 7 feet to 11 feet.
„ 7.—Lump phosphate from surface to 6 feet.
Granular phosphate none.

To a depth of 3 feet the lump phosphate can be picked out, and will not need washing. For the next 7 feet washing will probably be advisable, although not necessary. Below that depth washing will be necessary until the granular phosphate is reached. This will never require washing, and can be exported in bulk in its present condition.

It is understood that the latest estimate of the amount of phosphate on Phosphate Hill is 12,000,000 tons.

No prospecting has been done in other parts of the island, but it is known that phosphate exists at Murray Hill, as it was from there that the original samples were taken. Even in the unlikely event of phosphate in paying quantities not being found in other parts of the island, it is evident that sufficient has already been discovered to give employment to the Christmas Island Phosphate Company for a very considerable period.

The quantity of phosphate said to be shipped from the island from November 1899 to October 1900 amounted to 28,017 tons, and it is estimated that 175,000 tons will be shipped in 1901.

XVI.—SUGAR.

Sugar Industry of Pernambuco, Brazil.

Foreign Office Annual Series, No. 2591.

The sugar industry, which has thrived for several consecutive years, appears to be about to pass through as severe a crisis as has been experienced in British Guiana, the West Indian Islands, and elsewhere. Nor will the return of Cuba to the field of competition in the production of cane sugar make matters easier for the Brazilian sugar planters of this and neighbouring states.

Diffusion.—In the 1894 report on the trade of Pernambuco, mention was made of the system of extracting the juice of the cane by diffusion. The factory mentioned (Cucaú) was then, and is still, the only one that has tried the system of diffusion. From a commercial point of view it has proved a terrible failure, and the company has become bankrupt, after expending some 9,000,000 milreis, or at 9*d.* exchange, which is taken as the average at which gold has been expended on it, over 300,000*l.*

Although this money includes the cost of a narrow-gauge railway some 44 miles long, with nine locomotives and sufficient rolling stock, yet the value of the whole concern could not be estimated at more than 100,000*l.* But this does not prove that the diffusion process is a failure in Brazil, and the consul is inclined to modify his views in this respect. He finds that the question of fuel, which he thought so vital, is partly solved in the present case by the long distance traversed by the railway, and, therefore, the wood fuel can be procured from far or near to the factory with practically little difference to the cost. Also, he finds that four boilers are fed with the megass and only two burn wood, instead of three with megass and three with wood, which makes a large difference in the calculation of cost of production. The process of diffusion is continuous, and if the diffusers are stopped through lack of cane, breakdowns, holidays, or any other cause, the juice commences to invert and the loss is very considerable, as the diffusers hold about 87 tons of cane; but this difficulty has not produced such ill effects as expected. Of course, in diffusion, a great deal more water has to be evaporated than by the crushing process, as about 30 per cent. of water is added to the cane in the diffusers, and this entails larger clarifiers, eliminators, and vacuum pans. The juice, already heated in the diffusers, commences boiling at a density of about 6° Beaumé, as compared with the juice from the cane by crushing at about 10° Beaumé. But on the whole the extra amount of sugar obtained, amounting to about 2 per cent. more than by the crushing process, may compensate for the extra initial expenditure on machinery and extra cost of manufacture.

Vast sums have been expended on improved machinery, but nothing whatever has been attempted in agricultural interests. No experiments have been made to improve the quality of the cane, or to discover new varieties, by planting from the seed and selecting the strongest and healthiest canes. No scientific examination and analysis of the various soils are attempted, and nothing has been done in the way of fertilising the impoverished fields.

Sugar Industry of Bohemia.

Foreign Office Miscellaneous Series, No. 554.

Various attempts to produce sugar from the maple tree were made in Bohemia during the early years of the last century which led, however, to no commercial result.

In 1331 the first factory for producing sugar from beetroot was erected by permission of Prince Thurn and Taxis at Dobrowitz, others followed in rapid succession, and in the year 1836 factories were working at Smidar, Schlan, Sadska, Peček, Neuhof, Libesnic, Vysocan, and other places.

The sugar industry to-day is the most important in the Dual Monarchy, and especially so in Bohemia, which alone produces nearly half of the total amount manufactured in Austria-Hungary. There are in all 213 raw sugar factories in work, of which Bohemia has 130.

The methods employed in the raw sugar factories are the "Robert" system, with triple saturation and mechanical filtration. In 1885 the factories of Podebrad and Nimburg were the first to adopt lime and carbonic acid as means for filtration, which is now universally used instead of the expensive spodium process. The beetroots are conveyed from the yard to the factory by means of water channels into which they are thrown and by which much labour is saved; nearly all the factories are built where there is an abundance of water and in close proximity to the lines of railway.

The factories vary in size from those built with a capacity of dealing with 200 tons of beet daily (24 hours) to that of

the largest at Laun, which can work 1,100 tons. The greater number are constructed with a capacity of from 400 to 800 tons daily. The working expenses in a raw sugar factory, here known as the "Regie," under the most favourable circumstances, are about 3*d.* per cwt. of beet, the average work at from 3*d.* to 5*d.*, and in small factories at 7*d.* per cwt. of beetroot. The refuse beet is sold to the farmers for cattle food, and is therefore an important by-product.

The sugar refining industry has reached a high state of perfection. There are in Bohemia 20 refineries which not only can refine the whole of their own raw sugar, but also a portion of the Moravian. The largest refineries are capable of refining on an average about 70,000 tons of raw sugar each per annum, and are at Schönpriesen, Aussig, and Nestomitz, on the northern frontier, and Skrschivan in the east.

The natural advantages for the cultivation of beet, with an abundance of coal and water, give to Bohemia its prominent position in the sugar industry, and its ready means of water transport to Hamburg as well as by railway makes it by far the most important in Austria-Hungary.

The sugar produced in Austria-Hungary was :—

Year.	Quantity.	Refined.
	Tons.	Tons.
1898—1899	1,041,768	871,108
1899—1900	1,098,546	841,965

In Bohemia :—

Year.	Quantity.	Refined.
	Tons.	Tons.
1898—1899	494,283	433,018
1899—1900	551,338	426,116

The sugar exported from Austria-Hungary was :—

Year.	Quantity.	Refined.
	Tons.	Tons.
1898—1899	140,270	519,873
1899—1900	134,043	516,016

From Bohemia :—

Year.	Quantity.	Refined.
	Tons.	Tons.
1898—1899	80,997	285,872
1899—1900	76,746	292,028

The total amount of molasses produced in Austria-Hungary in 1899–1900 was 252,606 tons, of which Bohemia gave 115,655 tons. The export of this article is trifling, being in all only 264 tons; it is principally used locally for the manufacture of spirits.

The total export of sugar from Austria-Hungary to the United Kingdom during the campaign, 1899–1900, was :—Raw sugar, with average polarisation of 90° and under 99°·3, 92,051 tons; refined, with average of 99°·3, 221,809 tons. There was also exported to Hamburg, 27,766 tons of raw and 23,219 tons of refined sugar, a large proportion of which also went to the United Kingdom, statistics of which I could not obtain.

The total amount of sugar produced for the present campaign in Austria-Hungary was: raw, 1,063,000 tons, a falling-off on the previous 1899–1900 campaign of about 35,000 tons. The production of Bohemia was 495,000 tons, as against 551,338 tons in 1899-1900.

The export from Austria-Hungary for the first eight months of the present campaign, that is to March 31, 1901, was: refined, 380,329 tons; refined, eight months, 1899–1900, 342,094 tons; raw, 44,224 tons; raw, eight months, 1899–1900, 63,144 tons. From Bohemia: refined, 172,598 tons; refined, eight months, 1899–1900, 146,685 tons; raw, 13,354 tons; raw, eight months, 1899–1900, 36,592 tons; the increase in the export of refined sugar this year being very marked and due in a very great degree to the increased imports into the United Kingdom, which imported from 1st April to 13th April 1901, no less than 137,756 tons, of which 81,464 tons was refined, as against a total of 56,285 tons during the same period of 1900, of which 33,753 tons was refined.

The greater portion of the sugar exported from Bohemia goes to the United Kingdom.

XVII.—BREWING, WINES, SPIRITS.

Arsenic in Beer.

Report of the Manchester Brewers' Association's Commission.

Drs. Lauder Brunton and Thomas Stevenson, Mr. A. Gordon Salamon, Dr. Luff, Mr. S. Buckley, and Mr. Fletcher Moulton, K.C., were appointed by the Manchester Brewers' Central Association a commission to inquire into the causes of the beer poisoning which occurred some time ago. Their final report, which was signed by all the members, states that in all 663 samples have been analysed. The manner in which the brewers carried out the previous recommendations made by the commission resulted in the beer brewed in Manchester being soon free from arsenic, and analyses of recent date proved that Manchester beer is now as pure as any produced in the United Kingdom in respect of its freedom from arsenic. In the preliminary remarks it is stated :—

"The analyses and investigations that we have made prove, finally and conclusively, that the presence of arsenic in injurious quantities in the Manchester beer was in all cases due to the sugars manufactured by Messrs. Bostock and Co. (Limited), and that such sugars derived their contamination exclusively from the employment in their manufacture of arsenicated sulphuric acid, suppplied by Messrs. Nicholson and Co. (Limited), of Leeds. No other cause of contamination to which the presence of arsenic in the observed quantities can be attributed has been discovered by us, nor is one believed to have been possible."

Notwithstanding that brewing from Bostock's sugar was discontinued on December 1, 1900, arsenic continued to be found in smaller quantities in brewings in Manchester and elsewhere, and this has caused a prolongation of the inquiry, for the commissioners say, "Although these traces of arsenic in beer (of which the presence was hitherto quite unsuspected) cannot be said to be directly poisonous, and are, perhaps, not even injurious to the human system, it is extremely desirable that they should be eliminated if it be possible."

Proceeding to give details and results of their analyses of beers, the commissioners say :—

"One hundred and sixty samples of beer have been analysed by us for the purpose of this inquiry. These samples were collected from all parts of the country, between November 22, 1900, and the end of February, 1901, the largest proportion, however, being derived from Manchester and the surrounding districts. Of these 160 samples, 84 were found to be quite free from arsenic when examined by the Reinsch test. The remaining 76 samples were found to contain arsenic in amounts ranging from 1-10th of a grain to 1-400th of a grain per gallon of beer. Of the 76 samples found to contain arsenic, 53 were traced to have been brewed with Bostock's sugar, and represented brewings previous to December 1, 1900. Of these 53 samples, 27 contained more than 1-20th of a grain of arsenic per gallon, a quantity which must be regarded as dangerous. Of these 53 samples, 37 were brewed in Manchester and district, and 16 were obtained from other parts of the country where Bostock's sugar had been used. The remaining 23 samples, which were not brewed from Bostock's sugar, were found to contain quantities of arsenic ranging from 1-30th to 1-400th of a grain per gallon of beer. Of these, 21 contained less than 1-70th of a grain per gallon. In our opinion these very

small quantities were, in every case, derived from the malt. The sample containing approximately 1-30th grain per gallon was clearly proved to be in all malt beer. Such also was the case in respect of a sample containing approximately 1-70th grain per gallon."

The report goes on to state the result of the examination of materials used in brewing. One hundred and thirty samples of brewing sugars were analysed, made up as follows:—Glucose, 63; invert sugar, 53; raw and purified sugar, 11; and caramels, 3.

"Eight samples of glucose were proved to have been made by Messrs. Bostock and Co. (Limited), and were probably delivered to breweries between September and November, 1900. All contained arsenic in quantities ranging from 0·027 per cent. to 0·075 per cent. The remaining 55 samples were independently obtained from brewing sugar factories, merchants, agents, brokers, and breweries, and represented normal output and deliveries, both British and foreign, of all such goods employed in brewing in this country. Of these 55 samples, five were ascertained to have been made by different makers previous to the contamination of Manchester beer by arsenic. They were found to be free from arsenic, with the exception of one, which contained an infinitesimal quantity, approximately about 1-600th of a grain per pound. The employment of even so large a proportion as 20 per cent. of this latter glucose for brewing purposes would have introduced an amount of arsenic into beer almost incapable of detection and certainly negligible. The remaining 50 samples were all found to be free from arsenic by the Reinsch test. Six samples were made by Messrs. Bostock and Co. (Limited), and were probably delivered into breweries between September and November, 1900. These samples all contained arsenic ranging in amounts from 0·021 per cent. to 0·067 per cent. The remaining 47 samples represented every known manufacture, and were independently obtained, as in the case of the samples of glucose. Of these, five were ascertained to have been manufactured previous to the detection of arsenic in Manchester beer. These five samples were all found to be free from arsenic by the Reinsch test. The remaining 42 samples were also found to be free from arsenic by the Reinsch test."

As to other materials, the commissioners report that raw and purified sugars, caramels and malt adjuncts, such as those made from rice, maize, sago flower, tapioca flower, and torrified barley, were all free from arsenic. In the case of hops, again, 11 typical samples were analysed, and all found to be free from arsenic.

"Nevertheless, there exists the possibility of minute quantities of arsenic being present in hops, and we are aware that such traces have been found to exist in hops by analysts of authority. It is conceivable, but extremely improbable, that they are introduced with the sulphur employed when the hops are upon the poles, but they are much more likely to be derived from the fuel and brimstone employed for kilning operations in the oast houses. The quantity of arsenic thereby introduced into beer must in any case be infinitesimal."

Reporting on preservatives, water-hardening materials and finings, the Commission found an infinitesimal quantity of arsenic in the former group, owing to gross negligence in manufacture, but in the samples submitted of finings for clarifying beer, there was no trace of arsenic. As to yeast they say:—

"Nineteen samples of yeast produced in Manchester breweries at or about the time of the outbreak were submitted to analysis. Of these, 10 contained arsenic in quantities ranging approximately from 1-4th grain to 1-200th grain per pound of pressed yeast. Analyses made during February last proved that the Manchester yeast had been freed from arsenic, and may to-day be regarded as quite pure. This statement applies even to those breweries in Manchester in which Bostock's sugars had previously been employed. The interesting discovery has been made that, if arsenic be present in worts, yeast will take up a very considerable portion of it. It would be rash to hazard the prediction as to whether this is due to mere mechanical absorption, or to any physiological or chemico-physiological action as between the arsenic and the yeast. The fact remains, however, that in a brewery in which only minute quantities of arsenic are to be found in the brewing materials, much more definite quantities of arsenic are to be found in the yeast."

Dealing next with 14 samples of barley, the commissioners report that five samples of the 14 contained minute quantities of arsenic, while one of unkilned barley contained approximately 1-400th of a grain of arsenic per pound. The remaining 10 were entirely free. From the results of the tests applied, the report says there can be no doubt that barley is liable to take up very minute quantities of arsenic when grown upon land manured with arsenicated fertilisers.

As to malt, 138 samples were examined. Of these, 97 were free from arsenic, and in the remainder it was found in quantities, varying from 1/39th of a grain per lb. to 1-300th of a grain. The samples which contained arsenic were mainly derived from maltings in the Midlands and the Northern counties, while those from the Southern and Eastern counties were entirely free. "The malt containing these traces of arsenic when brewed would, in all probability, impart only a minute quantity of arsenic to the beer, having regard to the removal of arsenic effected by yeast, and it is probable that such traces have existed for many years past. There can, however, be no doubt that it is quite practicable wholly to avoid this contamination, and no better evidence could be given in support of this statement than by reference to the malts supplied to-day to the breweries in and around Manchester, which are remarkably free from arsenic."

The importance of thoroughly examining this arsenical contamination led the commissioners to examine two products of the malting process not used in brewing, namely, malt culms and kiln dust, as well as the coal and coke used in malting. As to malt culms, they found cases where the malt was free from arsenic, yet the culms from the same malt were contaminated, while, in the case of 20 samples of kiln dust, 18 contained arsenic, in seven of them as much as 1 grain to 2 grains of arsenic per lb. being discovered. Arsenic was also found in minute quantities in the coal and coke used for malting.

Then follows a long list of recommendations, foremost among them being advice to brewers to make it a rule to require a written guarantee of freedom from arsenic with all purchases of brewing materials of every kind. The commissioners add:—Summing up the above results, it is clear that the most frequent source of arsenical contamination in beer is the use of malt which has been kiln-dried or malted with improper fuel containing arsenic. At the same time, the experience of the late outbreak has shown that precautions must be taken against the presence of arsenic in brewing sugars and other materials (except, perhaps, malt adjuncts), on account of the serious consequences of any carelessness in manufacture which might introduce arsenic. Notwithstanding the absence of arsenic in the samples of caramel which have been examined by us, it cannot be denied that the method of manufacture of caramel, and the fact that it is employed for imparting colour to beer (as well as spirits, sauces, and many other foodstuffs), admits of the possibility, though remote, of the introduction of arsenic in very small quantities in beer, and, therefore, although the amount of arsenic that could be so introduced would be infinitesmal, the brewer is recommended to obtain from the manufacturer a guarantee that the caramel has been tested and found to be free from arsenic."

In the case of hops the brewer should demand that the hop merchant should obtain from the factor, who in turn would obtain it from the grower, a guarantee that none but the purest "flowers" were used upon the poles, and that the fuel and brimstone employed for drying were free from arsenic. As to malt:—"Our investigations have shown that the contamination of malt culms and kiln dust is chiefly due to the use of coke, and especially of gas coke. Inquiries made of the principal maltsters in England have elicited the practically unanimous opinion that there is no necessity whatever to employ gas coke in the preparation of malt. Nor, indeed, is there any real necessity to employ coke at all for purposes of malting. If the use of coke were dispensed with, a good many malt kilns would have to be

structurally modified, but the brewer would not fail to appreciate the benefit of such alterations."

As regards the testing of beer for arsenic, the report goes on to say:—

We have now examined all the possible sources of arsenic in beer, and have pointed out the precautions necessary in each case. If such precautions be taken we believe that the beer will be brewed free from arsenic, and at no substantial increase of cost. And in view of the desire of the brewers that the beer supplied to the public should be free from all suspicion of containing arsenic, we recommend that the following should in future be adopted as the standard test and should be regularly applied. It is more stringent than the one recommended in our former report, but it is not too delicate for the standard of purity to which, in our opinion, the beer should attain.

This test (Reinsch) should be performed as follows:—

Take 200 c.c. of the beer in a porcelain evaporating dish, acidulate with 1 c.c. of pure concentrated hydrochloric acid, and evaporate till the volume of liquid is reduced to one-half. Then add a further 15 c.c. of the hydrochloric acid and insert a piece of pure burnished copper foil, a quarter of an inch by half an inch in size, and keep the solution gently simmering for an hour, replacing the evaporated liquid from time to time by distilled water. If at the end of an hour the copper remains bright and red, the beer is arsenic-free.

If a deposit is obtained on the copper, the foil should be removed, washed successively with water, alcohol, and ether, dried at a temperature not exceeding 100° C., and subjected to slow sublimation in a thin reduction tube, not less than two inches long and having an internal diameter of 0·15 inch, the upper portion of which should be warmed before the sublimation begins. For the purpose of the sublimation a small spirit-lamp flame should be used. If any sublimate is obtained, it must be examined under a magnifying power of about 200 diameters. Any sublimate which does not show well-marked octahedral or tetrahedral crystals is not to be considered arsenical. Mere blackening of the copper, or deposit thereon, does not demonstrate the presence of arsenic.

The addition of oxidising agents to decompose sulphites, and the use of reducing agents to decompose possible arsenates, is not recommended, as such a procedure is, in our opinion, unnecessary in the testing of beer, and introduces sources of error.

In all cases where the presence of arsenic is ascertained by this test, it is then desirable to estimate the amount by means of the Marsh test, which, although not giving an accurate quantitative determination, will, when properly applied, give an approximate estimation of the amount of arsenic present.

When arsenic is detected by the above test its quantity is best determined by the process of Marsh (Marsh-Berzelius), which should be performed as follows:—

The beer, preferably 50 c.c., is acidulated by the addition of 1 c.c. of pure concentrated hydrochloric acid, and gently boiled in a porcelain evaporating dish for a few minutes till frothing nearly ceases, cooled, and gradually introduced into a Marsh apparatus of 200 c.c. capacity, which is already giving off a gentle stream of pure hydrogen gas, evolved from pure zinc and diluted hydrochloric acid. The purity of the evolved gas is first tested by ascertaining that it yields no mirror after 15 minutes, when it has been passed through a drying tube charged with successive layers of cotton wool, lead carbonate, and spongy calcium chloride, and then heated to dull redness in a narrow glass tube drawn out to a capillary size just beyond the point of heating. The open point of the tube at which the gas escapes should be turned upwards at right angles, so that the amount of issuing gas can be regulated by seeing that when lighted the flame of the burning gas is just perceptible. The beer is introduced by means of a straight thistle funnel provided with a stopcock, so that the admission of liquid into the apparatus can be regulated without introducing air. From time to time a little pure concentrated hydrochloric acid is also introduced, so as to maintain the uniform evolution of gas.

The blank experiment having shown no mirror in the heated tube at the end of 15 minutes, the experiment is continued for half an hour after the commencement of the introduction of the beer. The arsenical mirror thus obtained is then compared with standard mirrors obtained by treating known quantities of arsenious oxide dissolved in water with the addition of pure hydrochloric acid under precisely similar conditions as to generation of gas, and the quantity of arsenic present is thus judged with great approximate accuracy. The mirror may subsequently be converted into crystals of arsenious oxide by sealing off the portion of the tube containing the mirror at each end by means of a blowpipe, and then gently heating the tube, by which means the mirror is volatilised, oxidised, and converted into crystals of the oxide. The mirrors obtained should be small, the comparison of small mirrors yielding more accurate results than where large mirrors are compared.

In conclusion, the commissioners state that if the precautions they recommend be observed they believe that beer will be brewed free from arsenic, and at no substantial increase of cost.

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 31st May	
	1900.	1901.
	£	£
Metals	2,835,636	2,419,189
Chemicals and dyestuffs	496,625	524,246
Oils	849,070	913,142
Raw materials for non-textile industries.	4,948,216	5,023,837
Total value of all imports	43,876,427	42,426,759

Summary of Exports.

Articles.	Month ending 31st May	
	1900.	1901.
	£	£
Metals (other than machinery)	4,506,390	3,475,413
Chemicals and medicines	863,553	810,923
Miscellaneous articles	3,261,695	3,640,013
Total value of all exports	24,715,930	23,556,712

Imports of Miscellaneous Articles for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Cement ..... Tons	9,820	22,422	21,115	38,436
China and earthenware. Cwt.	36,214	35,973	86,772	82,605
Drugs ..... Value £	..	..	99,455	103,861
Glass:—				
Sheet ..... Cwt.	108,184	105,860	58,400	58,970
Plate ..... „	30,981	56,414	33,421	61,170
Flint ..... „	46,861	48,449	93,734	86,168
Bottles ..... Gross	151,278	151,206	64,766	68,581
Other ..... Cwt.	25,908	19,130	45,907	38,340
Glue, size, gelatin „	19,807	21,911	44,048	45,575
Leather, unmanufactured. „	117,619	101,387	731,322	669,525
Oil seed cake .... Tons	31,022	34,179	200,828	216,132
Paints and pigments. Value £	..	..	118,129	122,393
Paper, pasteboard Cwt.	580,351	566,395	403,711	391,843
Scientific instruments. Value £	..	..	56,969	71,345
Soap and soap powder. Cwt.	15,335	23,158	20,499	26,265
Zinc manufactures. „	37,915	46,091	49,834	51,558

Imports of Metals for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Copper :—				
Ore ..... Tons	11,331	10 540	173,572	155,277
Regulus ..... „	8,963	8 531	360,129	329,550
Unwrought..... „	7,303	6,000	552,885	433,263
Lead, pig and sheet „	16,919	17,553	283,798	228,076
Pyrites ..... „	65,832	52,736	107,623	91,672
Quicksilver ..... Lb.	89,370	83,575	10,186	11,239
Silver ore..... Value £	..	..	76,222	84,943
Tin ..... Cwt.	34,019	36,135	229,248	212,901
Zinc..... Tons	7,364	5,913	157,769	109,233

Imports of Oils for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Cocoa-nut..... Cwt.	43,548	30,579	53,358	37,917
Olive..... Tuns	1,049	2,081	44,873	78,625
Palm ..... Cwt.	94,914	111,426	117,864	125,727
Petroleum :—				
Illuminating.. Gall.	16,660,024	20,436,254	328,003	345,891
Lubricating... „	3,178,291	2,757,009	102,156	90,612
Seed..... Tons	3,322	3,611	87,835	91,103
Train, &c. ..... Tuns	1,327	1,060	27,583	20,924
Turpentine ..... Cwt.	14,153	32,230	26,873	42,412

Imports of Raw Material for Non-Textile Industries for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Bark, Peruvian .. Cwt.	3,721	2,882	7,442	7,824
Caoutchouc ..... „	39,917	39,511	568,001	510,926
Gum :—				
Arabic ..... „	5,389	9,752	13,297	19,168
Lac, &c. ..... „	9,366	16,332	30,945	48,758
Gutta-percha .... „	8,843	5,153	108,404	77,696
Hides, raw :—				
Dry..... „	104,250	32,032	271,426	90,790
Wet ..... „	49,387	64,952	121,454	149,620
Ivory ..... „	495	1,057	22,613	36,514
Manure :—				
Guano ..... Tons	4,547	134	30,200	737
Bones..... „	5,097	7,494	22,792	30,614
Paraffin..... Cwt.	50,795	25,679	70,129	33,205
Linen rags ..... Tons	1,984	1,263	17,952	12,393
Esparto ..... „	13,691	22,387	55,089	92,002
Pulp of wood..... „	40,509	40,750	223,703	236,719
Rosin..... Cwt.	112,078	94,224	26,245	21,465
Tallow and stearin „	229,499	164,746	305,204	211,823
Skins :—				
Goat ..... No.	1,405,112	2,054,751	134,870	184,367
Sheep..... „	795,715	1,586,497	85,900	133,791
Nitrate of soda... Tons	8,833	8,960	72,837	75,302
Phosphate of lime „	30,979	24,435	52,649	31,895

Imports of Chemicals and Dyestuffs for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Bleaching materials „	32,325	31,628	16,884	10,468
Soda compounds... Cwt.		26,494		8,878
Borax, &c. ..... „	1,037	25,335	1,133	14,842
Brimstone ..... „	69,859	6,717	13,667	1,804
Nitrate of potash.. „	19,366	23,704	16,605	19,650
Chemicals, other Value £	..	..	131,116	152,516
Cutch and gambier. Tons	1,675	1,140	33,820	23,822
Dyes :—				
Alizarin.....Value £	..	..	18,922	23 874
Aniline and other „	..	..	50,302	47,289
Indigo ..... Cwt.	1,544	1,410	24 785	22,702
Bark..... „	44,243	49,903	19,002	20.202
Valonia ..... Tons	5,837	1,883	31,364	19,242

Exports of Metals (other than Machinery) for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Brass..... Cwt.	10.712	8 272	54,232	46,667
Copper ..... „	51,863	88,016	205,525	333,397
Lead..... Tons	5,268	5,340	94,987	84,641
Plated wares... Value £	..	..	30,354	37,217
Tin ..... Cwt.	9,506	9,881	66,185	60,632
Zinc..... „	13,314	23 248	14,422	19,288

Exports of Drugs and Chemicals for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Alkali ..... Cwt.	356,060	..	109,750	..
Bleaching materials „	112,604	57,181	33,095	19,180
Copper sulphate .. Tons	5,876	4,949	143,902	116,258
Chemical manures „	24,585	26,860	179 893	191,135
Medicines..... Value £	..	..	114,531	125,115
Soda compounds :—				
Ash..... Cwt.	..	95,243	..	20,612
Caustic ..... „	..	99,313	..	50,482
Bicarbonate .... „	..	22,516	..	7,843
Crystals ..... „	..	20,815	..	3,465
Sulphate..... „	..	52,318	..	3,895
Other sorts ..... „	..	25,831	..	13,810

Exports of Miscellaneous Articles for Month ending 31st May.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Gunpowder.... Lb.	641,900	Cwt.3,459	14,980	10,402
Candles..... „	2,130,300	1,591,600	37,820	28,697
Caoutchouc..... Value £	..	..	125,890	114,205
Cement..... Tons	49,015	34,682	89,030	67,425
Products of coal. Value £	..	..	178,967	109,757
Earthenware ... „	..	..	140,716	164,357
Stoneware ..... „	..	..	16,485	15,580
Glass :—				
Plate ..... Sq. Ft.	192,471	Cwt.5,793	11,615	9,085
Flint ..... Cwt.	8,452	9,859	21,033	23,210
Bottles ..... „	85.191	72,155	41.354	36,874
Other kinds ... „	23.152	17,710	23,572	19,229
Leather :—				
Unwrought.... „	12,793	10,662	140,694	114,250
Wrought.....Value £	..	..	33,809	39,959
Seed oil ..... Tons	5,012	4,673	126,186	121,574
Floorcloth.....Sq. Yds.	2,464,800	2,468 900	118,301	124,444
Painters' materials Val. £	..	..	189,180	195,233
Paper..... Cwt.	96,901	83,889	146,540	151,514
Rags..... Tons	5 872	5,473	38.059	32,021
Soap ..... Cwt.	85,247	69,802	91,800	77,011

Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—PLANT, APPARATUS, and MACHINERY.

Applications.

9901. E. E. Glaskin. Improvements relating to automatic governors for mixed gases. May 13.

10,303. O. Michael. A new or improved crucible furnace. Complete Specification. May 17.

10,332. E. Snowden. An improved construction of steel or iron stills for gas works, tar works, and the like purposes. Complete Specification. May 18.

10,412. The Actiengesellschaft für Zink Industrie vormals W. Grillo and M. Schroeder. Process for the production of porous contact substances or masses for use in catalytic processes. May 20.

10,421. J. Davis. Improvments in apparatus for calcining and disintegrating ores or other minerals, applicable to all processes where the alternate exposure of the material to heat and atmospheric air is of advantage. May 20.

11,082. E. G. Scott. Improvements in the distillation of ammoniacal and other liquors, and in apparatus therefor. May 29.

11,273. C. Schumann. Improved means and apparatus for emptying acid carboys by the pressure of air. June 1.

11,429. H. H. Lake.—From N. B. Powter, United States. Improvements in apparatus for separating liquids of different densities. Complete Specification. June 4.

11,435. E. A. Sperry. Improvements in concentrators. June 4.

11,603. E. C. H. Pape and W. S. Henneberg. Improvements in and connected with filters. Complete Specification. June 6.

11,935. E. S. Beaven. Improvements relating to the cleansing of furnace gases and means for use therewith. June 11.

12,044. Royles, Ltd., J. J. Royle, and O. M. Row. Improvements in or relating to evaporators. June 13.

Complete Specifications Accepted. *

1900.

10,156. C. Tellier. Apparatus for the production of ice and cold. May 30.

10,925. W. P. Thompson.—From The Firm of Litzelmann and Tailfer, France. Apparatus and method of decomposing alkaline and other amalgams. May 22.

11,268. J. Gore. Apparatus for compressing gas or air. May 30.

11,301. A. Gibb. Furnaces for melting or smelting. May 30.

11,852. Meldrum, Bros. Ltd., and J. S. Orton. Kilns and appliances for calcining, heating, and drying mineral substances. June 5.

12,431. G. B. Schwerin. Extraction of water or other liquid from mineral, vegetable, and animal substances. May 30.

12,789. W. G. Atkins. Construction and arrangement of filtering apparatus. May 22.

13,780. E. Paul. Heaters, condensers, evaporators, and the like. June 19.

14,013. J. Prentice and A. W. Prentice. Working of automatic refrigerating apparatus. June 5.

14,026. J. Keith. Gas or air compressors or pumps. June 12.

14,364. K. Enzinger. Filter presses and material for use in connection with the same. June 12.

14,592. R. C. Sayer. Apparatus for boiling and evaporating water. June 19.

19,003. J. Laidlaw. Centrifugal drying machines. June 12.

1901.

1153. A. J. Boult.—From A. Roller, Maschinenfabrik, Germany, and A. Schiff, Russia. Drying apparatus. June 5.

4780. P. E. Domergue. Apparatus for regulating the density of liquids. May 22.

5559. R. A. Sloan.—From W. H. Martin, Holland. Evaporators. June 12.

6737. M. Güttner. Centrifugal separators. May 30.

6801. F. W. Parker. Processes and apparatus for cooling or condensing fluids. May 22.

7330. B. J. B. Mills.—From B. Lande, United States. Furnaces. June 12.

* *See* note (*) on previous page.

II.—FUEL, GAS, and LIGHT.

Applications.

9890. J. Y. Johnson.—From The Deutsche Continental Gas Gesellschaft, Germany. Improvements in the treatment of residue obtained in the treatment of coal gas (for obtaining cyanogen compounds therefrom) for the purpose of obtaining Prussian blue. May 13.

10,013. G. Westinghouse. Improvements relating to the production and utilisation of gas. May 14.

10,023. J. A. Rey and J. M. B. Rey. Improvements in apparatus for burning hydrocarbons, alcohols, and other combustible liquids for heating purposes. Complete Specification. May 14.

10,024. L. Breittmayer. An apparatus for extracting the naphthaline contained in illuminating and heating gases. Complete Specification. May 14.

10,183. C. Busch. Improvements in apparatus for generating acetylene gas. May 16.

10,336. H. Koppers. Improved method for the heating of by-product-saving coke-ovens. Complete Specification. May 18.

10,337. H. Koppers. Improved coke-oven adapted to be worked with or without saving of by-products. Complete Specification. May 18.

10,585. R. Gray. Improvements in gas producers. May 22.

10,632, F. C. W. Timm. Improvements in or relating to the manufacture or production of oxygen gas. Complete Specification. May 22.

10,725. W. L. Wise.—From R. Klinger, Austria. Improvements in acetylene gas generators. Complete Specification. May 24.

10,867. C. D. Abel.—From Siemens and Halske, Aktien Gesellschaft, Germany. *See* Class XX.

10,868. C. D. Abel.—From Siemens and Halske, Aktien Gesellschaft, Germany. *See* Class XX.

10,869. C. D. Abel.—From Siemens and Halske, Aktien Gesellschaft, Germany. Manufacture of incandescence bodies for electric lighting. May 25.

10,913. S. Nobel. Improvements in and connected with the economising of fuel and the prevention of smoke. May 28.

10,923. G. L. Hogan. Improvements in acetylene gas generators. May 28.

11,145. G. A. Bronder. Improvements in gas retort charging apparatus. Complete Specification. May 30.

11,167. G. W. Morrison. An improved process for the treating and moulding of carbide of calcium and carbides containing calcium, with the view of controlling the production of gas therefrom. May 30.

11,187. H. Ledger. Improvements in illuminant mantles for gas or other burners, applicable for general lighting purposes. May 31.

11,218. F. C. von Heydebrand und der Lasa. Artificial fuel. Complete Specification. May 31.

11,255. G. R. Hislop. Improvements in incandescent gas burners. June 1.

11,356. R. J. Milbourne. Improvements in gas purifiers. June 3.

11,466. G. F. Jaubert. Improvements in the preparation of oxygen gas. Complete Specification. June 4.

11,504. J. B. Leroux and P. J. Carmien. Improvements in apparatus for burning mixtures of air and inflammable vapour for heating and incandescent lighting. Complete Specification. June 5.

11,519. C. Jousset and W. R. McKay. Improvements relating to the manufacture of artificial fuel. June 5.

11,532. F. Ferguson and E. Blau. Improvements relating to apparatus for the production of carburetted air. June 5.

11,682. A. A. Masters. Improvements in and relating to lighting and heating. June 7.

11,714. H. Kinsey, G. Challenger, and J. H. Nott. Improvements in apparatus for generating and storing acetylene gas. June 7.

11,785. H. Lux. Improvements in and relating to incandescent gas burners. June 8.

11,790. T. E. Pye. Improvements in apparatus for generating, storing, and delivering acetylene gas. June 8.

11,804. G. Smith. Improved acetylene gas machine (without generators). June 10.

11,825. S. S. Chandler and S. B. Chandler. Improvements in gas purifying apparatus. June 10.

12,137. J. Wilson. Improvements in and in the manufacture of mantles for incandescent gas lighting. June 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

3429. R. Harrison. Production of combustible gases from peat, spent tan, and similar substances, and appliances in connection therewith. May 30.

4877. J. F. Wittemann. Treatment and utilisation of fermentation gas. May 22.

9088. A. M. Plaissetty. New process for the incandescence of gas. May 22.

10,432. G. Bower. Apparatus for mixing and burning inflammable gas and hydrocarbon vapours and air for the production of light, heat, and power. June 12.

10,514. A. S. Bower. Apparatus for carburating and increasing the pressure of combustible or incombustible aeriform fluids for the purpose of heat, light, and power. May 22.

11,865. S. Biheller. Burners for incandescent gas lights. May 22.

12,523. H. Holm. Carbonisation of peat and apparatus therefor. June 19.

13,204. H. Gerdes. Pressure regulating apparatus for gas generators. June 12.

13,205. H. Gerdes. Gas generators. May 30.

13,259. H. Hill. Manufacture of mantles for incandescent lighting. May 22.

13,814. J. Y. Johnson.—From The Deutsche Continental Gas Gesellschaft and J. Buel, Germany. Manufacture of gas and coke. May 22.

14,138. C. Hennings. Apparatus for the production of acetylene gas. June 12.

14,426. J. P. Johnston. Apparatus for use in the manufacture of gas. May 22.

14,627. F. Hasselmann. Method of producing fuel from moor earth and moor moss. June 19.

14,673. C. Joly. System for the preservation of liquid air and other liquefied gases in the liquid condition. May 30.

1901.

1703. F. M. Bennett and J. O. Fowler, jun. Appliances for producing perfect combustion of gas. June 12.

6344. G. R. Bond, A. C. Buell, and C. D. Washburn. Acetylene gas generators. May 30.

8275. A. J. Boult.—From Société Lumière Boule, Belgium. Incandescent or Bunsen gas burners. June 19.

8281. A. J. Boult.—From Société Lumière Boule, Belgium. Mantle supports for incandescent gas burners. June 29.

8877. M. Graham. Hot coke conveyors. June 5.

8881. C. J. Johnson. Apparatus for manufacturing gas. June 12.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, PETROLEUM.

COMPLETE SPECIFICATION ACCEPTED.

1901.

5047. J. Wetter.—From Aktiengesellschaft für Theer- und-Erdol Industrie. Germany. Process for obtaining pure hydrocarbons, such as fluorene and phenanthrene contained in coal-tar, and for recovering certain by-products. June 19.

IV.—COLOURING MATTERS AND DYESTUFFS.

APPLICATIONS.

9999. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of anthracene derivative. May 14.

10,277. H. H. Lake.—From K. Oehler, Germany. Improvements relating to the manufacture of disazo-colouring matters. Complete Specification. May 17.

10,728. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of a brown colouring matter. May 24.

10,729. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of contact bodies for use in catalysis especially intended for use in catalytic process for the manufacture of sulphuric acid. May 24.

10,833. W. P. Thompson.—From Chemische Fabrik Opladen vormals Gebr. Flick Gesellschaft mit Veschränkter Haftung, Germany. Improvements in a process for the reduction of indigo. Complete Specification. May 25.

10,886. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of colouring matters of the anthracene series and of intermediate products for use therein. May 25.

11,022. A. Rahtjen. Method of preparing monobromeindigo and dibrome-indigo as well as monochlore-indigo and dichlore-indigo as well as monochloremono-bromeindigo. Complete Specification. May 28.

11,162. G. W. Johnson.—From Kalle and Co., Germany. Manufacture of a black dye containing sulphur. May 30.

11,163. A. Meyenberg, R. J. Lévy, and The Clayton Aniline Company, Limited. Improvements in the manufacture and production of colouring matters containing sulphur. May 30.

11,358. B. Willcox.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new halogenised bodies, new indigo colouring matters, and of indigo. June 3.

11,359. L. Aloy. Improvements in the manufacture of dyeing compositions. Complete Specification. June 3.

11,533. A. Thilmany. Improvements in or connected with the manufacture of composite colouring matters or pigments. Complete Specification. June 5.

11,624. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of colouring matter for the direct dyeing of black or blackish shades. June 6.

11,733. R. J. Urquhart.—From The Chemische Fabriken vormals Weiler-ter Meer, Germany. Improvements in the manufacture of dyestuffs. Complete Specification. June 8.

11,753. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of new azo dyestuffs, and of an intermediate product for use therein. June 8.

11,766. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new azo-colouring matters, and of intermediate products for use therein. June 8.

11,839. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture and production of new azo-dyestuffs. June 10.

12,021. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of an amidophenol derivative, and new intermediate products relating thereto. June 12.

12,185. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new colouring matters of the anthracene series. June 14.

12,202. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of rhodamine derivatives. June 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

10,316. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of colouring matters of the anthracene series and of intermediate products relating thereto. May 30.

12,517. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of dyestuffs containing sulphur and material for use therein. June 12.

12,804. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of black azo dyestuffs for cotton. June 12.

12,819. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of azo-colouring matters and material for use therein. June 12.

14,879. O. Imray.—From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of new rhodamine sulphonic acids. June 12.

1901.

7560. A. Haagen. Manufacture of colouring matters. June 12.

V.—TEXTILES: COTTON, WOOL, SILK, ETC.

APPLICATIONS.

11,287. H. Ferguson and J. Keefe. *See* Class XII.

11,288. H. Ferguson and J. Keefe. *See* Class XII.

11,858. A. J. Boult.—From C. Masse and La Société Francaise de Ramie, France. Improvements in or relating to the treatment of ramie, china-grass, and other fibrous material. Complete Specification. June 10.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

9505. J. M. Ross and J. Schneider. Apparatus for use in mercerising yarns. May 22.

11,749. L. Lederer. Manufacture of acetylcellulose. May 30.

1901.

3861. J. Imray.—From Heberlein and Co., Switzerland. Manufacture of lustrous and variously coloured threads and strips of textile materials. May 30.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

APPLICATIONS.

9847. E. Knecht. An improved method of discharging dyed textile fabrics. May 13.

11,853. C. Wolf. Improvements relating to apparatus for the dyeing, bleaching, and other similar treatment of fibrous materials. June 10.

11,942. J. Wezel. Improvements in the preparation of art chromo and other colour printing papers. Complete Specification. June 11.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

13,027. W. Ruttenan and Co. Machine for and method of dipping and dyeing skins, fabrics, cotton, wool, or other substances. May 22.

14,174. C. L. Jackson. Kiers for bleaching or otherwise treating textile and other materials. June 12.

14,518. L. Schreiner. Dyeing of textile fabrics. June 5.

15,413. A. G. Green, A. Meyenberg, and The Clayton Aniline Company, Limited. Process of dyeing black. June 12.

15,486. W. W. L. Lishman, T. W. Haughton, J. J. Kirkpatrick, and The Lishman Process Bleaching Company, Ltd. Kiers for containing liquors for boiling, bleaching, and dyeing purposes. June 19.

1901.

2182. J. Müller. Process for dyeing gloves and the like. May 30.

VII.—ACIDS, ALKALIS, AND SALTS.

APPLICATIONS.

10,224. S. Trivick. Process for the production of certain acid compounds. May 16.

10,226. H. R. Angel. Improvements in the manufacture of caustic soda as a bye-product from the reduction of refractory ores. May 17.

11,312. J. R. Leaver and H. A. Leaver. *See* Class X.

11,365. G. W. Bell. Improvements in or connected with the purification of brine. June 4.

11,436. R. Pearson. Improvements in the production of metallic nitrides and of ammonia. June 4.

11,497. F. P. Gutierrez. Improvements in and connected with the manufacture of common salt (chloride of sodium). June 5.

11,500. A. Sansone. Improvements in the manufacture of chlorine and chlorides and in the production of electric energy therewith. June 5.

11,772. J. W. Weston. Improvements in the manufacture of salt and apparatus therefor and for other purposes. June 8.

11,792. H. Briegleb. Improvements in and relating to the manufacture of sulphuric anhydride. June 8.

11,919. E. Teisler. Improvements in processes for preventing the escape of silicon fluoride in the decomposition of phosphates. Complete Specification. June 11.

11,999. A. Boake, Roberts, and Co., Ltd, and R. Stewart. Improvements in the manufacture of hydrogen peroxide. June 12.

12,019. G. B. Ellis.—From Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. Improvements in the manufacture of salicylic acid. June 12.

12,083. C. D. Abel.—From Actiengesellschaft für Anilin Fabrikation, Germany. Improved manufacture of phosphorus oxychloride. June 13.

12,093. H. W. Hemingway. Improvements in and in connection with the separation of sulphuretted hydrogen from gaseous and other fluids containing the same, and in the desulphurisation of sulphuretted hydrogen. June 13.

12,250. G. W. Johnson.—From The Chemische Fabrik Griesheim-Elektron, Germany. Improvements in and connected with the manufacture or production of chromium compounds and alkalis. June 15.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

3866. A. W. Lawton. Manufacture of common salt. June 5.

9350. J. Y. Johnson.—From Stassfurter Chemische Fabrik vormals Vorster und Grüneberg, Germany. Manufacture or production of cyanides. May 22.

9351. J. Y. Johnson.—From Stassfurter Chemische Fabrik vormals Vorster und Grüneberg, Germany. Improvements in the production or manufacture of cyanides. May 22.

9352. J. Y. Johnson.—From Stassfurter Chemische Fabrik vormals Vorster und Grüneberg, Germany. Improvements in the manufacture or production of cyanate of potassium. May 22.

9710. H. F. Kirkpatrick-Picard. Manufacture of the haloid compounds of cyanogen. May 30.

10,925. W. P. Thompson.—From The Firm of Litzelmann and Tailfer, France. *See* Class I.

11,678. S. R. Adcock. Process for the production of cupric oxide from natural sulphates or carbonates of copper. June 5.

12,180. J. Raschen, F. J. Norman, and W. G. Luxton. Apparatus for the manufacture of cyanides from sulphocyanides. June 19.

13,263. A. Gutensohn and H. H. Price. *See* Class X.

14,021. A. Shearer. Manufacture of alkaline chromates and bichromates. June 12.

14,572. E. M. Hall. Process for obtaining pure alumina from bauxite. June 19.

1901.

5239. W. H. Wheatley.—From The Clyde Chemical Co., Ltd., New South Wales. Means employed for the extraction of oxide of chromium from its ores, and its subsequent treatment to obtain soluble salts. June 12.

7999. A. Zanner. Manufacture of concentrated sulphuric acid. May 22.

8230. J. Y. Johnson.—From the Atmospheric Products Co., United States. Manufacture of nitrogen compounds from atmospheric nitrogen. June 12.

VIII.—GLASS, POTTERY, AND ENAMELS.

Applications.

10,550. E. Lee. Improvements in printing tiles and other objects by means of collotype blocks, with natural colours, or with other designs or patterns, and in apparatus therefor. May 21.

11,595. F. T. Brearley. Improvements relating to prismatic plate glass. June 6.

12,017. L. Stein and W. Storr. Process for uniting pieces of glass, porcelain, or like material, for mosaic work and the like. Complete Specification. June 12.

Complete Specifications Accepted.

1900.

12,070. F. W. Gaunleit and J. H. Lloyd. Production of designs upon glass and other surfaces. June 12.

23,579. W. F. Stiel. Compressing moulds for manufacturing glass or enamel facing plates. June 19.

23,580. W. F. Stiel. Glass or enamel facing plates, and process of manufacturing same. June 19.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Applications.

9892. H. E. Carter. Improvements in and relating to the manufacture of artificial stone. May 13.

10,084. S. E. Boivie. An improved process for the manufacture of artificial stone. Complete Specification. May 15.

10,124. H. Higgins. Improvements in or relating to apparatus for drying, seasoning, and impregnating wood and other substances. May 15.

10,297. C. von Forell. Improvements in processes for the manufacture of Portland cement. Complete Specification. May 17.

10,448. G. F. Lebioda. Improved apparatus for impregnating wood and the like with preservative or other fluids. Complete Specification. May 20.

10,622. P. Timofeeff. Improvements in pavements. May 22.

10,634. A. Skrobanek. A new or improved process for the manufacture of wood-like material. May 22.

10,857. A. M. Clark.—From The Firm of Fellner and Ziegler, Germany. Apparatus for the manufacture of cement and the like. May 25.

10,958. E. H. Hurry and H. J. Seaman. Improvements in process of manufacturing Portland cement and apparatus therefor. May 28.

11,045. J. Pfister. Improvements in processes of dyeing or preserving timber. Complete Specification. May 29.

11,715. C. Lorenc. Improvements in the manufacture or production of mortar, and the treatment of stone, and the construction therewith of masonry work. Complete Specification. June 7.

11,724. A. Tracy. Improved construction of fireproof flooring. June 8.

11,927. H. H. Lake.—From S. Giussani, Italy. An improved process for the preservation of wood.—Complete Specification. June 11.

12,073. S. Sborowitz.—Process for producing imitation marble. Complete Specification. June 13.

12,207. E. Serre and E. Oulmière. Improvements in cement or mortar.—June 14

12,272. H. H. Lake.—From The Firm of Fried, Krupp Grusonwerk, Germany. Improvements relating to the manufacture of artificial stone blocks, slabs, and the like. June 15.

Complete Specifications Accepted.

1900.

12,513. H. H. Lake.—From Wachtel and Co., Germany. Manufacture of artificial sandstone. May 22.

13,467. T. M. Thom and A. C. Oakes. Manufacture of artificial stone. May 30.

15,940. W. Schwarz. Manufacture of limestone. June 19.

1901.

2861. V. Barreto. Apparatus for slaking lime. June 19.

8282. F. Boas. Manufacture of building materials. June 12.

9739. F. A. Beny and J. Heinrigs. Apparatus for slakin lime. June 19.

X.—METALLURGY.

Applications.

9903. The British Aluminium Company, Ltd.—From A. Hutchinson Cowles, United States. Improvements in obtaining volatile elements from their ores and compounds. Complete Specification. May 13.

9927. S. Cowper-Coles and Co., Ltd., and S. Cowper-Coles. Improvements in or relating to the deposition of metals or compounds. May 14.

9990. A. Reynolds. Improvements in metallurgical converters. May 14.

10,008. E. Schilz. Improvements in the cyanide process of gold extraction. May 14.

10,039. R. McAllister. Utilization of steel slag. May 15.

10,105. C. H. T. Havemann. An improved process for the treatment of mixed ores and compounds for the extraction or separation of lead and zinc therefrom. May 15.

10,198. S. R. Adcock. Improvements in obtaining copper from mixed sulphates. May 16.

10,351. G. G. M. Hardingham.—From F. Heberlein, Switzerland. Improved means applicable for use in the separation of volatile from non-volatile metals, metalloids, and other metalliferous substances. May 18.

10,415. T. H. Cobley. Improvements in the treatment of copper ores for the production of copper salts, copper oxides, copper and iron paint, and metallic copper. May 20.

10,668. Sir H. S. Maxim. Improvements relating to the smelting of iron and other ores and to furnaces therefor. May 23.

10,670. H. C. Bull and A. Watling. Improvements in or connected with the extraction of gold and other metals from sea-water and in apparatus therefor. May 23.

10,705. A. G. Betts. Improvements in or relating to the coating of aluminium or its alloys. Complete Specification. Filed May 23. Date applied for Oct. 24, 1900, being date of application in United States.

10,859. H. Goldschmidt. A new and improved process for welding metals. May 25.

10,964. H. Batt. An improved method of and apparatus for the treatment of ores for the extraction of the metals therein. May 28.

10,983. E. D. Wassell. Improvements in and relating to he manufacture of wrought iron. Complete Specification. May 28.

11,312. J. R. Leaver and H. A. Leaver. Improvements in the treatment of tinning residues containing chlorine. June 3.

11,319. H. R. Angel. Improvements in the concentration of zinc from slags and ores. June 3.

11,339. J. Armstrong. Improvements in or connected with smelting or refining zinc, cadmium, or antimony. June 3.

11,480. P. Auchinachie. The hardening of metals, particularly iron and steel. June 5.

11,547. J. R. Leaver and H. A. Leaver. Improvements in the treatment of flux skimmings, residues from zinc galvanizing. June 6.

11,687. O. Schramm. Improved process for hardening iron. Complete Specification. June 7.

11,763. R. Hutchinson and H. Mitchell. An improved composition for hardening and toughening steel and cast iron. Complete Specification. June 8.

11,832. R. Dietrich. Process for the production of highly carburised steel. Complete Specification. June 10.

11,933. H. S. Blackmore. Improvements relating to the reduction of metals and to the production of alloys of the same. Complete Specification. June 11.

12,006. H. R. Beringer. A process for the treatment of residue and low-grade ores containing copper, tin, and other metals. June 12.

12,268. O. Massenez. Improvements relating to the manufacture of steel. June 15.

12,274. W. B. Middleton and the Non-Injurious White Paint Syndicate, Ltd. *See* Class XIII.

Complete Specifications Accepted.

1900.

10,915. J. L. Babé and A. Tricart. Process for extracting zinc, and apparatus therefor. May 30.

11,602. E. Martin. Manufacture of sheets or plates of aluminium covered with silver. May 22.

12,823. F. L. Saniter, J. L. Smith, R. Bedford, and the South Durham Steel and Iron Company, Ltd. Open-hearth steel furnaces. June 19.

13,263. A. Gutensohn and H. H. Price. Process of eliminating the sulphur from sulphide ores. May 30.

13,299. J. L. Smith, R. Bedford, jun., and the South Durham Steel and Iron Company, Ltd. Open-hearth steel process. May 30.

13,952. H. H. Lake.—From Société Fonderia Milanese D'Acciacio, Italy. Manufacture of steel. June 12.

22,699. H. A. Frasch. Process of recovering and separating metals from their ores and concentrates thereof. June 19.

1901.

1758. H. H. Lake.—From A. G. Betts, United States. Refining of lead. May 22.

4696. A. Rónay. Process of making briquettes from comminuted or pulverulent ore, including purple ore. June 12.

6330. A. J. Lustig, L. Kahn, and I. Lehman. Casting of ingots. May 22.

8097. R. B. Wheatley. Manufacture of metallic alloys. June 3.

8153. W. B. Johnson.—From The Pittsburg Reduction Company, United States. Process for the purification of aluminium. May 30.

8175. E. Herter. Method of and means for casting and simultaneously refining raw zinc and other metals, excepting iron. May 30.

8904. H. H. Lake.—From W. E. Simonds, United States. Alloys, and processes for the production thereof. June 5.

9124. W. S. Mather. Crucibles for smelting. June 12.

XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

Applications.

10,191. A. d'Arsonval and G. Vaugeois. Improvements in and in apparatus for the manufacture of the plates of electric accumulators. Filed 16 May. Date applied for 27 Dec. 1900, being date of application in France.

10,459. V. G. Apple. Improvements in storage batteries. Complete Specification. May 20.

10,482. A. A. Govan. A cell for electrolysis. May 21.

10,505. T. A. Edison. Improvements in storage batteries. Complete Specification. May 21.

10,879. W. K. L. Dickson. Improvements in voltaic cells. May 25.

10,903. E. J. Plummer. Improvements relating to the electrolysis of salt. May 28.

10,974. P. M. Justice.—From Castner Electrolytic Alkali Company, United States. Improvements in and connected with electrodes for electrolytic cells. Complete Specification. May 28.

10,975. P. M. Justice.—From Castner Electrolytic Alkali Company, United States. Improvements in electrodes for electrolytic cells. Complete Specification. May 28.

10,976. P. M. Justice.—From Castner Electrolytic Alkali Company, United States. Improvements in electrolytic cells. Complete Specification. May 28.

11,600. J. J. H. Hunte.—From J. Lürgen, Germany. Improvements in or relating to the formation of positive plates for electric accumulators or secondary batteries. June 6.

11,619. E. W. Jungner. A method of producing electrodes for electric accumulators. Complete Specification. June 6.

11,625. The Electrical Power Storage Company, Ltd., and W. H. Hanson. Apparatus for applying plastic material to matrices more especially intended for use in applying the paste to the grids in making secondary battery elements. June 6.

11,702. B. J. Lürgen and The D. P. Battery Company, Ltd. Improvements in and relating to electric accumulators. June 7.

12,186. G. W. Johnson.—From The Chemische Fabrik Griesheim-Elektron, Germany. Improvements in the electrolytic production of lead dioxide. June 14.

12,195. W. P. Thompson.—From R. J. Gülcher, Germany. Improvements in and in the construction of electrodes. June 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

12,531. C. von Sedneff. Positive electrode for accumulators. June 19.

1901.

3192. P. Juriè. Electric furnaces. May 22.

4494. W. P. Thompson. — From Akkumulatoren und Electricitäts Werke Aktiengesellschaft (formerly W. A. Boese and Co.), Germany. Process for making electrodes for accumulators. June 19.

XII.—FATS, OILS, AND SOAP.

APPLICATIONS.

9897. B. Crowther, Jun. Improvements in or relating to the manufacture of soap. May 13.

9969. A. J. Boult.—From C. Wacker. United States. Improvements in or relating to apparatus for extracting oil from fish or other material. Complete Specification. May 14.

10,384. J. B. Scammell and E. A. Muskett. Improvements in solidifying and toughening oils. May 20.

10,629. H. H. Lake:—From J. S. Stewart Wallace, Holland. Improvements in and relating to the treatment of mineral or vegetable oils or the distillates or residuals thereof. May 22.

10,637. O. Gillon. Process for the use of soda salt in the manufacture of soap. May 22.

10,663. C. Daeschner. Process for separating the resinous parts without their decomposition from mineral oils the like. May 23.

10,825. C. A. McKerrow. Improvements in the preparation of fullers' earth, silicates, and siliceous matter for use in the treatment of oils, fats, and greases. May 25.

11,012. J. Snowdon. An improvement in lubricants. Complete Specification. May 28.

11,074. G. Dangoise and La Société Generale Belge de Deglycerination. An improvement in separating glycerine from oils and fatty matters. May 29.

11,155. E. G. Scott. Improvements in melting tallow and like fatty bodies. May 30.

11,165. T. Weyl. Manufacture of compounds of fatty acids and their derivatives with ozone. May 30.

11,287. H. Ferguson and J. Keefe. The manufacture of an improved detergent for the treatment of raw silks and silk yarns in the gum. June 1.

11,288. H. Ferguson and J. Keefe. The manufacture of an improved detergent for treating raw wools and wool-yarns. June 1.

11,289. H. Ferguson and J. Keefe. The manufacture of an improved laundry detergent. June 1.

11,419. The Cotton Seed Company, Ltd.—From J. C. W. Stanley, United States. Improvements in or relating to the bleaching of oleaginous matter. Complete Specification. June 4.

11,941. A. J. Boult.—From L. Grassie, United States. Improvements in or relating to the treatment of cotton seed. June 11.

12,200. The Shell Transport and Trading Company, Ltd., and P. Dvorkovitz. Improvements in the purification of mineral oils. June 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

14,480. F. Kinneavey. Preparation for washing and scouring. May 22.

23,804. A. Klumpp. Manufacture of soap. June 19.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, ETC.

APPLICATIONS.

9920. J. Thame and The South Western Rubber Company, Ltd. Improvements in and relating to the treatment of crude rubber. May 13.

10,696. C. Wallis and R. E. M. Lagerwall. Improvements in roller mills, applicable for the manufacture of paint, white lead, pigments, printing ink, confectionery, or the like. May 23.

10,865. J. Lones, E. Holden, and J. Lones. New or improved processes or combination of processes for the manufacture of a white pigment from zinc, and for utilising residual products obtained during the conducting of the said processes or combination of processes. May 25.

10,962. C. A. Day.—From The Coleman International Ship and Pile Coppering Company, United States. An improved anti-fouling coating for metal structures. Complete Specification. May 28.

10,963. C. A. Day.—From The Coleman International Ship and Pile Coppering Company, United States. An improved anti-fouling coating for metal structures. Complete Specification. May 28.

11,149. G. E. Heyl-Dia and The Dialene Rubber Company, Ltd. Improvements in desulphurizing vulcanized rubber. May 30.

11,238. E. Edwards.—From Oxylin-Werke Actien-Gesellshaft, Germany. Improvements in rubber-coated materials and in the method or process of preparing or treating the same. Complete Specification. May 31.

11,337. F. J. Corbett. An improved apparatus for manufacturing white lead. Complete Specification. June 3.

11,533. A. Thilmany. *See* Class IV.

11,569. G. Rigg. Improvements in and connected with the manufacture of zinc oxide. June 6.

11,658. A. O. Gill and W. S. Gill. A solvent for paint and varnish. June 7.

12,022. J. W. Worsey and J. H. Lancashire. Improved process for treating zinc oxide. June 12.

12,274. W. B. Middleton and The Non-Injurious White Paint Syndicate, Ltd. Improvements in the treatment of zinc and other ores, chiefly for the direct production of zinc white, and in furnaces therefor. Complete Specification. June 15.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

9037. A. M. Plaissetty. Collodion of aluminium nitro cellulose for the obtaining of inexplosibles and uninflammable products. May 22.

19,871. J. Hommel. Heat-insulating paint. June 12.

1901.

7625. J. Welter.—From The Elektricitäts Aktiengesellschaft vormals Schuckert and Co., Germany. Process for hardening colophony and other soft resins. May 22.

8995. A. Bente. Process and apparatus for the manufacture of soot from tar. June 5.

XIV.—TANNING, LEATHER, GLUE, AND SIZE.

APPLICATIONS.

10,152. C. A. Allison.—From The Scott Leather Machine Company, United States. Improvements in apparatus for treating and colouring hides and skins. Complete Specification. May 16.

11,092. M. C. Lamb and P. Spence and Sons, Ltd. Improvements in and connected with the manufacture of leather and the preservation and curing of skins. May 30.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

10,308. W. P. Thompson.—From A. Bloch and W. Reuter, United States. Apparatus for the treatment and preparation of fur. June 12.

1901.

9025. L. B. Trant, G. Soldani, and P. V. San Martin. Tanning. June 5.

XV.—MANURES, Etc.

Complete Specification Accepted.

1901.

9514. T. Storer and R. McAlley. Treatment for fertilising purposes of the distillery refuse known as burnt or pot ale. June 12.

XVI.—SUGAR, STARCH, and GUM, Etc.

Applications.

10,113. W. P. Thompson.—From H. A. J. Manoury, France. Improvements in processes of refining sugars of low quality. May 15.

10,461. J. Y. Johnson.—From The Cereal Sugar Company, United States. Improvements in and apparatus for refining sugar. Complete Specification. May 20.

10,861. B. J. B. Mills.—From The Société Anonyme "Trust Chimique," France. The treatment of feculent and amylaceous matters, and of natural products containing these matters. May 25.

10,884. C. Steffen. Improved process of obtaining very pure concentrated saccharine juices and nutritious or pulp poor in water from beetroots without the use of water. Complete Specification. May 25.

11,442. C. B. Duryea. Improvements in the method of manufacturing thin boiling or modified starch. Complete Specification. June 4.

Complete Specifications Accepted.

1900.

9356. M. Duniczewski. Apparatus for manufacturing lump sugar. May 30.

1901.

4199. A. Classen. Process for converting cellulose into sugar (dextrose). June 12.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

10,015. H. J. Haddan.—From J. C. Riley, United States. A new or improved process for ageing liquors. Complete Specification. May 14.

10,074. S. N. Pinkus and S. B. Schryver. Improvements in the manufacture of preparations containing ferments. May 15.

10,127. F. E. Bradley. Improvements in the drying of malt so as to prevent its contamination with arsenic. May 16.

10,287. C. F. von Schlichtegroll. Improved process of rectifying spirit by the action of refrigeration and filtration. Complete Specification. May 17.

10,356. T. Haynes, jun. An improvement in malt kilns. May 18.

10,741. F. Singer. An improved machine for turning green malt. May 24.

10,813. W. J. Menzies. Improvements in pressing machinery for the treatment of brewers' and distillers' grains, hops, and the like. May 25.

11,035. T. Hancock. Improved method of obtaining diastase and preparing it for use. May 29.

Complete Specifications Accepted.

1900.

9991. G. Valentine. Utilisation of pressed yeast. May 30.

12,459. A. Eckardt. Brewing process for the production of high or low fermenting beer-wort. June 12.

12,949. J. W. Bennett. Bennett's anti-bacteria system and appliances for the use of brewers of ale, beer, and porter or stout during the processes of cooling, fermentation, and racking. June 12.

19,946. R. Flessa. Process for producing colouring malt. June 12.

1901.

3194. B. E. R. Newlands. *See* Class A.

8363. O. Imray.—From Joshua Brothers Proprietary, Ltd., Victoria. Method of accelerating the maturing of whisky, brandy, and other strongly spirituous liquors. May 30.

9514. T. Storer and R. McAlley. *See* Class XV.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

10,283. H. W. Potter. An improved animal food and process or method of preparing the same. May 17.

B.—Sanitation; Water Purification.

9982. L. Gathmann. Improvements in water purifying apparatus. Complete Specification. May 14.

10,346. J. W. Welch, H. W. Heywood, and T. R. Wollaston. Improvements in apparatus for the treatment of sewage. May 18.

10,404. W. C. Easdale. Improvements in filter bed construction and means of liquid distribution. May 20.

10,838. A. J. Boult.—From E. Vial, Belgium. Improvements in or relating to the treatment of sewage. May 25.

11,353. C. H. Koyl. Improvements in apparatus for softening and purifying water. Complete Specification. June 3.

11,354. C. H. Koyl. Improved process for the purification of water. Complete Specification. June 3.

11,366. T. Waite. Improvements in apparatus for softening, filtering, and purifying water. June 4.

11,944. J. C. Miller. Process and apparatus for sterilizing and cooling liquids. Complete Specification. June 11.

12,015. J. Weck. Improvements in sterilizing apparatus Complete Specification. June 12.

C.—Disinfectants.

9936. M. Condron. An improved preservative compound. May 14.

10549. C. Billing. New or improved antiseptic or detergent. May 21.

11,286. H. Ferguson and J. Keefe. The manufacture of an improved disinfectant detergent for household and personal purposes. June 1.

11,670. J. Baxeres de Alzugaray. Improvements in and connected with the manufacture of disinfecting and antiseptic fluids and compounds. June 7.

12,120. V. A. M. L. Beck. Insect destroying powder. June 14.

Complete Specifications Accepted.

A.—Foods.

1900.

9648. J. M. Rodoconachi and W. M. Briggs. New kind of food preparation. May 30.

9649. M. Phillips and O. Müller. Process for the treatment of cotton pods or shells for the manufacture of food for animals. May 22.

12,676. J. Jensen. Process for the preservation of eggs. June 5.

1901.

3194. B. E. R. Newlands. Drying or roasting or smoking of malt, hops, hams, or other alimentary matters. June 5.

7545. L. O. Ferson. Food products and processes of preparing the same. May 22.

7620. G. C. Marks.—From Force, Société Anonyme. France. Process for preserving butter and animal fats, May 22.

B.—Sanitation; Water Purification.

1900.

12,619. R. G. Brooke. Apparatus suitable for purifying fluids. June 5.

1901.

6121. B. Seufer. Processes for eliminating iron from water and aqueous solutions. May 1.

6931. J. Fischel. Treatment of liquid waste products. May 30.

7806. H. H. Lake.—From J. M. A. Lacomme and W. Lauder, United States. Apparatus for use in the purification of water. May 22.

8088. A. G. Brookes.—From W. Trippe, Germany. Treatment of waste liquors from the manufacture of sulphite cellulose in order to render the same useful, and to obtain certain products therefrom. May 30.

C.—Disinfectants.

1901.

7719. J. Welter.—From The Aktiengesellschaft für Theer-und Erdöl. Industrie, Germany. Treatment of phenols to render them soluble in water. June 12.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

10,099. F. Rymkiewicz. Process for drying and condensing pulps and plastic substances. May 15.

10,213. E. Zühl. A new or improved process for the manufacture of a celluloid-like material. Complete Specification. May 16.

10,535. C. I. Goessmann. Improvements in the manufacture of paper and other fabrics. Complete Specification. May 21.

11,453. H. H. Lake.—From F. B. How, United States. Improvements in apparatus for making carbon paper. Complete Specification. June 4.

Complete Specifications Accepted.

1900.

9053. F. Wittstock. Coloured mosaic paper. May 22.

11,751. E. Zühl. Method of producing celluloid. May 30.

13,131. J. N. Goldsmith and The British Xylonite Company, Limited. Manufacture of celluloid. May 30.

17,048. F. Haenle. Metallised paper. May 30.

1901.

8072. E. Zühl. Process for the manufacture of a celluloid-like substance. May 30.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

10,867. C. D. Abel.—From Siemens and Halske Aktien Gesellschaft, Germany. Manufacture of thorium, zirconium, and elements of the ytterite group. May 25.

10,868. C. D. Abel.—From Siemens and Halske Aktien Gesellschaft, Germany. Manufacture of metallic thorium, zirconium, and elements of the ytterite group, and the production of coatings of the said metals upon other bodies May 25.

11,229. C. Bachadour. Purgative preserve, extract, decoction, and syrup made from rose-leaves. June 31.

11,301. H. L. F. Moulton.—From A. Verley, France. Improvements in the manufacture or production of bodies for use in perfumery and confectionery. June 1.

11,328. J. Mayer. An improved process for the production of tea extract. June 3.

11,474. M. Ekenberg. Improved manufacture of coffee extract. Complete Specification. June 4.

11,783. J. Y. Johnson.—From The Vereinigte Chininfabriken Zimmer and Co., Germany. Acidyl derivatives of the cinchona alkaloids and their manufacture. June 8.

Complete Specifications Accepted.

1900.

14,213. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of organic bromo compounds. June 19.

14,491. A. M. Clark.—From F. Blum, Germany. Process for the preparation of prophylactic immunifying or curative substances. June 12.

1901.

526. G. W. Johnson.—From C. F. Boehringer and Soehne, Germany. Manufacture or production of homologues of xanthin. June 19.

6537. R. Reitmeyer.—From J. Volmar, Germany. Manufacture of saccharin. May 22.

XXI.—PHOTOGRAPHY.

Applications.

9983. H. H. Lake.—From R. B. West, United States. An improved sensitised paper for photographic printing May 14.

10,524. S. E. Page.—From The Grenier Art Company United States. Improvements in photographic fabrics. Complete Specification. May 21.

10,721. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The preparation of new chemical compounds, and their use as developing means in photography. May 24.

11,232. W. P. Thompson.—From La Société Anonyme de Photographie Automatique Inalterable et Instantanée, France. Improvements in apparatus for rapidly producing photographs. May 31.

11,719. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Process of rendering silver-haloid layers sensitive to rays of different degrees of refrangibility. June 7.

11,866. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of photographic developers stable in the dry state. June 10.

Complete Specification Accepted.

1900.

10,795. A. Sauve. Apparatus for the simultaneous production of triple negatives, especially for colour photography. June 19.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

9984. J. E. Bloméu. Improvements relating to the manufacture of high explosives. Complete Specification. May 14.

9991. D. Hickie. Controlling the temperature of explosives. May 14.

10,784. C. Helcké. Improvements in or connected with the manufacture of nitroglycerin. May 25.

10,994. A. J. Boult.—From Fritiof Säfström, Sweden. Improved ignition composition for matches. May 28.

11,065. R. Hawkins. Improvements relating to the manufacture of explosive compounds. May 29.

11,212. J. S. Potts. Improvements in matches. Complete Specification. May 31.

11,400. E. Edwards.—From W. Kent, United States. Improvements in smokeless powders. Complete Specification. June 4.

11,711. W. Friese-Greene. Improvements in the manufacture of explosives. June 7.

11,959. F. Bale. Improvements in the manufacture and production of matches and striking compositions, and in the substances and processes employed therein. June 12.

COMPLETE SPECIFICATIONS ACCEPTED.

1900.

17,809. L. Varicas. Set time and automatic explosive liquidised gas shell. June 3.

1901.

2339. D. Bachrach. Nitrocellulose and similar compounds, and process of making the same. May 30.

5264. R. W. Scott. Explosive charges for guns. April 24.

XXIII.—GENERAL ANALYTICAL CHEMISTRY.

COMPLETE SPECIFICATION ACCEPTED.

1900.

11,101. F. W. Le Tall.—From M. Walker, United States. Measurement of the magnetic properties of iron and steel. May 23.

PATENTS UNCLASSIFIABLE.

APPLICATIONS.

10,827. S. Bornett. A process for the production of products of crystallisation in the form of bars. May 25.

10,926. K. Koch and A. Stelling. A new article of manufacture, consisting of felt manufactured from metal. May 28.

11,407. R. Oblath. Improvements relating to the manufacture or construction of rubies. June 4.

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