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Thursday, Feb. 27th, 1902:—
Mr. Wilton P. Rix. "Preparation and Behaviour of Insoluble Frits for Pottery Glazes."

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Every Candidate for admission as a Member of the Society must be proposed according to the form set out in Rule 26. He must be proposed by one or more Members to whom he is known personally. The proposal form must be sent duly signed to the General Secretary, who shall lay the same before the Council at its next Meeting. If the majority of the Members of Council present are in favour of his election, the Candidate shall be declared duly elected, and his name shall be entered in the Register of the Society. At whatever period of the year a new Member may be elected, he shall be required (unless the Council shall determine otherwise) to pay his subscription for that year; having done which he shall be entitled to receive all the numbers of the Society's Journal for that year.

The Nomination Paper must take the following form, and may be obtained from the General Secretary, or from any of the Secretaries of Local Sections:—

The Society of Chemical Industry.

We, the undersigned Members of the Society of Chemical Industry, hereby propose
(Candidate's Name in full) ....
as a fit and proper person to become a Member of the Society.
For Names only of Members recommending the Candidate. } ....
....
And I, the said Candidate for Membership, hereby declare that if elected I will do my best to support the Society, and will observe its Bye-laws.
Signature of Candidate......................................
Address to which the Candidate wishes the Society's publications to be sent, and which is to be entered in the Society's Register. } ....
....
Profession or Business Occupation ....
Date......................................

(*Extract from Bye-laws—Rules* 27, 29, *and* 30.)

Each Member shall pay an annual subscription of Twenty-five Shillings, due on the First of January in each year; except that any Member may pay a life composition fee of Twenty Pounds in lieu of an annual subscription. He shall also pay, on receipt of notice of election, an entrance fee of One Guinea in addition to his first year's subscription; except that should a Member who has paid an entrance fee resign and subsequently seek re-election the Council may dispense with a further entrance fee.

The Journal of the Society.

In order that members may be supplied with information at as early a date as possible, the Council has ordered that the Journal shall be issued twice a month, commencing January 1902, and that it shall contain abstracts of French and United States, as well as British, patents.

Subscriptions for 1902.

Members are reminded that the subscription of 25*s*. for 1902, payable on January 1st next, should be sent in good time to the Hon. Treasurer (Mr. Samuel Hall), in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation should reach the General Secretary not later than January 15th, 1902.

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.

Communications.

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.

Arsenic Committee.

The Joint Committee of the Society of Chemical Industry and the Society of Public Analysts will present their report at the meeting of the London Section on January 6, 1902, and at the next meeting of the Society of Public Analysts.

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Deaths.

Gilbert, Sir J. Henry, F.R.S., Harpenden, Herts. Dec. 23.
Tait, T. S., Inverurie, N.B.

London Section.

Meeting held on Monday, December 2nd, 1901.

MR. OTTO HEHNER IN THE CHAIR.

THE LEMON OIL INDUSTRY.

BY HERBERT E. BURGESS, F.C.S., AND J. F. CHILD.

After giving an account of the early history and uses of the lemon, the authors went on to say:—

The two principal lemon-growing districts now are Sicily and the Riviera, but the Tyrol, Spain, Portugal, and the countries on the south coast of the Mediterranean also supply a very considerable quantity of the fruit.

The climate for the cultivation of the lemon should be moist and sheltered, and the soil naturally drained. The cultivated trees of Sicily demand such an amount of moisture as can be obtained only at the mountain slopes, and the structure of the mountains is peculiarly favourable to this end. The loose stones of which they are mostly composed absorb the rain water through their innumerable pores and fissures, and afterwards allow it to swell forth in springs lower down. In this way the irrigation, which is absolutely indispensable for the members of the citron tribe during the dry season, is obtained.

The whole north and east coast, from the Bay of Castella round by Messina to Avola, is an almost endless succession of orchards. In the interior of Sicily, practically no lemon, or, indeed, other trees are cultivated.

A young lemon tree will begin bearing when five years old, and will then produce about 200 lemons. When 15 or 20 years old it will average 1,000 fruit a year, and a full grown tree will produce from 3,000 to 5,000. A lemon tree can be made to flower early by suitable irrigation, and so bear fruit (the summer crop), which is nearly all exported, and fetches high prices, but it is said, however, to be detrimental to the tree, and not to be indulged in too many times in succession, the trees being allowed to take their natural course, and to produce fruit ripening in December.

The cultivation of the lemon and production of its essential oil is probably the chief revenue-bearing industry of Sicily. In compiling their commercial statistics the Italian Board of Trade do not specify the lemons separately from the oranges, and there is no reliable information as to the value of the industry from the exports, but the extent of the industry in Sicily may be gathered from the statistics of the export from the two principal towns, viz., Palermo and Messina.

Palermo exported, in 1899, oranges and lemons, 56,853,720 kilos. Out of this amount there were imported to the United Kingdom, 11,018,470 kilos. In 1898 Palermo exported 51,759 kilos. of essential oils, and in 1899, 72,620, an increase of 40 per cent. In 1899, of raw lemon juice, 767,620 kilos.; citrate of lime, 301,380 kilos. Messina exported, 1899, oranges and lemons, 59,809,682 kilos.; of essential oils, 363,312 kilos.; citric acid, 111,070 kilos.; citrate of lime, 1,060,000 kilos.; concentrated lemon juice, 2,147,719 kilos. Catina exported, in 1899, 53,451 tons of oranges and lemons, valued at £250,316.

Although Sicily and the Riviera are the chief producing districts of the lemon, there is apparently no good reason why our own colonies should not encourage the cultivation of this fruit. Australia has for many years supplied its own requirements in oranges and lemons. The lemon tree has also been cultivated in New South Wales, Paramatta, Florida, California, and Jamaica, with varying success. Dr. James Neish, in an article in the Journal of the Jamaica Agricultural Society, July 1901, in speaking of the partial failure to grow lemons in that island, says: "There is no reason why this discouraging episode should deter others from a similar attempt if carried out on better lines, and, in point of fact, the cultivation of lemons has been recently attempted in the island, but on a smaller scale than at first." There are to be seen, in the galleries of the Imperial Institute, samples of lemon, Seville orange and Tangerine orange oils, which have been actually expressed in Trinidad, Dominica, and Jamaica. The West Indies supply us with the majority of limes and lime oil. Why should they not supply us in like manner with lemons?

The extraction of the oil from the fruit differs from the usual method pertaining to essential oils, inasmuch as the majority of such oils are obtained by distillation processes; that of lemon oil is made almost exclusively by expressing the rind of the nearly ripe fruit. There are two methods adopted for this purpose. First, the scorzetta, in which the lemons are cut in halves, and then pulped by girls with the aid of a peculiar scoop. An experienced worker, by rapid turns of the spoon ejects the pulp from the rind, at the rate of 24 halves per minute. After the lemons are pulped, the peel is allowed to soak in water from 2—4 hours. This part of the process is of great importance, as, in the soaking, the rind absorbs water, and becomes tough and brittle—a characteristic of great importance to the rind-presser. The halves are then passed on to men who do the pressing. They pick them up, one at a time, with the right hand, and let the half lemon-rind fit the fingers like a cap, gently pressing it against a sponge held in the left hand. The sponge, when saturated, is squeezed into an earthen vessel, and the water allowed to separate from the oil. The oil so obtained is filtered, and put into large copper bottles, ready for exportation. The only difference in the other process, viz., the taglio-in-tre, is that the lemon is cut into three longitudinal pieces in such a way that there is practically no acid pulp left in the rind. The process of expression is the same as in the scorzetta process. Of recent years a machine has been introduced into some factories, but the general opinion is that the hand-expressed oil is cheaper and better in all respects, and for this reason several firms who had introduced the machines into their factories are stated to be now abandoning them for manual labour. This machine must not be confused with the Bergamot one, which is practically a grinding mill for taking off the outside rind and so breaking the oil vesicles, but is one which carries out the Scorzetta process, and requires only two girls to work it. It is possible that in some of our colonies, where the expense of male labour might be a drawback to the profitable manufacture of this oil, such a machine would solve the difficulty, although in Sicily, where men's labour is so cheap, the use of the machine offers no advantage.

As regards the yield of oil, 110 kilos., or, say, about 1,000 lemons will yield from 14—16 ounces. A man working by the Scorzetta process will cut 12,000 lemons a day, and his companion will press 3,000, and for this work they will each get 2½ lires *per diem*, equal in our money to 1*s.* 10*d.*

Constituents of the Oil.—Up to the present time the following substances have been reported to be present in oil of lemon:—Pinene, phellandrene, limonene, citral, octyl and nonyl aldehydes, geranyl acetate, geraniol, methyl anthranilate, citronellal, citraptene, and a resin.

Limonene, citral, geranyl acetate, geraniol, and citronellal are all well-known substances, and their chemical and physical constants can be found in most text books, but we would draw attention to the aldehydes, nonyl and octyl—as these have only quite recently been shown to be present by one of us—*see* "Proceedings of the Chemical Society," 1901, No. 240. These aldehydes, though present in very small quantities, play, in our opinion, a most important part in the aroma of this oil. The crystalline body, and a detailed account of how it is obtained, can also be seen in the "Proceedings of the Chemical Society," 1901, No. 240. Although a similar substance has been obtained by Crismer, yet the interesting feature about it is its method of isolation by means of acetone.

Quite recently we have been able to prove the presence of the terpene, lævo-pinene, also another terpene which we have not yet been able to identify, owing to the very small quantity. We are still working on the subject. The pinene had the following constants:—Boiling point, 154° C. at 752 mm.; sp. gr., 0·859 at 15° C.; rotation, – 7° 30′; refractive index, 1·4678, at 15·5° C.; nitrosochloride, m. pt., 100° C.: hydrochloride, m. pt., 124° C.; although the rotation and gravity of these are not what are generally given for pinene, owing to the smallness of quantity we were unable to purify it any further, but we think the very characteristic hydrochloride is quite sufficient to prove the identity of this terpene with pinene.

Had it not been for Dr. Young's pear-shaped dephlegmator with 12 bulbs, possibly this would never have been isolated. The fractionation from an ordinary distillation flask is quite useless. Messrs. Schimmel have been unable to find any pinene in oil of lemon, even on fractionating large quantities, but to be quite certain that this pinene did not get into the oil by illegitimate means, we carefully fractionated some oil that one of us actually saw pressed in Palermo, and we were able to identify this same terpene, so that pinene is apparently a natural constituent of the oil.

The resin was obtained by dissolving the residue, left after the distillation of lemon oil, in hot acetone, which has the property of taking up the resin as well as other matters in the oil, but on cooling the resin is thrown out as an amorphous powder while the other substances remain dissolved. One or two treatments of this kind will produce a fairly clean sample. So prepared, it gives a melting point of 64°—67° C. Of the many substances that have been said to be present in oil of lemon, we have been unable to confirm citronellal and phellandrene.

Examination of the Oil.—For the examination of lemon oil as to its genuineness, the determination of its specific gravity, optical rotation, and citral content, combined with the rotation after distilling the first 10 per cent., are usually considered sufficient, but we have found that these determinations alone are by no means adequate to detect adulteration, as we shall endeavour to show later.

The specific gravity given in most text books, including the British Pharmacopœia, is 0·857—0·860 at 15° C., but in our opinion, 0·857 is too high. We should suggest 0·856—0·859, as 0·860 is seldomed obtained, 0·856 being quite an ordinary gravity for an undoubtedly genuine oil. The gravity varies according to the season when the fruit is pressed, and is always found to be higher late in the season than in the earlier part. An explanation of this may be that as the lemons are left on the tree to ripen, they lose part of their terpenes. The flavoured portion remains in the lemon, and being less volatile and of considerably higher gravity than the terpenes, increases the gravity of the oil.

Mr. Jacob, German consul at Messina, says, "I had hitherto always found that the gravity of this essence varies from 0·857 to 0·861, the last-named figure not being very frequent. I find that all lemon oil of last season had a very low specific gravity, and that the gravity of 29 samples varies from 0·8536 to 0·8588, and does not exceed the last-named figure." Messrs. Fratelli de Pasquale and Co. state: "All the lemon oils we have ever examined have always given us a specific gravity under 0·858, 0·8575—0·8577 being the normal. We have never examined or heard of any oil having 0·860, and we are sure no regular lemon oil has ever existed giving the said character, and this year's crop especially was of high rotation, and of a low specific gravity." Undoubtedly this season's oil has been of a lower gravity than previously.

Optical Rotation.—The rotation was taken in a tube 100 mm. long, by means of a polarimeter employing the sodium light. A variation of temperature between 15° and 20° makes only a slight difference, and for all practical purposes may be ignored. We have found that a range of α_D + 58° to + 63° is quite compatible with a genuine oil. At the earlier part of the season an oil may have a rotation of 68°, but, of course, such an oil is not usually met with on the market, as it would all be bulked with oil of a lower rotation. The rotation also varies considerably for the district in which the fruit is grown.

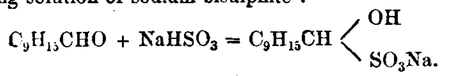
Refractive Index.—We have found a determination of the refractive index to be of considerable value in the examination of this oil, as in many others. Much has been said and written about the worthlessness of such a constant in essential oil analysis. This may be true of some oils, but it by no means bears out our experience in the case of lemon oil. Where one has a range of 1·4733 to 1·4830, or say 7 in the third place of decimals, and where 90 per cent. of the oil has a low refractive index, and the other 10 per cent., a very high one, we maintain, and have found, it to be a very useful constant. There is one important reason why probably the refractive index has not found favour and use among workers in essential oils, viz., that the temperature at which the observations have been made has in many cases not been recorded, and so a comparison is impossible. We at first found this a serious drawback. Another reason has been the difficulty of getting a suitable instrument which is quick and reliable. The difficulty and tediousness of determinations by the hollow prism method are well known. After trying many instruments, we have found the Abbé Refractometer the most suitable for this kind of work.

With regard to the rotation of the first 10 per cent. distillate, the British Pharmacopœia, 1898, directs that "if 100 volumes be fractionally distilled, the 10 volumes first collected should not produce a rotation differing by more than two degrees from that produced by the original oil." This is, in our opinion, incorrect and absolutely worthless, as all oils of undoubted purity give a greater difference than two degrees. This we have proved conclusively, both in the laboratory and on a manufacturing scale. We quite recognise that the rate of distillation, length of distilling head, and temperature of the distilling bath play an important part in the nature of the distillate.

Citral Content.—The next determination, that of citral, is in some respects the most important, and at the same time the most difficult. The strength (not flavour) of lemon oil is in a large measure due to the amount of citral present; but the fineness of aroma is due to the alcohols and esters present, and, as we have previously stated, to the other two aldehydes.

There are three methods generally employed, viz.:—

(1) By the addition compound formed on shaking with a strong solution of sodium bisulphite:—

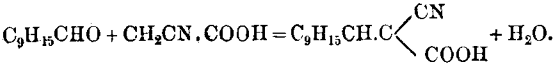


$$C_9H_{15}CHO + NaHSO_3 = C_9H_{15}CH\left\langle\begin{matrix}OH\\SO_3Na.\end{matrix}\right.$$

(2) By the formation of an oxime with hydroxylamine in alcoholic solution and estimation of the amount used by titration of the unused hydroxylamine:—

$$C_9H_{15}CHO + H_2NOH = C_9H_{15}CH:NOH + H_2O.$$

(3) By the condensation compound formed with cyanacetic acid in alkaline solution:—



$$C_9H_{15}CHO + CH_2CN.COOH = C_9H_{15}CH.C\left\langle\begin{matrix}CN\\COOH\end{matrix}\right. + H_2O.$$

(4) By the formation of a soluble sulphonic acid with sodium-sulphite, using phenolphthaleïn as an indicator:—

$$C_9H_{15}CHO + 2\ Na_2SO_3 + 2\ OH_2$$
$$= C_9H_{17}\ (NaSO_3)_2COH + 2\ NaOH.$$

(1) *The Sodium Bisulphite Method*, as directed in text books, is not to be recommended, and a strongly acid solution should be avoided. A 40 per cent. solution of sodium metabisulphite is most convenient. The method is as follows:—To 25 c.c. of lemon oil add 75 c.c. of 40 per cent. sodium metabisulphite solution and 25 c.c. of sodium sulphite (made by exactly neutralising the sodium bisulphite solution with a 10 per cent. solution of sodium hydrate) in a suitable flask. (*The Analyst*, August 1900.) The temperature is then raised to about 70° C., and the solution thoroughly shaken for one hour. We find that the addition of sodium sulphite is indispensable, owing to the fact that it forms a soluble sulphonic acid compound, and a clear meniscus is thus obtained. The oil is then raised into the measuring burette, and the diminution in volume gives the amount of total aldehydes. A correction must be made for the solubility of the terpenes in solution. We subtract from the reading 0·35 c.c., but recommend each worker to determine this correction for himself.

This method may also be used for solutions containing high percentages of citral, but using from 5 to 20 c.c., and measuring in a Hirschsohn flask or modification of it.

(2) *Hydroxylamine Method.*—A freshly prepared alcoholic solution of hydroxylamine hydrochloride is used, containing one gram of hydroxylamine-hydrochloride in 20 grms. of 80 per cent. alcohol. 10—12 c.c. of this solution are diluted to 250 c.c., and 25—50 c.c. are titrated for hydroxylamine in the usual manner, with N/10 sodium hydrate solution, using first methyl orange and then phenolphthaleïn as indicators.

A second equal amount of the strong hydroxylamine-hydrochloride solution is mixed with a weighed quantity of lemon oil, and diluted with absolute alcohol until a clear solution is obtained. After the addition of 0·5 grm. of sodium bicarbonate, the mixture is introduced into a 150 c.c. flask and heated on the water-bath for 45 minutes under a reflux condenser. After cooling, the product is washed into a 250 c.c. flask with distilled water, care being taken to wash out the condenser as well as the flask. After thoroughly shaking out, the watery layer is adjusted to 250 c.c., and 25 c.c. pipetted off. For a determination of the unused hydroxylamine in this, one drop of methyl orange is added and then diluted hydrochloric acid is cautiously run in from a burette until a faint rose colour is developed. The colour is then titrated back with N/10 sodium hydrate solution, phenolphthaleïn added, and titration continued until the development of a rose colour. The difference between the blank and the experiment will be the amount of hydroxylamine used, and the percentage may be calculated by the following formula:—$C = \frac{1·52 \times A}{G}$, where C = the percentage of aldehyde, A = number of c.c. of N/10 sodium hydrate solution used, and G = weight of oil taken. This is, in our opinion, an excellent method if one only knows the approximate percentage of citral present. If known, the correct quantity of hydroxylamine and sodium bicarbonate can be added, when good results may be obtained. The method as we have described it above gives good results with lemon oils.

(3) The procedure in this case, as suggested by Parry, is as follows:—200 c.c. of the oil are concentrated to 25 c.c. in a Wurtz flask under a pressure which should not exceed 15 mm. The 175 c.c. of terpenes contain a little citral, but the quantity is so small that it may be neglected. 10 c.c. of the residue, of which the specific gravity is known, are shaken with a solution of 5 grms. cyanacetic acid and 5 grms. ordinary KOH in 30 c.c. water in a small absorption flask with a long graduated neck, and the non-absorbed residue read off as usual. (The specific gravity of citral is so near that of the concentrated oil used that it may be taken as identical.) In our opinion this method is practically useless, as it is impossible to read the meniscus owing to resinous and slimy matter. Messrs. Schimmel have pointed out this same difficulty. We have found that,

if the residual oil be steam-distilled, it is possible to get a fairly clean meniscus, and it might be worth while for anyone wishing to use this method to adopt our suggestion. Anyhow, it is a long and tedious process, and we fail to see any advantage in it. The readings are invariably too high, and this is not surprising when commercial citral, containing 84 per cent. of aldehyde, gives 100 per cent. by this method. Another obvious disadvantage is having to use a concentrated solution.

(4) Professor Tiemann, in his elaborate work on the aldehydes of lemon-grass oils has, among other important reactions, shown that if citral be agitated with a neutral solution of sodium sulphite, a sodium salt of the sulphonic acid results, as shown by the above formula. By taking advantage of the sodium hydrate formed by this reaction, we have used phenolphthaleïn as an indicator to show when all the citral has been combined; and the fact that the above sodium salt is quite soluble, has, in our opinion, made the estimation of citral both accurate and simple. The method we have adopted is as follows :—A saturated solution of sodium sulphite is prepared, and, if acid, is neutralised with a solution of sodium hydrate until a faint pink colour is permanently maintained with phenolphthaleïn. To 50 c.c. of such solution 25 c.c. of the oil are added, and two drops of an alcoholic solution of phenolphthaleïn. The whole is then heated on a water-bath to nearly boiling point, constantly shaking. A deep red colour almost at once appears, which shows that the action has commenced. A few drops of sulphurous acid are then cautiously added, and this is continued until no further colour is produced after a further addition of SO_2. The oil is then measured as described in the first method. The obvious advantage of this method is that the end of the reaction may be ascertained to a certainty, while the above bisulphite method depends on the continual shaking for a period of not less one hour.

Table 1 shows a comparison of these various methods.

TABLE 1.

Showing Comparison of the Four Methods for Determination of Citral.

Nominal Percentage of Citral in Lemon Terpenes.	Sodium Bi- and Mono-sulphite. I.	Hydroxyl-amine. II.	Cyan-acetic Acid. III.	Sodium Sulphite. IV.
2·5	2·2 (2·1)	2·6 (2·4)	..	2·2 (2·1)
5·0	4·4 (4·2)	5·0 (4·8)	..	4·0 (4·2)
7·5	6·5 (6·3)	7·1 (7·2)	..	6·4 (6·3)
10·0	8·6 (8·6)	9·6 (9·6)	..	8·4 (8·6)
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Commercial citral.......	84	..	100	84
Terpeneless lemon oil ...	46·5	46·6	62	45·5
Lemon-grass oil.........	75	..	97	75

The figures in brackets show the calculated citral present.

Our conclusions with regard to these various processes are that the bisulphite with sulphite (No. 1) and the sulphite (No. 4) are the two most reliable, and give almost theoretical results if carried out on the lines we have suggested.

The percentage of citral in lemon oil is certainly not 7—8 per cent., but much nearer 4 per cent. We have shown elsewhere that 7—8 per cent. is a highly improbable figure, and our latest investigations confirm our opinion. Still, if a genuine oil, having a gravity of 0·860, can be obtained, then 7—8 per cent. would be quite reasonable, but such oils are not easily obtainable, though a small quantity of such oil was recently submitted to us, which did contain 7·4 per cent. of citral.

Adulteration.—The following are the chief adulterants :—Lemon terpenes, turpentine, lemon grass citral, and, less commonly, distilled lemon oil, cedarwood oil, and stearin. The Italians are as great offenders in this direction as our own merchants. In many cases a considerable amount of scientific knowledge is shown in the preparation of these sophisticated oils, and a great deal may be accomplished by means of a specific gravity balance and a polariscope. Of these adulterants the most frequent and the most difficult to detect are the lemon terpenes obtained during the manufacture of terpeneless oil.

A few figures that we obtained from the British Consul for Sicily and the German Consul at Messina will show to what a serious extent this adulteration goes on at the actual centres of production. There were imported into Palermo, of turpentine :—

—	1900.	First half of 1901.
	Kilos.	Kilos.
United Kingdom	505,000	..
France	70,000	30,600
Russia...............................	132,000	80,000

Messina imported lemon- and orange-oil terpenes, in 1900, 843 kilos from England and 4,205 kilos. from Germany; as well as 4,932 kilos. of French and 25,064 kilos. of American turpentine.

Besides the quantity of terpenes imported, there are in Sicily annually worked up into terpeneless oil about 4,000 lb. of lemon oil. This would give about another 3,600 lb. of terpenes.

These figures show that Sicily uses about 15,745 lb. of terpenes and 605,101 lb. of turpentine yearly.

We have been unable to make any comparison between the amount of turpentine used in Sicily and other purely agricultural islands, like the Isle of Wight, but this enormous importation of turpentine is very suspicious, because there exists no legitimate purpose for which it can be used in the quantities imported. It cannot be employed in manufactures, because there are none in Sicily, and it cannot be used in any art, unless it be the art of adulteration. The large quantity of terpenes also imported into Sicily is more suspicious still, because for this there exists no reason whatever, save a dishonest one; there being no purpose for which it can be used other than that of sending it back to this country, plus a small amount of new oil.

Of these adulterants, the lemon terpenes are the most difficult to detect, chiefly for the reason that natural oil consists of about 90 per cent. of such, and so a considerable amount may be added without altering the constants of an oil to any appreciable extent. Turpentine is still used, but not to so large an extent as formerly. It may easily be detected on distilling and examining the rotatory power of the first 10 per cent. of distillate, which would have a much lower rotation than the original oil. Citral from lemon grass oil is a very common adulterant, and is always added to bring up the strength of an oil adulterated with terpenes. Even the purest commercial citral has a distinct and unpleasant smell, resembling lemon grass oil. Stearin is alleged to be used for increasing the gravity of an oil. We have never found any oil so adulterated, and should think it highly improbable that such adulteration is practised. Distilled lemon oil would have practically the same effect as the addition of terpenes.

A Scheme for the Examimation of Lemon Oil:—

(1) Determination of the sp. gr. at 15° C.
(2) „ „ opt. rot. in 100 mm. tube.
(3) „ „ ref. index at 20° C.
(4) „ „ citral content, 25 c.c. (total aldehydes).
(5) Fractional distillation.

The following is the method of distillation we adopt, and, if carried out carefully, will show any adulteration.

One hundred c.c. of the oil to be examined are put into a distilling flask having three bulbs blown in the neck, and fitted with cork and thermometer. This is connected up to a condenser with a suitable receiver having two vessels graduated at 10 c.c. and 80 c.c. A Brühl's apparatus answers the purpose very well. The whole is exhausted, and a pressure of not more than 15 mm. should be obtained. The flask is then gently heated by means of an oil bath. The first 10 c.c. should not take less than seven minutes to distil. The next vessel is put into position, and the distillation continued until 80 c.c. have distilled over. The pressure is then relieved, and the residual oil in the flask is distilled over with steam. The quantity so obtained should be carefully noted. The three fractions are examined by the polariscope and refractometer. (NOTE.—We have used the Zeiss form of instrument for this purpose, as being one that most analytical chemists have, and a comparison of results is thus facilitated.)

The following is a typical distillation :—

No. of c.c.	Time of distilling.	Pressure.	Boiling Point.	Rotation α_D	Zeiss at 20° C.
	Mins.	mm.			
10	7	14	64	+ 55° 30′	72·0
80	36	13	71	+ 70° 40′	72·4
7·5	..	..	..	+ 10° 56′	82·5

Table 2 gives the results of five pure oils by this method.

TABLE 2.

A Comparison of various Types of Lemon Oil, all of undoubted Purity.

—	Particulars of Oils.	Physical Constants of Original Oils.	Distillation.			Citral content of 3rd fraction.
			No. of c.c.	Rotation α_D in 100 mm. Tube.	Zeiss Readings.	
						Per Cent.
I.	Genuine oil of good quality. (Messina.)	Sp. gr. = 0·857 Rotation = + 63° 12′ Ref. index = 1·4755	10 80 7·5	+ 55° 30′ + 70° 40′ + 10° 56′	72·0 72·4 82·5	42
II.	Genuine oil (Palermo). Not typical.	Sp. gr. = 0·857 Rotation = + 61° 27′ Ref. index = 1·4757	10 80 7·5	+ 54° 6′ + 67° 45′ + 19° 13′	72·2 72·4 81·5	35
III.	Genuine oil of very high gravity.	Sp. gr. = 0·862 Rotation = + 58° 30′ Ref. index = 1·4771	10 80 7·0	+ 59° 41′ + 67° 16′ − 6° 0′	72·0 72·7 90·6	64
IV.	Genuine oil of high gravity.	Sp. gr. = 0·860 Rotation = + 60° 36′ Ref. index = 1·4769	10 80 7·0	+ 50° 6′ + 68° 43′ − 3° 20′	72·2 72·6 88·6	64
V.	Genuine oil, new crop, 1901.	Sp. gr. = 0·858 Rotation = + 61° 28′ Ref. index = 1·4754	10 80 7·0	+ 51° 47′ + 69° 2′ + 8° 14′	71·8 72·3 81·9	40

100 c.c. of oil taken in each experiment.—Zeiss readings (Butyro-refractometer) corrected to 20° C.

No. 1 is a very typical oil of last season's pressing.

No. 2. An oil expressed in Palermo, and of undoubted purity, being pressed, coppered, and sealed in the presence of one of us (Child).

No. 5. A new oil of good quality and freshness.

No. 3 and 4. A genuine oil of very high quality, and only very exceptionally met with.

TABLE 3.

Showing Oils adulterated with Terpenes, Turpentine, and Citral

—	Particulars of Oils.	Physical Constants of Original Oil.	Distillation.			Citral content of 3rd fraction.
			No. of c.c.	Rotation α_D in 100 mm. Tube.	Zeiss Readings.	
						Per Cent.
I.	Lemon oil, 90 per cent. Turpentine, 10 per cent.	Sp. gr. = 0·858 Rotation = + 57° 45′ Ref. index = 1·4752	10 80 8·0	+ 45° 48′ + 64° 56′ + 18° 25′	69·6 71·6 81·2	34
II.	Lemon oil, 90 per cent. Lemon terpenes, 10 per cent.	Sp. gr. = 0·856 Rotation = + 64° 2′ Ref. index =	10 80 7·5	+ 56° 21′ + 71° 37′ + 18° 19′	71·6 72·1 81·2	34
III.	Lemon oil, 80 per cent. Turpentine, 20 per cent.	Sp. gr. = 0·859 Rotation = + 53° 1′ Ref. index = 1·4741	10 80 8·0	+ 40° 9′ + 59° 5′ + 22° 41′	68·1 71·1 81·0	32
IV.	Lemon oil, 75 per cent. Terpenes, 23 per cent. Citral, 2 per cent.	Sp. gr. = 0·857 Rotation = + 62° 56′ Ref. index = 1·4753	10 80 7·5	+ 53° 50′ + 70° 30′ + 13° 3′	71·9 72·4 83·6	52
V.	Lemon oil, 50 per cent. Terpenes, 46 per cent. Citral, 2 per cent. Cedar wood oil, 2 per cent.	Sp. gr. = 0·856 Rotation = + 62° 5′ Ref. index = 1·4753	10 80 7·5	+ 53° 24′ + 70° 15′ + 5° 12′	71·8 72·5 90·5	34
VI.	Distilled oil.	Sp. gr. = 0·855 Rotation = + 63° 0′ Ref. index = 1·4751	10 80 9·0	+ 58° 16′ + 70° 10′ + 15° 5′	72·1 72·8 89·1	9

100 c.c. of oil taken in each experiment.—Zeiss readings (Butyro refractometer) corrected to 20° C.

Table 3 shows the effect of terpenes, turpentine, and citral. The oil used for making these mixtures is in each case No. 1 in Table 2.

No. 1 shows an increased gravity, which is always the case with oils adulterated with turpentine. The difference between original rotation and the first 10 per cent. is 12°, whereas in pure oils it should be from 7° to 8°.

The Zeiss number for the first fraction is about three lower than for a genuine oil, and the citral content of the third fraction shows 34 per cent. instead of 42 per cent.

No. 3. Similar differences are shown, but in a more marked degree.

No. 2 oil shows a rather high rotation, and the third fraction at once points to addition of terpenes, the rotation being high and the refractive index low. The citral content is also lower than that of a good oil.

No. 5. This oil has constants closely resembling that of a natural oil, but the third fraction, which has a rotation of + 5° 12′, shows on comparing it with a genuine oil, that whereas the Zeiss number should be 82·5, that of the adulterated oil is 90·5, and, therefore, the rotation should be *minus* not *plus* 5.

Terpeneless Oils of Lemon.— We do not propose to discuss the manufacture of terpeneless oils of lemon. We shall confine ourselves to the properties and examination of such oils; but it will not be out of place to give some reasons why these terpeneless oils have found such favour among mineral water manufacturers of late years. First, lemon oil contains roughly 90 per cent. of terpenes; in the presence of fruit acids these readily oxidise, and soon develop a smell and taste of turpentine. This, of course, is a serious objection to the use of natural oil, whereas, if these terpenes are removed, as in the case of terpeneless oils, there is nothing to deteriorate or change. Second, a most important advantage is its solubility, which has increased in a similar proportion to its concentration, say, from 12 to 20 times. This enables the mineral water maker to obtain a bright and clear drink instead of the muddy mixtures one often sees in waters prepared from natural oil. The relative yield of terpeneless oil is from 4 to 6 per cent., depending on the gravity of the natural oil used. The price should not be relatively higher, as the cost of manufacturing this article is, to a large extent, counterbalanced by the sale of the unused terpenes. These, as we have shown, find their way back to Sicily, and it is one of the worst worries of the works chemist to see that these identical terpenes, plus a small percentage of new oil, do not return to him as genuine oil of lemon. It is regrettable, in our opinion, that the Sicilian authorities do not see their way clear to prohibit the import of these terpenes, or impose a duty on them sufficient to make such adulteration unprofitable.

The failure of the Sicilian authorities to take measures to suppress these frauds is a danger to the essential oil trade of Sicily. Lemons are being cultivated in many of our colonies, and Sicily has no secure monopoly of the lemon trade.

For the examination of this class of oils a determination of the specific gravity, rotation, refractive index, aldehydic content and solubility, and in some cases, a determination of esters and alcohols after the removal of citral, is in most cases sufficient.

These constants are determined under the same conditions as in lemon oil.

On referring to Table 5, it will be seen that the specific gravity does not vary two in the third place of decimals, so that 895 to 899 may be safely given as the limits. In the third column is the optical rotation. This should not vary beyond the limits of −5° to −8°·30′. A rotation of less than −5° would indicate the presence of terpenes. On the other hand, a greater minus rotation would show that some of the earlier and sweet-scented fractions have been left out. It is of great importance when considering this factor that the next column be examined, as here will be found the refractive index which will indicate both the presence of terpenes and added citral in a clumsily adulterated oil. The limits for refractive index may be given as 1·4810 to 1·4822.

The percentage of total aldehydes should not be greater than, say, 50 per cent., or less than 40 per cent., when determined by either the sulphite or a combination of bisulphite and sulphite methods, Nos. 1 and 4, as previously described. Anything above 50 per cent. would suggest addition of lemon grass citral.

The unabsorbed portion should always have a greater minus rotation than the original oil.

As regards solubility, the usual method of determining this, *i.e.*, by observing the solubility in alcohol of definite strength is of little practical value to the essence maker, who requires to know the quantity of the weakest spirit that will dissolve a given quantity of oil. To ascertain this we have adopted the following method:—

Take 1 c.c. of oil, to which add 20 c.c. of 94 per cent. alcohol. Distilled water is run in from a burette until a permanent milkiness ensues. The number of c.c. of water used is what we term the "solubility number."

We have added a column giving the calculated refractive index of the absorbed aldehydes. This is only of value to show when a so-called terpeneless oil is only a mixture of partly concentrated lemon oil and lemon grass citral. Idris has shown such adulteration to be quite common. ("Proceedings of the Pharmaceutical Conference," 1899.) Referring to No. 6 oil on the table, an oil prepared by a well-known German maker, it will be seen at once that the percentage of total aldehydes is something like 12 per cent. higher than generally pertains to oils of other makers. On the other hand, the calculated refractive index of the aldehydes shows a difference of 0·0013. No. 7, an oil by the same maker, but about 25 per cent. cheaper, contains 6 per cent. more aldehyde than the better quality oil. Undoubtedly this addition must be due to lemon grass citral, and yet the calculated refractive index of the aldehydes remains the same. We investigated this matter, and fractionated 500 c.c. of oil, No. 4 in Table 4., and found, by rejecting Nos. 1 and 11—15, we obtained an oil as shown by No. 4*a*, which gives a calculated refractive index the same as Nos. 6 and 7. No. 10 shows an oil, to

TABLE 5.

Showing a Comparison of various Terpeneless Oils of Lemon.

No. of Sample.	Specific Gravity at 15° C.	Optical Rotation a_D, 100 mm.	Refractive Index.	Per Cent. of Aldehydes.	Refractive Index of Non-aldehydes.	Rotation of Non-aldehydes.	Solubility Number.	Calculated Refractive Index of Aldehydes.	Remarks.
I.	0·897	− 8° 19′	1·4818	43	1·4784	− 14° 12′	5·8	1·4886	Genuine terpeneless oil.
II.	0·896	− 6° 28′	1·4816	46	1·4783	− 11° 35′	5·7	1·4854	Ditto.
III.	0·898	− 8° 6′	1·4812	47	1·4778	− 14° 40′	5·9	1·4850	Ditto.
IV.	0·897	− 7° 54′	1·4820	47	1·4790	− 13° 37′	6·0	1·4854	Ditto.
V.	0·897	− 4° 33′	1·4833	32	..	..	..	..	Oil made from an old lemon, oil adulterated with turpentine.
VI.	0·897	− 7° 30′	1·4807	59	1·4763	− 18° 19′	9·2	1·4837	Celebrated German make, best quality.
VII.	0·900	− 6° 24′	1·4816	65	1·4773	− 17° 0′	10·0	1·4837	Ditto second quality.
VIII.	0·897	− 1° 30′	1·4867	83	1·4819	..	13·3	1·4876	Commercial citral.
IX.	0·896	− 1° 29′	1·4836	80	1·4787	..	16·5	1·4848	So-called terpeneless lemon grass oil.
X.	0·898	− 4° 40	1·4820	58	1·4769	..	8·8	1·4839	An attempt to imitate No. VI.
IV*a*.	0·897	− 5° 50′	1·4808	52	1·4776	..	7·8	1·4837	Terpeneless lemon oil used in No. X.
XI.	..	..	1·4860	90	..	..	21·2	..	Citral used in No. X.

Note.—All refractive indices corrected to 20° C.

every 100 c.c. of which 10 c.c. of citral have been added. This mixture conforms in a satisfactory manner with No. 6. In fact, with but slight alteration in the mixture of fractions for No. 4*a*, an exact imitation could be obtained.

TABLE 4.

Showing Fractionation of 500 *c.c. Terpeneless Oil of Lemon No.* 4.

Number.	Quantity in c.c.	Rotation α_D, 100 mm.	Refractive Index. $[N]_D$, 20° C.	Solubility No.	
I.	17	+ 12° 14′	1·4773	7·5	
II.	24	+ 11° 12′	1·4782	7·7	*
III.	32	+ 4° 24′	1·4790	7·9	*
IV.	32	− 1° 38′	1·4800	8·2	*
V.	32	− 5° 27′	1·4806	8·2	*
VI.	30	− 7° 0′	1·4809	8·2	*
VII.	28	− 8° 12′	1·4813	8·2	*
VIII.	46	− 9° 9′	1·4817	7·8	*
IX.	48	− 9° 43′	1·4819	7·4	*
X.	48	− 10° 41′	1·4820	7·1	*
XI.	30	− 11° 34′	1·4819	6·8	*
XII.	40	− 13° 4′	1·4817	6·4	
XIII.	32	− 15° 45′	1·4815	5·7	
XIV.	23	− 20° 22′	1·4815	4·9	
XV.	16	− 27° 30′	1·4828	4·1	

We have to express our thanks to the proprietors of the London Essence Co., in whose laboratories our investigations have been carried out, and to Mr. T. H. Page, B.Sc., our assistant, for valuable and careful analytical work in connection with this paper.

DISCUSSION.

Mr. E. J. **PARRY** said that he regarded the paper as especially interesting, inasmuch as it recorded not merely laboratory experiences, but a study of the oils in their native purity on the spot. To accept the facts put forward by the authors would be to upset a great many preconceived ideas on the subject of lemon oil. It seemed to him that a number of the authors' deductions from the analytical point of view had their origin in the fact that they had examined oils of abnormally low specific gravity. They considered 0·857 to 0·860 high, and perhaps it was so at the present; for, in a season like the present one, very few oils went above it, and the general average was so low that it was not safe to base any deductions on the examination of oils from 0·857 to 0·858. The gravities down to 0·853 of this year were strongly indicative of lemon terpenes, of which the production was much more than the figures in the paper indicated. He knew of five makers whose factories were in regular work, and another was about to start. In Sicily the terpene fetched as much as 2*s*. 6*d*. per pound when lemon oil was dear, or very nearly the price of the lemon oil; and in addition to being re-imported to England, it was used as an adulterant of bergamot oil. Then the maximum figure of 0·858, only represented this year's samples and must not be noticed seriously. He had repeatedly examined samples of Messrs. Fratelli de Pasquali's oils with gravities of 0·860 to 0·862. Consequently, the authors' statement that they had never seen an oil of over 0·858 was inexplicable. Schimmel (2) gave as the average gravity 0·857 to 0·863. Of course the latter figure was but rarely met with, but oils of 0·861 were, in normal years, common in the market. The standard of the British Pharmacopœia was clearly absurd, because one met with pure samples, having a difference figure, for the rotation, of 4 to 5. The authors had passed over the fact that Crismer had isolated the crystalline body referred to, and Dr. Schmidt had examined it and identified it with Tilden's limettine. Neither did they make any mention of linalyl acetate, nor to the previous discovery of pinene by Tilden, who had shown that it was present in this oil. Schimmel had experimented and found only 0·016 per cent. distilling below 170°, and this had none of the characters of pinene; therefore, either Tilden's sample was adulterated or Schimmel had failed to discover a body conclusively proved to be present. It seemed hardly possible that in fractionating 50 kilos. they could pass over more than the merest trace of pinene: was pinene, therefore, a regular or only an occasional constituent of the oil? He took it that the crystalline body that the authors had prepared was clear evidence of its presence. The important point was the estimation of citral in the oil. The cyanacetic acid method to which reference has been made was due to Tiemann. It gave a very cloudy reading, but if an accurate estimation was required, he contended that after steam distillation this method was the most correct. It seemed to have escaped the authors' notice that one could not completely extract citral by bisulphite of sodium except with great difficulty. Tiemann pointed out that it required three shakings before one could get the last traces out. In a special sample of lemon grass oil the cyanacetic method left 18 per cent. of non-absorbable residue, but not a trace of citral. By the ordinary bisulphite method he only absorbed 76 per cent., which contained a large quantity of citral. In the case of lemon oil with only 6 per cent. it would be much more difficult to take out the last traces; indeed Tiemann's method showed that one could not easily get it out with bisulphite of soda. With regard to the refractive index, as the authors had pointed out, much adverse criticism had been passed on it. One point to be observed was that one could get no further information by it than by the polarimeter; and a polarimetric examination would give far safer indications to work on. The authors' last point was the terpeneless oil of lemon. The inference that he drew from that table was that the special sample, of German make, was not a genuine terpeneless oil, but was one with added citral. He had known a sample of this oil contain 62 per cent. of citral as against 59, and had personally prepared samples with 60 per cent. He, therefore, could not accept the authors' statement as to the amount of citral in terpeneless oil of lemon.

Mr. A. C. **CHAPMAN** said that chemists who worked in the laboratories of essential oil factories on large quantities of material, as the authors had, were at an advantage as compared with chemists working in other laboratories, as they could isolate and establish the identity of compounds, which would be impossible when working on a smaller scale. The authors of this paper had taken full advantage of their opportunities in this respect as they had recognised two new compounds and had further established the existence of pinene in certain samples. The aldehydes of which they had spoken were especially interesting, because they added two to the comparatively short list of fatty compounds known to exist in essential oils. With regard to pinene, its existence was affirmed years ago, then denied, and it was now conclusively proved by the authors to exist in certain samples at least. The sample of mixed aldehydes which was passed round had a strong lemon smell. Had the authors had an opportunity of comparing it with samples of the synthetic aldehydes? Then, these bodies underwent, as was well known, rapid transformation when exposed to the air, and he would like to know whether there had been any failure to obtain them from old samples of oil. Also what influence the acids formed by their oxidation had on the flavour and smell of the oil. The authors had brought a somewhat scathing indictment against the Italians in general and the Sicilians in particular, in regard to immorality in their dealings with lemon oil. Their position was, he thought, a somewhat peculiar one. The essential oil distiller was, in fact, the manufacturer of the very product he most feared as an adulterant, and Mr. Burgess had pointed out that the cost of the terpeneless oil need not be relatively more than the cost of the original oil, because of the value of these terpene by-products. The terpeneless oil makers were, therefore, in the position of being the suppliers of the adulterant they most feared, and they sold it apparently in the hope that their own chemists would be clever enough to prevent its return to their own works masquerading as lemon oil. The only proper course was to get rid of this product altogether, and thus remove much responsibility from the shoulders of their own chemists and much temptation from the path of the "guileless Sicilian." The apparatus referred to had been devised by Mr. Burgess and himself, not with reference to any particular method of estimating citral, but to confer greater accuracy on absorption processes in general. If the sulphite method had any value, then by using this piece of apparatus, a much greater degree of accuracy could be obtained. And that brought him to a point which had been raised by Mr. Parry. He had said that the citral could not be extracted unless the solution had three or four shakings, and with this he quite agreed, but with the Hirschsohn flask the proper amount of shaking could not be given, whilst with this apparatus it could. He was glad to hear Mr. Parry say that he considered 8 per cent. of citral in lemon oil too high; but he must dissent from one of his remarks, namely, the alleged inability of the sodium bisulphite method to give good results. Mr. Burgess and himself had independently prepared solutions of pure citral in pure distilled lemon terpenes. His own results satisfied him that by the use of the new apparatus and sufficient bisulphite he could get figures closely in accordance with the amount which had been added. At that time he hoped to undertake himself a critical examination of some of these processes for the estimation of citral; but Messrs. Burgess and Child had followed the matter up, and he thought that their results had abundantly justified their work, and would be of great value to all who were interested in the chemistry of lemon oil.

Dr. J. Lewkowitsch desired to raise a point not directly connected with the paper, but still of interest to users of the Zeiss apparatus. In Tables 2, 3, 4, and 5 the refractometric constants were expressed differently; the limits of the Zeiss butyro-refractometer were evidently reached in the case of the oils in Tables Nos. 4 and 5; in fact the butyro-refractometer figures would have been much over 100. He would like to enlist the sympathies of the authors of this paper to help to induce the maker to construct a butyro-refractometer which would show higher refractions to cover such cases as tung oil and rosin oils. At present, chemists who possessed the butyro-refractometer were obliged to buy two instruments instead of one.

Mr. J. F. Child, in reply, said that with reference to Mr. Parry's remarks on the pure samples of lemon oil, the time of year when collected and the districts in which they were gathered would be important factors. Those collected by himself were obtained about the middle of December of last year. Mr. Parry had spoken of the use of terpene for the adulteration of bergamot oil. This he doubted, because around Reggio the lemons were of a round character, and were ground in the bergamot machine, and the oil so obtained was used to adulterate, not the terpenes obtained from lemon oil. With regard to the sp. gr. 0·860 to 0·862, his Company had made offers to the principal Sicilian firms for supplies at these gravities and were unable to obtain any. With reference to the distilling the first 10 per cent. of the oil, it must be understood that the shape of the flask and the temperature of the oil bath were important matters. They had adopted this form of flask from Schimmel's books, but had enlarged it so as to suit the 100 c.c. distillation. Mr. Parry seemed somewhat sceptical as to the presence of pinene, but he would like to point out that without the pear-shaped fractionating head of Dr. Young, it would have been impossible to separate pinene from the lemon oil terpene.

They had never recommended the bisulphite, *i.e.*, the acid bisulphite, process, because one could only get the so-called addition compound which, it was true, would, on heating, decompose into citral and a sulphonic acid; this citral might again be taken up if the shaking were continued, but it made the method unreliable. They had recommended the addition of sulphite, which formed the soluble compound. This made the method identical with the sulphite and bicarbonate, and the specially recommended sulphurous acid and sulphite ones. A great advantage in this latter method was, as had been shown, that the indicator being present showed distinctly the beginning and end of the reaction. The bisulphite, bicarbonate, and sulphurous acid in the presence of sulphite were simply used to neutralise the sodium hydrate formed, and thus prevent the decomposition of the compound. With regard to taking up all the citral in the first shaking of the latter process, using their method, they had some citral made from the terpeneless lemon oil, the unabsorbed portion of which they had treated with bisulphite. A small amount of compound was formed, which was washed and decomposed. The little drop before the meeting represented the aldehyde obtained. The refractive index of that sample was 1·4601; it was a mixture of the two new aldehydes, and could not contain much, if any, citral. They always recommended that the refractive index should be taken in conjunction with the rotation, as it was not a sure guide by itself. With regard to citral in terpeneless oil of lemon, Mr. Parry claimed to be able to make a concentrated lemon oil containing a higher percentage than that obtained by Mr. Burgess and himself. So could they by fractionation, but to do so they would have to reject other important flavouring portions. He had there a sample of the unabsorbed oil from a terpeneless oil such as he had spoken of, which, if examined, would be found to have a very fine flavour. It had high refractive index, and a minus rotation, showing that the presence of terpenes could not exist to any appreciable extent. They had examined it for the amount of esters and alcohol (calculated as geraniol and geraniol acetate). The figures given left a considerable portion unaccounted for, and they were still investigating this matter. As a proof that lemon oil did not generally contain more than 4 per cent. of citral, Mr. Parry's own method might be taken. He had himself recommended a method for the estimation of citral in lemon oil in which he first concentrated the oil by fractionation. They themselves had, both on a manufacturing scale and in the laboratory, concentrated considerable quantities of lemon oil. They had never been able to obtain more than from 4 to 6 per cent. of flavouring matter which did not contain more than 50 per cent. of citral. The terpenes on examination only gave very small quantities of the new aldehydes of a very low refractive index showing absence of citral. Calculating this back, it would be seen that this method gave from 2 to 3 per cent. of citral for lemon oil.

A question was asked as to dispersion. They had taken it for several oils, but had not found it of much value, and it made the operation a long and tedious one. They had neglected it, though he thought that in some cases it might be of great importance.

They had not yet prepared the synthetic aldehydes, but recognised the great importance of doing so, and hoped shortly to continue the investigation of these aldehydes, more particularly the polymerised form, which should be of great scientific as well as commercial interest. They had examined many lemon oils, and had always been able to obtain these aldehydes from them, and therefore believed them to be a natural constituent of the oil. On the other hand, they had not examined very old oils, but did not think the aldehydes oxidised so readily as Mr. Chapman has stated. With regard to the lemon terpenes, the only reply seemed to be that it was against the principles of every manufacturing chemist to unnecessarily waste any of his bye-products, and while there was such a demand for these terpenes, they would continue to fetch comparatively high prices. The authors, themselves, would not care to suggest to their employers the remedy proposed by Mr. Chapman. If the Sicilian authorities stopped the importation of these terpenes, then the chemist would have to find other uses for them, and he would not have far to seek. One of their objects in bringing this communication before the Society was to show a method of detecting such adulteration.

Manchester Section.

Meeting held on Friday, December 6th, 1901.

DR. H. GROSSMANN IN THE CHAIR.

VANADIUM: ITS EXTRACTION AND USES.

BY H. PROCTER SMITH, F.C.S.

The attempts which have recently been made to use metallic vanadium for various purposes have created considerable interest in the metal, and a consideration of the methods adopted for its separation and the effects it produces from one who has had considerable experience may not be unwelcome.

The metal has a most interesting history; originally discovered by Del Rio in the lead ores of Zimapan, Mexico, and named erythonum, and discredited by Collet Descobla in 1805 as an impure oxide of chromium, it was rediscovered in 1830 by Sefstra, in the celebrated iron of Taberg in Sweden, and named vanadium.

In the same year Wohler confirmed Del Rio's discovery, and Sefstra handed over his research and materials to Berzelius, who made an extensive investigation and classed it with chromium and molybdenum. Finally the metal of Berzelius was shown by Roscoe to be a nitride or an oxide, according to the method of preparation, and that it belonged to the antimony group of the nitrogen family. Up to the present its application has been principally confined to the preparation of vanadium compounds for colouring purposes.

The metal occurs principally as vanadates of lead and copper, lime and alumina, the two former being the chief sources of the metal.

Vanadium is a silvery white metal of very high melting point, about 2,000° C., and low specific gravity of 5·5. When heated in oxygen it takes fire and forms the pentoxide V_2O_5. It ignites in chlorine forming the tetrachloride, and on heating in nitrogen is converted into the mono-nitride.

Hot hydrochloric acid is without action on the pure metal, but hot sulphuric acid dissolves it, forming a yellow solution, and nitric acid readily dissolves it, forming a deep blue solution. There are five oxides of vanadium, analogous in composition to the oxides of nitrogen; of these the highest member of the series, the pentoxide, is the most stable, and is obtained by the action of nitric acid upon any of the lower oxides. Chlorides, oxychlorides, a bromide, oxybromides, and sulphides of the metal are also known.

The preparation of the metal in a pure state is attended with many difficulties owing to the readiness with which it oxidises at or above redness in air or water-vapour, and in the process to be described the extraction of *pure* vanadium has not been attempted so much as the extraction of the metal in alloy with iron, of which the ore contains some quantity. The ferro-vanadium thus obtained is intended to be used in steel making, it being long known that the presence of vanadium in wrought-iron increases its ductility in a remarkable manner.

Process.—The ore treated was an impure vanadate of lead from Spain. The process may be divided into two chief portions, thus:—(1) Treating the ore for the separation of lead and preparation of a precipitate containing the iron and vanadium as oxides. (2) The reduction of the mixed oxides to the metallic state forming a ferro-vanadium alloy.

If it is desired to obtain the pure metal, this can be done by treating the oxides of vanadium and iron in such a manner as to separate the two metals before reduction.

The process is at present by no means perfect, but very satisfactory results and yields have been obtained, and with further experience and practice there is no reason why it should not become a success. The chief difficulty in obtaining vanadium or alloy of iron and vanadium from the oxide is owing to the exceedingly high melting point of the metal, the difficulty of reduction, and, as before stated, the tendency of the metal to oxidise and pass into the slag.

Ore.—The ore is of a very friable nature and crushes easily. It varies from light yellow to brown in colour, and is associated with galena, veins of the two minerals running side by side. From the analysis appended below it will be seen to consist of vanadate of lead with silica, oxide of iron, lime, manganese oxide, &c.

The ore is first crushed to a fineness such that it will pass through ⅛-inch mesh sieve, and fused with bisulphate of soda or potash. For this purpose the residue from the "nitre pots" of the sulphuric acid manufacturer may be very conveniently and economically used. This residue consists of bisulphate of soda or potash with a varying quantity of free sulphuric acid, and is known commercially as "nitre cake." This fusion may be effected in an iron vessel similar to a Pattinson de-silverising pot or in a small reverberatory furnace with a good hard sand bottom and a large tapping hole. In practice I have found the latter furnace to give good results if worked carefully and am taking one of this type as an example. The proportion of "nitre cake" to ore depends of course on the composition of the latter, but with some similar to that of analysis given—2 parts of nitre cake to 1 part of ore—give the best results.

Vanadium Ore.

	Per Cent.
Lead oxide	34·15
Lead sulphide (galena)	1·43
Vanadic acid (V_2O_5)	11·49
Iron peroxide	14·64
Silica	35·67
Manganous oxide	0·77
Lime	0·32
Zinc oxide	0·62
Carbonic acid	0·42
Molybdic acid	trace
Silver	3½ oz.
	99·51

The "cake" is thrown into the furnace, which is closed until the "cake" has almost melted, and the ore then added as rapidly as possible and the whole mass thoroughly mixed and agitated. A violent effervesence at first takes place, which soon subsides, and as the mixing of the fusion is continued it gradually thickens and is ready for tapping out. With charges of about 1 cwt. of ore and 2 cwt. of nitre cake and a moderate heat, it takes from 20 minutes to half an hour. Too high a temperature must be carefully avoided, as in that case the mass thickens too rapidly and does not allow the molten bisulphate sufficient time to thoroughly attack the ore. When decomposition is complete the tapping-hole of the furnace is cleared and the molten material either run into thin cakes on the floor or run into an iron or copper ladle, and then cast into thin cakes on a level surface. The cooled mass presents a lemon to orange yellow appearance and is very deliquescent, turning in the course of a few hours quite green on the parts exposed to the air, and by gradually absorbing more moisture, becomes white.

This "fusion cake" is crushed preparatory to its treatment with water. This is accomplished by a pair of rolls, being reduced to such a size that it can pass through a ¼-inch sieve.

Lixiviation.—The crushed "fusion cake" is put into a stout wooden vat, water added, and metallic iron in some form or other—a convenient form for this is old boiler plates, which may be suspended in the liquid by means of stout copper wires. The water, in order to facilitate the lixiviation, is heated by blowing steam through, and the whole is kept well stirred for 3 to 4 hours.

The contents of the vat now present a greenish-white appearance, which, on allowing to stand for from 4 to 6 hours, separates into a heavy white precipitate, leaving a clear solution of varying colour from dark green to blue. This is siphoned or steadily pumped into another vat, leaving behind the heavy white sediment. The action of the bisulphate of soda during the fusion appears to be to convert the lead, vanadium, iron, &c., into sulphates.

The vanadium sulphate combines with the sodium sulphate forming $Na_2SO_4 + (VO)_2(SO_4)_3$, the yellow mass previously referred to. $VO.OH.SO_4$ is also present and probably some HVO_3.

Treating with water, owing to the insolubility of lead sulphate, forms a very ready means of separating that metal. The insoluble precipitate left behind contains therefore all the lead as sulphate, almost all the silica, and any undecomposed ore which might be present. The clear solution siphoned over contains sulphates of the vanadium and iron and any manganese present, together with traces of silica.

The purpose of putting the scrap-iron into the vats during lixiviation is to reduce any vanadium present as vanadate to the vanadic salt. A little of the vanadium generally appears to be present in this state, and if allowed to remain so would not, in the subsequent precipitation with caustic soda, come down completely and hence be lost.

To the clear siphoned liquid is now added, in small quantities at a time, a solution of caustic soda (a 25 per cent. solution forms a very convenient strength) when a black or grey precipitate is thrown down. The addition of soda is continued till a drop of the filtered liquid, brought into contact with a drop of weak caustic soda solution on a white porcelain tile, remains perfectly clear, showing that precipitation is complete.

The precipitate contains the iron and vanadium as hydrated oxides and requires filtering. This is done by pumping into a filter-press, and, as the precipitate is of a very bulky gelatinous nature, it is necessary to pump it very slowly, and, in order to obtain full cakes and thorough washing of the precipitate, the frames to contain the cakes should not be more than half or three quarters of an inch thick. The clear filtrate coming from the press consists chiefly of a solution of sulphate of soda. It may be tested for vanadium, which would probably be present as sodium vanadate, by acidifying a little with hydrochloric acid and then adding some hydrogen peroxide, when a red coloration is produced by the presence of only minute traces of vanadium. This coloration is probably due to the formation of a hexavanadate. The precipitate in the press requires washing until the wash waters are free from any soluble sulphates, which can be tested for from time to time with a solution of barium chloride. As the washing proves a somewhat lengthy operation, many experiments have been made with a view to reduce this time, and consequently oxides have been prepared containing varying quantities of soda salts.

The precipitate when taken from the press is emptied into shallow iron trays. It has a greenish-black appearance which very rapidly turns to a brownish-yellow owing to the oxidation of the precipitated oxides of vanadium and iron to the pentoxide and peroxide respectively. The trays are put into an oven and dried. As the precipitate, under the best conditions of filtering and washing, contains 80 per cent. of moisture, the fuel and time spent in drying are very considerable for only a small yield of dry oxide.

The resulting oxide, if thoroughly washed, is a black compact brittle material having about the following composition when dried.

	Per Cent.
Vanadic oxide (V_2O_5)	18·0
Iron peroxide	77·5
Silica	1·4
Moisture loss, &c.	3·2

A similar oxide produced by only three hours' washing, on drying analysed as follows :—

	Per Cent.
Vanadic oxide (V_2O_5)	17·7
Iron peroxide	57·5
Silica	1·3
Moisture	6·1
Soda salts (soluble in water)	17·2

This oxide had a reddish-brown appearance, and in drying crumbled into a fine powder, which appeared to absorb moisture from the atmosphere.

Reduction.—The result of reducing the above oxide is to produce an alloy of iron and vanadium.

It may be reduced in two ways :—(1) By means of carbon and aluminium in an electric furnace. (2) By means of finely divided aluminium alone.

(1) *In Electric Furnace.*—The following description and sketch (Fig. 1) will give an idea of the furnace used.

A is a plumbago crucible, standing on an iron plate, B, which is connected to the negative pole. C a brass electrode connected to the positive pole and containing a carbon rod D. This is suspended from pulleys and counterpoised by weight E, and can be thus lowered or raised to make or break the electric arc at will. F, pipe to carry away fumes, &c., this can be connected to a flue G, connected with a stack. H a wooden screen covered with sheet iron to protect it from the heat and containing a glass window J of claret coloured and dark green glass. Behind this the operator can stand to observe the progress of the reduction, and still be protected from the heat, and any molten particles ejected during the reaction.

After a great number of trials with varying proportions of the vanadium and iron oxide, charcoal and aluminium, as well as the addition of other materials, in order to obtain most perfect reduction and a more fluid slag, I adopted the following mixture as giving the best results.

	Parts.
Dried oxide	8
Powdered wood charcoal	2
Aluminium (in small pieces)	1

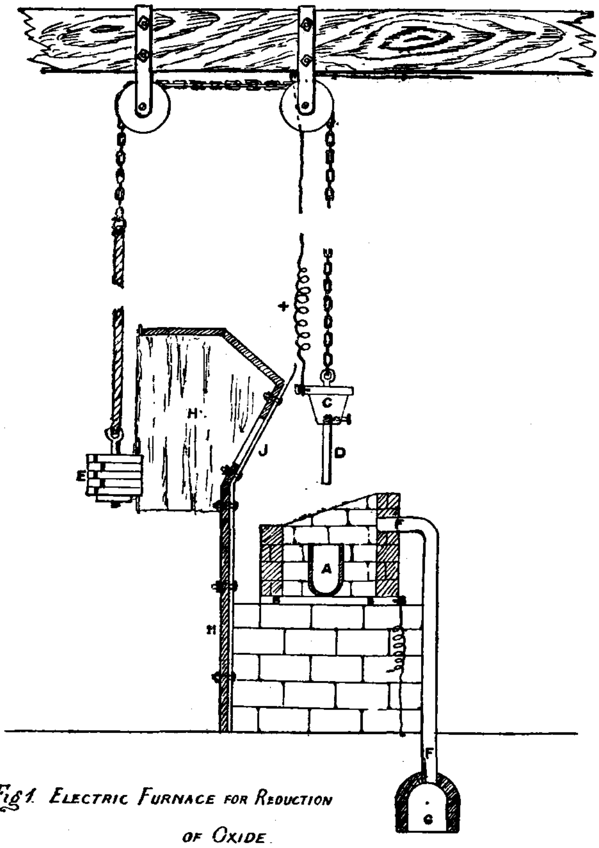


Fig 1. Electric Furnace for Reduction of Oxide.

The oxide and charcoal are intimately mixed together, and after striking an arc in the crucible are slowly added in small quantities at a time, care being taken not to break the arc, otherwise difficulty will be found in establishing it again. The mixture under the heat of the arc decreases much in bulk and gradually fuses, and when in a fused state the aluminium is added in pieces one at a time. After each addition, a violent reaction occurs, evolving much light and heat, and occasionally a little of the molten material is ejected from the crucible. This is especially noticeable if there is a large proportion of soda salts present in the oxide. After all the aluminium has been added, and the reaction appears to have finished, the arc is broken and the crucible allowed to cool. A crucible can seldom be used more than once, as it is much burnt away, and if the arc has not been kept quite in the centre of the pot, it is probable that a hole will have been made in the crucible somewhere.

On breaking the crucible, a button of metal is found in the bottom, weighing about one-third as much as the oxide used. It is fairly brittle, has a fine white granular fracture (very like that of ferro-chrome) and has approximately the following composition:—

	Per Cent.
Vanadium	16·0
Iron	70·0
Silicon	2·0
Aluminium carbon, &c.	12·0

On the top of the button is a thin layer of grey metal which in a short time crumbles to a grey powder. It contains a considerable quantity of aluminium, but I have not had an opportunity for further examination. The slag is greenish-grey to black in colour and crystallises in black plates. It is extremely hard, and some varieties of it scratch glass easily. I have not fully examined any of the slag, but it contains a certain amount of vanadium and iron as well as a large quantity of alumina. When the oxide to be reduced contains soda salts, *i.e.*, chiefly sulphate of soda, this is evidently reduced to a sulphide and an odour of sulphuretted hydrogen is perceived from the slag on standing in a damp place.

(2) *By means of finely divided Aluminium.*—Goldschmidt pointed out in a paper, read some time ago, that by the great heat evolved in burning aluminium to the oxide, the reduction of some of the most refractory oxides containing metals of high melting points might be effected, especial attention being drawn to the reduction of such oxides as Cr_2O_3, WO_3, &c. Vautin patented a process for the reduction of lead and preparation of aluminium sulphide from pure galena by burning a mixture of galena and aluminium. In a paper read at the Iron and Steel Institute's meeting last year, the heat produced by burning aluminium and oxide of iron was used as a means of heating pieces of metal for the purpose of welding.

This reaction can be used in the reduction of the mixture of iron and vanadium oxides to the metals; the intense heat developed being also sufficient to melt the alloy produced and allow it to run and form a button under the slag.

The method adopted was as follows:—

A large fireclay (or plumbago) crucible was lined with calcined magnesia, which was moistened and pressed in tightly with wooden rammers, the crucible being allowed to dry thoroughly before using; the thickness of the lining being from half to three-quarters of an inch.

The crucible A (see Fig. 2) should stand on a layer of charcoal, some firebricks be built up around it, and the space between crucible and bricks filled in with small pieces of coke. This is in order to keep any cold draughts as much as possible away, otherwise the crucible may crack and part of the material would be lost. At one side or above the crucible, a funnel-shaped hopper B, made of sheet iron, is placed, in which is stored the reduction mixture of oxide and finely granulated aluminium. It is closed by a ball C, attached to a chain D, and can be raised to allow more mixture to run into the crucible. Dense fumes are given off during the operation and it is necessary to have some means of *rapidly* removing them, for which purpose a large sheet iron hood E, connected to a flue F attached to a chimney stack, is provided.

An oxide of the following composition was obtained from the earlier processes—

	Per Cent.
Vanadic oxide	16·6
Iron oxide	58·2
Silica	1·0
Moisture	5·6
Soda salts	18·2

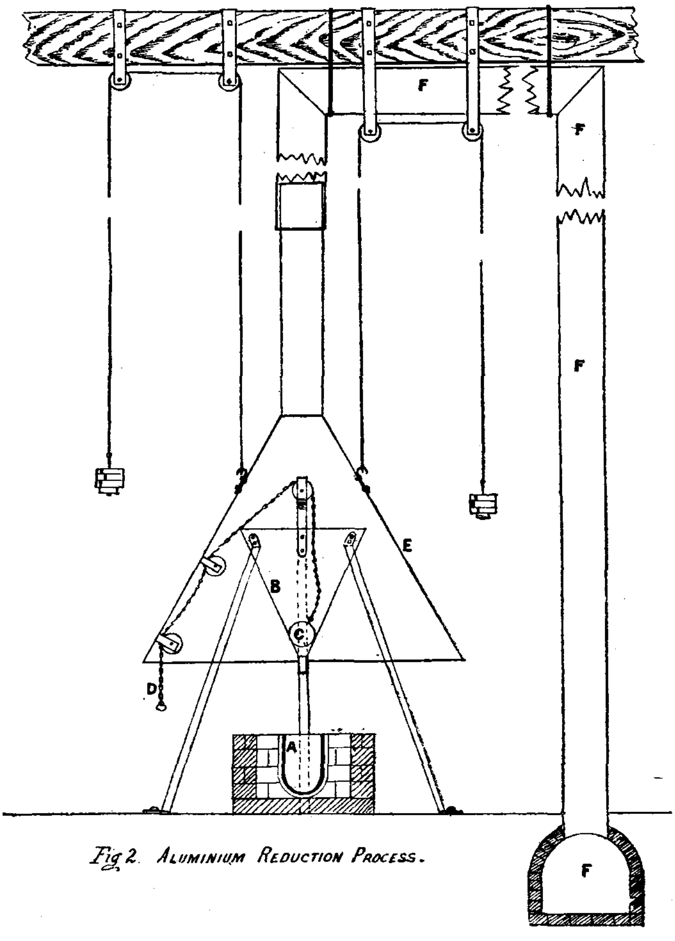


Fig 2. ALUMINIUM REDUCTION PROCESS.

It was then mixed with granulated aluminium in the following proportion:—oxide, 15 lb.; granulated aluminium, 4½ lb. The prepared crucible was placed in position and half filled with this mixture, the remainder being put into the hopper. The ignition of the material is one of the most difficult parts of the operation, as a very high *initial* heat is required to start it. For this purpose about 10 grms. of a deflagrating mixture of sodium peroxide and metallic aluminium or magnesium is required of about the following proportions:—sodium peroxide, 23 parts; aluminium dust, 5 parts. This mixture must be made just before use owing to the deliquescent nature of the sodium salt.

The "ignition mixture" is placed in a cone-shaped heap on the top of the "reduction mixture" in the crucible and ignited by means of a red hot iron bar. This ignites the whole mass and flames and dense fumes are evolved from the crucible. As soon as the reaction subsides a little, the feeding in of the reduction mixture is commenced from the hopper by means of the ball and chain, and arranged so as to run into the crucible in a small but continuous stream. The whole crucible presently becomes white hot and a pair of blue spectacles is required to conduct the operation. The contents are now in a violent state of ebullition, and occasionally small quantities of the molten material are ejected from the crucible. When the hopper is empty the crucible is allowed to cool, and on inverting, the fused mass of metal and slag can be turned out. The crucible may, after relining with magnesia, be used for future operations. The *time taken in this operation is five minutes*, and this may be taken as the average time for a similar quantity. It is therefore a great saving of time when compared with the electric furnace reduction.

On breaking the fused mass a brittle metallic button is obtained from the bottom. It is very easily broken, and in appearance crystalline, imilar to antimony, but has a slightly yellower lustre, nd is of the following composition:—

	Per Cent.
Vanadium	14·9
Iron	58·1
Aluminium	26·0
Silicon	1·3

The slag, which comprises more than two-thirds of the mass, is black in colour, of a glassy nature and filled with holes. It is exceedingly hard and scratches glass.

Uses.—The alloy of vanadium and iron is used in steel-making in the same manner as ferro-chrome, ferro-nickel, &c., and quite as much care is required as in adding chromium to steel, owing to the ease with which vanadium is oxidised.

The following experiments with vanadium steel were made by Professor Arnold, of Sheffield.

A sample of tool steel of the following composition was made:—

	Per Cent.
Carbon	1·20
Silicon	0·26
Sulphur	0·02
Phosphorus	0·02
Manganese	0·07
Vanadium	0·56

Also another, similar to the above, but containing *no vanadium* and *only* 0·07 *per cent. silicon.*

The following results were obtained on mechanically testing the steels:—

——		With Vanadium.	Without Vanadium.
Tensile strength	tons per sq. in.	72·42	62·50
Elastic limit	„ „	50·75	35·70
Elongation	per cent. in 2 in.	6·25	8·00
Reduction of area	per cent.	5·90	7·80

Here, although the tensile strength and elastic limit of the steel was much increased, the ductility was *apparently* a little decreased. Also, on testing a sample of the hardened vanadium steel against a tungsten steel, containing 1·2 per cent. carbon and 3·0 per cent. tungsten on hard chilled white iron, the vanadium steel when tempered to a deep straw was equal to the quenched and untempered tungsten steel.

Other experiments have shown that malleable iron was affected as below:—

——	Tensile Strength.	Elongation.
	Tons.	Per Cent.
Malleable iron	24·5	19
Ditto + 0·50 per cent. vanadium *forged bar*	39·0	12
Ditto + 0·50 per cent. vanadium *annealed*	33·7	32

Mild steel containing vanadium gave the following results:—

——	Tensile Strength.	Elongation.
	Tons.	Per Cent.
Original steel	30	17
Ditto + 1·0 per cent. vanadium	61	14
Ditto + 1·0 per cent. vanadium *annealed*	45	20

Vanadium thus considerably increases the tensile strength in steel, and, when annealed, gives an increased ductility; the experiments thus proving that vanadium steel is exceedingly hard when hardened, and very soft when annealed. It should prove of great service in the manufacture of armour-plates, ordnance, armour-piercing shells, tools, &c.

Estimation of Vanadium.—This paper would not, I think, be complete without giving an easy, quick, and reliable method of estimating vanadium in ores or alloys.

In Ores containing Lead, Copper, Iron, &c.—Sufficient of the ore is dissolved, so that about 0·05 grm. of metallic vanadium is present, in a small quantity of concentrated hydrochloric acid. It is diluted to about 250 c.c. with cold water and sulphuretted hydrogen passed through until no further precipitate comes down. It is then allowed to settle, filtered, and the precipitate redissolved in a small quantity of nitric acid and evaporated until most of the acid is expelled. It is then diluted and sulphuretted hydrogen again passed until precipitation is complete. The precipitate is then filtered and washed with water. The two filtrates are combined and evaporated to dryness in a large platinum crucible. This contains all the vanadium and iron, the precipitate consisting of copper, lead, &c.

If lead only is present, it may be separated by evaporating the hydrochloric acid solution of the ore with sulphuric acid, diluting, and allowing to stand to precipitate and settle the lead sulphate and filtering. The filtrate contains all the vanadium and iron, and is evaporated to dryness, as far as possible, in a platinum crucible, as above.

The dry mass is then fused, with an excess of a fusion mixture of two parts of sodium carbonate (dry) and one part nitre, for 20 or 30 minutes, care being taken to regulate the flame so as to prevent the mass frothing over the sides of the crucible. When cool it is extracted with hot water, filtered through a double paper into a large flask, and precipitate washed with hot water. The filtrate is slightly acidified with dilute sulphuric acid and warmed very gently to expel most of the nitrous fumes. When this is done cold water is added until bulk is about 400 c.c., and about 3 grms. of crystallised sulphite of soda. When dissolved, the contents of the flask are heated slowly to boiling, until no smell of sulphur dioxide is evolved. It is then titrated, while hot, with a N/20 solution of permanganate of potash until a faint pink tinge is *permanent for half a minute.* During the boiling and titration of the solution care must be taken to keep dust or any kind of organic matter out, otherwise the vanadium will be reduced to a lower oxide, and thus give results too high.

As a partial check on a titration, the titrated solution may be again reduced by a fresh quantity of sodium sulphite and boiled until free from SO_2, and permanganate added. This should agree within 0·10 c.c. with the former one.

In Alloys such as Ferro-vanadium Steels, &c.—Sufficient quantity to contain about 0·05 grm. vanadium is dissolved in just sufficient hydrochloric acid, oxidised with potassium chlorate, evaporated in a platinum crucible, fused with fusion mixture and treated as in the former case.

The reactions which occur are as follows:—The vanadium, after fusion with fusion mixture containing nitre, is converted in a soluble vanadate, and thus separated from the iron. This on acidifying with sulphuric acid and treatment with sulphite, is reduced from the pentoxide of vanadium (V_2O_5) to the tetroxide (V_2O_4), and on adding the permanganate of potash is again oxidised to the pentoxide.

$$2KMnO_4 + 5V_2O_4 + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 5V_2O_5 + 3H_2O.$$

Thus, 102·5 *grms. vanadium* require 16 *grms. oxygen.*

∴ 102·5 *grms. vanadium* are equivalent to 112 *grms. iron.*

∴ *Iron value* of *permanganate* × 0·914 = *vanadium value.*

The titration is a beautiful reaction and remarkably sensitive, gradually passing from purple, through blue and green, to yellow, when one drop of permanganate in excess turns it pink.

Aluminium may be used for the reduction, but the vanadium is then reduced to the trioxide. Zinc reduces it to the dioxide, requiring of course different factors for the permanganate.

In bringing this paper to a close, I must tender to Mr. E. L. Rhead, of the Technical School, Manchester, my heartiest thanks for the valuable suggestions he has made and the help he has given me in revising the paper. Also to Mr. E. Coles, of Llanelly, for making the drawings to illustrate it.

Discussion.

Dr. **Grossmann** said that it was of importance that the study of those elements which were considered rare should not be neglected, because what was considered a rare element now might become a comparatively common element by some fortunate discovery of new deposits. Many of the rare elements had important properties, which would be of great value if the sources of their supply were more abundant, and one of the most interesting of the rare elements was certainly vanadium. It was many years since he worked on vanadium himself, but if he remembered right, the test with permanganate which Mr. Procter Smith proposed was not a new one, but had been used for a long time. He regretted the absence of data from which the yield on a given quantity of raw material could be calculated. As far as he could follow the results given, the loss in vanadium seemed to be very large.

Dr. **Gerland** said that the alloy produced by the author's process must necessarily contain much impurity, probably in sufficient quantity to affect the properties of the vanadium-iron. The reduction of the precipitate containing 17 per cent. of sodium sulphate required much aluminium, and the sodium sulphide produced would cause a considerable loss in vanadium. The estimation of vanadium with permanganate had been described by himself (Ber. 1877, 1513 *et seq.*). Vanadium, though widely distributed, occurred usually in very small quantities, whilst large deposits were extremely scarce. Brown and yellow iron ores often contained it; in fact, Sefström discovered it in Swedish iron in 1830. Some sixty years ago a blast furnace near Homburg, in Hesse-Cassel, was fed with pisolitic iron ore (stated to contain 1 per cent. of vanadic acid) and charcoal, producing good iron, in which no vanadium was ever found. About 1 per cent. of it had recently come within his own experience in a specimen of coal from the Cordilleras of Chile; and a number of stray examples were to be found of its cropping up in many other materials, such as clay and bricks.

Sir Henry Roscoe's paper in the Proceedings of the Royal Institution, Feb. 1868, mentioned Alderley Edge as the source of the raw material for his vanadium research. It actually came from Mottram St. Andrews, about four miles from Alderley Edge. A Keuper sandstone of light colour, much lighter than that from Alderley, was to a small extent impregnated with copper. This was also worked by the Alderley Edge Copper Mining Co., and consisted of carbonate and arseniate. For the extraction it was mined, crushed, and digested with hydrochloric acid, and the solution separated and treated with scrap iron to precipitate the copper.

Hard by this greenish-speckled rock was a white soft sandstone with numerous small black specks, containing small quantities of cobalt, nickel, and a little copper. To extract these the crushed ore was moistened on his (the speaker's) advice with the iron liquor from the treatment of the copper ore, and furnaced at a low heat. The resulting sand was then washed with water, which dissolved out the cobalt, nickel, and copper chlorides. On carefully neutralising the solution with chalk, a brown deposit, consisting principally of ferric hydroxide, with a small amount of vanadiate, was produced. This precipitate accumulated for a long time, but was watched all along with much interest.

The late Mr. Down, manager of the Alderley Edge Mining Co., about this time handed him a sample of ore from a new locality, Harmer Hill, near Shrewsbury, showing groups of dark green crystals, which were recognised as vanadiates of lead and copper. These promised to be a more convenient source for the element, and a promising one, as the quantity of the deposit seemed to be large.

The deposit of the new mineral near Shrewsbury was several times visited by him, and he brought back a quantity of the ore for examination, and separated from it, in addition to the vanadium, a group of rare earths, which he considered to be new. Prof. Bunsen, his revered teacher, kindly examined a small sample spectroscopically and also pronounced them new. A short address on them, and on the ore, was delivered by himself at the Liverpool Meeting of the British Association in 1871.

Some time afterwards, Mr. Mellor, of the Magnesium Metal Company at Patricroft, acquired the Shrewsbury deposit, and this, he believed, formed the principal raw material for all the vanadium compounds they made. The mine gave out sooner than was expected, and had been filled up long since. This, then, was the first and only venture for working vanadium ores on something like a manufacturing scale. Mr. Mellor very kindly offered him the refuse for extraction of the new earths, but he had meanwhile accepted an engagement with Messrs. F. Steiner and Co., and had little time to attend to it. Altogether about 4 to 5 grms. were collected; but this quantity proved insufficient for an exhaustive examination, especially as it appeared to be a mixture of at least three elements.

The use of vanadium must be very limited. The largest amount seemed to have been consumed by the calico printers and dyers for aniline blacks, and in this application it had to compete with copper and cerium compounds. He had discovered meta-vanadic acid in 1877 (Ber. **9**, 872), and obtained it in microscopic scales of a beautiful orange-yellow colour, like mosaic gold, with a fine lustre and great permanency, but found no appreciation for it. Vanadium, however, formed a number of compounds with such varying properties that some of them would probably be found of technical value. He had also worked on the sulphates, corresponding to the three oxides, of which the two latter were already known.

In 1871, 32*l.* per lb. was offered to him for ammonium vanadiate, but since the Magnesium Metal Company had taken up the extraction, the price had come down to under 1*l.* per lb. If, however, a demand should arise, new sources were sure to be discovered, and, considering the rapid way thorium material had been procured as soon as there was a demand for Welsbach mantles, they might expect an ample supply of vanadium ore to be forthcoming.

Newcastle Section.

Meeting held on Thursday, November 28th, 1901.

MR. W. L. RENNOLDSON IN THE CHAIR.

THE ACTION OF LIGHT ON COLOURED BRASS LACQUERS.

BY HARRY SMITH, F.I.C.

Polished brass speedily tarnishes when exposed to the air, especially in places where coal gas is burned. The discolouration may be prevented by the application of lacquer to the metal surface, and a great variety of articles are protected in this manner.

Lacquer for brasswork is known as "hot" or "cold" lacquer, according to whether it is applied to the heated metal or not. "Cold" lacquer made from a solution of nitrocellulose is sometimes used, and is spread upon the cold metal with a brush as in ordinary varnishing.

"Hot" lacquer is very largely employed, and in this process the fluid is gently and repeatedly brushed over the heated surface of the metal until a sufficiently thick and durable coating is obtained. The art of "hot lacquering" is not easily acquired, but very beautiful results are obtained by skilful workmen; the temperature to which the brass must be heated, and the time which must elapse between successive coatings are points which require careful attention, otherwise the last applied layer may dissolve and remove some portion of the previous coat, producing a streaky uneven appearance. The experiments which I am about to describe were made with "hot" lacquers, but would also apply to the process of cold lacquering.

Brass lacquer is usually a solution of seedlac in methylated spirit. Other resinous bodies are sometimes used with seedlac, such as mastic and sandarac, but seedlac is the essential ingredient, as it produces an exceedingly hard and tough coating upon the metal. It is necessary to use a good quality of seedlac, which should be fresh, old lac is apt to produce a brittle film and will not adhere to the brasswork with the proper tenacity. When the lacquer is applied to the heated brass, alcohol is first driven off and then the seedlac fuses, producing a hard lustrous film upon the surface of the metal.

Brass lacquer is sometimes made almost free from colour to preserve the original appearance of polished brass, but it is generally coloured so as to produce a golden or coppery tint, in fact, the colour may vary from the palest lemon yellow to a deep claret. The colouring matters which are usually employed for this purpose are of vegetable origin, such as saffron, turmeric, dragon's blood, sandal or sanderswood, and gamboge, these colouring matters are preferred to those derived from coal tar as they are supposed to be of a more permanent nature. In order to determine to what extent this belief is founded on fact, I have lacquered a number of brass tubes, in some cases the lacquer was tinted with vegetable colouring matters and in others with aniline colours. The lower half of each tube was then wrapped in several layers of black paper to protect that portion from the action of light, and the tubes were placed under a skylight with an east aspect, thus they received the diffused light from the sky, and the direct rays of the sun until a little after noon each day. The brass tubes were exposed in this way from November 3, 1900, until the last day of September 1901, and as the summer of 1901 has been unusually fine and sunny, these severe conditions have produced some very striking effects on the colouring matters which were employed.

The lacquers were made by first dissolving the colouring matter in the methylated spirit and then adding the seedlac, mastic, and sandarac, the total weight of the three latter amounted to 8 per cent. of the whole. After gentle shaking for 24 hours the liquid was decanted from the insoluble residue (which is very large in the case of some of the vegetable colouring matters), and the lacquer was then allowed to settle until perfectly clear and fit for use. In the following tables only the percentage of colouring matter is stated, for the sake of clearness, it must be understood that this includes the woody fibre and other inert substances in the dyestuff.

Experimen	Weight per Cent.	Colouring Matter.	Appearance.	
			Before Exposure.	After Exposure.
32	1·02	Turmeric	Pale lemon yellow.	Colour entirely gone, considerably blackened.
2	1·65	"	"	"
3	3·30	"	Deeper lemon yellow.	"
31	8·18	"	Golden yellow	"
30	16·36	"	Deep gold....	"
26	2·45 0·82	Turmeric ... Sandalwood.	Red gold.....	Colour entirely gone, slightly blackening.
34	6·13 1·02	Turmeric ... Sandalwood.	Copper.......	"
33	4·09 2·04	Turmeric ... Sandalwood.	Copper.......	"
29	2·45 0·6	Turmeric ... Saffron	Pale lemon yellow.	Colour entirely gone, considerably blackened.
41	6·13 1·02	Turmeric ... Dragon's blood	Red gold.....	Greatly faded, and slightly blackened.
40	2·04 1·02	Turmeric ... Dragon's blood	Pale Copper..	"
5	0·4	Saffron	Palest golden yellow.	Colour entirely gone, considerably blackened.
13	0·5	Sandalwood..	Palest copper.	Colour gone, no blackening.
14	1·3	Sandalwood..	Pale copper ..	Colour gone, no blackening.
36	4·09	" ..	Deep copper..	"
35	8·18	" ..	Deep ruby....	A little paler.
24	0·32 0·6	Sandalwood. Metanil yellow	Red gold	A little paler.
37	2·04	Dragon's blood	Deep red gold	Much paler.
38	4·09	"	Deep copper .	"
39	8·18	"	Deepest copper	"
25	0·6 0·34	Dragon's blood ..... Metanil yellow	Red gold.....	A little paler.
19	0·17	Metanil yellow	Greenish-gold	Faded.
20	0·7	"	Gold.........	"
8	0·4	Annatto	Palest gold ..	Brownish black.
9	1·25	"	Pale gold	Black, greenish metallic lustre.
11	0·4	Gamboge	Pale gold	Scarcely perceptible fading, no darkening.
12	1·25	"	"	"
15	0·17	Auramine	Greenish-gold	Faded.
16	0·34	"	Gold.........	"
21	0·17	Primrose.....	Pale greenish-gold.	Faded.
22	0·68	"	Greenish-gold	"

If we summarise these results, we find :—

1. That the vegetable colouring matters, Turmeric, Sandalwood, Saffron, Dragon's Blood, and Annatto, all fade completely when exposed to sunlight.
2. That Turmeric, Saffron, and Annatto also turn the lacquered brass black when it is exposed to sunlight.
3. That the aniline colours, Metanil Yellow, Auramine, and Primrose, are more suitable for colouring yellow lacquers than Turmeric, Saffron, and Annatto, because they do not turn the brass black.
4. That Dragon's Blood is more suitable for copper-coloured lacquer than Sandalwood, because it does not fade so quickly or so completely.
5. That Gamboge is more suitable for the manufacture of yellow and golden lacquers than Metanil Yellow, Auramine, Primrose, Turmeric, Saffron, and Annatto, because it scarcely fades at all, and does not blacken the brass.

Mr. W. M. Jones read a "Note on a peculiar Blackening of a Chrome-Yellow Pigment."

New York Section.

Meeting held on Friday, November 22nd, 1901.

DR. V. COBLENTZ IN THE CHAIR.

THE MANUFACTURE OF NITRIC ACID.
PART II.

BY C. W. VOLNEY, PH.D.

(See this Journal, June 1901, 544—546.)

To ascertain the influence which a vacuum of 600 mm. has on the mixture of concentrated sulphuric acid and sodium nitrate, I repeated the experiment recently described* under vacuum indicated by 610 mm. to 650 mm., or under absolute pressures of 150 mm. to 110 mm. Table 1 gives the observations which were made during the

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* This Journal, June 1901, 544—546.

reaction in the retort and distillation of the acid. Reviewing the temperatures, we find that the decomposition of the nitrate under 110 mm. pressure occurs also in two phases; the first phase represents the reaction of free sulphuric acid on the nitrate, which takes place at about 74° of the salt mixture; the second phase shows the reaction of the sodium polysulphate on nitrate, occurring at about 85°. We see that during the first phase the temperature of the oil-bath shows below 100°, and during the second phase up to 145°.

TABLE 1.

Time.	Temp. of Oil Bath.	Temp. of Salt Mixture.	Temp. of Distilling Acid.	Indicated Vacuum.	Pressure.	Remarks.
AM.	° C.	° C.	° C.	mm.	mm.	
10.0	13·5	18	11	610	150	No reaction.
11.0	21	21	14	610	150	”
11.30	13·5	20	12	630	140	”
PM.						
12.7	16	20	15	630	130	”
1.0	18	17	12	630	130	Heat applied.
1.7	32	18	20	650	110	Reaction commences; gas develops.
1.19	66	35	22	650	110	Distilling acid shows.
	72	54	22	650	110	” ”
	76	55	22	650	110	Foaming in retort.
	83	68	23	650	110	Boiling, &c.
	95	73	24	650	110	Distilling.
1.28	102	74	24	650	110	Much foaming; fire lessened.
1.30	102	74	24	650	110	Distillation free and regular.
1.34	101	74	24	650	110	Salts molten and boiling.
.40	99	74	24	650	110	Heat increased.
	100	78	28	650	110	Distillation lessens; heat increased.
1.50	122	80	28	650	110	Slight show of yellow vapours, which ceases.
1.55	128	83	28	650	110	Foaming again.
2.0	140	86	28	650	110	” ”
2.5	140	86	28	650	110	Salt mass thickens; distillation lessens.
2.10	145	85	27	650	110	” ”
2.15	140	96	27	650	110	Salt mixture quietly flowing
2.20	130	100	24	650	110	Distillation lessens.
2.25	128	101	23	650	110	” ceases.
2.30	128	101	23	650	110	” ”

It is remarkable that the distilling vapours of the monohydrate during both phases show a boiling point lying below 30°. And it may be stated, as a general result of these observations, that under an indicated vacuum of 650 mm. or 110 mm. pressure the decomposition of the sodium nitrate by sulphuric acid is performed by an outside application of heat below 140°, the distilling acid going over below 30°. It follows that under such conditions a monohydrate of almost absolute strength and purity could be produced. The difficulties which necessarily would attend the practical working of such a process cannot be under-estimated. The evils which attend the use of concentrated sulphuric acid culminate in the foaming, which frequently gets beyond all control, so that a part of the contents in the retorts are run into the coolers or receivers. As already stated, the inventors of the vacuum process have had recourse to the use of sulphuric acid of 60° Bé., instead of the concentrated acid; and as the resulting nitric acid shows a strength of only about 40° Bé., a redistillation from concentrated sulphuric acid is necessary, if an acid of higher specific gravity is wanted.

But with the use of sulphuric acid of 60° B. the process in the retort is not the same as when concentrated acid is used; and as there appears to be no clear reference to this fact in the literature of the subject, the following references to experimental work may serve to explain it. The change in the process is evidently due to the introduction of water into the reaction; the acid of 60° B. containing about 20 per cent. of water. Reference has been made to the action of the concentrated sulphuric acid on sodium nitrate in two phases.* In the first phase the free sulphuric acid acts on the nitrate, forming a trisulphate, $NaH_3, 2SO_4$; in the second phase this trisulphate decomposes the remaining nitrate with formation of "bisulphate"—

$$NaH_3, 2SO_4 + NaNO_3 = HNO_3 + 2NaH, SO_4.$$

As has been shown, this reaction requires the higher temperature, under 760 mm. pressure and *in vacuo;* and, as the anhydrous trisulphate forms at that higher temperature an oily liquid, the excessive foaming, repeatedly observed, is entirely owing to the state of this salt. But *this trisulphate, on addition of water, dissolves at once into* disulphate and hydrated sulphuric acid—

$$NaH_3, 2SO_4 + H_2O = NaH, SO_4 + H_2SO_4, H_2O.$$

By the use of an acid of 60° B., or in consequence of the water in that acid, the decomposition of the nitrate in the retort is performed at once with formation of the "disulphate" at a lower temperature, and with less foaming than attends the reaction of trisulphate.

The process referred to needs experimental verification; and for this reason, as well as for comparison between the distillation of the mixture of sodium nitrate and sulphuric acid of 60° B., I have treated equivalent quantities of the same, 85 parts sodium nitrate, and 120 parts sulphuric acid of 60° Bé. in the previously described apparatus and manner. Table 2 shows what was absorbed during the distillation under atmospheric pressure, together with the temperatures of oil-bath, or the heat applied to the retort, the temperature of the contents of the retort and that of the distilling acid.

Table 3 shows, in addition to the same observation, the indicated vacuum and corresponding pressures under which the distillation takes place.

TABLE 2.

Distillation of Sodium Nitrate and Sulphuric Acid, 60° *B., under Atmospheric Pressure.*

Time.	Temp. of Oil Bath.	Temp. of Salt Mixture.	Temp. of Distilling Acid.	Remarks. 755 mm. pressure.
PM.	° C.	° C.	° C.	
1.55	17	16	17	No reaction.
3.0	20	16	17	Heating commences.
	45	23	..	No reaction.
	70	40	25	”
	105	63	60	Gas develops; reaction commences.
	115	63	60	Gas develops steadily,
3.18	126	81	..	” ” rapidly.
	..	85	68	Distillation; no foaming.
	130	89	70	” ”
	136	92	72	” ”
	141	95	74	Contents of retort boil; distillation.
	..	..	75	Violent boiling.
	..	..	76	”
	146	113	103	Distillation good.
3.30	..	118	111	
	150	120	118	Distillation rapid; contents of retort boil quietly.
3.35	150	120	118	” ”
3.40	154	121	118	Distillation steady.
3.46	154	121	119	”
4.0	154	123	118	”
4.10	154	123	118	”
4.15	160	125	103	Distillation lessens.
4.30	150	125	98	”
4.45	150	126	78	Distillation ceases.

Note.—Throughout the operation the fire was not lessened, but steadily kept up.

Reviewing the results of these distillations, we find that in the operation under atmospheric pressure the greatest portion of the acid distils at about 118°, the contents of the retort showing thereby a temperature of 121°; and that under a pressure of 120 mm. the same acid distils at 74°, and the retort contents showing thereby 78°. In both cases the remaining salt cake consists of sodium bisulphate with water of crystallisation.

The observations show also that the distillation under 755 mm. pressure produces no foaming, and can be smoothly carried out; and that the foaming, which occurs under 120 mm. pressure, does not interrupt the work.

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* Journal of the American Chemical Society, xxiii.

TABLE 3.

Distillation of Sodium Nitrate with Acid of 60° ***B.*** *in vacuo.*

Time.	Temp. of Oil Bath.	Temp. of Salt Mixture.	Temp. of Distilling Acid.	Indicated Vacuum.	Pressure.	Remarks.
NOON.	° C.	° C.	° C.	mm.	mm.	
12.0	13	13	14	..	770	No reaction.
PM.						
1.0	16	14	14	506	165	„
1.35	16	14	14	650	120	Some gas bubbles, probably air, escapes.
1.45	46	17	14	650	120	Heat applied.
	65	22	16	650	120	Reaction commences.
	84	24	16	650	120	Gas develops.
	95	28	..	650	120	More gas develops.
2.0	96	33	..	650	120	„ „
	120	37	23	650	120	„ „
	126	40	..	650	120	„ „
2.7	133	57	24	650	120	Gas develops fast; liquid foams; not rising much.
	..	65	..	650	120	
	147	71	..	650	120	.. ..
	..	77	40	650	120	Foaming, rising.
	153	84	..	650	120	Violent boiling; distillation good.
	156	85	..	650	120	
	163	87	50	650	120	.. ..
2.20	164	87	72	650	120	Distillation good.
2.25	167	88	74	650	120	Foaming and boiling, not rising too high.
2.30	167	79	74	650	120	
2.34	167	77	74	650	120	Foaming less; salt boiling; distilling.
2.45	163	77	74	650	120	
2.50	163	77	74	650	120	Salts separating on bottom of liquid; distilling; boiling, not foaming.
2.55	163	76	74	650	120	
3.0	164	76	74	650	120	
3.15	176	76	74	650	120	Distillation lessens.
	178	78	63	650	120	„ ceases.
4.20	180	79	50	650	120	

The produced acid shows no difference in quality or quantity, and has a sp. gr. of 1·38 = 39·8° Bé. at 21° C.

The decomposition of sodium nitrate *in vacuo* by concentrated sulphuric acid thus takes place at such a low temperature, that a pure monohydrate of nitric acid is produced, and this vacuum is especially serviceable in the second phase of the process (see *ante*). With an acid of 60° Bé. a distillation *in vacuo* offers less advantage over distillation under atmospheric pressure, as the water present in that acid effects the decomposition with formation of bisulphate at sufficiently low temperature to prevent any serious decomposition of nitric acid of the concentration obtainable by the operation.

The redistillation of the weaker nitric acid over concentrated sulphuric acid tends, undoubtedly, to considerable increase in cost, and the promoters of the different methods of manufacture are making claims to advantages which, in either case, will have to be ascertained by the practical manufacturer to be fully appreciated.

The experimental work detailed above will also explain the principles upon which certain other proposed improvements in the manufacture of nitric acid are based.

R. E. Chatfield* proposes to make strong nitric acid by distilling one molecule of sodium nitrate with two of concentrated sulphuric acid, at temperatures between 100° and 130° C. This reaction affords the trisulphate and nitric acid, which can be distilled even below 100°, and a strong pure monohydrate is produced. It is difficult, however, to see how this process can carry a patent.

The chemical works "Rhenania"† add to the hot bisulphate in the retorts appropriate quantities of sulphuric acid of 60° Bé, and then the necessary quantity of sodium nitrate. This is with a view to transform the bisulphate into polysulphates, and then reconvert it into bisulphate by the aid of the sodium nitrate, thus enabling them to distil highly concentrated nitric acid. As they propose to do this without letting the retorts cool down, they claim the advantages of continuing the operation in heated retorts and the use of the dilute sulphuric acid.

As the polysulphate does not form so long as water is present, this process would appear to furnish only bisulphate and free sulphuric acid, as any polysulphate in the retort would be transformed into bisulphate and sulphuric acid on the introduction of acid of 60° Bé. The free acid would then decompose the freshly added nitrate, and nitric acid would be distilled from the mixture, so that any saving would seem to arise from using the heated retors over again.

I have no data on which to form an opinion of this process in practical working. It would seem to require a special method of opening and closing to enable the sulphuric acid and nitrate to be introduced into the hot retorts.

In former times, when smaller retorts were used, and when there was not much need of highly concentrated and pure nitric acid, and when a charge consisted of only 200 or 300 pounds of nitrate, it was customary to continue the distillation until a neutral sulphate remained as nitre cake, the amount of sulphuric acid being calculated for the forming of the neutral salt only. As a result, the nitric acid was very impure, red, and seldom of great strength.

This practice is now, I believe, universally abandoned, as the nitric acid of to-day is the more valuable article. It appears from the following, however, that in some cases the acid sulphate, resulting in the usual, regular manufacture of nitric acid as retort residue, is still used for the production of neutral sulphate and nitric acid.

For this purpose the Netherfields Chemical Works, Glasgow, advise me of the process which they use at their works* for the manufacture of neutral sulphate and strong nitric acid. Mr. William Garroway, the inventor, states that the process has been in operation for the last three years with satisfactory results. The acid sulphate or bisulphate, resulting from the manufacture of sulphuric or nitric acid, is mixed with appropriate quantities of sodium nitrate and heated in a properly constructed retort oven, and when at a proper temperature a spray caused by an air-blast and weak nitric acid is blown over the salt mixture, so that the gases—nitrogen oxides—are thereby mixed with water-gas and atmospheric air. By conducting these gases through a series of condensing tubes and regenerating towers, the description of the process states that ultimately the strong 96 per cent. nitric acid can be run off for use.

The neutral sulphate remaining from this operation furnishes nitre cake, which Mr. Garroway's analysis shows to have 98·36 per cent. of sulphate and, practically, no impurities.

TINFOIL AND BOTTLE CAPS MANUFACTURE.

BY RAFAEL GRANJA.

Tinfoil.—The size or fineness of tinfoil is expressed by the number of square inches covered by one pound of foil. The three principal varieties of tinfoil are pure tinfoil, composition foil, and German foil.

The pure tinfoil contains tin exclusively, while composition foil contains a core of lead in the middle covered on both sides by a thin coat of tin. To recognise those two foils, a drop of nitric acid is put upon the sample examined. If pure tin, metastannic acid is formed instantaneously, and a white spot appears; if composition, the coat of tin is also attacked, but the layer of metastannic acid is so thin that the lead will show through it, thus presenting a black spot.

For the making of pure tinfoil the tin is first melted in cast-iron pots, and cast into slabs of about 19 by 13 and 1 in. thick. Those slabs are cooled and then passed through the breaker. The breaker is a large rolling mill of the ordinary pattern. By successive passages and reducing each time the space between the rolls, the slab of tin is transformed into a sheet having little more than the original width, a thickness of about $\frac{1}{16}$ in. and a length of about 25 ft. This sheet tin is wound tightly upon a spool which is then brought to the roller mill.

C is the spool supported by bracket D. S is a tank containing soapsuds which flow on the sheet of tin and prevent

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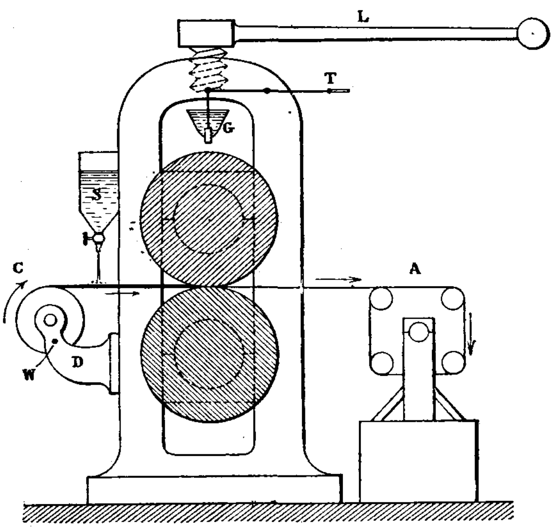
* R. E. Chatfield, Eng. Pat. 16,512, 1891.
† Ger. Pat. 166,962, March 8, 1898.

* Private communication from Mr. William Garroway, Glasgow, August 6, 1901.

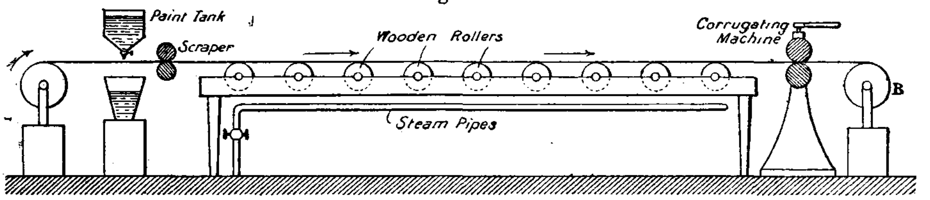
Fig. 1.



its sticking to the rolls. A is the reel on which the foil is wound as it comes out of the rolls. The pressure between the rolls is regulated by a lever L, which the workman can handle with one arm while he winds the foil with the other.

While the pressure between the rolls is one of the elements which determine the thickness of the foil, no very thin foil could be produced unless another element were added, which is a high tension of the sheet between the spool and the rolls. In order to obtain it a screw W presses on the brass rim of the spool, and while the rolls draw the tin the resistance created by the screw holds it back. A reluctance so to speak, of the tin to go through the rolls is thus created, and the thinness of the foil is increased. A third element also comes into play, which is the heat developed by the high friction of the bearings. Under its influence, the parts of the rolls which are nearer to the bearings become hotter than the middle parts and consequently expand more. If the rolls were perfectly flat, they would tend to become concave, the pressure between them would be less in the middle than on the ends, and a foil wavy and undulated on the edges would be produced. In order to avoid this defect, the rolls are ground slightly convex, so that when they have taken their normal expansion they will be perfectly flat, and the pressure even, all along the line of contact.

Fig. 2.



In order to control still better such evenness of pressure a gutter, G, filled with cold soapsuds is suspended above the rolls. The bottom of this gutter is perforated with small valves which are manœuvred by touches, T, each valve having its touch so that the whole resembles a piano keyboard. If a line of waviness is noticed at any time in the foil coming out of the rolls, the workman plays on one or several touches above that line, the valves are opened, cold water trickles over that part of the rolls which was too hot, cools it, and smoothness is restored.

For the making of composition foil, an ingot of lead is first broken to a thickness of about $\frac{1}{2}$ in., and a length of 3 ft. It is then sandwiched between two sheets of tin about $\frac{1}{8}$ in. thick, when the whole is passed through the rolls of the breaker at high pressure. The three sheets are thereby welded into one, the lead being in the middle and the tin on both faces. This composite sheet is then passed through the breaker, wound on spools, and rolled as with pure tin foil.

Foils of pure tin can be rolled up to 10,000 sq. in. per lb. and composition to 7,000, but with German foil as high as 14,000 can be reached. This German foil is made of tin alloyed with a few per cent. of foreign metals. The composition of the metal is supposed to be a secret. This foil is readily recognized by its special thinness, which equals that of tissue paper, and by its appearance, being bright on one side and dull on the other. The ingots of metal for German foil are first broken, and the resulting sheet tightly wound upon the spools. Then 2 spools are set together behind the rolling mill, and the 2 sheets are passed at once through the rolls. Two sheets of pure tin would weld under those circumstances, but the composition of the metal is such that it will not weld. To further prevent welding, soapsuds are injected between the 2 sheets of metal as they enter the rolls. As 2 sheets are fed to the mill, two foils come out of it, and as 7,000 can be obtained for a single foil, 14,000 can be reached with 2. The faces of the foils which were in contact with the rolls are bright, while the internal faces are dull.

Those different qualities of tin foil as they come from the rolling mill need only to be folded, piled up, pressed, and cut to the desired size, to be made into a saleable article. However, further work may be demanded, for instance, painting, corrugating, and printing. For the painting, the foil is wound on a drum and passed on a long table of wooden rollers, provided with steam pipes, which promote quick drying of the painted foil. Varnish of the desired shade dissolved in fusel oil is caused to flow on the foil at the head of the table, and is spread evenly by a rubber scraper. The foil then progresses slowly to the other end of the table, which it reaches in a dry condition, the fusel oil having all evaporated, and it is wound on a second drum. If corrugating is desired, the corrugating machine is interposed between the drum B and the drying table.

The corrugating machine is composed essentially of 2 rollers, 1 made of steel, and engraved, and the other made of compressed paper. Smooth foil passed between those 2 rolls will come out corrugated. Finally the foil may be printed, which is done by passing in the last resort through a printing press as if it were paper.

Bottle Caps.—For the manufacture of bottle caps, a composite sheet is first rolled having a core of lead, and on each face a coat of tin hardened by 1 per cent. antimony From 3 to 6 of these sheets are passed through a first punching and stamping machine. The bunches of caps thus produced are then passed in a series of stamping machines, the shape of dies and matrices being such that the sides of the caps are drawn more and more, while the heads of the caps remain nearly unchanged.

Under the pressure and friction the caps would weld into one, but the 1 per cent. antimony is sufficient for preventing such occurrence. The bunches of caps having thus acquired their shape, they are taken in hand by girls who pick the caps apart, a tedious and delicate work. The caps are then polished on a small lathe with an ivory tool, dressed and cut, painted, and stamped if desired.

Nottingham Section.

Meeting held at Derby on Tuesday, November 26th, 1901.

MR. L. ARCHBUTT IN THE CHAIR.

ROSIN GREASE.

BY L. ARCHBUTT, F.I.C.

Rosin grease is made by stirring together rosin oil and slaked lime. One method of preparation consists in stirring the rosin oil with about three-fourths of its weight of slaked lime made into a cream with water. The mixture rapidly solidifies, expelling the superfluous water, which is run off, and the grease is then diluted by gradually stirring in a further quantity of oil, until the desired consistency is attained. The oil used for diluting may be rosin oil, petroleum oil, or neutral coal tar oil ("grease oil"). Grease is also made by mixing dry slaked lime with rosin oil, without water, also by mixing the lime with mineral or coal tar oil, and adding the rosin oil afterwards. Cheap greases are thus prepared, which are used to lubricate the iron axles of colliery trucks and ordinary road vehicles.

The formation of rosin grease has been attributed to a supposed property possessed by the unsaturated hydrocarbons contained in rosin oil of combining directly with lime and other bases (see Mills' "Destructive Distillation," 4th Edit., 1892, p. 97). This, however, is not the true explanation. Crude rosin oil invariably contains a considerable percentage of resin acids (undecomposed colophony), which have distilled over with the hydrocarbons, and it is the combination of these acids with the lime, forming a soap when the rosin oil and the lime are stirred together, which is the real cause of the formation of rosin grease. Refined rosin oil, which has been freed from resin acids, is incapable of forming a grease with lime; on the other hand, the larger the percentage of resin acids contained in the crude rosin oil, the stiffer the grease which can be formed. This was proved by the following experiments.

The percentage of resin acids in three samples of commercial rosin oil was determined by dissolving a weighed quantity of each oil in ether, shaking out with soda, and weighing the resin acids recovered from the soda solution. The following results were obtained :—

Description of Rosin Oil.	Resin Acids.
	Per Cent.
"Pale refined"................................	0·14
"Medium"................................	5·80
"Hard-run"................................	21·30

The acids extracted from each sample were quite hard and brittle.

20 grms. of each of the above oils were stirred with about 45 c.c. of a cream of lime, containing about 15 grms. of slaked lime. The mixture made with the 'pale refined" oil containing only traces of resin acids, did not perceptibly thicken on stirring, and formed no solid grease on standing, emulsified when stirred, but separated into its constituents when left undisturbed. Both the other mixtures set. The "hard-run" oil gave a much harder and more solid grease than the "medium" oil; the former grease when pressed between folds of blotting paper, gave a fairly hard cake, that from the latter squeezed out and spread over the paper in a layer of buttery consistency. Very much less lime than the above suffices to solidify the oil. Thus 10 grms. of the "hard-run" oil referred to above were mixed with 0·75 grm. of lime cream, containing only 0·25 grm. CaO, or not much more than enough to neutralise the resin acids present. This mixture was stirred for five minutes and then left. The lime readily mixed with the oil, forming a thick creamy emulsion, which did not separate on standing. The emulsion gradually thickened, and in three days was a stiff paste, which gradually got stiffer. 10 grms. of the same oil were next stirred with 3·0 grms. of lime cream, containing 1·0 grm. CaO, or about 4½ times the amount necessary to neutralise the resin acids. This mixture set hard in about five minutes, and could then be lifted, bottle and all, by means of the stirring rod. Another mixture containing 0·5 grm. CaO, or rather more than twice the quantity necessary to neutralise the acids, set in about 20 minutes.

Some of the same "hard-run" oil was purified in the laboratory by dissolving in ether, extracting the acids by shaking with soda, and then recovering the neutral oil by evaporating off the ether. With the oil thus purified, all attempts to form a grease with lime failed. The neutral rosin oil, freed from the resin acids, showed no tendency to emulsify with the cream of lime, and on standing they soon commenced to separate. These mixtures have been kept in stoppered bottles for eleven months, and have occasionally been stirred and vigorously shaken together, but, as you see, the contents of the bottles are quite fluid, and show no tendency even to form permanent emulsions.

The formation of a grease with lime is, therefore, not due to a property possessed by the hydrobarbons of rosin oil. It is due to the accidental presence of free resin acids in crude rosin oil.

Discussion.

Dr. J. Lewkowitsch wrote that, naturally, neutral rosin oil, free from acids, could not combine with lime. As the author had pointed out, the combination existed merely between the rosin acids and the lime, and the soap so formed possessed—in consequence of its physical condition —in a high degree the power of occluding, or if one liked to call it so, of emulsifying and consequently holding firmly combined, a large amount of neutral rosin oil. The soda soap possessed the same property to a high degree, as was well known to refiners of rosin oil; but since the soda soap was soluble in water, the emulsion could be readily broken up, and hence refining by means of caustic soda was a feasible technical process. It was well known that for the making of rosin grease unrefined oils were used as they come from the still, and as he could state from practical experience, such oils contained any proportion of rosin acids up to even 50 per cent.

Mr. R. M. Caven thought that Mr. Archbutt had made it quite clear why distilled rosin oil formed a grease with alkalis. He was astonished that the statement could be made that turpentine formed a compound with calcium hydroxide. There was evidently nothing in the nature of turpenes or other hydrocarbons which permitted them to unite with bases.

Mr. F. J. R. Carulla, in allusion to the use of resin spirit for varnish making, and the variations in the solidification period mentioned in the paper, asked whether there was any remedy for non-drying varnishes. He recently had occasion to use a varnish that had been bottled for 25 years and kept well stoppered. It was originally a quick-drying varnish, but now dried very slowly. Was this due to some molecular change, and, if so, could its original nature be restored?

Mr. Archbutt, in reply to Mr. Carulla, said that the drying of varnishes was an obscure subject which had very little connection with his paper. A good deal depended upon the condition of the surface to which the varnish was applied, as well as upon atmospheric conditions. Some varnishes lost their drying power with age.

Journal and Patent* Literature.

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* 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.

Engine; The Waste-Heat Auxiliary ——. F. H. Mason. Consular Repts., Aug. 13, 1901, 358—365.

It is a well known fact that a large proportion of the energy delivered to any type of steam engine by its boiler is lost in the exhaust; in any case, there is a dead loss of 80° F. of temperature fall, *viz.*, the difference between the temperature of the condenser (140° F.) and that of the circulating water (60° F.). The waste-heat auxiliary engine devised by Prof. E. Josse, Royal Technical High School, Charlottenburg, Berlin, utilises this heat for evaporating a liquid which boils at a much lower temperature than water. On account of its viscosity, its lubricating qualities, and ease of pressure control at the temperatures in question, liquid sulphur dioxide has been adopted; at 140° F. and 60° F. its vapour pressures are 156 and 41 lb. per square inch respectively. The SO_2 is evaporated by the exhaust steam in an "atomiser," and, after doing work in the cylinder of an engine adjacent to the main steam engine, escapes into a condenser, from which it is pumped back into the "atomiser." The whole apparatus must be air- and water-tight, and be capable of resisting a pressure of at least 150 lb. per square inch.

Tests on a waste-heat engine utilising the energy of the exhaust of a 150-H.P. Görlitz triple-expansion steam engine, delivered an additional energy of 34·2 per cent. of that of the main engine, *i.e.*, reduced the steam consumption per indicated horse-power hour from 11·2 to 8·36 lb.

A similar engine of 175 H.P. has been put in operation at the Berlin Electrical Works, Markgrafen Strasse, which adds 41·7 per cent. of the working energy of the compound steam engine from the exhaust of which it derives its energy. The nett cost of the engine was 49,700 marks, a gas plant of equal power costing 47,625 marks—a difference so slight as to be negligible in the face of the great economy.

— J. W. H.

PATENTS.

Gases from their Mixtures; Method of and Apparatus for the Separation of ——. R. P. Pictet, Geneva, Switzerland. Eng. Pat. 19,254, Oct. 27, 1900.

The patentee describes minutely the application of his invention in separating the constituent gases of air, but states that it may be used for separating other mixtures, or for separating solid, liquid, or compressed carbonic acid from air to which chimney gases, &c., may be added. In the separation of air, the following processes and arrangements are adopted, *viz.*:—(1.) The air is filtered, compressed, and freed from water. (2) The dry and compressed air is cooled to the temperature of liquefaction, namely, about − 194° C. (3) The condensed and liquefied air is filtered, to remove the solid carbon dioxide which it holds in suspension. (4) The filtered liquid air is evaporated again to separate, first, the more volatile constituent, nitrogen, and afterwards the less volatile constituent, oxygen. (5) The evaporation of the liquid air is utilised for the liquefaction of the compressed air to be separated into its constituent gases. (6) An automatic arrangement enables the pressure of liquefaction to be regulated, to ensure a continuous working of the apparatus. (7) An arrangement, which does not need attention when once adjusted, permits the separated nitrogen and oxygen to leave the apparatus in such condition as to have the required commercial or industrial value, and also permits the escape of mixtures of gases too nearly resembling atmospheric air. The first filtration of the air is preferably effected through cotton wool placed between two perforated plates, through which the air also passes. After compression of the air, water is removed. The exchanger and liquefier contain respectively corresponding numbers of sections and trays or troughs, the gas produced in each tray or trough of the liquefier being led through the whole length of the channel of the corresponding section of the exchanger, so as to abstract heat from the gaseous mixture to be cooled and liquefied. In the liquefier, the liquefied gaseous mixture is made to pass from tray to tray, to be gradually evaporated and fractionally distilled. The gas outlets of the exchanger are provided with valves or sluices, enabling the collection, in varying degrees of purity, of the separated gases, and the discharge of gases which it is not desired to collect, to be effected without interference with the flow of the liquefied gaseous mixture in the liquefier. The solid carbonic acid is removed by one or more filters, arranged in conjunction with the liquefier, so as not to interrupt the process. Each filter consists of a chamber containing partitions, with cotton wool, filter paper, or the like, interposed (see also this Journal, 1901, 985).—R. A.

Fluids [Milk, &c.]; Apparatus for Equalising the Temperature of ——. T. T. Sabroe, and H. J. T. Hansen, both of Aarhus, Denmark. Eng. Pat. 13,128, June 27, 1901.

This apparatus is stated to be especially applicable for the treatment of pasteurised milk, which, when taken from the pasteurising apparatus, requires to be reduced in temperature before it is conveyed to the cream separator, the object of the apparatus being to employ the heat abstracted from the pasteurised milk to warm the cold milk delivered to the pasteurising apparatus. The apparatus consists essentially of a central plate of corrugated or other suitable form, and having a liquid passage or conduit on each side of it. The two liquids are delivered at the top, on opposite sides of the plate, and are collected separately at the bottom without being mixed.—R. A.

Separating Mechanical Admixtures from Liquids, and Apparatus therefor. E. Füllner, Silesia, Germany. Eng. Pat. 19,237, Sept. 26, 1901.

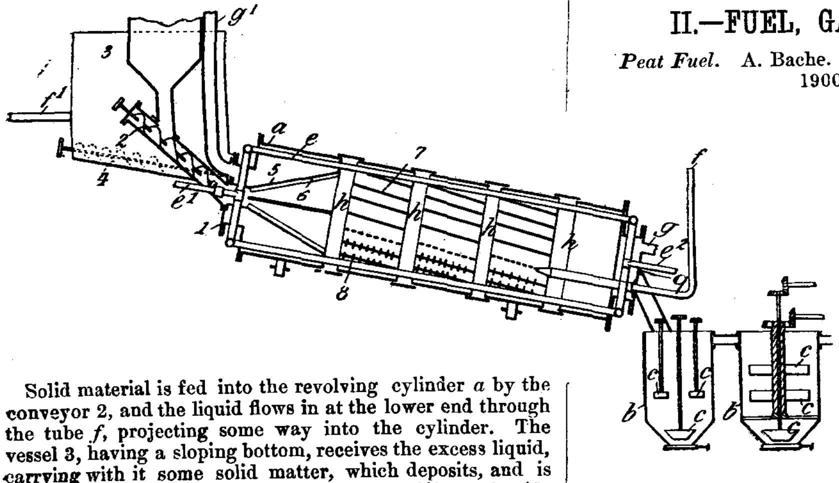
The separation is effected by means of an endless filter cloth, which is carried round a perforated drum rotating in the liquid to be purified. The drum is open at one or both ends, at the deepest point of which its interior is in free communication with the outside of the liquid-containing tank, through a perforation in the wall of the latter. The liquid, by its natural hydrostatic pressure, passes through the filter cloth and the perforated drum-casing into the interior of the drum, and deposits its admixtures on the slowly moving filter cloth, which then passes through a scraping apparatus, while the clarified liquid is discharged through the open ends of the drum. When the liquid to be purified contains little or no fibrous constituents, fibrous materials, such as cellulose, &c., are added to it, to form a fibrous coating on the filter cloth, and so assist the clarifying action.—R. A.

Evaporating or Concentrating in vacuo; Apparatus for ——. J. B. Alliott, and J. McC. C. Paton, both of Bloomsgrove Iron Works, Nottingham. Eng. Pat. 21,571, Nov. 28, 1900.

Evaporating or concentrating vessels, whether used singly or in multiple effect, are constructed with their covers and an adjacent section of their vapour pipes detachable from the apparatus and from each other, so that these parts can be removed for the purpose of cleansing the apparatus from deposits, &c. Clean parts may also be substituted for those removed. The joints of the removable parts may be provided with a suitable packing. When a multitubular heating drum is employed in the apparatus, it is secured by a single central bolt, and can be withdrawn when the bolt is released and the cover removed. The drum may be seated on a suitable packing, and be connected with a steam or vapour inlet. An air or ammonia pipe extends from beneath the drum nearly to its top.—R. A.

Liquid and Gas; Process and Apparatus for Treating Materials with ——. P. Naef, New York, U.S.A. Eng. Pat. 17,054, Sept. 25, 1900.

The form of apparatus shown is adapted for the extraction of gold from ores by cyanide or chlorine; but is also applicable for the extraction of glue from bones, the manufacture of bicarbonates, and for heating and cooling such fluids as beer, in presence of a suitable gas.



Solid material is fed into the revolving cylinder a by the conveyor 2, and the liquid flows in at the lower end through the tube f, projecting some way into the cylinder. The vessel 3, having a sloping bottom, receives the excess liquid, carrying with it some solid matter, which deposits, and is returned to the cylinder by the conveyor 4; f' is the liquid-outlet pipe. Heating or cooling liquid is passed through the pipes e, e' being the inlet and e^2 the outlet. A conical funnel, 5, having rims, 6, arranged on helicoid lines, receives the sediment from the vessel 3. The partitions, 7, are in sections, and are perforated, corrugated, or of wire gauze, arranged in an inclined position, to facilitate lifting the material through the liquid, so as to expose it to the action of the vapour or gases. When radial partitions are used, they are bent so as to move the material, and do not run in the direction of the axis. Vapour or gas is admitted by the tube g, and has outlet at g'. When the rotation of the cylinder is arrested, the sludge is permitted to flow into the settling tank b, provided with stirrers, and having a valved bottom, and communicating with other vessels. The sludge may be run alternately into different settling tanks, the supernatant liquor be withdrawn for return to the cylinder, and water for lixiviation be added in its place.

In the application of the apparatus in the manufacture of bicarbonate, this is dissolved by aid of heat in presence of carbon dioxide; the liquor is settled in a continuous manner, and is then passed through another cooling cylinder, into which the gas is also passed. The apparatus is stated to offer special advantages in the treatment of beer, which may be first cooled in presence of air and afterwards replaced by carbon dioxide. There are 85 claims.—E. S.

Pulverising and Separating Mineral and other Substances; Machinery or Apparatus for ——. C. W. Kitto and H. H. Kitto, both of Acton, Middlesex. Eng. Pat. 20,040, Nov. 7, 1900.

The apparatus is of the type in which the pulverising is effected by a ball impelled around a circular path or track by a pair of rotary discs, the discs being driven from a common shaft which extends co-axially through the discs and the path or track. The discs are held upon longitudinally adjustable dome bearings by springs, which do not encircle the shaft as usual, but are disposed, at suitable distances apart, around it, the arrangement being such as to enable the discs to be brought closer together, to compensate for the wear of the ball, without removing the shaft. Bolts are passed through each disc and bearing, and, in conjunction with the springs, serve to hold the discs evenly on their bearings, and to facilitate the relative adjustment of the discs. Each bearing is carried by, but is separate from, a longitudinally adjustable clutch sleeve, rotating with the driving shaft, and can be adjusted for wear or renewed, without dismantling the apparatus. The casing of the apparatus is made of boiler plate, &c., bent to the form of the path or track, and is caused to grip the latter by drawing the edges of the plate together. The driving discs are provided with beaters or distributors, which tend to throw the material from the bottom of the casing towards the ball path, and the driving shaft, with the ball and discs, has some freedom of movement longitudinally under the control of springs.—R, A.

II.—FUEL, GAS, AND LIGHT.

Peat Fuel. A. Bache. Proc. Inst. Civil Eng. **147**, 1900—1901.

The author gives an account of the views of various Scandinavian writers on the use of peat as fuel. The peat bogs of Sweden are estimated to be equivalent to 3,000,000,000 tons of coal. In almost all furnaces peat can be substituted for coal and for 10 years, in Martin-steel and glass furnaces, brick and tile kilns, &c., this has been done. The lowest price of coal in Sweden is 16*s.* 8*d.* per ton, the equivalent price of peat being 9*s.* 3*d.* At Haarlem, in the province of Holland, the peat bog lies below the level of the sea, and is worked by floating excavators; these excavators draw 1 ft. of water, and deliver the kneaded peat at the rate of the equivalent to 100—200 tons of air-dried peat (25 per cent. of water) per day, to the drying floors, at a cost of 3*s.* per ton; the peat is spread out in a layer 2 ft. thick, which subsides in two days to less than half that thickness. It is then cut up into blocks 3 ins. × 3 ins. × 5½ ins., which are placed on end. Peat briquettes have been made at Ironowka, near St. Petersburg, for the past 10 years; the peat mull is crushed between rolls, sifted through sieves of 0·4 in. mesh, and conveyed to the drying chamber, where the proportion of water is reduced to from 15 to 20 per cent. The drying chamber contains a series of cast-iron trays, one above the other, heated by exhaust steam; the mull is conveyed along these trays, being stirred at the same time, and is delivered finally to the press, the open-ended cylinder of which contains a sufficient number of briquettes to provide enough friction to allow of the pressure required. To make good and durable briquettes, the peat must contain 18 per cent. of water. The heating power of such briquettes is probably one-third greater than air-dried peat, and the cost of manufacture is twice as great. They have the advantages of being easily stored and of withstanding moisture; they can be stacked out of doors without cover. The coking of peat does not yield such valuable residual products as is the case with wood. Air-dried peat containing 25 per cent. of water, yields 40 per cent. of peat charcoal; its calorific power is, before coking, 6,300 B.T.U. per lb., after coking, 5,040 B.T.U.; there is thus a loss of 20 per cent. of heating value. Peat powder so thoroughly dried as to expel the combined water, has proved to be a good fuel at Jönköping, and has the same evaporative power as coal. The special difficulty of employing peat charcoal for iron smelting has been its friability; it also occasionally contains phosphates. The principal by-products are recovered in the following percentages on the dried peat at the Stangfjord Electro-Chemical Works:—Creosote oil, 4 per cent. (2*l.* per ton); ammonium sulphate, 0·4 per cent. (8*l.* per ton); acetate of lime, 0·6 per cent. (6*l.* per ton); wood spirit, 0·2 per cent. (35*l.* per ton). At the Trent Valley Peat Fuel Works, Kirkfield, Canada, the peat is raised by a dredger and conveyed to the works, where it is filled into bags of coarse felt, 6 ft. × 6 ft. 4 ins., which, a dozen at a time, are squeezed by a hydraulic press at a pressure of 4,000 lb. per sq. in.; the press takes 8 tons at a time, the operation lasting 12 minutes. The pressed stuff then passes through a crushing machine, where all the larger pieces of peat are broken up and fed into the upper end of a sloping cylinder, 5 ft. in diameter and 30 ft. long, which slowly revolves, and terminates at its lower end in a firing chamber, wherein the temperature is about 1,500° F. (? 500° F.). The yield is about 3 tons of dried peat per hour. The dried peat contains 12 per cent. of moisture, and is finally compressed into cylinders $1\frac{1}{3}$ ins. × $1\frac{1}{2}$ ins. The cost is reckoned at 6*s.* 5*d.* per ton, including royalty. A ton of peat yields 460,000 more British thermal units of heat if first coked and burnt as peat charcoal than if burnt direct as peat. The relative composition of peat charcoal and peat is given by Mr. Tylvad as follows:—

——	Peat.	Peat Charcoal.
Carbon	42·7	76·5
Hydrogen	4·0	3·5
Oxygen	27·4	11·9
Nitrogen	1·6	1·3
Water (absorbed)	21·9	2·0
Ash	2·4	4·6

The flues and combustion chambers for boilers, &c. should be increased 8—27 per cent. over the usual dimensions for coal, when intended for burning peat. Peat charcoal yields excellent gas for lighting and heating; that from peat itself is too largely mixed with carbon dioxide to be of service for gas engines.—J. W. H.

Furnace Combustion; Phenomena of ——. O. Boudouard. Bull. Soc. Chim. 1901, **25**, [18], 833—840.

The author has studied the conditions controlling the reaction $2CO \rightleftarrows CO_2 + C$ in the gas producer, the blast furnace, and the cupola. In the producer, where the object is to convert the whole of the carbon into carbon monoxide, experiments described in this and previous papers (this Journal, 1899, 278, 279, 498, 742) point to the following as the best conditions:—A high temperature; the reduction of carbon dioxide by carbon begins at about 550° C., and is only complete at 1,000° C.; a porous fuel in a finely divided state and a very slow stream of air and gas. In the blast furnace, where the reduction of oxide of iron is effected by carbon monoxide, and part of the carbon dioxide formed is again reduced to carbon monoxide by the fuel, any of this gas escaping from the furnace represents so much fuel wasted. Here, again, temperature, porosity, and draught are the important factors. Peculiar deposits of carbon (containing a small percentage of iron) found in the furnace are attributed to the decomposition of carbon monoxide into carbon dioxide and carbon by long and quiet contact with oxide of iron at comparatively low temperatures (300°—400° C.). Such deposits, forming in fissures, joints, and pores of the furnace wall wherever there are particles of iron oxide, eventually destroy the structure. In the cupola, where the fusion of metal, not reduction, is the object in view, complete combustion of carbon to carbon dioxide must be aimed at, and reduction of carbon dioxide to carbon monoxide avoided. The conditions therefore are, a dense fuel in large pieces exposing the smallest possible surface, a quick draught to lessen the time of contact of the carbon dioxide with the carbon, a low air pressure and much divided stream, and a rapid cooling of the carbon dioxide formed. A hot-air blast is detrimental.—T. A. L.

Gas; Modern Practice in the Manufacture and Distribution of ——. H. E. Jones. Proc. Inst. Civil Eng. 1901, **146**, [4], 1—27.

The author first refers to recent important improvements in the consumption of gas, and then treats of the whole subject of the manufacture and distribution of gas under the following headings. Labour-saving machines; residuals; enrichment of gas; capital expenditure in gas undertakings; works of manufacture; distribution. The paper is accompanied by seven diagrams.—A. S.

Acetylene; Dissolved ——. M. Fouché. La Nature, 1901, 289. Proc. Inst. Civil Eng. 1901, **146**, [4], 68.

Claude and Hess (this Journal, 1897, 524) have found that at a pressure of 10 atmospheres, 1 litre of acetone will dissolve 240 litres of acetylene gas, with a slight increase in volume, and that the solution is non-explosive (see also this Journal, 1897, 787, 788). When using the dissolved acetylene for illuminating purposes, the pressure is reduced from 10 atmospheres to that of a column of 2 or 3 inches of water, by means of a special reducing valve attached to the iron cylinders in which the solution is distributed for consumption. The gas is forced through a dry meter, with a manometer attached, and is consumed in a special burner In the original, the various types of cylinders are described and illustrations are given of the apparatus.—A. S.

Air Carburetted with Benzol in Fischer's Apparatus; Behaviour of ——, *and the Limits of Explosion of Admixtures with Air.* F. Ulzer. Mitt. d. tech. Gewerbe-Museums, Vienna, 1901, **11**, [7, 8, 9, and 10], 158.

A measured quantity of the gas was passed through U-tubes placed in freezing mixtures, and the benzene which separated, was weighed. The initial temperature of the gas was 20° C., and the benzene always contained a small proportion of water.

Temperature.	Volume of Gas.	Weight of Benzene.	Weight of Benzene per 100 litres of Gas.
° C.	Litres.	Grms.	
0	109	11·10	10·18
− 5	111	12·85	11·57
−10	126	14·9	11·82
−20	157	20·05	12·77

A mixture of 74 volumes of carburetted air and 26 volumes of air, or of equal volumes of the two, is not explosive; whilst 72 volumes of carburetted air and 28 volumes of air, or 52 volumes of carburetted air and 48 volumes of air, form explosive mixtures.—T. A. L.

Naphthalene Stoppages [*in Gas Plant*]; *Removal of* ——. J. f. Gasbeleucht. 1901, **44**, [41], 756.

Sartorius, acting on Bueb's advice, used a Standard washer, having two compartments, charged with anthracene oil, for washing gas at Aschersleben, and found that this treatment completely prevented naphthalene stoppages, which had previously caused great trouble, especially at the inlet and outlet of the gasholder. But Bueb's further recommendation to use the anthracene oil, after removal from the washer, for diluting the tar in the collecting (hydraulic) main, proved a source of frequent blockages owing to thickening of the tar in the main itself and in the tar wells. This recommendation was disregarded, and the anthracene oil from the washer was run direct into the tar for disposal.

Rother had had less trouble from naphthalene stoppages at Spandau during the past winter than in previous years. He had brought into use during the winter a *setting in* which the ascension pipes were 2 metres longer than those hitherto used, and the hydraulic main consequently 2 metres higher. Since the use of this setting, naphthalene stoppages in the works had ceased, though stoppages occurred in a main laid under a bridge in the town. These were removed by evaporating crude xylene into the gas.—J. A. B.

Coal and Coke; Arsenic in ——. A. C. Chapman.
See under XXIII., *page* 1241.

Sulphur in Fuel; Determination of the Total ——. R. Dubois.
See under XXIII., *page* 1241.

PATENTS.

Fuel; Manufacture of ——. P. R. de F. D'Humy, Liverpool. Eng. Pat. 19,087, Oct. 25, 1900.

Peat or other decayed vegetable matter is mixed with combustible binding materials in the wet state, and then heated "until a chemical separation of the carbon constituents of the mass has taken place." The mass is then pressed and cooled.—R. S.

Peat; Carbonisation of ——. H. Holm, Denmark. Eng. Pat. 20,893, Nov. 19, 1900.

An improvement on the process and apparatus described in Eng. Pat. 12,523, 1900 (this Journal, 793, 1901). In this case, the carbonising vapours ("smoke") travel in a closed circuit, being in one part of its circuit heated in a closed chamber, outside which fresh hot vapours from the generator constantly pass, while in the carbonising chamber its temperature is again reduced. Valves are provided which automatically act to admit more vapours into the circuit if the pressure diminishes, or to allow some to escape if the pressure increase.—R. S.

Peat; Treatment of ——. H. H. Lake, London. From P. Jebsen, Norway. Eng. Pat. 16,347, Aug. 14, 1901.

To make peat charcoal, the peat is carbonised in thin layers in vertical or nearly vertical retorts, each retort being provided with a central flue, so that the peat layer is heated from both inside and outside. The retorts extend downwards from the heating space into a cooling chamber, from which a discharge shoot opens into a lower room. Above the retorts there is a drying floor, heated by the gases leaving the heating chamber. These gases are caused to pass through zigzag channels under the floor.—R. S.

Fuel Briquettes, Blocks, or Pieces; Manufacture of ——. F. Chailly, New York. Eng. Pat. 17,863, Sept. 6, 1901.

The briquettes are formed from comminuted combustible material and an agglomerating substance or binder composed of a mineral containing calcium and an agglutinating viscous or gelatinous substance, or solution of such a substance, or a soluble silicate, or silicic acid. A special composition claimed, consists of a mixture of a comminuted combustible material, anhydrous gypsum, dextrin or starch, soluble silicates or silicic acid, and residues of mineral oils, commonly called black oil.—R. S.

Coke; Apparatus for the Manufacture of ——. J. H. Darby, Wrexham. Eng. Pat. 3586, Feb. 19, 1901.

To prevent the coke having a dark appearance, due probably to the combined action of air and water when cooling, a fixed or portable hood or cover is placed at the exit of the coke oven, and is provided with water jets for thoroughly drenching the coke while it is practically out of contact with the air.—R. S.

Gases of High-caloric Value from Low-caloric Fuel Material; Process for the Production of ——. A. J. Boult, London. From D. Turk and the Actien Gesellschaft "Lauchhammer," both of Riessa a/Elbe, Germany. Eng. Pat. 23,662, Dec. 27, 1900.

Gas from lignite, peat, or other material, which yields a large proportion of steam and carbon dioxide, is passed through carbonaceous fuel previously raised to incandescence by an air-blast, and the steam and carbon dioxide are thereby decomposed. The gases produced during the application of the air-blast to the carbonaceous fuel are not mixed with the gas.—J. A. B.

Gas Producers. W. J. Crossley, Manchester, and J. Atkinson, Marple, Cheshire. Eng. Pat. 19,377, Oct. 30, 1900.

A circular grate, leaving an annular opening for the escape of ashes, is revolved slowly, either continuously or at intervals, with the object of keeping the fuel in uniform condition. The air for combustion passes through the annular space between an outer shell and the lower part of the producer, to the grate opening, and thereby absorbs heat from the producer shell. The gas formed, passes away through a central internal pipe which extends downwards from the lid of the producer, and fuel is fed into the space between this pipe and the lining of the producer. Volatile constituents of the fuel are gasified by the heat from the effluent gas, communicated through the central pipe. A metallic lining may be employed in the producer at about the level of the mouth of this pipe, in order to conduct heat upwards to the fresh fuel.—J. A. B.

Gas Generators. P. A. Fichet and R. M. J. Heurtey, both of Paris. Eng. Pat. 16,356, Aug. 14, 1901.

The walls of the generator incline inwards at its lower part, and form a support for the fuel. At the top of the bed of fuel, air is admitted, and at the bottom there is a hollow cone, in which the gas formed, collects, and escapes by a lateral pipe. Below this cone is a hopper discharging on to a revolving or swinging plate, which is operated so as to discharge the slag or cinders into water contained within a closed casing surrounding the outlet hopper.—J. A. B.

A Fixed Gas for Lighting, Heating, or any other Purpose; Process and Apparatus for Making ——. E. B. Cornell, Philadelphia, and W. C. Alderson, Overbrook, Pa., both in U.S.A. Eng. Pat. 17,171, Aug. 27, 1901.

Steam is produced in a boiler, within the setting of which are retorts to which the steam and crude petroleum are admitted in regulated proportions. The retorts each comprise a core and annular space, through which the products circulate, and are thus brought into close contact with the hot outer walls. The outlet pipes are of smaller size than the inlet pipes to the retorts, with the object of retarding the escape of the gas, which is finally passed through condensing pipes to the gasholder. The retorts are maintained at a temperature of at least 1,800° F., whereby the steam is stated to be completely decomposed, and the gas formed to consist substantially of carbon monoxide, ethane, and methane. $1\frac{3}{4}$ lb. of crude petroleum to 1 lb. of steam are said to give good results.—J. A. B.

Blast-Furnace Gases, and Apparatus therefor; Impt. in Charging Motor Engines Worked by ——. B. H. Thwaite, London. Eng. Pat. 21,733, Nov. 30, 1900.

The gases are mixed, on their way to the engine, with an amount of air insufficient to produce a combustible mixture, the remainder of the air necessary for combination being drawn into the cylinder, by the suction of the piston, along with the gas and air already mixed. The first portion of air is admitted at the fan delivering the gas to the purifiers, &c., the supply being regulated by a suitable valve, which valve, in one modification of the apparatus, is controlled by governors actuated by a vane or wheel situated within the gas main and set in motion by the current of gas therein.
—C. S.

Gas; Apparatus for the Manufacture of ——. F. W. C. Schniewind, New York City. Eng. Pat. 17,510, Aug. 31, 1901.

The apparatus is intended for carrying out a method of gas manufacture for which an application for Letters Patent has been made. The method is an improvement on one protected under Eng. Pat. 10,588 of 1900 (this Journal, 1900, 814). A water-gas generator supplements the apparatus there described, and is provided with connexions and valves by which the water-gas is passed into the gasholder, which receives the richer gases produced in the retorts, while the producer gas, which is made intermittently with the water-gas, is mixed with the poorer gases which are used for heating the retorts. The illuminants, removed from the poorer gases by washing with oil, serve to maintain the illuminating power of the mixture of water-gas and the richer gases from the retorts. Thus the employment of the water-gas generator greatly increases the bulk of gas obtained suitable for illuminating purposes. —J. A. B.

Illuminating Gas and Coke; Manufacture of ——, and Apparatus therefor. O. Ruppert, Essen-Ruhr, Germany. Eng. Pat. 18,513, Sept. 16, 1901.

In order to improve the quality of the coke, the inventor carbonises coal in vertical retorts, in which the weight of the superincumbent coal prevents swelling of that below. In order to avoid reduction of the quality of the gas, as soon as the gas evolved is found to be poor in illuminants, it is no longer discharged into the condensers, but into a chamber which communicates with the passages for heating the retorts, where it supplements the supply of producer-gas. The construction of the settings of retorts, with the distributing chamber and heating passages, is described in detail by reference to diagrams.—J. A. B.

Washers for Producer or other Gases. C. Humfrey, Hartford, Cheshire. Eng. Pat. 22,644, Dec. 12, 1900.

In washers, each consisting of a chamber partly filled with water and provided with dashers for producing a spray, the invention consists in having pairs of dashers, the blades of which are curved in opposite directions, so that they throw up liquid towards one another and thereby produce an extremely intimate admixture of the washing liquid in the form of spray with the gas. The liquid tends to rise at the ends and to fall at the centre. To obviate this, extensions or side chambers are provided at the ends, and are connected with an extension at the centre, whereby the liquid is continually brought back to the centre, and the water remains practically level.—R. S.

Washing and Cleansing Gas from Ammonia and other Impurities; Apparatus for ——. R.W.B. Creeke, Fife, N.B. Eng. Pat. 5948, March 21, 1901.

In revolving brush scrubbers, instead of using cylindrical brushes having brushing fibres on the outer surface only, the gas being passed across the surface at right angles to the longitudinal axis of the cylinder, each brush has concentric rings of brushing fibres, and the gas is caused to pass along the outside surface of the brush in the longitudinal direction, and then back again through one of the annular passages, past an interior concentric cylindrical brush, or *vice versa*. A series of three such compound brushes is shown arranged in a tower, with suitable gearing for driving all the brushes from one shaft. A diagram also shows how twelve cylinders may be combined.—R. S.

Calcium Carbide; Process and Apparatus for the Production of ——. D. de Vulitch and J. D'Orlowsky, both of Paris. Eng. Pat. 21,214, Nov. 23, 1900.

The process consists in fusing a mixture of carbon and an excess of lime in an electric or other suitable furnace, and discharging the molten mass out of contact with the air, into a bath of a suitable hydrocarbon. The latter is decomposed into carbon and hydrogen, the former of which combines with the excess of calcium to form more carbide. The carbide combines, during crystallisation, with hydrocarbon, whereby the former is rendered capable of subsequently resisting the humidity of the air.—R. S.

Acetylene Gas; Apparatus for Generating ——. C. Busch, Paris. Eng. Pat. 10,183, May 16, 1901.

A carbide container, hermetically closed except at its base, which is perforated in order to admit water and allow of the escape of the gas formed, while retaining the lime, which serves to purify the gas, is placed within a bell, which dips into a vessel containing water. The bell is provided with a burner and tap, and an air-discharge cock.—J. A. B.

Acetylene; Apparatus for Generating ——. G. Belin, Brussels. Eng. Pat. 17,566, Sept. 2, 1901.

A generator containing carbide is connected by two passages—through the lower of which water enters and through the upper of which gas escapes—with the lower of two compartments of a vessel. The upper compartment serves as a water reservoir, and communicates with the lower by a pipe reaching nearly to the bottom of the latter. Water passes from the reservoir to the lower compartment until it reaches the inlet passage to the generator, whereupon the gas evolved, passes into the upper part of the lower compartment and forces water back into the reservoir, until the gas is drawn off for consumption.—J. A. B.

Vapour-burning Apparatus; Impts. in ——. A. Kitson, London. Eng. Pat. 20,210, Nov. 9, 1900.

The inventor claims: Incandescence-lighting vapour-burning apparatus, in which oil is vaporised in a horizontal or nearly horizontal tube enclosed in a casing, the vapour issuing from the tube, drawing air into a mixing chamber and forming therewith a combustible mixture, which is apportioned, by the aid of distributing appliances, to the main burner or burners above the vaporising tube, and to one or more auxiliary burners adapted to heat the vaporising tube, as by projecting flame underneath and along the same. The pipe conveying the combustible mixture to the burner is combined with a frame, provided with burners and so connected with the said pipe that it (the frame) can be moved so as to bring a mantle, mounted over any of the burners, into operative position during action, without more than a momentary interruption of the illumination. —C. S.

Producer Gas Burners for Steam or other Boilers. C. Humfrey, Hilderstone, Hartford, Cheshire. Eng. Pat. 22,643, Dec. 12, 1900.

The burner is a combination of a number of tubes fixed into a tube plate and projecting into or towards the flue or other place to be heated, and of a surrounding chamber, the effect being to form a number of narrow passages in juxtaposition, through one set of which (*e.g.*, the tubes) the producer gas is passed, whilst the air is admitted through the other set, in order to facilitate admixture.—C. S.

Gas and Vapour Burners; Impts. in ——. A. Hayes, Salt Lake City, U.S.A. Eng. Pat. 5757, March 19, 1901.

The method claimed is for vaporising and burning hydrocarbon oils, by forcing them, in the state of fine spray, through a flame, mixing the resulting vapour with air to form a gas, and conducting this gas to a burner.

The apparatus consists of a mixing tube; means for forcing a fine stream or spray of oil into the mixing tube; means for maintaining a vaporising flame within the mixing tube, about the spray or stream of oil; means for supplying air above the flame to mix with the vapour generated from the oil; and means for supplying a fixed combustible gas to the mixing tube.—C. S.

Igniting Devices for Automatic Gas Lighting; Impts. in ——. W. P. Thompson, Liverpool. From A. Simonini, Brooklyn, U.S.A. Eng. Pat. 13,579, July 3, 1901.

The lighter comprises, in combination, a preliminary heater of platinum black; an igniter, consisting of a body of fibrous material surrounding the heater, the said body being impregnated with a solution of thorium nitrate, 99 per cent.; cerium nitrate, 0·98 per cent.; and rhodium chloride, 0·02 per cent.; and an intermediate heater, formed on the upper portion of the fibrous body by means of a solution of thorium nitrate, 99 per cent.; cerium nitrate, 0·8 per cent.; and rhodium chloride, 0·2 per cent., the last-named solution being applied in gradually decreasing quantity from the upper end of the fibrous body toward the lower end thereof. —C. S.

Bunsen Burners; Impts. in ——. C. M. Kemp, Baltimore, and G. H. Denny, New York, U.S.A. Eng. Pat. 16,162, Aug. 12, 1901.

The claims include the combination, in a Bunsen burner, of a gas-supply tube and tapered valve thereon, with a cap threaded on the gas tube and provided, in the centre of its top, with a port through which the said valve projects; also a cylindrical sleeve fixed on the cap and extending nearly to the top of same; a burner tube, mounted on the gas tube, free to rotate on the same, and provided with air-inlet ports arranged opposite said valve; means for preventing the vertical movement of the burner tube; and a projection on the sleeve, extending into a vertical slot in the burner tube, which sleeve is snugly fitted into the burner tube, and serves, when raised and lowered, to diminish and increase the operative area of the air-inlet ports.—C. S.

Hydrocarbon Burners; Impts. in ——. R. G. Groves, Wolverhampton. From The Pan-American Light Co., New York, U.S.A. Eng. Pat. 19,799, Oct. 4, 1901.

The invention consists of a hydrocarbon lamp, provided, above the burner or burners, with a vaporiser comprising a number of superposed (substantially parallel) tubes, so arranged as to form a zigzag passage for the hydrocarbon, the inlet tube being farthest from, or least exposed to, the heat from the burner, in order to obviate the decomposition of the liquid hydrocarbon. The vapour outlet is arranged to discharge upwards into an upright mixer, for mixing it with atmospheric air drawn in by injection. The vaporising tubes decrease in diameter from below upwards, to allow for the expansion of the hydrocarbon and protect the incoming oil from the intense heat of the burner. The lamp has preferably two primary igniters, one for gas and the other for a combustible liquid, such as alcohol. In the latter case, the igniter consists of a tubular guide with ignition apertures, and of an interior sliding trough containing the igniting liquid and fitted with apertures which register with those in the guide tube. Some of the tubes of the vaporiser are fitted with devices for scraping off any accumulations of carbonaceous or other obstructive matter.—C. S.

Incandescence Mantles for Gas Lighting. O. A. Berend, London. From J. R. Schauer, Weipert, Bohemia. Eng. Pat. 22,470, Dec. 10, 1900.

An incandescence mantle for gas lighting, made from fabric manufactured by a twill-knitting machine.—H. B.

Incandescence Bodies for Electric Glow Lamps. A. Just and R. Falk, Vienna. Eng. Pat. 22,599, Dec. 11, 1900.

There are claimed incandescence bodies composed of a mixture of zirconium, with zirconium oxide, without the addition of any other metals or oxides. The materials are moistened with water and pressed into moulds.—H. B.

Incandescent Gas Burners. The Portable Gas Fountain Syndicate, Ltd., Westminster. From J. Thovert, Lyons. Eng. Pat. 23,605, Dec. 24, 1900.

Inside the mixing tube of a Bunsen burner is arranged a disc with helically inclined blades (the device resembling a propeller), which cause the current of gas and air to sweep round and become more thoroughly mixed. To produce a still more perfect mixing of the gas and air, there may be arranged concentrically, at the lower end of the mixing tube, two annular ascending and descending passages, which cause the gases to take a zigzag course before passing up the mixing tube.—H. B.

Self-igniting Incandescent Gas-Lights. A. Rosenberg, Berlin. Eng. Pat. 23,701, Dec. 28, 1900.

When platinum solutions are applied to unburnt fabric, they generally spread over a greater surface than is desired. To obviate this, no aqueous solution is used, but a solution of an organic platinum compound in fatty or ethereal oils, similar to those used in the ceramic art for the production of platinum and gold lustres. Such solutions do not spread on the fabric.—H. B.

Electric Lighting, Heating, and Resistance Bodies; Manufacture of —— [*Nernst*]. W. Boehm, Berlin. Eng. Pat. 11,529, June 5, 1901.

The materials, such as the rare earths or alkaline earths, to be used in making the glow-bodies, are subjected to a very strong heat, such as that of the electric arc, to cause them to become as dense as possible. They are next finely ground, made into a paste, and formed into rods or other suitable shapes. By subjecting the formed bodies to the heat of the arc, they become coated with a glaze, and contract to the extent of about 20 to 30 per cent. of their diameter. It is preferable to fuse the ends of the bodies into a ball form in the electric arc, to still further increase their density and to give them a larger contact surface. The ends may first be coated with a difficultly fusible substance, and then fused in the electric arc.—H. B.

Burners for Oil Lamps having Incandescing Mantles. L. C. Nielsen, Copenhagen. Eng. Pat. 1585, Jan. 23, 1901.

The claim is for a ring or the like, which may be revolved, for regulating the air-inlet orifices as required.—H. B.

Incandescent Electric Lamps and Heaters; Manufacture of Filaments and other Conductive Bodies for ——. W. E. Sander, Berlin. Eng. Pat. 14,411, July 15, 1901.

There are used, as the bases of the filaments, the metals of the rare earths, such as thorium and zirconium, and of the alkaline earths, and uranium, and certain combinations of these metals, particularly the hydrogen and nitrogen compounds. The reduction of the oxides is effected by heating them highly, either *in vacuo*, in an atmosphere of neutral gas, or in the presence of hydrogen or nitrogen, along with a suitable reducing agent, such as magnesium. The metallic bodies are found in an amorphous form inside the retort; but at the same time gaseous products are formed, which can be deposited on heated articles suspended inside the retort. The solid products are made into a paste with cellulose solution, extruded into filaments, dried, carbonised in a neutral atmosphere, and then highly heated *in vacuo* or in a neutral atmosphere. In utilising the gaseous products, any suitable filament is suspended in an enclosed space, from which oxidising gases are excluded, the filament is caused to glow, and the gaseous bodies are introduced. These deposit themselves, with partial decomposition, first on the hottest parts of the body, so that the filament gradually acquires equal cross-section in all parts.—H. B.

Incandescent [*Oil*] *Lamp Burners.* A. Schapiro, Berlin. Eng. Pat. 19,502, Sept. 30, 1901.

This relates to burners of the circular wick type, in which the flame is regulated by moving up or down a cylinder situated within the inner wick-tube, so that the surface of the stationary wick, from which the oil is to evaporate, can be increased or diminished. The claim is for attaching the flame-spreader rigidly, by means of a rod, to the movable cylinder, so that as the latter moves up or down, the flame spreader moves with it.—H. B.

Incandescent Lighting; Bunsen Burners for use in ——. G. Ihle, Berlin. Eng. Pat. 19,247, Sept. 26, 1901.

To produce as perfect a mixing as possible of the gas and air, the mixing tube of an ordinary Bunsen burner is divided longitudinally into several separate parallel chambers, into which there open as many separate gas-nozzles leading from a common mixing chamber. The burner head is common to all the mixing tubes.—H. B.

Incandescent Gas Burners. J. Mallol, Birmingham. Eng. Pat. 19,776, Nov. 3, 1900.

Centrally within the burner head is fixed an inverted ribbed cone, which is heated by the flame and itself imparts heat to the stream of combustible gas passing up between the ribs. To prevent the flame from flashing back, a wire-gauze cone, surrounding the heating cone, is fixed within the burner head. The mantle support rises from a socket in the base of the heating cone. Air is admitted to the mixing tube from below, the air inlets consisting of perforations in a horizontal annular disc surrounding the gas inlet nipple.—H. B.

Candles and other Illuminants for Producing Effective Christmas Tree and other Illuminations, with Process for Manufacture of same. E. Oelbermann, Cologne-Ehrenfeld, Prussia. Eng. Pat. 19,286, Sept. 27, 1901.

The claims made are, the use of a very thin wick as compared to the cross-section of the fatty substance, both being as free as possible from sodium, and incorporating with the fatty substance materials which will produce coloured flames. Metallic salts which are soluble in the fatty substance are used; those which, like barium and strontium, give scoriaceous products are not suitable, nor are those which give rise to small local explosions. For red the inventor employs lithium salts, and for green, salts of copper. —H. I.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, PETROLEUM.

Pyrogenetic Contact Reactions of Organic Compounds. W. Ipatiew. Ber. 1901, **34,** [14], 3579—3589.

The decompositions of organic compounds under the action of heat may be of two kinds: (1) Decompositions produced by the action of heat alone; (2) contact decompositions, produced by the action of heat, but necessitating the presence of some foreign substance, with which the vapours come into contact. According to the nature of the contact substance, these decompositions take place at lower temperatures, and the products vary, not only quantitatively, but even qualitatively.

The decompositions of anhydrous ethyl alcohol have been studied. The alcohol was boiled in a flask and the vapours passed through a hard glass tube contained in a furnace. The products passed through a large flask cooled with water, a reflux condenser, a coiled tube, then two receivers cooled by ice and salt, thence through bromine into a gasometer. Before the experiment the air was expelled from the flask by alcohol, and from the tube by carbon dioxide. The temperature was determined by a Le Chatelier's pyrometer in the middle of the tube. In order to obtain comparative results about 150 grms. of alcohol were always distilled over per hour.

Alcohol is decomposed to the extent of traces only at 700° C., at 800° C. the decomposition is more marked, and at 820°—850° C. it is considerable. In order to estimate the aldehyde produced, the contents of the first and second flasks were distilled, the distillate which passed over below 70° C. was diluted with ether, ammonia gas passed through, and the crystalline compound weighed. The increase in weight of the wash-bottles was added to the aldehyde so found. At 820°—830° C. about one-fifth of the alcohol destroyed is decomposed into ethylene and water, and four-fifths into hydrogen and aldehyde. About one-third of the aldehyde remains undecomposed; the remainder is further converted into methane and carbon monoxide. The higher the temperature the more aldehyde is produced, but also the more is destroyed. In a platinum tube a similar decomposition occurs, but apparently at a lower temperature.

In the presence of zinc in the form of rods at 620°—650° C., alcohol is decomposed principally into aldehyde (about 80 per cent. of the alcohol destroyed); the gases are very rich in hydrogen, and little ethylene is obtained. In the presence of zinc dust (purified, and dried at 150° C. in carbon dioxide) at 550° C. about half the alcohol is decomposed into ethylene, and half into aldehyde, which is then partially destroyed. Brass turnings at 650° C. produced principally aldehyde.

Ethyl ether requires a much higher temperature than alcohol for its decomposition; of 145 grms. distilled over zinc at 750° C., 33 grms. were decomposed, 10 grms. of acetaldehyde and 3 grms. of ethylene being obtained.

The author considers that the greater production of aldehyde by oxidisable metals (zinc) is explained by the following equations, in which "Me" = metal: $C_2H_5.OH = C_2H_4 + H_2O$; $Me + H_2O = MeO + H_2$; $C_2H_5.OH + MeO = Me + CH_3.CHO + H_2O$.—A. C. W.

Fluorene; Manufacture of ——. Actienges. f. Theer- und Erdöl-Ind., Berlin. Ger. Pat. 124,150, July 1900. Zeits. angew. Chem. 1901, **14,** [44], 1118. (See also Eng. Pat. 5047, 1901; this Journal, 1901, 796.)

This process for separating fluorene from the mixture of hydrocarbons in which it is contained is based on the observation that, when fused with caustic potash at about 280° C., fluorene forms a solid potassium compound:—$C_{13}H_{10} + KOH = C_{13}H_9K + H_2O$. When the melt is removed from the boiler, the potassium compound of fluorene settles down in the form of a brown mass, which quickly solidifies; this is separated from the upper layer of hydrocarbons, consisting principally of anthracene and phenanthrene, and is decomposed by treatment with water. The fluorene so recovered is then purified by distillation or by recrystallisation, and is employed for the manufacture of dyestuffs.—J. F. B.

Carbolic Acid; Determination of Crude ——. G. Schacherl.
See under XXIII., *page* 1251.

Carbolic Acid; Determination of Crude ——. F. Seiler.
See under XXIII., *page* 1251.

IV.—COLOURING MATTERS AND DYESTUFFS.

Benzylamine; Conditions of Formation of ——. Action of Ammonia on Benzyl Chloride. R. Dhommée. Comptes Rend. **133,** [17], 636—638.

The author has investigated the effect on the amounts of benzylamine, dibenzylamine, and tribenzylamine produced, by variations in the nature of the solvent, in the molecular weight of the solvent, the strength of the ethyl alcohol (when used as a solvent), the temperature, the proportion of solvent to a constant amount of ammonia, the proportion of ammonia to a constant amount of solvent, and the total amount of solvent and ammonia in a constant proportion to one another. The only really considerable influence is exerted by the last factor, and the author recommends using, with 100 grms. of benzyl chloride, 15 litres of alcohol containing 105 grms. of NH_3 (*i.e.*, saturated with the gas). The materials are left in contact at the ordinary temperature for about five days; the yield is 42·6 grms. of benzylamine (with about 6 grms. of dibenzylamine and 1 grm. of tribenzylamine).—J. T. D.

Chromophoric Groups. H. Rupe and D. Wasserzug. Ber. 1901, **34,** [14], 3527—3531.

The authors have prepared from *m*-nitro-acetophenone, condensation products containing double linkings in different positions in the molecule with a view of determining their chromophoric effect and their influence when these products are combined with other radicles.

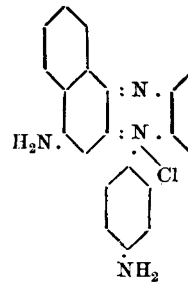
m-Nitrobenzylidene-m-nitro-acetophenone, $1.3.O_2N.C_6H_4.CH{:}CH.CO.C_6H_4.NO_2 1.3$, is best obtained by boiling together in alcoholic solution equimolecular proportions of *m*-nitro-acetophenone and *m*-nitrobenzaldehyde together with a few drops of soda lye. The condensation product crystallises from acetic acid in pale yellow needles melting at 210° C., insoluble in water, alcohol, ether, and light petroleum spirit, and soluble in acetic acid, acetone, benzene and chloroform. On reduction with stannous chloride and hydrochloric acid, the hydrochloride of the corresponding diamino compound is obtained. This yields a diacetyl derivative melting at 150° C. and crystallising in small white needles. The diamine gives a tetrazo compound which combines with various naphthol sulphonic acids and their derivatives, yielding direct cotton dyestuffs which, compared with the corresponding dyestuffs from benzidine, are redder in shade.

p-Dimethylaminobenzylidene-m-nitro-acetophenone is obtained by condensing *p*-dimethylaminobenzaldehyde and *m*-nitro-acetophenone in alcoholic solution with gaseous hydrochloric acid. The product crystallises from glacial acetic acid in thin red needles melting at 165° C. It is sparingly soluble in water and ether, more easily in glacial acetic acid, acetone and benzene, and dyes wool and silk a golden-yellow colour. The corresponding amino compound obtained by reduction with stannous chloride and hydrochloric acid gives a diazo compound which, after combination, yields dyestuffs giving bright shades on wool, silk and tannined cotton.

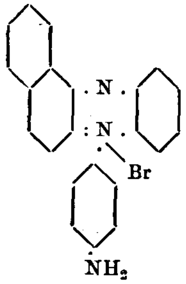
The condensation product 3.4-*dihydroxybenzylidene-m-nitro-acetophenone*, obtained from protocatechualdehyde and *m* nitro-acetophenone, forms small yellowish crystalline needles from dilute alcohol, which melt at 217° C. The product is a strong mordant dyestuff, and gives orange shades on an alumina mordant, and a reddish-brown shade on chromed cotton or wool, which is particularly fast to soap. The effect of the presence of the nitro group is very marked; the condensation product from protocatechualdehyde and acetophenone only dyes chrome mordants a pale yellow. The nitro condensation product gives a diacetyl compound melting at 179° C. and a diethyl derivative melting at 103° C.—T. A. L.

Rosinduline No. 14. F. Kehrmann and E. Ott. Ber. 1901, **34**, [12], 3092—3098.

The six isorosindulines hitherto unknown contain the amino group in the phenyl nucleus attached to the azonium nitrogen, and, with a view of completing the series, and investigating the effect of the introduction of the amino group into this part of the molecule, two further isomerides have been prepared. The starting point for No. 14 is 2.4′-diaminodiphenylamine, obtained from Nietzki and Witt's dinitrodiphenylamine, melting at 211°·5 C., by reduction with stannous chloride and alcoholic hydrochloric acid. The dihydrochloride of the base forms colourless crystals, which turn violet on exposure to light. On boiling this salt in alcoholic solution with 4-amino-1.2-naphthoquinone, condensation takes place, and on distilling off part of the alcohol, the dyestuff crystallises out on cooling. The yield of 13.6-diaminophenylnaphthophenazonium chloride (Ber. **31**, 3076; this Journal, 1899, 130)—



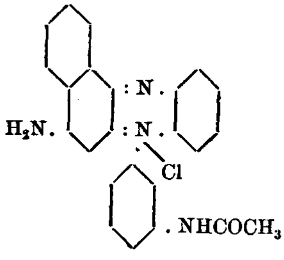
is about 65 per cent. of the theoretical. The product is stirred with acetic anhydride to a thin paste, and allowed to stand 24 hours at the ordinary temperature, when only the amino group in the phenyl nucleus is acetylated. The chloride of this substance separates from alcohol in large bronzy brick-red crystalline plates slightly soluble in cold water and alcohol. The dilute alcoholic solution has a strong brick-red fluorescence, whilst the solution in concentrated sulphuric acid is bluish-green. On boiling with acetic anhydride and sodium acetate, the diacetyl compound is obtained. The chloride of this product separates from alcohol in orange-yellow, greenish-golden, shining crystalline plates, and dissolves in concentrated sulphuric acid with a violet colour, becoming orange on dilution with water. The orange-red alcoholic solution does not fluoresce. The monacetyl compound described above, when diazotised in concentrated sulphuric acid solution, treated with alcohol and ferric chloride solution, yields an iron double salt, which is decomposed by washing with brine, giving acetylisorosinduline chloride. The salt is purified by dissolving in alcohol and precipitating with ether, and separates in golden-yellow crystalline plates, soluble in alcohol and water. The solution in concentrated sulphuric acid is dirty violet, becoming yellow on dilution with water. On hydrolysis with dilute sulphuric acid and precipitation with sodium bromide, a brownish crystalline powder of isorosinduline bromide No. 14 is obtained, having the formula—



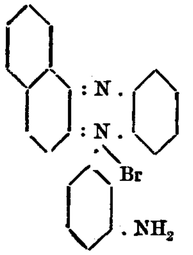
The product dissolves in concentrated sulphuric acid with a violet colour, becoming golden-yellow on dilution with water.—T. A. L.

Rosinduline No. 15. F. Kehrmann and P. Nüesch. Ber. 1901, **34**, [12], 3099—3104.

By boiling together in alcoholic solution 4.1.2-aminonaphthoquinone and 2.3′-diaminodiphenylamine with a drop of dilute hydrochloric acid, condensation takes place, and the resulting product is converted into the diacetyl compound. This, on boiling with hydrochloric acid in alcoholic solution, gives 6.12-diaminophenylnaphthophenazonium chloride, which, on treatment with acetic anhydride at the ordinary temperature, yields the monacetyl derivative—



from which the amino group can be removed by the diazo reaction. The product is converted into the bromide and then hydrolysed with sulphuric acid yielding 12-aminophenylnaphthophenazonium bromide—

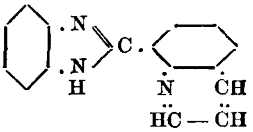


which separates from alcohol in dark yellowish-brown greenish metallic granules very sparingly soluble in water. The solutions are non-fluorescent and have a bitter taste. Concentrated sulphuric acid dissolves the product with a pure violet colour.

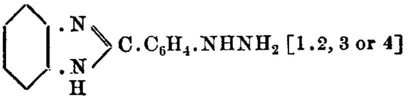
A comparison of the spectra of dilute alcoholic solutions of this product and of that described in the foregoing paper with phenylnaphthophenazonium shows that whilst the introduction of an amino group in the *m*-position (Rosinduline No. 15) has very little effect indeed, the presence of the same group in the *p* position increases the absorption of the less refrangible rays.—T. A. L.

β-Aminophenylbenzimidazoles; Comparison of the Three ——. *VII.* Bol. Miklaszewski and St. v. Niementowski. Ber. 1901, **34**, [12], 2953—2974.

In continuation of previous work on a new series of anhydro compounds, in which several derivatives of (*β*)-*o*-aminophenylbenzimidazols were described, the authors have prepared the isomeric (*β*)-*m*- and (*β*)-*p*-derivatives. The investigation of the azo dyestuffs herefrom has been indicated by Lauth, and undertaken, at any rate as regards the *p*-series, by F. Muttelet, so that the authors have confined themselves in the paper more particularly to two series of compounds, the quinobenzimidazoles—



and the hydrazinobenzimidazoles—



The aminobenzimidazoles are obtained by reducing the corresponding nitrobenz-*o*-nitranilides, and, with a view of characterising the three isomeric compounds, the authors have prepared the hydrochlorides and nitrates, acetyl and benzoyl derivatives, thioureas, azo dyestuffs and hydrazines, together with certain hydrazones, and also the quinobenzimidazoles. In general the aminophenylbenzimidazoles are colourless crystalline substances, insoluble in water, readily soluble in organic solvents. They have decided basic properties, and give well-crystallised salts with two equivalents of an acid. The nitrates and hydrochlorides are readily soluble in water and alcohol, whilst the latter salts are sparingly soluble in concentrated hydrochloric acid. The acetyl and benzoyl derivatives are the sole products of the action of the corresponding acid anhydrides on the *m*- and *p*-compounds, no condensation to di-anhydro compounds taking place as in the case of the *o*-derivative. It is also to be observed that the action of benzoyl chloride and soda lye does not decompose the imidazole ring. Carbon bisulphide in alcoholic solution forms, with the *m*- and *p*-derivatives, the corresponding thioureas in the same way that primary aromatic amines react. The products are yellow substances readily soluble in alkalis. On diazotising the aminobenzimidazoles at 0° C. in strongly acid solution, the resulting compounds combine readily with alkaline *β*-naphthol solution, yielding red azo dyestuffs. When the diazo salts are reduced with stannous chloride and hydrochloric acid, the corresponding hydrazines are obtained. These substances are yellow in colour, soluble in concentrated alkali lye, and precipitated from these solutions by carbon dioxide. The hydrazines are also soluble in acids, giving well-crystallised hydrochlorides and nitrates. The bases are precipitated by ammonia from solutions of these salts. The hydrazines reduce ammoniacal silver, but are very stable towards Fehling's solution, and condense with aldehydes, ketones, and ketocarboxylic acids to the corresponding hydrazones. These products are crystalline yellow substances, insoluble in water, soluble in alcohol, and do not possess sharp melting points. They are decomposed into their components by boiling with mineral acids, and certain of them, when treated with concentrated nitrite solution, give a pink coloration. The quinoline bases from the three isomeric (*β*)-aminophenylbenzimidazoles were obtained by means of Skraup's reaction, but only one of the two possible derivatives was formed from the *m*-compound. The new quinolines are similar to that from the *o*-compound which has been obtained previously; both contain a molecule of water of crystallisation, are soluble in organic solvents, and form diacid salts; for example, $C_{16}H_{11}N_3 2HCl$.—T. A. L.

Anthragallol; Autoxidation Products of ——. M. Bamberger and A. Praetorius. Monatsh. Chem. **22**, [6], 587—589.

The authors have commenced an investigation into the changes undergone by anthragallol when a current of air is passed through the alkaline solution for a considerable time. A yellow substance was isolated, which crystallises from alcohol, ether, and water in fine plates, melting at 197°, and which dissolves in alkalis with crimson colour. The behaviour of this body is similar to that of oxyjuglone and hydroxy-α-naphthoquinone acetic acid (Liebermann, Ber. **33**, 1900, 572), but the constitution is not yet ascertained. It has the empirical formula $C_{12}H_{10}O_5$; gives a red-violet silver salt, $C_{12}H_8O_5Ag_2$; a monomethyl ether crystallising in yellow needles, melting at 135° C.; and a crystalline orange-coloured derivative with phenylhydrazine.

During the oxidation of anthragallol in this manner, hydrogen peroxide is freely formed.—H. L.

Luteolin and Digitoflavone; Identity of ——. H. Kiliani and O. Mayer. Ber. 1901, **34**, [14], 3577—3578.

The flavone detected in *digitalis* leaves, to which the name Digitoflavone was given (Ber. **32**, 1184; this Journal, 1899, 677), is now shown to be identical with Luteolin. On fusion with potash it yields protocatechuic acid. On benzoylation it gives a tribenzoate melting at 201° C., whilst a tetrabenzoate is obtained, melting at 200°·5 C., from so-called digitoflavone or from its tribenzoate by the action of benzoylchloride and pyridine (Deninger's method). Comparative mordant dye tests further showed the identity of the Luteolin and digitoflavone, so that the latter term is superfluous.—T. A. L.

Amino-azo Compounds; Fatty Aromatic ——. B. Prager. Ber. 1901, **34**, [14], 3600—3606.

Fatty aromatic compounds, such as benzene-azo-aceto-acetic ester, in their enolic form, are analogues of *o*-hydroxy-azo compounds, but hitherto no fatty aromatic analogues of amino-azo compounds have been described. The author has obtained two representatives of this class of bodies by combining diazobenzene chloride with *β*-aminocrotonic ethyl ester, $CH_3.C(NH_2):CH.CO_2C_2H_5$, and its N-methyl derivative. The reaction takes place most readily in hydrochloric acid solution, the hydrochloride of the new compound crystallising out and readily yielding the base on neutralisation with ammonia. The amino-azo compounds have a decided basic character, but the amino group is only loosely attached, since, if the acid solution of the benzene-azo-aminocrotonic ethyl ester be allowed to stand some time, ammonia is split off and benzene-azo-aceto-acetic ester is formed. The action of alkalis or boiling with water produces the same decomposition. Aromatic amino-azo compounds react with *p*-nitrobenzaldehyde, forming nitrobenzal compounds or else triazines, whereas benzene-azo-aminocrotonic ester and *p*-nitrobenzaldehyde condense with elimination of alcohol, the reaction taking place at the ordinary temperature, yielding a neutral compound, $C_{17}H_{14}O_4N_4$, which, on heating with alcoholic sulphuric acid, readily decomposes into *p*-nitrobenzaldehyde and benzene-azo-aceto-acetic acid. In the case of the N-methylated derivative, condensation with *p*-nitrobenzaldehyde also takes place, but neither water nor alcohol is eliminated. The reaction product, $C_{20}H_{22}O_5N_4$, splits off methylamine on heating with alcoholic sulphuric acid, but neither nitrobenzaldehyde, benzene-azo-aceto-acetic acid nor its ester are formed. No amino-azo compound could be obtained from diazobenzene and diethylamino-crotonic ester, $CH_3.C[N(C_2H_5)_2]:CH.CO_2C_2H_5$, and the basic compound, $C_{22}H_{29}ON_5$, formed in presence of sodium acetate, is characterised by its stability, although only a small yield of the product is obtained.

Benzene-azo-aminocrotonic ethyl ester is formed by mixing 150 c.c. of a seminormal diazobenzene chloride solution with 10 grms. of *β*-amino-crotonic ethyl ester dissolved in 20 c.c. of alcohol. The solution solidifies after 15 minutes to a yellow crystalline magma, which, after filtering, is washed with brine, dissolved in water, and precipitated as base with ammonia. The pure product melts at 103° C. the yield being 9·4 grms.

By adding 3·49 grms. of this substance to a warm (20° C.) solution of 2·26 grms. of *p*-nitrobenzaldehyde in 140 c.c. of alcohol, and allowing the whole to stand about 30 hours, a yellow crystalline precipitate (2·9 grms.) separates. This, after crystallisation from benzene (420 c.c.), melts at 177° C., and has the formula $C_{17}H_{14}O_4N_4$. As stated above, when heated with alcoholic sulphuric acid, it decomposes into *p*-nitrobenzaldehyde and benzene-azo-aceto-acetic acid. The combination of diazobenzene chloride and β-methylaminocrotonic ethyl ester in aqueous hydrochloric acid solution, yields benzene-azo-methylaminocrotonic ethyl ester hydrochloride, the orange-coloured base, $C_{13}H_{17}O_2N_3$, separating from alcohol and melting at 114° C.—T. A. L.

Dyestuffs; Sulphonated Oxyazo ——, and their Salts.

P. Sisley. Bull. Soc. Chim. 1901, **25**, [18], 862—867.

THIS investigation of Orange II. (diazosulphanilic acid and β-naphthol), Chrysoïn (diazosulphanilic acid and resorcinol), and Crystal Ponceau (diazo-α-naphthylamine and G-salt, 2.1′.3′-naphthol disulphonic acid), has shown that the free acids of these dyestuffs are strong acids decomposing mineral salts in aqueous solution. The precipitate formed on adding hydrochloric acid to a hot concentrated solution of Orange II. is principally the undecomposed sodium salt. To determine the amount of decomposition by hydrochloric acid of various strengths, 2 grms. of Orange II. were cohobated with 50 c.c. of boiling acid for 10 minutes, left cold for 24 hours, and the precipitate collected and calcined. The sodium sulphate determined in the ash was taken as representing the undecomposed sodium salt. With acid containing 2 per cent. of hydrochloric acid (*i.e.*, 4·28 times the quantity theoretically necessary for complete decomposition), there was no precipitate and presumably little decomposition. With acid containing 2·5 per cent. HCl the decomposition, according to the table given, amounted to 46·2 per cent., and gradually rose, with increasing strength of acid, until, with 20 per cent. HCl, 97·4 per cent. of the dyestuff was decomposed. The isomeric α-naphthol derivative Orange I. is entirely decomposed by 2 per cent. acid, and Chrysoïn by 3 per cent. acid under the same conditions. The *pure acid of Orange II.* was prepared by dissolving the crude product in a mixture of 1 vol. of alcohol and 2 vols. of ether, filtering, and adding 12 vols. of ether. The precipitated acid was redissolved in a little hot alcohol, and reprecipitated with ether in the form of reddish-brown crystalline needles having the formula $C_{16}H_{12}SO_4N_2 4H_2O$. It is very soluble, 1 litre of water at 19° C. dissolving 174·4 grms. and 1 litre of dilute hydrochloric acid (10 grms. of HCl per litre) dissolving 69·7 grms. of the organic acid. With acid of twice this strength the solubility falls to 9·7 grms. per litre. If sodium chloride solution (8 per cent.) be added to an equal volume of a 4 per cent. solution of the Orange acid, the sodium salt of the dyestuff is precipitated and the filtrate is found to be strongly acid. Sodium sulphate is decomposed in the same way. This remarkable property appears to be common to all the oxyazo dyestuffs. The decomposition of the mineral salt by the organic acid takes place even in the presence of five equivalents of hydrochloric acid. Commercial Orange II. consists of the sodium salt, and when recrystallised from alcohol separates in plates having the composition $C_{16}H_{11}N_2SO_4Na2\frac{1}{2}H_2O$. Dissolved in saline solutions (*e.g.*, in water containing 15 grms. of sodium chloride per litre) it crystallises therefrom in needles containing $5H_2O$. The anhydrous salt, the lower hydrate, and the higher hydrate have different solubilities in water and also in brine. Saturated solutions in water at 19° C. contain respectively 19·56, 32·01, and 41·16 grms. per litre reckoned as anhydrous salt. In this respect Orange II. resembles sodium sulphate. The author has observed the same phenomena with Croceïne Orange, Croceïne Scarlet 3 B N, and Croceïne Scarlet 8 B. He has further prepared the calcium, magnesium, and ferrous salts of Orange II. by double decomposition from the sodium salt or by the action of the free Orange acid on the appropriate chlorides or sulphates. All these salts crystallise with $2\frac{1}{2}H_2O$, and are closely analogous in crystallographic properties. The aniline salt of Orange II. is obtained by adding aniline hydrochloride or the theoretical quantity of aniline to a solution of the Orange acid. The product is easily crystallised from a mixture of alcohol and water (2 : 1), the crystals resembling sublimed alizarin. This salt remains unchanged in sulphuric acid containing 50 grms. of sulphuric acid per litre. Its solubility in water at 24° C. is 0·508 grm. per litre.

The free acid of Chrysoïn, $C_{12}H_{10}SO_5N_2$, crystallised from dilute hydrochloric acid forms steel-grey needles very little soluble in cold water (2 grms. per litre at 19°·5 C.). Taking advantage of the ready decomposition of mineral salts by Chrysoïn acid, a number of its salts have been prepared and are described.

The free acid of Crystal Ponceau, a disulphonic acid, is peculiarly difficult to prepare. E. Knecht (this Journal, 1888, 623) added one equivalent of sulphuric acid to a dye-bath containing this dyestuff as sodium salt, with the object of setting free the dyestuff acid. The bath dyed wool no better than before the addition. Nothing can be concluded from this experiment, because the sodium salt was not really decomposed. The author finds that even from its solution in strong sulphuric acid the sodium salt is reprecipitated on dilution with water. He succeeded, however, in preparing the free acid by suspending 200 grms. of the calcium salt in 2 litres of alcohol, and adding the equivalent 32 grms. of sulphuric acid. After boiling for 10 minutes and cooling, he obtained by filtration 43 grms. of calcium sulphate—the theoretical quantity. The filtrate, after distilling off most of the alcohol, was diluted with water, acidified with hydrochloric acid and boiled. On cooling, the dyestuff acid separated in small red crystals. Dried in the air they have the composition $C_{20}H_{14}S_2O_7N_2.9H_2O$, are very soluble in water (209·6 grms. per litre at 23° C.), and mineral acids diminish the solubility less than usual. From a solution in normal sulphuric acid, sodium chloride at once precipitates the dyestuff as sodium salt. The dyestuff acid also decomposes acid sodium phosphate and sodium bisulphate. The above method of preparation is a general one, and has enabled the author to obtain many sulphonic acids otherwise difficult to prepare. Commercial Crystal Ponceau, consisting mainly of the sodium salt, contains some calcium salt which can only be removed by several crystallisations from dilute alcohol. The pure sodium salt—

$$C_{20}H_{12}N_2S_2O_7Na_2.7H_2O,$$

has a deep brownish-red colour. Its solubility in water at 23° C. is 55·7 grms. per litre. Mineral salts diminish the solubility without causing the formation of any other hydrate such as that described above in the case of Orange II. The calcium, barium, and ferrous salts have also been prepared, and all crystallise with $7H_2O$. The calcium salt separates on mixing a saturated aqueous solution of calcium sulphate with the equivalent quantity of the sodium salt. But when the calcium salt is dissolved in alcohol and water (1 : 2), and the equivalent of sodium sulphate is added, the reverse reaction takes place, the whole of the calcium being precipitated as calcium sulphate. Here the direction of chemical change is determined by the nature of the solvent, and we have a neat illustration of the laws of Berthollet.

—T. A. L.

Benzene-azo-β-naphthylauramine.

R. Möhlau and K. P. Graelert. Ber. 1901, **34**, [13], 3384—3386.

THE action of tetramethyldiaminodiphenylcarbinol on *o*-amino-azo compounds (Ber. **34**, 881; this Journal, 1901, 570) yields azo-auramines containing the chromophoric groups N : N and N : C, and formed from the azoleucauramines by loss of hydrogen, which takes place on heating them in alcoholic solution. In the case of benzene-azo-β-naphthylauramine, since this compound itself readily undergoes decomposition under these conditions, it was found necessary to conduct the transformation in presence of a very small quantity of the reagent (alcohol). For this purpose, one part by weight of benzene-azo-β-naphthyleucauramine was heated on the water-bath with 1·5 parts of alcohol under a reflux condenser for 14 hours, when a sample, on warming with acetic acid, remains red, and does not turn blue or green. The resulting product, after recrystallisation from a mixture of acetic ester and methyl alcohol, formed scarlet rhombic prisms with a bluish reflex, melting at 180° C. The yield was 15 per cent. of the weight of the leuco compound. The auramine is readily soluble in benzene, chloroform, and acetic ester, sparingly soluble in acetone and alcohol, and very slightly soluble in ether and methyl alcohol. On heating with 5 per cent. sulphuric acid, it is readily decomposed into benzene-azo-β-naphthylamine and tetramethyldiaminobenzophenone. The azo auramine is reduced in alcoholic solution by sodium amalgam to its leuco compound, when, on adding acetic acid, the solution turns blue.

—T. A. L.

Euxanthone and Alizarin; Methylation of ——, by Means of Dimethyl Sulphate. C. Graebe and R. H. Adèrs. Annalen, **318,** [2 and 3], 365—370.

Euxanthone was dissolved in excess of dilute caustic soda and shaken with dimethyl sulphate (considerably more than 2 mols.). The yellowish-white precipitate was washed with water to disappearance of alkaline reaction, and then recrystallised from alcohol. The pure 7-methyl ether was obtained, melting at 130·5° C. The colourless dimethyl ether remains in the mother-liquor, from which it is precipitated by water; it is, however, better obtained from the sodium salt of the monomethyl ether. This salt is obtained by precipitating a solution of the 7-methyl ether in hot alcohol by a solution of the calculated quantity of sodium in alcohol. The sodium salt (dried at 100° C.) was heated on the water-bath for 30 – 60 minutes with rather more than 1 mol. of dimethyl sulphate, the product extracted with alcohol, and the solution precipitated by water. After crystallisation from petroleum spirit, the dimethyl ether is colourless, and melts at 149·5° C. Colourless euxanthone monomethyl ether is obtained on heating the dimethyl ether on the water-bath with sulphuric acid of 90 per cent. strength, and precipitating with water. This ether melts at 240° C.; it is soluble in dilute alkalis.

Xanthone gives a considerable quantity of xanthene on heating with zinc dust, euxanthone a very small quantity. It was now found that euxanthone dimethyl ether gives only a small quantity of a reddish substance, consisting chiefly of xanthene.

Alizarin 2-methyl ether is obtained by dissolving alizarin in a solution of 2 mols. of caustic soda, evaporating to complete dryness, heating with dimethyl sulphate, treating with water, and recrystallising from alcohol. The melting point is 230°—231° C. (corr.). An attempt to convert the ether into an anthrol methyl ether, by heating with zinc dust, was unsuccessful; anthracene was almost the only product. The sodium salt of the monomethyl ether, obtained as a red precipitate by the addition of the calculated quantity of sodium ethylate to a hot alcoholic solution of the ether, remained unchanged on heating with methyl iodide and dimethyl sulphate. In attempting to prepare the silver salt of the monomethyl ether by acting on the sodium salt with alcohol and silver nitrate, a mixture of the ether, silver oxide, and sodium nitrate separated.

—A. C. W.

Chloranthranilic Acids; Two New ——. P. Cohn. Mitt. d. tech. Gew. Museums, Vienna, 1901, **11,** [7, 8, 9, and 10], 178—182.

By oxidising the two isomeric chloracet-*o*-toluidines with permanganate in neutral solution, the author has obtained two new chloranthranilic acids, which he now further characterises by preparing a number of azo-dyestuffs from them, and also by converting them into the corresponding 4- and 6-chlorosalicylic acids. Tables are given showing the characteristics of the various azo dyestuffs, from which it appears that the azo derivatives of the 2.6-acid are much more soluble and bluer in shade than those from the 2.4-acid, but yellower than the dyestuffs from anthranilic acid itself. On boiling the diazo solutions of the 2.4- and 2.6-acids with water, the former yields 4-chlorosalicylic acid, crystallising from water in white needles, melting at 207° C., whilst the latter gives 6-chlorosalicylic acid, having a similar appearance and melting at 166° C. This acid is readily soluble in water, alcohol, and most organic solvents, is scarcely volatile with steam, and gives the salicylic reaction (violet coloration) with ferric chloride. The salts are well crystallised, and with the exception of the silver salt are very easily soluble in water.—T. A. L.

Mordant Dyestuffs. Noelting. Chem. Zeit. 1901, **25,** [80], 860.

The following new facts in the substituted anthraquinone (diphenylene diketone) series of dyestuffs, show that some modification of Liebermann and v. Kostanecki's theories have now become necessary. (1) Hystazarin—

$$C_{14}H_6O_2(OH)_2\ [2:3]$$

is a mordant dyestuff, but inferior to alizarin—

$$C_{14}H_6O_2(OH)_2\ [1:2],$$

where one hydroxyl is adjacent to a chomogenic CO-group. (2) Quinizarin, $C_{14}H_6O_2(OH)_2$ [1 : 4] is also a mordant dyestuff, but much weaker than alizarin. (3.) Anthrarufin. $C_{14}H_6O_2(OH)_2$ [1 : 5], and anthraflavic acid—

$$C_{14}H_6O_2(OH)_2\ [3:7]$$

are not mordant dyestuffs, but when the two positions, 1·5 and 3·7 are occupied, in the same compound, as in anthrachrysone, $C_{14}H_4O_2(OH)_2$ [1 : 3 : 5 : 7] the latter shows mordant-dyeing properties which are increased by the entry of two sulphonic groups. (4) Nitro groups confer mordant-dyeing properties, or increase the same if already exhibited. Thus, chrysophanic acid—

$$C_{14}H_5O_2(OH)_2CH_3\ [1:8:2]$$

and chrysazin, $C_{14}H_6O_2(OH)_2$ [1 : 8], are not mordant dyestuffs, but tetranitrochrysazin—

$$C_{14}H_2O_2(OH)_2(NO_2)_4\ [1:8:2:4:5:7]$$

and dinitro-anthrachrysone, tetranitro-anthrachrysone, and dinitro-anthrachrysone disulphonic acid all have this property. Nitro groups also increase the mordant-dyeing properties of azo dyestuffs.—R. L. J.

Dinitro-ortho-anisidine: Chemical Reaction in which one of the Products continues the same Reaction. R. Meldola and J. V. Eyre. Proc. Chem. Soc. **17,** [241], 185.

The authors give further evidence in support of the constitutional formula assigned to the dinitroanisidine already described (this Journal, 1901, 572). The 3:4:6-triaminoanisole and the corresponding 3 : 4-diamino-6-acetaminoanisole, both form azines on condensation with benzil, which are described. The view put forward (*loc. cit.*), that the eliminated nitro group comes out in the form of nitrous acid, is confirmed by quantitative determinations of the amount of diazo-oxide formed from known weights of dinitroanisidine in acetic acid solution with accurately titrated solutions of nitrite. The latter was added to the extent of one-fourth of the theoretical quantity required on the assumption that one molecule of dinitro-anisidine requires one molecule of nitrite. In all cases the diazo-oxide produced, as determined by the quantity of azo-β-naphthol compound formed, was far in excess of that capable of being produced by the nitrite added, thus proving that the nitro group eliminated by the nitrous acid continues the process of diazotisation.

PATENTS.

Amido-benzoic Acids, and of Anthranilic Acid and Colouring Matters therefrom; Manufacture of Substituted ——. G. B. Ellis, London. From F. A. Pertsch, Avully, Switzerland. Eng. Pat. 19,202, Oct. 26, 1900.

Salicylic acid is converted into 5-nitro-2-amino-benzoic acid. This product is acetylated and reduced, when it yields 5·2-aminoacetamino-benzoic acid, which, after diazotisation, combines with phenols, amines, and their derivatives forming azo dyestuffs. These, after hydrolysis, may again be diazotised and combined, giving disazo dyestuffs. If the 5·2-aminoacetamino-benzoic acid be diazotised in alcoholic solution and boiled, acetamino-benzoic acid is formed, which, after hydrolysis, yields anthranilic acid.—T. A. L.

Colouring Matters of the Anthracene Series; Manufacture and Production of ——. B. Willcox, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 20,719, Nov. 16, 1900, Second Edition.

The halogen derivatives of mono- or di-amino or alphyl-amino-anthraquinones or their sulphonic acids are treated with ammonia, or with substances which produce ammonia during the reaction. If necessary the products may be subsequently sulphonated by any suitable method. For instance, 10 kilos. of crude anilino-anthraquinone sulphonic acid suspended in 200 litres of water are treated with 12 kilos. of bromine and well stirred for 12 hours, when the bromo-anilino-anthraquinone sulphonic acid is precipitated by adding salt. After filtering off and drying, 10 kilos. are mixed in an autoclave with 50 kilos. of aqueous ammonia (20 per cent. of NH_3) and heated for about six hours to 200° C. When cold, the new dyestuff is salted out and gives, on chrome mordanted wool, greyish-blue to bluish-black shades.—T. A. L.

Indigo; Impts. in the Manufacture of an Initial Material for the Production of ——. B. Willcox, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 21,821, Dec. 1, 1900.

The specification relates to the preparation of phenylglycocoll *o*-carboxylic acid, by causing the neutral salts of anthranilic acid and of chloracetic acid to react on one another in aqueous solution at about 40° C. for some days. The acid salt which separates is then filtered off and employed for the production of Indigo.—T. A. L.

Indigo Leuco Compounds into Indigo, and the Application thereof to Covering Textile Fibres; Impts. in the Conversion of ——. B. Willcox, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 23,338, Dec. 20, 1900. Second Edition.

The patentees find that sulphur reacts with Indigo leuco compounds such as indoxyl, indoxylic acid, and Indigo-white, the leuco compound being converted into Indigo. The reaction can be carried out in the presence or absence of textile fibre, and if the textile material be impregnated in parts with sulphur, light and dark blue effects are produced when the fibre is passed through an Indigo-vat. The method is also applicable for printing Indigo shades, for which purpose a mixture of sulphur and indophor (indoxylic acid) is printed on the material. On subsequent steaming, Indigo is formed directly on the fibre.—T. A. L.

Sulphide Colour [Blue], and of its Leuco Compound; Manufacture of a ——. R. B. Ransford, Upper Norwood. From L. Cassella and Co., Frankfort-on-Main, Germany. Eng. Pat. 21,310, Nov. 24, 1900.

The blue dyestuff obtained, according to Eng. Pat. 16,247 of 1900 (this Journal, 1901, 889), by heating *p*-dialkylamino-*p*-dihydroxydiphenylamine with sulphur and alkaline sulphides cannot be successfully purified by the process given in the specification. The patentees now find that if the crude melt be neutralised with hydrochloric acid the products formed separate as leuco bases, and, after well washing with water, the hydrochloride of the pure leuco blue may be dissolved with dilute hydrochloric acid and salted out from this solution, the impurities remaining undissolved. The soluble leuco hydrochloride may be oxidised to blue by air in an alkaline solution, or its aqueous solution may be oxidised with other reagents, such as ferric chloride. The aqueous solution of the leucohydrochloride dyes tannined cotton, and may then be subsequently oxidised. It is also suitable for dyeing wool. Moreover, the blue dyestuff gives bright blue shades fast to light, washing, and chlorine on unmordanted cotton from baths containing caustic alkalis or alkaline sulphides.

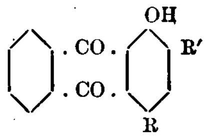
—T. A. L.

Colouring Matters [Bluish-green] therefrom; Impts. in the Manufacture of Sulphonated Aldehydes and ——. G. B. Ellis, London. From the Société Chimique des Usines du Rhône anc. Gilliard P. Monnet et Cartier, Lyons, France. Eng. Pat. 21,365, Nov. 26, 1900.

By sulphonating *m*-toluic aldehyde at 0°—5° C. with fuming sulphuric acid (60 per cent. SO_3) it yields *o*-sulphotoluic aldehyde. This substance readily condenses with dimethylaniline, its homologues or derivatives, to form bluish-green dyestuffs fast to alkalis.—T. A. L.

Dyestuffs of the Anthracene Series; Manufacture or Production of ——. H. E. Newton. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat, 21,897, Dec. 3, 1900.

According to Eng. Pat. 23,927 of 1894 (this Journal, 1895, 1041) dyestuffs are obtained by reacting with aromatic amines on hydroxyanthraquinones. The patentees now describe the manufacture of new dyestuffs by treating with primary aromatic amines, derivatives of erythro-oxyanthraquinone, having the general formula—



where R represents halogen or nitro group, and R′ halogen or a sulphonic acid group. For instance, dibromo-erythro-oxyanthraquinone and *p*-toluidine give a condensation product which, on sulphonation, yields a product dyeing blue shades on unmordanted and greenish-blue on chrome-mordanted wool.—T. A. L.

Dyestuffs containing Sulphur; Impts. in the Manufacture or Production of ——. H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 21,898, Dec. 3, 1900.

The patentees find that the products obtained by treating various aromatic compounds with sulphur and alkaline sulphides react like thiophenols, in that they are capable of alkylation or alphylation. The process can be carried out with the dyestuffs themselves or when dyed on the fibre, employing the usual reagents, which react in a neutral or alkaline solution, such as ethyl or benzyl chloride, the salts of alkyl sulphuric acid, alkyl sulphates, chloracetic acid or its amide. Moreover, certain ammonium derivatives, such as dimethylbenzylphenyl ammonium chloride, serve as alkylating agents for these products, and, owing to their solubility in water, are distinct from other alkylating agents. They are also inodorous.—T. A. L.

Monobrom-Indigo and Dibrom-Indigo, as well as Monochlor-Indigo and Dichlor-Indigo, and Monochlor-monobrom-Indigo; also Methods of Dyeing and Printing with the said Halogen-Indigos; Method of Preparing ——. A. Rahtjen, Hamburg, Germany. Eng. Pat. 11,022, May 28, 1901.

Indigo in the dry state, or in presence of a solvent or diluent, reacts with bromine or chlorine at a low temperature, forming the products enumerated in the title. The new derivatives are very similar to Indigo, and dye from a vat in the same way. They may also be sulphonated, although not so readily as Indigo itself.—T. A. L.

Greenish-Black Sulphurised Directly Dyeing Dyestuffs for Cotton; Manufacture of ——. A. G. Bloxam, London. From Chemische Fabrik Brugg, A. G., Brugg, Switzerland. Eng. Pat. 16,876, Aug. 22, 1901.

Benzene sulphonic acid, or one of its homologues or salts, or a corresponding sulphochloride or sulphamide is fused with sulphur and an alkaline sulphide. The resulting products dye cotton greenish-black shades from baths containing sodium sulphide, carbonate and chloride or sulphate.—T. A. L.

V.—PREPARING, BLEACHING, DYEING, PRINTING, AND FINISHING TEXTILES, YARNS, AND FIBRES.

Finishing Materials containing Starch and Starch Products. D. Saare. Zeits. Spiritusind. 1901, **24**, [44], 451. (See also Fürth, this Journal, 1901, 242.)

The author discusses the composition of certain typical starch glazes which have passed through his hands. In the first group are starches brought into a pasty condition by the aid of alkalis. One of these was a slimy, pasty mass with a tinge of grey colour; it was odourless and had the composition: water 80·5, starch 16·5, ash 1·9 per cent., with an alkalinity of 1 per cent. caustic soda. Such a mixture would require to be neutralised by sulphuric or acetic acid before use. Another had been prepared with soda and then treated with acetic acid; analysis showed: water 93·03, starch 6·9, ash 0·08 per cent.; it had an acid reaction and yielded 0·14 per cent. of acetic acid on distillation with steam.

Another group of glazes contain calcium and magnesium chlorides, the object of which is, partly to increase the liquefaction of the starch and partly to act as antiseptic and loading. One of these was a uniform, pasty, and cloudy mass in the form of a viscous fluid; it contained: water 64·00, calcium chloride 21·67, magnesium chloride 0·06, and starch 14·27 per cent. Those made with an admixture of magnesium chloride as well as calcium chloride appeared in the form of transparent, gelatinised, wax-like pieces. Two samples showed the following composition:—

——	1.	2.
	Per Cent.	Per Cent.
Water	50·17	61·49
Calcium chloride	16·49	18·43
Magnesium chloride	4·31	7·44
Starch	29·03	12·64

Those finishes which contain calcium chloride possess the disadvantage of rusting iron, a drawback which does not occur with those prepared with magnesium chloride only.

This type of size is largely used by felt-hat makers under the name of "vegetable glue."

"Lucon" may also be cited as representing another class. It forms a yellowish-white, lumpy powder with an odour of tallow. Its composition was found to be:—water 8, starch (partly dextrinised) 54, tallow 5, salt, clay, and sand 25, soda-ash 8 per cent. The aqueous solution gave a red coloration with iodine solution. Lastly, the group of soluble starches is dealt with. These products give clear solutions instead of pastes with hot water, gelatinising on cooling to a milky paste. In the case of dextrins the addition of 10 per cent. of glucose is considered an advantage. Glucose makes the goods softer and more pliable, and does not fill, but if used alone, the cloth has a greasy feel; dextrin on the other hand, makes the goods hard and fills them. A finish of this kind appeared as a viscous, milky, sweetish mass, with a slight odour of potato farina; it contained: water 27·8, potato starch 1·4, dextrin 54·9, and sugar (glucose) 13·8 per cent.—J. F. B.

Wool; Action of Caustic Soda on ——. C. E. Washburn. Textile World, Aug. and Sept. 1901.

The author has subjected various woollen yarns to the action of caustic soda solutions of different concentration, for longer or shorter periods, at different temperatures, and has examined the tensile strength and behaviour towards dyestuffs of the yarns before and after treatment. His results agree closely with those obtained by Buntrock (this Journal, 1898, 452). The maximum increase in tensile strength is obtained with solutions of 82° Tw. (42° B. or 1·41 sp. gr.) at 13° C., by which in five minutes, for example, a yarn of 40 strands having a tensile strength of 42·0 lb. is converted, after treatment and washing with 1 per cent. sulphuric acid, into a yarn showing a tensile strength of 57·0 lb.

After treatment with the caustic soda, a whiter yarn is obtained by washing with a 1 per cent. solution of sulphuric acid than by using dilute nitric or hydrochloric acid, which colour the wool yellow. Ammonia can be used in the place of acid, but it acts much slower. If the caustic soda is not entirely removed from the wool before drying, the yarn is harsh and brittle.

The author likewise tried the effect of adding glycerin to the lye, with results similar to those obtained by Buntrock. (This Journal, 1898, 452.)

The combined action of formaldehyde and caustic soda somewhat lowered the tensile strength of the fibre.

Yarns which had been treated for five minutes with lyes of 82° Tw., when dyed in the same bath with untreated yarns, were found to show the following saving:—

Mordant Dyes.—Dyestuff saved = $\frac{1}{3}$ the amount used; time saved: 40 per cent.

Basic Dyes.—Dyestuff saved = $\frac{1}{8}$ the amount used; time saved: 10 per cent.

Acid Dyes.—Dyestuff saved = $\frac{1}{5}$ the amount used; time saved: 30 per cent. Indigo, too, is taken up better by the treated wool than by untreated.

The author then shows that by weaving untreated and treated yarn together, or by printing woollen cloth with caustic soda thickened by gum tragacanth paste, and then dyeing, various colour effects may be produced, the wool which has been acted upon by caustic soda acquiring a deeper shade in the dyebath than the untreated wool.

The chemical change brought about in the wool fibre by the action of caustic soda, and subsequent washing in dilute sulphuric acid, appears to be the removal of 84·45 per cent. of its sulphur, since the author finds untreated wool to contain 3·42 per cent. sulphur, while wool after treatment contains only 0·53 per cent. This sulphur is in part removed by the caustic soda in the bath, and partly evolved as sulphuretted hydrogen during the washing with dilute sulphuric acid.

In determining the best temperature, it was found that if the bath was cooled to 5° C. caustic soda began to separate, and the yarn began to lose its strength. The author is of the opinion that if it were possible to maintain the concentration of 82° Tw. at 0° C., the tensile strength of the yarn would be even more increased than at 15° C.

A machine is employed, containing three compartments; the first, for caustic soda, should be copper-lined; the next compartment, containing lukewarm water, should be the same size as the first, and built of iron or wood. Between the first and second compartments, is a pair of squeezing rolls. The third compartment is copper lined, and contains 1 per cent. sulphuric acid solution. After passing from the acid bath, the wool may be either washed by hand, or may be passed into a fourth compartment.

The skeins of yarn are to be linked into a continuous chain, and the speed of the machine so regulated that exactly five minutes would be required to pass this through the caustic bath. The material to be treated should be thoroughly scoured and wetted before passing into the caustic soda of 82° Tw. (at 15° C.) in the first bath. After leaving the fourth compartment, the yarn may be dyed or dried in the usual manner.—H. I.

PATENTS.

Artificial Filaments, Threads, Horse Hair and the like; Manufacture of ——. F. Lehner, Zurich, Switzerland. Eng. Pat. 20,461, Nov. 13, 1900.

In this process two or more threads of cellulose, nitrocellulose, or cellulose derivative (prepared as in the artificial silk process, but somewhat thicker than such filaments) are

allowed to run together through a glass fork, immediately after their formation, and whilst they are still in a condition to mutually combine into a perfectly solid single thread.
—R. L. J.

Textile Fibres or Filaments from Solutions of Cellulose, or of other Material from which Fibres or Filaments can be formed; Improved Apparatus for Production of ——. C. F. Topham, Kew Gardens, Surrey. Eng. Pat. 23,157, Dec. 18, 1900.

The apparatus consists of a cylindrical vessel containing a filter formed with an internal passage communicating with external grooves which are covered by the filtering medium, and thus securing a large surface. The filtered material collects in the vessel and passes out through an arm (which enters the vessel a short distance so as to form a trap for air-bubbles) to the forming nozzle, which is specially designed to facilitate cleaning.—R. L. J.

Thread from Cellulose Solutions; Manufacture of ——. J. Imray, London. From E. Bronnert, Niedermorschweiler, Alsace, and M. Frémery and J. Urban, both of Dremmen, Aachen, Germany. Eng. Pat. 4303, Feb. 28, 1900.

Cellulose, dissolved in ammoniacal cupric oxide solution or zinc chloride solution, is expressed into sulphuric acid solution containing from 30 to 65 per cent. of real acid. A weaker acid solution produces threads deficient in tenacity, and acid stronger than the above-mentioned limits attacks their substance, 50 per cent. acid being the optimum for most purposes.—R. L. J.

Asbestos; Treatment of ——, *for rendering it Waterproof, and increasing its Fire-resisting Qualities.* M. Raphael. Eng. Pat. 11,856, June 10, 1901.

See under IX., *page* 1212.

Fabrics; Treating ——, *with Liquids.* G. H. France, Bradford. Eng. Pat. 18,079, October 11, 1900.

An improved method and apparatus for discharging, drying, bleaching, or otherwise treating textile fabrics, particularly heavy cotton tissues, which are liable to be creased in such operations.

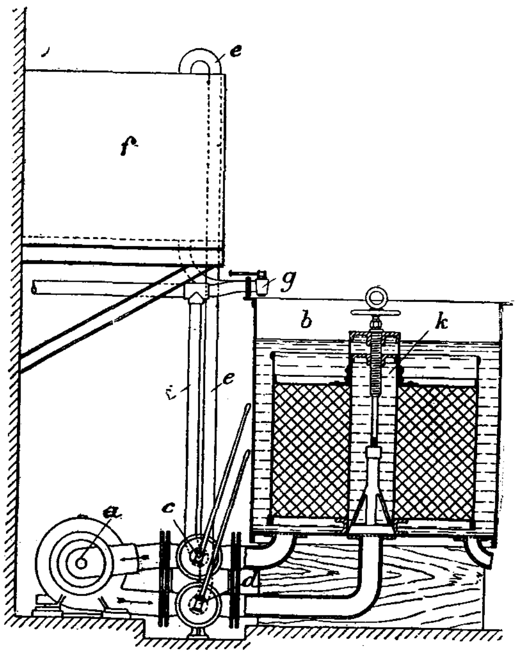
The bath employed in treating the tissues is contained in a long vat, the back of which is inclined at an angle of 45°, while its front is vertical. A revolving winch is mounted at a suitable height above it. The "pieces" of tissue to be treated are fastened together end to end, and then passed in a continuous manner through a guide eye over the winch into the vat. After a considerable length of tissue has been received in the vat, the end first fed into this is passed up between a pair of guide bars over the winch and down again into the vat. This is repeated until the tissue has arrived at the exit end of the machine, when it is withdrawn by a pair of squeezing rollers. After passing through a cooling or rinsing tank, it may be taken to another machine for further treatment.—E. B.

Dyeing Apparatus. J. O. Obermaier, Rheinpfalz, Germany. Eng. Pat. 15,191, Aug. 25, 1900.

The object of this invention is to provide apparatus for dyeing cotton materials with certain dyestuffs, such as the sulphur dyestuffs, as much as possible in the absence of air, *for enabling the greater part of the dyestuff which it is* necessary to employ to be rapidly and economically recovered, and for rapidly and thoroughly rinsing the materials after dyeing.

Between a pump *a* (see figure) and a dye-vat *b* two three-way cocks *c*, on the suction pipe, and *d*, on the delivery pipe, are arranged. *c* is in communication through a pipe *i* with a water tank (not shown), and *d* is connected through a pipe *e* with a dye-liquor reservoir *f*. *g* is a water-supply cock, and there is a water-discharge cock at the bottom of the dye-vat. Over the orifice of the pipe connecting the three-way cock *d* with the dye-vat, there is placed a hollow distributing cone provided with openings for the escape of the liquid supplied. The cone is surrounded by a perforated drum *k*, screwed to a spindle.

At the beginning of the dyeing operation the dye-vat *b* is filled with water, and this is circulated by means of the pump, to expel all air from the material. The dyestuff is then added, and the bath is further circulated while dyeing takes place. When this operation is finished, the three-way cocks are turned and the dye-liquor is forced into the tank *f*. As it is necessary to employ concentrated solutions of the dyestuffs, from which only a small proportion is abstracted by the cotton, the greater part of the dyestuff employed, is thus recovered for use again. Moreover, since the recovery is effected very rapidly, namely, in about half a minute's time, inequalities in dyeing, due to mechanical deposits of dyestuff upon the materials caused by the decomposition of the dye-liquor by the air, do not result, as is the case when centrifugal and squeezing machines are used in recovering the excess of the liquor.



Dyeing Apparatus.

By again changing the position of the cocks *c* and *d* and opening the water-discharge cock, water is caused to flow through the materials from the inside to the outside of the drum. The materials are thus effectually rinsed, the operation being accomplished with most dyestuffs in one minute's time, whereas in the apparatus hitherto in use 1—2 hours have been required for the purpose.—E. B.

Shades Fast against Washing; Manufacture or Production on the Fibre of ——. H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 913, Jan. 14, 1901.

In place of using diazotised *p*-nitraniline for developing the "Benzonitrol" or "Nitrazol" colours, the patentees employ the diazo derivatives of the dichloro-anilines $NH_2 : Cl_2 =$ 1:2:4, 1:2:5, or 1:3:4. The shades obtained are remarkable for their fastness to washing. For example, Chicago Blue B, when developed with diazotised dichloro-aniline, yields a clear greenish blue, and Primuline, developed in the same way, gives a bright greenish-yellow shade which is fast to washing, whereas the yellow obtained with Primuline and diazotised *p*-nitraniline bleeds on to the interwoven white fibre when washed.—T. A. L.

Dyeing [*Sulphur Dyestuffs*] ; *Impts. in* ——. C. D. Abel. From The Actiengesellschaft für Anilinfabrikation, Berlin. Eng. Pat. 1285, Jan. 19, 1901.

Cotton materials which have been dyed with sulphur dyestuffs become tender on being stored, most probably as a result of the oxidising action of the air upon the dyestuffs, and the consequent formation of acids which attack the fibre. This damage is avoided by treating the materials, after dyeing, with alkaline oxidising mixtures, such as alkaline solutions of sodium peroxide, potassium ferricyanide, potassium permanganate, sodium hypochlorite, and potassium chromate, as in the following examples:—

1. A bath is prepared of caustic soda and sodium hypochlorite, so as to contain 1—2 c.c. of caustic soda-lye at 40° B. per litre, and ¼ per cent. (of the weight of the cotton) of active chlorine. The cotton material, previously dyed with Sulphur Black T, is introduced into the bath, which is then heated slowly to boiling. The material is afterwards rinsed and dried.

2. The material dyed with Immedial Black is boiled for about half an hour with 2 per cent. of its weight of potassium chromate and 5 per cent. of its weight of sodium carbonate. It is then washed and dried.—E. B.

VI.—COLOURING WOOD, PAPER, LEATHER, Etc.

PATENT.

Production of Designs or Representations ; Pigments and Dyes with relation to the ——. J. Stevenson, Edinburgh. Eng. Pat. 17,783, Oct. 6, 1900.

According to this invention designs are produced from permanent and non-permanent pigments or dyestuffs, either used together or superimposed. By the fading caused by light, exposure to the atmosphere or internal chemical action, the non-permanent colour is changed or disappears, whereby the production of many pleasing colour effects in the design is claimed.—H. I.

Printing Tinplate in Dead or Dull Colours ; Process of ——. H. Bayerthal, London. From Fr. Ewers, Lübeck. Eng. Pat. 11,745, June 8, 1901.

The tinplate is first spread all over with a ground colour made from a quick drying varnish mixed with any "earthen or metal" colour and glycerin or "stearine oil." The preparation is spread thickly by a rubber roller, to which "the surface of the coating will adhere owing to its flexibility, the result being a grainy rough appearance which makes the colours appear dead or dull." The coloured design is printed in colours such as are usually employed.—H. I.

VII.—ACIDS, ALKALIS, AND SALTS.

Nitric Acid Solutions; Some Physical Properties of ——. V. H. Veley and J. J. Manley. Proc. Roy. Soc. 1901, **69**, [452], 86—119.

The authors have plotted the contractions which occur on mixing nitric acid and water against the percentage composition of the diluted acid, and find changes of curvature at points corresponding to hydrates of nitric acid with 14, 7, 4, 3, 1½, and 1 mols. of water.

Similarly, in the case of the refractive indices, the most marked points of curvature correspond to the 14, 7, and 1½ hydrates.—J. S.

Condensing Towers for Noxious Gases. J. G. Clemmer. Eng. and Mining J. 1901, **72**, [15], 453—455.

The author recommends pipe towers packed with coke, instead of the usual brick or stone towers, for condensing gases. The former, in consequence of their thin walls, do not retain the heat of the gases from the furnaces, and they have a greater capacity per sectional area than brick towers. It is stated that one pair of pipe condensing towers, as shown in the accompanying figures, will condense as much gas as a brick or stone condenser, 6 ft. by 6 ft. in cross section. The towers described and illustrated are for the purpose of condensing noxious gases, but they may be also used, with slight modifications, for condensing hydrochloric acid. Fig. 1 is a side elevation of the towers, whilst the smaller diagrams show the details of construction, the figures giving the numbers of the different parts required being for a set of three pairs of pipe towers. The towers are built of ordinary sewer pipe, and are bound in with iron straps or clamps, several layers of roofing felt, coated with soft tar, being interposed between the pipe and the clamp; the latter is also coated with soft tar. If a pipe cracks, the crevice is filled with a mixture of China-clay and soft tar, of the consistence of putty, and the joint clamped. The lower section of the tower is packed with chemical brick crib work, so as to sustain the packing of coke in the upper sections. It is advisable to pack the joints between the sections of piping with one strand of asbestos rope, and to cover this with a layer of the mixture of China-clay and tar. The tops of the towers are closed, and the gases prevented from escaping at that point by inverted "chemical ware" cups sealed with the condensing water. The condensing water may be distributed from the spigot on to the perforated tops, in the openings covered by the inverted cups, or an oscillating trough may be used. The stones for the bases of the towers are hollowed out to provide a receptacle for the condensed gases, and a hole is drilled through from the bottom of the hollow part, for the removal of the condensed products. The stones are then treated with hot tar, to render them capable of resisting the action of the condensed gases. For draught, a steam jet controlled by a valve may be used in the outlet pipes, or the latter may be run down to the earth and connected to the chimney flues, with a damper between them for control.

Fig. 1.

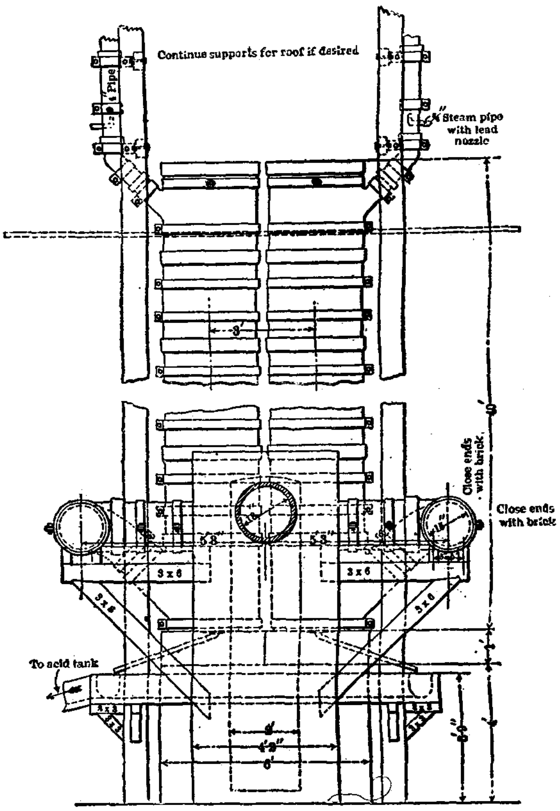
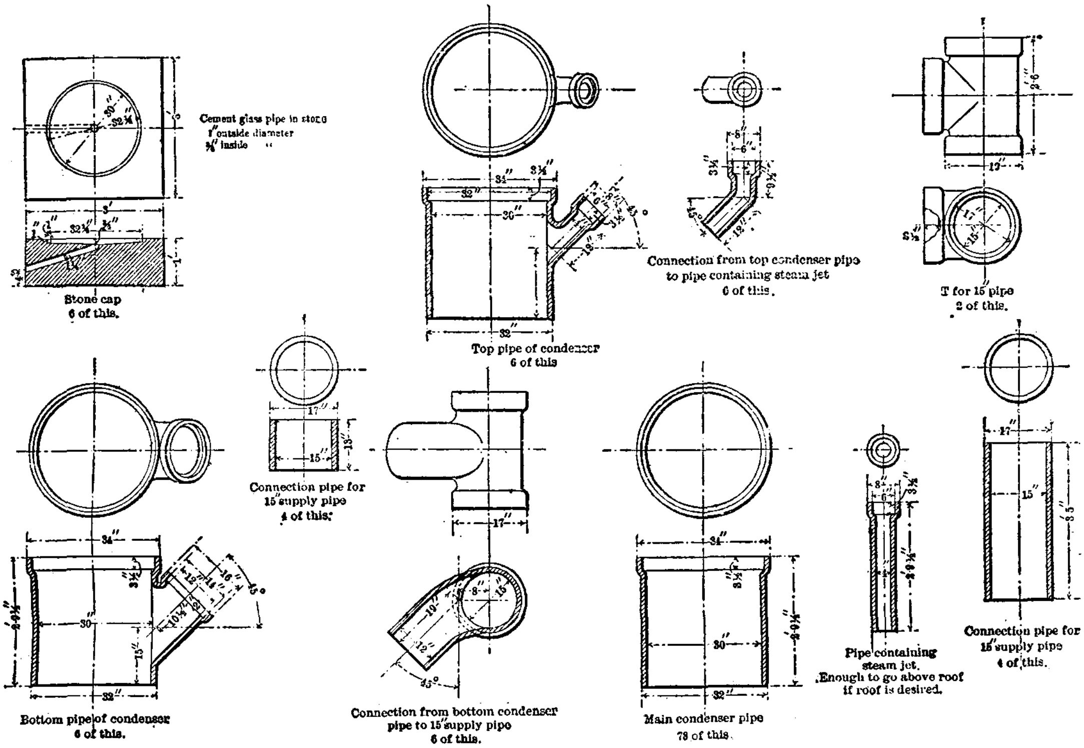


Fig. 2.



Stone cap
6 of this.

Top pipe of condenser
6 of this

Connection from top condenser pipe
to pipe containing steam jet
6 of this.

T for 15" pipe
2 of this.

Connection pipe for
15" supply pipe
4 of this.

Bottom pipe of condenser
6 of this.

Connection from bottom condenser
pipe to 15" supply pipe
6 of this.

Main condenser pipe
78 of this.

Pipe containing
steam jet.
Enough to go above roof
if roof is desired.

Connection pipe for
15" supply pipe
4 of this.

—A. S.

Nitric Acid ; Manufacture of ——. C. W. Volney.
See page 1189.

Boric Acid ; Determination of ——. Partheil.
See under XXIII., *page* 1244.

PATENTS.

Alkaline Chlorides ; Electrolytic Decomposition of ——, *and Apparatus therefor.* J. D. Gilmour. Eng. Pat. 18,397, Oct. 16, 1900.

See under XI. A., *page* 1220.

Alkali Salts (Chlorine and Caustic Soda and Potash) ; Decomposition of —— *by Electrolysis, and Apparatus therefor.* J. Greenwood. Eng. Pat. 22,406, Dec. 8, 1900.

See under XI. A., *page* 1220.

Liquid and Gas ; Process and Apparatus for Treating Materials with [Manufacture of Bicarbonates] ——. P. Naef. Eng. Pat. 17,054, Sept. 25, 1900.

See under I., *page* 1195.

Sulphuric Anhydride ; Manufacture of ——, *by the Contact Process.* O. Imray, London. From Farbwerke vormals Meister, Lucius und Brüning, Hoechst-am-Main, Germany. Eng. Pat. 1385, Jan. 21, 1901.

It is found that, although at a temperature of 400° C. or slightly over, the oxidation of SO_2 in presence of air and catalysing agents is practically complete, with no reverse action, yet the progress of the reaction is very much slower than at the temperature of, say 500° ; but, at the latter temperature there is a tendency for the SO_3 formed to split up into SO_2 and O. The present invention consists in conducting the process, first at the higher temperature to obtain the advantage of the far greater speed of reaction, and in completing it at the lower temperature, at which no reversal takes place. This may be done in two ways : first, before the single contact hitherto used, a smaller contact is inserted with a reacting temperature for the burner gases of about 530° C., where about three-fourths of the SO_2 is rapidly oxidised ; the gases are then cooled, either by admission of cold non-acting gases, or by an external set of pipes, to about 430°, and passed into the second contact chamber for completion of the process. Second, the contact chamber is enlarged, the gases entering at about 530° and leaving the chamber at about 430°, similar means for cooling as described before being used.

The following list of substances available as catalytic agents is given :—

1. The metals of the platinum group.
2. The oxides and sulphates of Fe, Cr, Ni, Co, Mn Ur, Cu.
3. The oxygen compounds of Al, Be, Zr, Ce, Di, La, Th, Ti, Si, and of nearly all rare metals.
4. Mixtures of No. 1 with one or more substances of Nos. 2 and 3.
5. Mixtures of two or more of the substances mentioned under Nos. 2 and 3.

Agents Nos. 2, 3, and 5 are efficacious only at high temperatures, hence, one of these may be substituted for platinum in the hotter part of the contact chamber ; while for the low temperature contact zone, substances Nos. 1 and 4 only are found to be applicable. Reference is made to Eng. Pat. 3166, 1888, German Pat. 113,932, and to Eng. Pat. 285, 1899 [this Journal, 1899, 1124].—E. S.

Nitrogen and Nitrous Oxide ; Manufacture and Use of ——, *from Atmospheric Air.* R. Marston, Leicester. Eng. Pat. 19,074, Oct. 25, 1900.

Air, previously freed from carbon dioxide, is passed, with a regulated proportion of hydrogen or of such bodies containing hydrogen, as ammonia, into a heated retort containing copper, iron, or other oxidisable metal. It is stated that thus a mixture is obtained of nitrogen with nitrous oxide and other nitrogen oxides, which latter are removed by passage of the gases through scrubbing towers containing iron shavings, sulphur, or other substances capable of similar action, followed it may be by treatment with a ferrous and then with an alkaline solution so as to leave a mixture containing only nitrogen and nitrous oxide. This mixture of gases is stated to be superior as an anæsthetic to nitrous oxide alone. The separation of nitrous oxide from its mixture with nitrogen may be effected either by solution of nitrous oxide in water and its subsequent liberation by heating; or by subjecting the mixture to sufficient pressure in a suitable vessel, to liquefy the nitrous oxide, which is then withdrawn. Apparatus for adjusting the proportions of air and hydrogen or ammonia passed into the retorts is described.—E. S.

Brine; Purification of ——. G. N. Vis, Schweizerhalle, Switzerland. Eng. Pat. 276, Jan. 4, 1901.

Brine containing other sulphates, or calcium or magnesium compounds besides gypsum, is treated with sodium sulphate in such proportion as to form with them magnesium or calcium sulphate and sodium chloride. Then slaked lime is added to precipitate magnesium hydroxide, any calcium sulphate thus formed being also precipitated, as the brine is supposed to have been originally saturated with that sulphate. The brine thus freed from magnesium salts and from calcium halogen salts is treated with ammonium carbonate in the proportion necessary to decompose the gypsum present and precipitate calcium carbonate. The brine, freed from the precipitate, is evaporated until only about 15 per cent. of its original volume remains, the salt deposited being removed. The ammonium salt used is then contained in the mother liquid as sulphate, and the liquor is used for the treatment of fresh brine, to which slaked lime is added, and, if necessary, sodium sulphate. The brine thus treated is stirred for several hours, then filtered, and carbon dioxide is passed in until no further precipitate forms. The ammonium salt originally used is thus recovered continuously in the mother liquors.—E. S.

Cyanogen Compounds; Manufacture of ——, *from Gas Mixtures containing Ammonia.* E. R. Besemfelder, Charlottenburg, near Berlin. Eng. Pat. 4,323, Feb. 28, 1901.

The ammoniacal gases produced by the destructive distillation of organic nitrogenous matter, preferably in the continuous manner described in Eng. Pat. 13,830, 1899 (this Journal, 1900, 725) are passed through a rope or chain tower fed with cold, strongly alkaline liquid, whereby ammonium salts are decomposed with liberation of ammonia; the gases are then dried by passage through scrubbers containing caustic lime before being led to the cyanogen generators. These contain incandescent coke, assumed to have been in previous use. The contained gases are expelled by "gas residue," that is, the inflammable gas collected in holders from the cyanogen absorbers. The generator is then heated or "blown hot," by compressed air, and the gases of combustion are blown out by "gas residue," after which the purified ammoniacal gases are admitted. When the temperature falls too low for the reaction to go on, the process is repeated. The generators are used in series, as of four, each of which is separately connected to a common chimney flue, and to a cyanogen absorber, and is fitted with pipes for admission of "gas residue," and of compressed air, for blowing the coke hot when needful. They have ashpits and a carbon feed, and are worked in a continuous cycle.—E. S.

Solids and Liquors; Heating for the Purpose of Decomposing, Drying, Calcining, or Distilling them. Process and Apparatus for ——. P. Naef, New York, U.S.A. Eng. Pat. 17,053, Sept. 25, 1900.

The apparatus is described with especial reference to the conversion of sodium bicarbonate of the ammonia-soda process into carbonate. The bicarbonate is charged by means of a hopper and conveyor into the upper end of an inclined revolving cylinder, having a hopper connection at its opposite end to the upper end of a similar, but reversely-inclined, lower cylinder, having a discharge hopper, on which a furnace or fire-box may be mounted for the combustion of fuel. Or a pipe conveying combustible gas may enter the lower end of the bottom cylinder, inside which the gas is burned by aid of air heated by passage through the discharging hopper. Both cylinders contain series of perforated agitating devices, arranged in sections, radially off-set from each other, varieties of which are shown. Carbon dioxide is passed from a heater into the upper cylinder, so as to flow in opposite direction to the passage of the bicarbonate, and has exit from the upper end into a dust chamber, and thence to a washer or absorber for collection of the ammonia. By means of suitably connected and valved pipes, the heated carbon dioxide is also circulated through the heater and the lower cylinder, the carbon dioxide liberated in which goes partly through the connecting hopper into the upper cylinder, and partly to the heater for circulation. Thus the bicarbonate is converted into carbonate in an atmosphere of heated carbon dioxide, partly extraneous and partly derived from the bicarbonate.
—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Glass; Plasticity and Adhesiveness of Diamond-cut ——, *at ordinary Temperatures.* J. Piccard. Ber. **34,** [14], 3635—3639.

It is certain that glass possesses a certain amount of plasticity (capacity of movement of the molecules beyond the limit of elasticity) not only on heating, but also at ordinary temperatures; but it is questionable whether it combines with this the property of adhesiveness (union of separated parts, capacity for being welded). The question was, *e.g.*, whether a fine crack in a piece of glass could be made to disappear at ordinary temperatures so that the rigidity would be restored, if not completely, at least to a considerable extent. To answer this question, the author has experimented by means of diamond cuts.

When a scarcely visible scratch made on a glass plate with a good diamond is examined under the microscope, it is seen to consist of two distinct parts. On the surface is a channel 0·02 mm. broad and 0·01 mm. deep, formed partly by splintering, partly by the plastic movement of the glass to either side; this plays a subordinate part in the breaking. From the bottom of this channel, a fine capillary slit extends perpendicularly into the glass to a depth of 0·05—0·1 mm. This crack, which at first extends only to one-tenth or one-twentieth of the thickness of the plate, weakens it, and by a tap, or snapping action, is widened till the plate breaks. In this capillary slit where the breaking process goes on, we also see the beginning of the re-uniting or healing process.

The natural, rounded, crystal edge of a "cutting" diamond—as contrasted with a "writing" diamond, made from a sharp splinter—produces a jagged scratch on a glass plate, and causes an uneven break. Hence it is necessary to use a mechanical device, so that the diamond produces a uniform clean cut.

The author takes a strip of clear window or plate glass about 70 cm. long, and exactly 10 cm. wide, and on one side, with any diamond, divides it into 14 numbered squares, and on the other side makes a fine cut with the special diamond along the whole length of the strip, which is then broken so as to give 12 good pieces, 10 cm. long and 5 cm. wide, and each divided into equal squares by the fine diamond cut. These are placed in two groups of six pieces each, and used the one for the immediate, the rest for the supplementary strength tests.

For the strength test they are clamped horizontally, near to the cut, which is on the upper surface, between two carefully levelled metal plates, and the free ends loaded with a tin box (fastened on by a stirrup) into which shot are poured till the plate breaks, and the weight of the shot, &c. noted. The shot must be added regularly, as the breaking depends not only on the weight but on the time during which it acts.

The other six pieces are loosely clamped with the cut downwards in a horizontal tin gutter, and the free ends either not loaded at all, or with 50, 100, 200-grm. lead plates, and allowed to rest. After one, two, or three days they are subjected to strength tests like the others. By this means a constant increase in carrying power is developed.

If the load necessary to break a glass 1·5—2·0 mm. thick is considered equal to 100, 30 represents that necessary to break a plate with the diamond cut, and after a 1—3 days rest, 36, or an increase of 20 per cent. Thus the adhesiveness, or at least the plasticity of glass at ordinary temperatures can be indicated numerically. To the question as to how deep the effect of the diamond cut extends in the glass, the answer is, that by carefully obliterating the cut on a plate with gypsum cement, the plate will attain the same rigidity as an uncut plate of the same thickness.

—W. C. H.

Glass; Method of Colouring ——. N. Meurer. Monit. Ceram. **32,** [14], 105.

According to the author a yellow, brown, red, or black colour can be imparted to glass by adding carbon, preferably in the form of sifted and slightly moistened lignite, to the charge of glass metal previous to melting. The pigmentary power of lignite is said to be three or four times as great as that of manganese peroxide, whilst the relative cost of the two ingredients is given as about 1 : 20.—C. S.

Pottery; Flowing Under-Glaze Colours in ——. J. Salt. Monit. Ceram. **32,** [18], 138.

The flowing of these printed colours (chiefly blue, black, and dark green) under the glaze when fired is produced by the aid of copper oxide added to the colours themselves, or by placing a mixture of lime and common salt in the saggar along with the printed ware. The presence of tin compounds and sodium chloride in the covering glaze may also produce a similar result.

For blue, the author prepares a flux of: Nevers sand, 20 kilos.; "Cornwall stone," 6; calcium carbonate, 4; borax, 3·5 kilos., strongly calcined in the biscuit kiln. The preparation applied to the ware is composed of: above flux, 2,500 grms.; copper oxide, 250; cobalt oxide, 2,500 grms., ground and passed through a fine sieve.

For black, 1·5 kilos. of dry ("sec") copper sulphate and 1·15 kilos. of potassium chromate are dissolved separately, each in 5 litres of water, and then mixed. After 25—30 minutes the precipitate is filtered, washed, and dried. The product, weighing 1·75 kilos., is gently calcined in the glaze furnace, finely ground, and washed, the weight being thereby reduced to 600 grms. When printed and fired under glaze, this preparation gives a handsome black, surrounded by a bluish-green aureole.

A second quality black is obtained by the following method:—Two fritts are prepared, the one (A) by intimately mixing 125 parts of copper oxide with 75 parts of chromium oxide; the second (B) by calcining in the glaze kiln: Fontainebleau sand, 100 parts; sodium phosphate, 100; lead oxide, 80; pegmatite, 70; calcium carbonate, 50; borocalcite, 20; kaolin, 40; and fluor spar, 20 parts. Of these ingredients, 700 parts of fritt A and 100 of fritt B are finely ground along with 75 parts of calcium carbonate and 25 of sand.

In many cases, however, the salt and lime mixture is to be preferred, provided care be taken not to place the saggars anywhere near other printed pieces in the kiln, especially such as are decorated with cobalt colours, since these latter are liable to alteration by the evolved gases. The mixture employed consists of: red lead, 100 parts; common salt, 50 parts; quicklime, 50; borax, 50 parts. It is best placed in a small receptacle near the top of the saggar, so as to obtain the most uniform distribution of the gases.—C. S.

PATENT.

Enamelled Articles; Manufacture of ——. J. Rapoport, Budapest. Eng. Pat. 18,014, Sept. 9, 1901.

In this process the powdered colours are applied to the object to be enamelled, in small heaps placed side by side, or in separate or mixed layers, and so arranged that in the kiln the colours shall be on the highest parts of the object, and will consequently when fused flow over it. More powder should be taken than is required to cover the surface treated, the excess being allowed to drain off.

—W. C. H.

IX.—BUILDING MATERIALS, CLAYS, MORTARS AND CEMENTS.

Chimney Construction; German Practice in ——. G. Lang. J. Gas Lighting, 1901, **78,** [2007], 1092.

As high chimneys, compared with other brickwork structures, occupy a very short time in building, it is important to so construct them that the mortar shall safely sustain the superincumbent weight, whilst yet "green." Lime mortar strengthened by an addition of Portland cement is usually considered best for expeditious work. Lime mortar alone becomes equally strong in time, and resists the heat better, but it does not harden quickly enough, and is therefore only used for building tall chimney stacks, when the work is to be done very slowly, and where a strong outside scaffolding is used. It is recommended to use a mixture of cement, lime, and sand, in the proportions 1 : 2 : 6 for the upper portion of the shaft, and 1 : 2½ : 8 for the bottom courses. The proportion of cement may be reduced if the lime is of a hydraulic nature, but should be increased if the sand is very sharp. For the cap of the chimney, the proportions should be 1 : 1 : 4. Mortar made of cement and sand alone is not suitable, as it does not resist heat well, and is attacked by the carbon dioxide in the flue gases, especially in the presence of moisture.

—A. S.

Building Stones and Mortar Materials for the Walls of Reservoirs at Gotha; Experiments on ——. Mitt. aus den Königl. t. Versuchs-Anst. zu Berlin, 1901, **19,** [2], 59—86.

An account, with tables of results of tests applied to building stones and mortar materials, is given, and of tests of resistance to tension, compression, and penetration of water, &c.

The following conclusions are drawn:—

(*a.*) The strength of trass-mortar is increased by an admixture of cement, especially when set in air.

(*b.*) The strength of mortar diminishes when very fine grained sand is used.

(*c.*) When "air-set," pure cement-mortar acquires tensile strength more quickly, and compressive strength more slowly, than when "water-set." This holds good also for cement-trass mortars as long as the cement exceeds the trass by a certain amount. A deficiency of water is unfavourable to the hardening of pure trass-mortar.

(*d.*) The difference in strength of air-set, as compared with water-set mortars, follows from the incomplete hardening in air; it increases with the age of the mortar. At first the difference is scarcely perceptible; the explanation offered is that the consolidation, in the early stages of the tests, is mechanical rather than chemical, and the water-content, in water-set mortars, hinders the development in the mortar tested of resistance to tension and still more to compression.

(*e.*) The impenetrability to water of trass-mortar is increased by an addition of cement, but it partly depends on the proportions employed.

The author mentions that (*c*) and (*d*) confirm his earlier observation (Mitth. **4,** 1900) that trass-mortar properly hardens only in presence of sufficient moisture. He quotes Bachmann's remark that, against the advantage of the slow hardening of trass-mortar must be set the possible disadvantage that a complete stoppage of hardening may occur through an insufficient supply of water.

The author adds that the preceding results of his are, as a whole, confirmed by practical observations referred to in "Le Ciment," made by Equer, on mortars used in the dry dock at Castigneau. He states that the mortar in the seams, where water had penetrated, deteriorated into a soft white powder. It is stated that Portland cement, when mixed with suitable sand, gives excellent results (*e.g.*, in Boulogne harbour). In any case greater care must be used in the employment of hydraulic lime.—W. C. H.

Lime and Cement Kilns; Quartz Shale versus Firebrick as Material for ——. P. Jochum. Thonind. Zeit. **25**, [94], 1587—1591.

The conditions exacted of fireproof materials for lining lime-kilns are:—Capacity to withstand a temperature of 1,000° C.; to exhibit neutral behaviour towards the lime dust, as well as towards the alkaline dust and the gases furnished by the fuel used in the kiln; and, in the case of shaft furnaces, sufficient mechanical tenacity to withstand the physical influences of the descending charge. Now, as quartz shale, by reason of its high percentage of silica, is peculiarly susceptible to the action of alkalis, it is evidently less suitable for the purpose in view than good firebrick containing 42—45 per cent. of alumina, provided the latter be present in the form of normal silicate and not simply the result of additions of bauxite made for the purpose of imparting a fictitious percentage of alumina.—C. S.

Slag Cement. W. K. Hatt. Chem.-Zeit. 1901, **25**, [82], Rep. 294. From Thonind. Zeit. 1901, **25**, 1336.

The author accepts the definition of Portland cement adopted by the Verein Deutscher Portland Cement Fabrikanten, namely, that it is a product obtained by crushing, after heating to the sintering point, a mixture of limestone, marl, chalk, or hydraulic limestone with clay, whilst slag-cement is a material prepared by the following processes: (1) Granulation of the slag; (2) drying of the slag; (3) mixture with a suitable proportion of slaked lime; and (4) grinding of the mixture. The Portland cement of the United States comprises those cements which are produced by the burning to the sintering point and grinding of artificial mixtures of limestone (or of marl, chalk, or hydraulic limestone), and clay (or slag sand).—W. G. M.

Incrustation from the Stone Gallery of St. Paul's Cathedral. E. G. Clayton. Proc. Chem. Soc. **17**, [241], 201—203.

Around what is known as the Stone Gallery, at the base of the dome of St. Paul's Cathedral, is a balustrade of Portland stone, surmounted by a heavy coping-stone of the same material. Much of the surface of the stone is greatly "weathered," and is coated by a stratum of a grey or black substance largely consisting of calcium sulphate, which in some places (especially on the under-side of the coping-stone) attains a thickness of three-quarters of an inch. This material, which is brittle and detachable with a knife, has a very rough and irregular surface, is stalagmitic in character, and, though differing in colour, in other respects resembles very closely some boiler deposits.

It is suggested that the presence of so much calcium sulphate is principally due to two centuries' solvent and weathering action of rain, charged with sulphurous and sulphuric acids derived from the gases and smoke of innumerable surrounding chimneys.

Asbestos. E. G. Clayton. Proc. Chem. Soc. **17**, [241], 203.

The following analyses are of four kinds of asbestos (one of which, A., was stated to be of English origin, the remainder being examples of the ordinary mineral).

Including the English specimen, all possess the general composition distinctive of true amphibole-asbestos. The English mineral consisted of moderately long greyish-green fibres, easily separable. B., C., and D., which had been pulverised before reaching the writer, were apparently associated with a little gypsum.

——	A.	B.	C.	D.
Water lost at 100°	1·19	0·87	0·86	..
„ „ 150°	0·05	0·13	0·00	..
„ „ a red heat	0·70	0·95	1·83	..
Total water	1·94	1·95	2·69	1·39
Silica (with traces of manganese oxide, alkalis, &c.).	60·88	48·48	59·82	61·74
Lime	11·11	6·19	5·91	6·91
Magnesia	10·84	35·52	28·27	25·72
Alumina	Trace	4·69	1·25	2·30
Iron oxide	15·23			
Sulphuric anhydride	..	3·17	2·06	1·94
	100·00	100·00	100·00	100·00

PATENTS.

Plaster or Artificial Stone; Composition for Use as ——. E. Bushman, Stapleton, N.Y., U.S.A. Eng. Pat. 19,597, Nov. 1, 1900.

The composition consists of oxide of zinc, pulverised flint, pulverised silica, borax solution, caustic soda, caustic potash, aluminium, asbestos, silicate of potash, silicate of soda, and water.—W. C. H.

Artificial Stone. S. E. Boivie, Ragnhildborg, Sweden. Eng. Pat. 10,084, May 15, 1901.

The process consists of mixing slaked lime with quartz or slate, nitric acid being then added to the mixture, which is immediately pressed into slabs, and subsequently submitted to steam pressure.—W. C. H.

Artificial Marble and the like; Manufacture of ——. O. Hertwig and E. Liebaug, Thuringia. Eng. Pat. 16,640, Aug. 19, 1901.

The artificial marble is made by intimately mixing, approximately in the following proportions: 16 parts of magnesite, 17 parts of magnesium chloride, 45 parts of flint or equivalent, 5 parts of kaolin or equivalent, and 17 parts of water, and pouring the mass on to glass or other polished plates.—W. C. H.

Wood; Preserving, Fireproofing, &c. ——. G. Feyerabendt, Tilsit, Germany. Eng. Pat. 15,402, July 30, 1901.

The method is to treat wood with a solution of sodium aluminate, to which some alumina has been added, under pressure, at a temperature of about 270° F. The wood may subsequently be washed, if necessary.—W. C. H.

Asbestos; Treatment of —— for rendering it Waterproof, and increasing its Fire-resisting Qualities. M. Raphael, Breslau. Eng. Pat. 11,856, June 10, 1901.

Asbestos articles, which are naturally hygroscopic, if for use at ordinary temperatures, are, by this process, coated with chrome glue, alum glue, or chrome alum glue, or are coated first with a solution of resin soap, and afterwards immersed in a solution of, say, calcium chloride, to form an insoluble calcium soap on the surface and in the pores. When the articles are to be subjected to high temperatures, they are coated with water glass, or a glass or porcelain enamel is burnt on to the surface.—W. C. H.

X.—METALLURGY.

Metals; Action of Ammonia on ——, at High Temperatures. G. G. Henderson and G. T. Beilby, J. Chem. Soc. 1901, **79** and **80**, [468], 1245—1256.

When platinum, gold, silver, copper, iron, nickel, or cobalt is exposed to the action of ammonia at temperatures ranging from 600° to 900° C., a large proportion of the latter is resolved into its elements, whilst the metal becomes disintegrated. The fracture of metals which have been exposed to this action is not crystalline, as has been previously stated, but spongy or cellular, giving the idea of sudden cooling while in a state of active effervescence. Iron and copper rods, a quarter of an inch in diameter, were completely penetrated to the centre by the ammonia in 30 minutes. Copper exposed for seven days to the action of ammonia at 800° C. became reduced to a fine spongy powder.

By the prolonged action of ammonia on platinum, very fine deposits of platinum black are produced on the surface of the more massive metal. The formation of spongy deposits on the outside of platinum crucibles heated by Bunsen burners, and the disintegration of the platinum wires of pyrometers exposed to furnace gases, may perhaps be accounted for by the presence of traces of ammonia in the combustion gases.

The absorption of small quantities of nitrogen by pure iron rendered it hard and brittle like steel. Malleable iron tubes exposed to the action of ammonia for seven days at 800° C. became so brittle that they could be broken like porcelain by a blow from a hammer. It is probable that some of the effects on the structure and properties of iron and steel, which are at present attributed to other elements, may be due to the presence of traces of nitrogen.

It may be stated that metals, in general, when exposed to the action of ammonia at high temperatures, are either converted into nitrides—wholly or partially—or else considerably changed in their physical properties, even although no nitrogen is permanently fixed by the metal.

The authors consider that the physical effects produced are due to the alternate formation and dissociation of the nitrides of the metals taking place between certain narrow limits of temperature, the reaction being turned in either one direction or the other, according as ammonia or hydrogen molecules preponderated in the gases which were in contact with the molecules of metal at and below the surface.

—A. S.

Metals ; Distilled ——. Kahlbaum. Chem.-Zeit. 1901, **25**, [84], 919.

This is a continuation of the author's work published in 1899. He has distilled 24, and examined closely into the properties of 9, metals. The object of the distillation is complete purification. The test of purity is absolute homogeneity and constancy of spectrum. If the spectrum of a metal is identical before and after distillation, absolute purity has been attained, in the author's opinion. In the case of a specimen of tellurium, 25 lines vanished after the first distillation, and 21 after the second ; other lines were diminished in intensity but not removed. The suppressed lines were those of foreign constituents.

The density and specific heat of distilled metals were re-determined, a cylinder of 1·5 c.c. volume being cast and turned from metal which had been distilled *in vacuo*, and then submitted to pressure for the purpose of determining the density. A copper cylinder was immersed in castor-oil and subjected to various pressures (on all sides) up to 20,000 atmospheres, the density being taken after each successive increment. At the highest pressure the metal was completely plastic. Whilst the density of ordinary copper increased at first rapidly, afterwards slowly, with each rise of pressure, that of distilled copper at first increased and then fell off. Hence it is assumed that the density is not a specific property of a substance, but the individual property of a given specimen. The specific heat is analogous, decreasing with increasing pressure, but the decrease is very slight.—W. G. M.

Blast Furnace; Calculation of the Composition of the Gases from, and of the Volume and loss of Blast in the ——. B. Osann. Stahl u. Eisen, 1901, **21**, 905. Through Chem.-Zeit. 1901, **25**, [82], Rep. 295—296.

Analysis of the flue-gases does not always give correct indications of the chemical reactions taking place in the furnace ; and consecutive tests may give results which differ markedly, without apparent cause, but really by reason of the fluctuation in the oxidising and reducing reactions of the furnace. The process of calculation used gives an average result ; it relates to the proportion of CO_2, CO, N, and H_2O in the flue gases. In the original paper a practical example is taken ; the composition of the gases is calculated, as also the heat required per 100 kilos. of pig-iron, the heat introduced with the blast, and the volumes both of blast used and of gases produced. A number of thermal balance-sheets are given, and a table is introduced showing the quantity of heat w, and the values of x and y (the proportions of CO_2 and CO), and the quantity of combined oxygen in the gases. Further calculations show the value of the oxygen available from the reduction of the ores. There is also a table showing the composition of the gases when different proportions of coke are used. In calculating the volume of blast used, the author recommends that the quantity of air blown into the furnace be referred to the ton of coke burnt, instead of using the proportion of the number of revolutions of the blowing engine to the quantity of pig-iron produced daily. Finally he calculates the loss of blast in the blowing engine.—W. G. M.

Cast Iron ; Influence of Aluminium on the Carbon in ——. Melland and Waldron. Chem.-Zeit. 1901, **25**, [82], Rep. 296.

Various proportions of aluminium (of 99 per cent.) were melted with pure Swedish charcoal pig in crucibles and poured into two ingots, one of which was cooled quickly, the other slowly. About 1·4—1·8 kilos. were used in each experiment, the charge being melted under charcoal. Up to 2 per cent. Al, aluminium was without influence on the proportion of carbon present, but with from 2 to 11 per cent Al, there was a reduction of 0·13—0·39 per cent. in the total carbon. In the rapidly-cooled specimens, 0·23 per cent. or less of Al was without influence on the proportion of graphite ; the separation of graphite then increased as the Al rose to 0·53 per cent., and then remained constant up to 1·78 per cent. Al, after which it decreased again. In the slowly cooled ingots, the effect of small proportions of Al was found to be irregular ; with from 0·23 to 1·78 per cent. Al, the metal was grey, and contained 3 per cent. of graphite, but with large additions of aluminium the graphite percentage was lower, being only 0·2 per cent. when 11·8 per cent Al was present. Aluminium cannot, like silicon, cause the separation of all the carbon present in the form of graphite. The quickly-cooled bars were very fine-grained and quite unlike the others in texture. A carbide, Al_4C_3 was found to have been formed at temperatures below the melting-point of cast-iron, a circumstance which may account for the low proportion of graphite in the high aluminium alloys. No hydrocarbon was however evolved from the finely-divided metal, and the examination of the etched polished surface gave no indication of its existence. The original strength of the metal was lowered by 60 per cent. by the addition of 0·25 per cent. Al. The metal containing 3·8 per cent. of Al bore a crushing stress of 121 kilo. per square millimetre even with the skin of white-iron removed.—W. G. M.

Open-Hearth and Converter Process ; A Combined ——. Stahl u. Eisen, 1901, **21**, 524. Proc. Inst. Civil Eng. 1901, **146**, [4], 41—42.

It has been observed at Nischne Salda in the Ural, that the pig iron smelted from very pure magnetic ores with charcoal is so low in silicon that, with the converter alone, it is impossible to bring out the charge at the proper heat. The difficulty has been overcome by first passing the metal through a Siemens regenerative furnace, where its temperature is raised to over 1,400° C. It is stated that by increasing the initial heat in the metal in this way, the time of blowing in the converter is shortened, and the charge is finished at a high temperature, the combustion of silicon going on through the whole period, instead of being completed in the first few minutes. The following table shows the nature of the changes which occur.

——	Silicon.	Manganese.	Carbon.
Blast-furnace metal ...	0·75	2·6	3·7
Metal superheated in Siemens furnace.	0·70	2·01	3·65
Metal blown 3 mins. in converter.	0·56	1·31	2·50
Metal blown 6 mins. in converter.	0·47	1·31	1·80
Metal blown 9 mins. in converter.	0·43	1·01	1·05
Metal blown 12 mins. in converter.	0·33	0·65	0·53
Metal blown 14½ mins. in converter.	0·026	0·31	0·25

If the silicon in the pig iron exceed 1 per cent., sufficient steel scrap is added in the open-hearth furnace to reduce it as nearly as possible to that amount. The table on next page shows analyses of such a charge at different periods.

When the silicon in the superheated metal amounts to 1·2 — 1·4 per cent., additions of 10—12 per cent. of cold scrap during the blow in the converter are necessary to control the heat developed. For the production of 1 ton of ingots by this method, there are required 1·1 tons of pig

——	Silicon.	Manganese.	Carbon.
Pig iron, 2·6 tons	1·96	3·45	4·40
Scrap, 1·3 tons	0·021	0·20	0·45
Bath (calculated)	1·31	2·37	3·92
„ (actual)	1·18	1·72	2·28
After blowing 4 mins. .	0·68	1·12	1·34
„ 8 „ ..	0·35	0·80	0·70
„ 12 „ ..	0·018	0·125	0·28
After addition of spiegel	0·02	0·275	0·33

iron and 1·18 cb. m. of wood consumed in the gas-producers of the Siemens furnace.—A. S.

Iron and Steel, Internal Strains of ——; and their Bearing upon Fractures. A. Wingham. Iron and Steel Institute, Autumn Meeting, 1901.

The author puts forward a series of hypotheses in an attempt to elucidate the sources and the character of the internal strains of metals generally, but more especially of iron and steel, and to throw light upon the cause of the sudden and unexpected breakages of metal used for machinery and for structural purposes.

A given mass of crystalline material tends to become more largely crystalline, and the crystals to become more compact, and any excess of the mother mass or eutectic existing between the minute crystals, which are first formed and which go to build up the larger crystals, must, when these are brought into closer contact with one another, be squeezed out into the larger channels which constitute the films between the facets of the larger crystals. During the slow cooling, after solidification, of a piece of steel, its constituents will tend to assume the simplest forms of chemical combination preferentially existing at normal atmospheric temperature, and its crystalline character will be developed to the greatest extent compatible with the existing conditions. The cooling, however, being more rapid than the process of adjustment, complete equilibrium is not attained at any given temperature, and hence, in the final condition of coldness, there exists in the metal a certain amount of latent effort to adjustment, which may be considered as a potential disruptive force. The metal, although apparently adjusted to the general conditions of pressure and temperature, &c., is gradually and persistently endeavouring to become more crystalline, and is accumulating an internal expansive pressure. This internal pressure is accentuated, and the tendency to readjustment favoured by various conditions, such as changes of temperature, vibration, oscillation, revolution, and jolting or jarring, especially of a tensile character, to which the metal is subject during its industrial life. The readjustment which takes place constantly, though slowly at the ordinary temperature, proceeds more rapidly under the accelerative influence of heat, so that a metal used at steam heat will develop, in a much shorter time, internal strains leading to flaws, and finally to fracture.

Ewing and Rosenhain (this Journal, 1900, 1115) have observed that the crystals of a piece of lead when strained under compression beyond the elastic limit, grow with arms and branches thrown out, and producing spaces between them, which, at a later stage, become filled up to form the face of the crystal. It is maintained that the medium for the development of this and other fern-like growths which have been observed, must be the eutectic; and if, from any cause the crystals are not completed, the metal becomes a mass of crystals welded or cemented by interpenetration with the eutectic. This hypothesis is stated to offer an explanation of the modifications of the character of a metal by mechanical treatment, such as the breaking down of crystalline structure and the production of a closer texture by hammering, &c., the interpenetration and binding together by welding and soldering, the power to bend, the disruption under stress, &c.

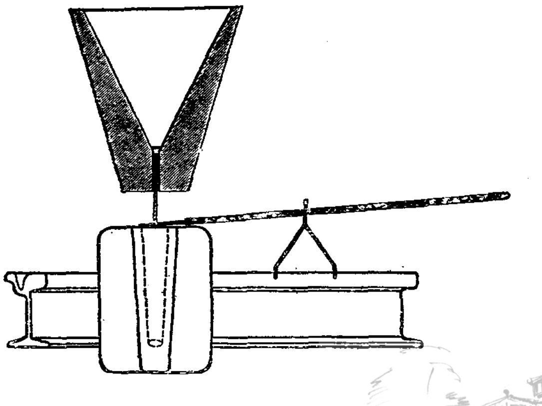
It is concluded that a metal, in order to possess durability and stability, should contain those constituents which do not lend themselves to after-crystallisation or separation. The fluidity or mobility of the eutectic is important in this respect, and the best conditions are fulfilled when the eutectic has a solidifying or rigidity point not far removed from that of the crystals which separate from it. In this way the minute crystals first formed quickly become cemented with the solidified eutectic, and a metal of fine grain texture results. This should be accomplished, however, with the minimum possible quantity of eutectic. The high elastic limit and strength of steels containing metals like nickel, &c., may be explained by the formation, with the less fusible nickel, of a high melting eutectic, which solidifies and hardens soon after the separation of the minute crystals of steel or nickel steel. The nature and number of the non-metal constituents of a complex metal also considerably influence the character of the eutectic; in general, these should be kept down to the lowest possible limit, so as not to produce any superfluous eutectic, which would facilitate the readjustment to crystalline form.

The effects of the various methods of thermal treatment of steels are explained in the following manner:—With sudden cooling to normal temperature, the natural preferential separation of the constituents is prevented, and there results a complex metal which is itself hard. The effect of annealing is to allow the natural stages of separation to take place, partly or wholly, according to the temperature and time occupied in the process. The effect of tempering is to allow sufficient release of the eutectic to take place to have a binding action upon the remaining hard particles, so that the brittleness resulting from the absence of a cementing agent is destroyed, whilst the hardness is not entirely obliterated, and, consequently, a tough and flexible steel is obtained.—A. S.

Welding Process: Automatic Tapping Arrangement for the Thermite ——. H. Goldschmidt. Zeits. für Elektrochem. 1901, **7**, [68], 935—943.

The author describes an improvement in the process for welding rails, broken shafting, &c. by means of the high temperature generated by the combustion of a mixture of aluminium powder and oxide of iron, "thermite." (See this Journal, 1899, 870, and 1900, 908.)

Formerly the weld was effected by pouring the contents of the crucible, consisting of an upper layer of molten corundum and a lower layer of molten iron (temperature about 3,000° C.) into the mould surrounding the parts where the joint was to be made. In this way the molten corundum first came in contact with the parts to be welded and sometimes gave rise to a bad joint. In the improved process, the "thermite" is fired in a conical crucible, lined with refractory material and provided with a small slightly tapering orifice in the bottom. This orifice is closed, before charging the crucible, by means of an iron disc, covered if necessary by a layer of sand, the thickness of which depends on the quantity of "thermite" being handled. A few seconds after the combustion is complete, the white hot molten iron which has collected at the bottom, melts the iron disc and is automatically discharged into the mould. A further improvement consists in allowing the molten iron to flow to the bottom of the mould by means of a specially provided channel and then to rise up round the joint which is to be made. If necessary a bye-pass can be made between this channel and the main chamber of the mould to allow



the molten corundum to flow over the upper part of the joint.

The paper is illustrated by seven figures, and detailed instructions and dimensions are given which are applicable to given conditions. The accompanying figure shows the general arrangement and also the means provided for tapping the crucible if for any reason the iron disc should not melt through after the combustion is complete. This consists of an iron rod, placed in the discharge orifice, which, when raised by means of the lever, displaces the disc. The illustrations refer chiefly to the welding of electric tramway rails and a ten-inch shaft.—J. S.

Cementation [Iron] of Finished Goods. C. W. Bildt. Chem.-Zeit. 1901, **25**, [82], Rep. 296. From Oesterr. Zeits. Berg- u. Hüttenw. 1901, **49**, 504.

The objects to be cemented are packed at some distance from one another—the distance depending upon the size of the objects and the degree of cementation required—in a mixture of 60 per cent. of wood charcoal and 40 per cent. of bone meal. They are placed in chests which are kept tightly closed throughout with a cover of loam, and heated to 1,110°—1,200° C. The objects should be made of refined-iron or of good mild steel, low in carbon, but not of Swedish iron, which, on account of the slag, contained in it, is liable to form blisters. The cemented steel goods do not require re-melting; they may be hardened and tempered, and will bear hammering hot or cold. For cutting-tools, the resulting material is said to be better than English cast-steel. Objects may be locally hardened by covering the parts not to be affected with clay. —W. G. M.

Soldering Experiments with the so-called Solder Paste "Ferrofix" for Cast Iron. Mittheilungen aus den Königl. t. Versuchs-Anst. zu Berlin, 1901, **19**, [2], 86—90.

The process of F. Pich, of Berlin, is described in Ger. Pat. 110,319. The surfaces to be soldered are freed from graphite during the process, and at the same time molten solder is brought into contact with the graphite-free cast-iron surfaces at a red heat in the furnace.

The solder consists of a paste of cuprous oxide, and borax as a flux. The latter melts on heating, and protects the surfaces and the cuprous oxide from oxidation. Stronger heat is then applied, and the cuprous oxide gives up its oxygen to the graphite, forming carbon monoxide and dioxide, and the metallic copper is left in a finely divided condition, and combines with the hard solder applied to the edges and unites them. The results of the experiments show that with care it is possible by this process to form soldered joints of cast-iron, practically equal to the material itself in strength.—W. C. H.

Ferro-Manganese; Cause of the Disintegration of —— when exposed to the Open Air. R. Dubois. Bull. Assoc. Belge des Chim. **15**, (7), 281—286.

The author reports an instance where a pile of ferro-manganese, left exposed to the open air for a couple of months, was found to have suffered disintegration, the mass shrinking to about one-half its original bulk. It was at first thought that this change was the result of oxidation, assisted by the action of carbon dioxide and water vapour, the more so, because the same change was particularly noticed in some pieces of the alloy lodged on a steam pipe. The chemical examination of the powder, however, showed that such was not the case, but that the alteration was due to the weathering of the portions richest in manganese, the powder containing 82·17 per cent. of Mn and 7·43 per cent. of Fe, compared with 79·99 and 11·94 per cent. respectively, in the original sample. Moreover, the presence of 0·135 per cent. of moisture in the weathered sample is sufficient to indicate that the disintegration could be effected by the mechanical action of frost.

As, in its altered condition, the ferro-manganese is practically useless for its intended purpose in the converter, it is evident that care should be taken to store it under cover.—C. S.

Nickel Steel, The Metallurgy of ——. A. W. Zdanowicz. Stahl und Eisen, **21**, 753; through Zeits. angew. Chem. 1901, **14**, [44], 1112.

According to Browne and Porter, the nickel steel used for shafting and other forgings contains 3·25 per cent. of nickel and 0·25—0·35 per cent. of carbon. The author has obtained good results with less than 2 per cent. of nickel, adding pure nickel just after re-carburisation; in many works the ferro-manganese and ferro-silicon are introduced into the bath at the same time. For shafts and forgings the bath should be fined down as far as possible; too high a percentage of manganese and silicon has a bad influence on the tenacity of nickel steel.

One of the main conditions in forging nickel steel is the possession of a sufficiently powerful steam hammer or hydraulic press, otherwise the texture of the interior remains crystalline, and the forging is crude and liable to crack diagonally. The effect of the blows must penetrate to the heart of the mass, and produce a blending of the metal and the pressure must be continued for a sufficient length of time; hence the hydraulic press is most suitable.

On cooling, a recalescence point occurs between 700° and 650° C., and a change of structure takes place; crystallisation of the steel begins as it solidifies, and the crystals are larger the more slowly it is cooled. At the recalescence point the crystallisation seems to stop; if the block be heated to a temperature above the recalescence point, smaller crystals will be found on cooling; by overheating the crystals are destroyed, and an amorphous structure is produced. According to Souther, an increase of 0·25 per cent. of carbon lowers the recalescence point of ordinary steel 138° C., and 5 per cent. of nickel lowers it 302° C.; nickel, therefore, has a considerable influence on the critical temperature of steel, the duration of recalescence is nearly proportional to the percentage of nickel. The nickel, therefore, probably forms at the critical temperature definite compounds with the iron or carbon, perhaps with both.

In heating or annealing, the temperature of the block must be kept slightly below the recalescence point. In order to compensate for the stresses produced in forging, and for the molecular disturbances, and to increase the contraction and elasticity, the annealing of the forgings is absolutely necessary. Unannealed forgings are liable to bend in working; the greatest care must be paid to the temperature whilst annealing. The nickel steel prepared by the author for shafts and forgings showed a tenacity of 55—60 kilos. per sq. mm. and 20 per cent. extension, with a good bending test. The unannealed pieces could be bent almost on themselves, and the fracture had a good, fibrous structure, whereas pieces annealed at temperatures above the recalescence point broke at a very small angle, and showed a totally altered structure.—J. F. B.

Cyanide Plant at the Athabasca Mine, British Columbia. Eng. and Mining J. 1901, **72**, [16], Canadian Supplement, 515.

The plan followed, comprises :—(1), Settling in tanks; (2) straight percolation, assisted by vacuum; (3) deposition on zinc shavings; (4) acid treatment for refining the product. The plant, which is capable of treating 35 tons daily, consists of one stock solution tank, diameter 4 ft., stave 6 ft.; two solution tanks, diameter 10 ft., stave 6 ft.; two settling tanks, diameter 14 feet, stave 10 ft.; five leaching tanks, diameter 18 ft., stave 4 ft.; two gold solution tanks, diameter 10 ft., stave 6 ft.; 24 zinc boxes, each 1 cb. ft. capacity; one clean-up tank, diameter, 6 ft., stave $2\frac{1}{4}$ ft.; one clean-up tank, diameter, 4 ft., stave 6 ft.; one vacuum filter box; one Hampton zinc lathe, one solution pump; and one vacuum pump. The tailings pass directly into the settling tanks, which are provided with automatic distributors, annular launders for overflow, and three side discharge doors each. The leaching tanks are fitted with filters and centre discharge doors, and are so arranged that the tailings can be sluiced out through the bottoms of the tanks. The zinc boxes are square sheet iron buckets; each one is independent of the others and can be handled by means of an iron bail. —A. S.

Gold Halides. F. Lengfeld. Amer. Chem. J. 1901, **26**, [4], 324—332.

By the repeated action of bromine upon finely divided gold, the chief product is aurous bromaurate, Au_2Br_4, mixed with some aurous bromide or gold (probably the latter). The bromaurate is a steel-blue solid, stable in the absence of water, but easily decomposed at the ordinary temperature by water and many organic solvents.

When chlorauric acid is left in a moderately dry atmosphere the compound $HAuCl_4.3H_2O$ is obtained. If this be kept *in vacuo* over phosphorus pentoxide, loss of water of crystallisation is accompanied by decomposition. On standing in air it rapidly deliquesces, but attempts to identify a definite compound failed. It was not found possible to isolate $HAuCl_4.4H_2O$, which Thomsen believed to exist.

Bromauric acid has the formula $HAuBr_4.6H_2O$, and not $5H_2O$ as stated by Thomsen. Kept over drying agents it loses three molecules of water of crystallisation, but can lose no more without decomposition.

In investigating the silver salts of halauric acids, the action of chlorauric acid upon silver carbonate was found to be a complicated one. Among the products formed are silver chloraurate, silver chloride, and auric chloride.

The main reaction between potassium chloraurate and silver carbonate, whether in aqueous or alcoholic solution, appears to be: $2KAuCl_4 + Ag_2CO_3 = K_2CO_3 + 2AgCl + 2AuCl_3$.—E. H. T.

Sulphide Ore Treatment (Phœnix Process). E. A. Ashcroft. Trans. Inst. Min. and Met. **9**, 378—418.

The Phœnix cyclic process is the joint invention of the author and Mr. J. Swinburne, and is conducted in four stages.

1. The mixed or complex sulphide ore is treated with dry chlorine gas at a temperature above 600° C. in a large tipping converter of the Bessemer type, whereby the chlorine displaces the sulphur and other elements combined with the metal and forms metallic chlorides. The crushed ore is introduced at the top of the converter into a bath of fused zinc chloride and chlorine is injected at the bottom, the temperature being such that free sulphur is produced and volatilises, together with a little sulphurous acid, for condensation and recovery. Chloride of sulphur is produced only at temperatures below 600° C. The decomposition is rapid and complete, the heat set free maintaining the bath at the proper temperature. Additions of ore are made until the converter is filled with fused chlorides, part of which is run out for the next treatment, whilst some is left in to form a bath for further quantities. Neither the silicates of iron and manganese in the ore nor the converter lining are attacked, the former remaining with the gangue suspended in the chlorides. The time for a trial treatment of 1,000 kilos. of ore, requiring four pourings, was 12 hours. The average temperature was 650° C., the chlorine consumed 470 kilos., and the sulphur condensed 100 kilos out of a total quantity of 210 kilos., the remainder having oxidised. The weight of the fused chlorides, chiefly lead and zinc with suspended gangue, was 1,260 kilos. All the gangue, and 92 per cent. of the total chlorides, remained in the converter, the remaining 8 per cent., chiefly of iron and zinc, being found in the condenser with the sulphur, from which it was separated by washing in water.

2. In this stage the lead is removed from the fused chlorides by the action of metallic zinc at about 450° C., any gold or silver having previously been thrown out by metallic lead. Any copper or antimony may similarly be removed. The separations may likewise be obtained by fractional electrolysis, in which case chlorine is set free and has to be recovered. Any iron which might be present to interfere with the ultimate electrolysis of the zinc chloride, can be removed at a stage in the converter treatment by blowing in air, whereby it is thrown out and remains suspended as oxide for separation in stage 3.

3. The heavy sands and oxides of iron and manganese are allowed to settle by standing in iron pots at a little below 400° C., and the pure fused zinc chloride decanted as far as possible. The residue may be distilled to complete the removal of the chloride, but preferably it is dissolved out by water and the solution evaporated until the fused salt is obtained. Alternatively, to obtain the oxides of iron and manganese free from gangue, they may be thrown out from the chlorides by a supplementary air treatment after the gangue has been removed as above (this Journal, 1901, 907).

4. The final stage is the electrolysis of the pure zinc chloride, and this may be effected in three ways: in solution with diaphragms; fused and externally heated; and fused and internally heated by the current. Of these the last named is preferred as being less expensive than the first, and without the difficulty of materials for the vessels in the second, since the fused chloride in the centre is surrounded by solid and cooled layers. The apparatus consists of a steel tank 6 ft in diameter, lined with 18-in. fire bricks, the inner layers being set in a special cement. The cathode is a mass of molten zinc, of approximately 1 ton, in the bottom of the tank, and connection is made by a steel block buried in the brickwork, and provided with five screwed copper tubular rods, through which the current enters, and through which air circulates to prevent overheating. The anode consists of 120 rods of hard carbon dipping about 6 ins. into the electrolyte, and suspended from a removable cement-lined iron plate. The zinc is tapped by removing a carbon plug, and fresh fused chloride poured in from above. The carbons last six months or longer. The purity of the zinc depends on the previous purification of the chloride, but it has practically been obtained with only 0·15 per cent. of lead and 0·02 per cent. of iron. The vats are sealed from the air to prevent dilution of the chlorine, which is drawn off for re-use in stage 1. The best results are obtained with the temperature between 425° and 525° C., and it is found advantageous as regards conductivity to have sodium or calcium chloride present in the electrolyte to an extent resulting in not more than 28 per cent. of zinc, instead of 48·5 per cent. for the zinc chloride alone. These chlorides are not decomposed and do not require renewing, but rather have a tendency to accumulate by the introduction of small quantities with the zinc chloride, a periodical removal of part of the electrolyte being then necessary. The chlorine gas evolved, is dried in towers packed with zinc chloride, and liquefied or kept under pressure, the oxygen with which it was diluted being then, with some of the chlorine, used for oxidising and precipitating the iron in the converter.

The working cost will depend largely upon the cheapness of power, of which the process is a large consumer, the computation at 2·2 volts (theoretically calculated) required for a ton of average ore, yielding about a quarter of a ton of zinc, resulting in 495 kilowatt-hours. Estimated items of cost are given based upon present experience. The merits claimed for the process are the high percentage of recoveries, the absence of undesirable by-products, the absence of wear and tear on the converter and vat linings and carbon anodes, and the general simplicity with the economy of automatic working.—A. W.

Tin Ores, especially Silicates or Tin-Slags; Lixiviation of——. Brandenburg and Weyland, Kempen. Ger. Pat. 123,764, Jan. 22, 1901. Through Zeits. angew. Chem. **14**, [43], 1095.

The ores or slags are boiled with an aqueous solution of sodium bisulphate, the solution filtered from the separated gelatinous silica, and worked up for tin or tin compounds.
—J. T. D.

Tinfoil and Bottle Caps Manufacture. R. Granja.

See page 1191.

Metals and Alloys; Analysis of ——. F. Ulzer. Mitt. d. tech. Gew. Museums, Vienna, 1901, **11**, [7, 8, 9, and 10], 162—165.

The alloy known as Namo metal consisted of: lead, 0·27; copper, 12·98; tin, 83·27; antimony, 3·15; iron, 0·25 per cent., and traces of phosphorus; whilst a sample of Babbit's metal gave: lead, 40·69; copper, 6·48; tin, 43·76; antimony, 8·82; and iron, 0·12 per cent. The alloy Magnolia had the following composition:—Lead, 76·60; antimony, 18·24; tin, 4·91; copper, 0·064 per cent., and a trace of iron. A phosphor bronze contains: lead, 14·62; copper, 4·58; antimony, 7·37; tin, 73·08; phosphorus, 0·12 per cent., and traces of iron.—T. A. L.

Nickel Alloys. H. Wedding. Verhandl. des Vereins zur Beförd. des Gewerbfleisses, 1901, 143. Proc. Inst. Civil Eng. 1901, **146**, [4], 50.

For the results obtained with nickel-iron alloys see this Journal, 1901, 127. Experiments dealing with the preparation of alloys of the four elements, iron, nickel, manganese, and carbon, showed that the proportion of manganese in the finished alloy did not at all correspond with the amount originally weighed out. In some of the casts, instead of 0·5 per cent. of manganese, there was, on the average, only 0·15; in others, instead of 1, there was only 0·33 per cent. In order to produce an alloy containing the proportion desired, it is proposed to melt the manganese first, and then introduce it into the mixture of the other metals.—A. S.

Nickel, Copper, and Aluminium; New Alloys of ——. Häntzschel. Verhandl. des Vereins zur Beförd. des Gewerbefleisses, 1901, 5. Proc. Inst. Civil Eng. 1901, **146**, [4], 49.

"*Nickel-aluminium.*"—In the production of this alloy, the difficulty experienced in making alloys of nickel and aluminium, owing to the great difference between the melting points of the two metals (1,450° and 600° C. respectively) has been overcome by the addition of copper, the melting point of which is intermediate between those of nickel and aluminium. The alloy obtained is thus really one of aluminium, nickel, and copper, consisting for the most part, however, of aluminium, as is shown by its low specific gravity—2·86. "Nickel-aluminium" can be rolled into plates and bars; in six tests, it showed an average ultimate tensile strength of 25·9 kilos. per sq. mm. (16·44 tons per square inch).

"*Minckin.*"—This is an alloy similar to, but containing more nickel than the so-called German silver; the latter contains about 26 per cent. of nickel. It is chiefly useful on account of its resistance to the corrosive action of weak acids and alkalis.—A. S.

Nickel-coloured Alloy; Manufacture of a ——. M. Ekker and J. Krajcsics, Budapest. Ger. Pat. 123,750, Sept. 25, 1900. Zeits. angew. Chem. **14**, [43], 1095.

The following ingredients are placed in layers in a crucible:—Copper, 375; phosphor-bronze, 2; nickel, 700; magnesium powder, 8; copper, 375; aluminium, 2; zinc, 800; cadmium, 15; zinc ashes, 2; copper, 750. After melting, 2 parts of powdered sal-ammoniac are well stirred in. The alloy does not oxidise in the air, keeps its lustre at 100° C., and resists corrosion by acids.—J. T. D.

Silver-coloured Alloy; Manufacture of a ——. M. Ekker and J. Krajcsics, Budapest. Ger. Pat. 123,919, Sept. 25, 1900. Zeits. angew. Chem. **14**, [43], 1095.

The following ingredients are placed in layers in a crucible:—Nickel, 600; copper, 2,000; zinc, 400; magnesium powder, 10; cadmium, 50; zinc ashes, 2; aluminium, 1. After melting, 1 part of powdered sal-ammoniac is well stirred in. The alloy does not oxidise in the air, keeps its lustre at 100° C., and is very resistant to acids.

—J. T. D.

Aluminium and Copper; Alloys of ——. L. Guillet. Comptes Rend. **133**, [18], 684—686.

Aluminium-copper alloys were formed by burning a mixture of metallic aluminium, finely granulated, with copper oxide; when the amount of aluminium was greater than that corresponding with the production of the substance $CuAl_4$, the mixture would not ignite. After reaction and cooling, the crude button was analysed, and also the residue (usually crystalline) left after treating it with dilute hydrochloric acid. The three compounds (Cu_3Al, $CuAl$, $CuAl_2$) described by Le Chatelier were all isolated. With the compound CuAl there was obtained always 2 or 3 per cent. of a silicide of copper and aluminium.—J. T. D.

Copper; Precipitation of ——, by Ferrous Salts. H. C. Biddle. Amer. Chem. J. 1901, **26**, [4], 377—382.

Investigation of the reduction of copper salts by ferrous salts in aqueous solution shows that the reaction is a reversible one, and that its direction is determined by the relative concentration of the ferrous, ferric, cuprous, and cupric ions. Theory indicates that copper will not be deposited in a solution containing many ferric ions (either originally present or formed during the reaction), and this is confirmed by the fact that ferrous chloride, or sulphate, does not precipitate copper from the corresponding copper salts, however long they may be kept in contact. On the other hand, reduction does take place if few ferric ions be present; *e.g.*, when an excess of ammonium carbonate is added to a solution of ferrous and cupric chlorides, the yellow liquid obtained, deposits, on standing, a slight but brilliant mirror of copper. The influence of the concentrations of the ions is also shown by the fact that if an excess of sodium carbonate be added to a solution containing cupric and ferrous salts, there is no deposition, but if only a slight excess of the carbonate be employed, the precipitated carbonates gradually change to a brick-red precipitate containing copper and basic ferric carbonate. Potassium bicarbonate reduces in a similar way, whether in slight or great excess; this is because the acid ferrous and cupric carbonates are more soluble than the corresponding normal salts. Their solubility is increased under pressure; consequently, if the action take place in a closed vessel containing carbon dioxide, the deposition takes place more readily. Copper from the cuprous state may be reduced in a similar manner.

—E. H. T.

Tungsten and Copper; Aluminium Alloys containing ——. Carl Berg, Eveking. Ger. Pat. 123,820; addition to Pat. 82,819 of Jan. 18, 1894. Through Zeits. angew. Chem. **14**, [43], 1095.

Tungstic acid is reduced, using cryolite as a flux, and aluminium is added to the melted metal so as to produce an alloy containing 10 per cent. of aluminium. Another alloy of aluminium with electrolytic copper and zinc is made, and the two alloys are melted together in such proportions that the resulting alloy may contain 91 to 92 per cent. of aluminium and 2 to 7 per cent. of zinc.

—J. T. D.

Platinum and Iridium; Determination of —— in Platinum Ores. Leidié and Quennessen.

See under XXIII., *page* 1242.

Furnace Combustion; Phenomena of ——. O. Boudouard.

See under II., *page* 1196.

Pyrite and Marcasite, and Methods of Distinction. H. N. Stokes.

See under XXIII., *page* 1241.

Vanadium: Its Extraction and Uses. H. Procter Smith.

See page 1183.

PATENTS.

Alluvial Deposits; Extraction of Metals and Minerals from ——. J. Mactear, London. Eng. Pat. 19,150, Sept. 25, 1901.

This is a kind of continuously working cradle for treating alluvial deposits or accumulations of gold, tin, and other metals and minerals, &c.—J. H. C.

Tin: Recovery from "Hardhead" or Slag. E. M. Bradford, Neath Abbey. Eng. Pat. 19,592, Nov. 1, 1900.

Sulphur (or a suitable sulphate or sulphide) is added to the material to be treated, and the mixture is strongly heated in a blast or cupola furnace, with or without the addition of lime or an alkali. The tin is volatilised as oxide and collected as fume.—J. H. C.

Iron and Steel; Process for Toughening, Hardening, or Annealing ——. W. Holzer, New York and London, and W. F. L. Frith, London. Eng. Pat. 13,352, July 24, 1900.

The metal is toughened by being heated in a bath of mercury within a closed receptacle, and allowed to slowly cool while in the bath. The temperature may be about 1,000° F. Further, an electric current may be passed through the bath while the metal is under treatment, *i.e.*, during the heating or cooling, which current should be intermittent.—A. W.

Steel; Production of ——. A. J. Boult, London. From E. Goldschmid, Frankfurt a/M., Germany. Eng. Pat. 21,552, Nov. 28, 1900.

The process is for the production of steel in Martin's furnaces, and consists in the application of a water-gas flame for heating, and fusing the charge in a twin furnace or a furnace with two hearths, so that the gases escaping from the first hearth pass over and heat the charge on the second hearth, the preliminary heating of the water-gas being unnecessary. The furnace can be arranged so that after tapping and recharging the first hearth, the air through the regenerator and the gas can be reversed, and the charge in the second hearth worked, whilst the gases therefrom pass over and heat the fresh charge on the first hearth.—A. W.

Iron or Iron Alloys; Manufacture of ——. F. C. Crean, Montreal. Eng. Pat. 17,639, Sept. 3, 1901.

The iron or alloy is made by mixing magnetite, such as black sand which has been freed from silica, &c., with molten cast iron or other suitable metal, the proportions varying according to the quality of metal required. The mixing may be done in a ladle by filling a tube, having a funnel opening at its upper end, with the magnetite in a loose granular state, passing its lower end to the bottom of the molten metal and gradually raising it to the surface, whereby the magnetite gradually falls into and becomes mixed with the metal.—A. W.

Iron; Manufacture of ——. A. Simon.
Eng. Pat. 20,513, Nov. 14, 1900.
See under XI. B., *page* 1221.

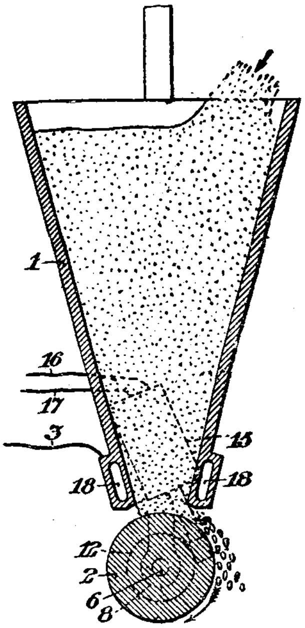
Gold; Extraction of ——, *from Sea-Water or other Solutions containing the same.* J. F. Duke, Downe. Eng. Pat. 22,389, Dec. 8, 1900.

The solution containing gold is submitted to the action of aluminium hydroxide, a gelatinous silica compound, or other substance, such as that obtained by neutralising an aluminium salt or a tin salt with an alkali, these substances reacting with the gold haloid salt to free the gold, and afterwards exerting a fining action upon the solution by settling and carrying down the gold. The gold may also be separated from the solution by the action of substances, such as protochloride of tin, used in conjunction with the aluminium hydroxide, &c., for the said purpose of rapid deposition.—A. W.

Ores and Concentrates [*Fe*]; *Methods of and Apparatus for Agglomerating Comminuted* ——, *and for reducing Metal therefrom.* M. Ruthenburg, Philadelphia, U.S.A. Eng. Pat. 13,867, July 8, 1901.

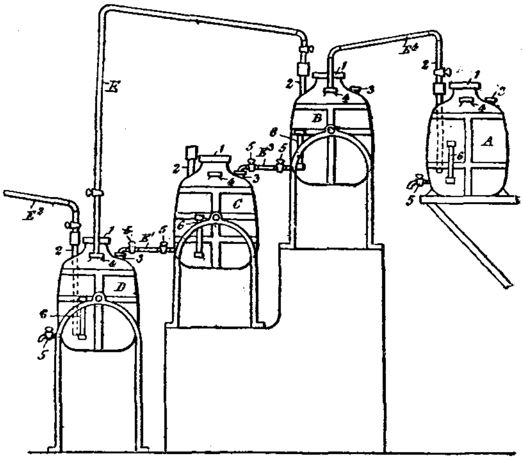
The comminuted ore or concentrate, either alone or mixed with a fluxing medium or a reducing agent or both, is brought together in the path of an electric current, whereby the particles of the mass are fritted and perhaps partially reduced, the product then being removed from the action of the current in coherent lumps suitable for smelting. The lumps may be conveyed to an enclosure and maintained at a temperature sufficient to complete reduction or until the effects of the reducing agent are equalised throughout the mass.

The apparatus, which may be constructed according to the accompanying sectional drawing, consists essentially, in combination or separately, of the parts as follows:—A hopper, 1, a cylindrical roller, 2, placed opposite the discharge of the hopper, the said roller being fixed upon a shaft, 6, which is mounted for rotation in bearings, and held upon, but electrically insulated from the hopper. The hopper and the roller are connected with and form the terminals of an electric current, so that the ore mixture in its descent passes through the heating circuit and is fritted by the action of the current into lumps which are discharged by the revolution of the roller. The roller revolves by means of worm gearing in engagement with a worm, 12, on the end of the shaft, and connected with an outside motor, 15, which motor may be electrically actuated through the conductors, 16, 17. The hopper may be water-jacketed at its lower end, 18, and is attached direct to the electrical conductor, 3. The current is conveyed to the other terminal or roller, 2, through the shaft, 6, from an outside collar attached thereto, and connected by a contact brush with the other conductor.
—A. W.



Copper Ores; Treating ——, *and Apparatus therefor.* R. Seeman, Ealing. Eng. Pat. 18,864, Oct. 22, 1900.

The treatment consists of leaching copper ores containing carbonate of copper or native oxide with a solution of ammonia, drawing off the clear ammoniacal solution of copper, and then distilling off the ammonia, the process being conducted in vessels of which the parts in contact with the ammoniacal solution of copper are made of earthenware or material unacted on by this solution.



The plant claimed is outlined in its preferential form in the accompanying drawing. It consists of four different vessels: A the safety vessel, B the mixer, C the settler, and D the still. Each can be closed hermetically, and is provided at the top with a central filling-opening with cap, 1, three side nipples, 2, 3, 4, adapted both for plugs and connecting pipes, and at the lower side with taps, 5, and gauges or inspecting glasses, 6. The vessel A is stationary, but the other three are mounted at different levels on trunnions connected with a fly-wheel, so that when disconnected they can be rotated, rocked, or tilted. When connected up, the whole of the apparatus is closed except the nipple 3 in the vessel A. The powdered ore and ammonia solution are placed in B, which is then plugged and rotated until sufficiently leached. Whilst settling, the connections are made, and the solution is then decanted through E^3 into C, whence, after further settling, it is run through E^1 into D. The ammonia is then volatilised by steam entering D through E^2, and the vapour passed through the pipe E and condensed in the mixer B, which has been charged with fresh ore, any ammonia escaping therefrom through E^4 being absorbed by water in the safety vessel A. The residue in D is cupric oxide, and is washed out through the central opening. The residue ejected from the mixer B may contain copper sulphide, and is smelted by any known process.—A. W.

Lead; Process for the Dezincing of Zinc Desilverised ——. T. Barton and T. B. McGhie, both of London. Eng. Pat. 19,273, Oct. 27, 1900.

To remove zinc from metallic lead a layer of caustic soda or other alkali is fused on the surface of the molten metal, whereby the zinc is oxidised, and can be removed with the alkali. A current of steam or steam and air may be used to accelerate the oxidation, whilst the alkali can be repeatedly used, after the separation of the zinc oxide by skimming or dredging, since a small quantity only of the latter is dissolved.—A. W.

Zinc; Treatment of Slags and By-Products containing ——. H. F. Kirkpatrick-Picard, London. Eng. Pat. 15,166, Aug. 25, 1900.

The slags, &c., are ground, roasted to oxide if necessary, mixed with carbonaceous material suitable for coking, such as bituminous coal, preferably formed into briquettes, and distilled under such conditions that they are first coked into coherent masses, and finally the zinc reduced and volatilised, while the residual molten slag, which may contain recoverable metals, is retained in the remaining coke.—A. W.

Composition or Flux; A New or Improved ——, for use as a Substitute for Borax. Louise Alpe, Birmingham. Eng. Pat. 19,326, Oct. 29, 1900.

The composition is prepared by mixing 85 parts of boric acid with five parts each of barium chloride, cryolite, and graphite. The joint to be brazed is covered with the composition, heated to dull redness, more of the flux is dusted on, and at the proper temperature, brass wire or spelter is applied, which melts and flows through the joint.—E. S.

Aluminium or its Alloys; Coating of ——. A. G. Betts, New York, U.S.A. Eng. Pat. 10,705, Oct. 24, 1900.

The surface of the aluminium or alloy is covered with a layer of dry fusible non-oxygen salt of copper or other suitable metal, which is then heated to fusion.—J. H. C.

Solder for Aluminium, &c. J. A. McLeod and H. E. Prevost, Haverhill, U.S.A., and H. Smith, Lowell, U.S.A. Eng. Pat. 19,836, Oct. 4, 1901.

This consists of: zinc, 25; aluminium, 6; and tin, 69 parts by weight.

The zinc and the aluminium are melted together, after which the tin is added and stirred into the molten mass.

—J. H. C.

Aluminium Alloy. A. Manhardt, Vienna. Eng. Pat. 19,403, Sept. 28, 1901.

12·05 parts of tin, 0·20 of antimony, 0·09 of phosphorus, 7·50 of copper, and 0·06 part of magnesium are melted together, then 100 parts of aluminium are added little by little, and finally 0·06 part of bicarbonate of soda and 0·04 part of sulphur. After skimming off the slag, the alloy is ready for use.—J. H. C.

Mineral and other Substances; Apparatus for Pulverising and Separating ——. C. W. and H. H. Kitto. Eng. Pat. 20,040, Nov. 7, 1900.

See under I., *page* 1195.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

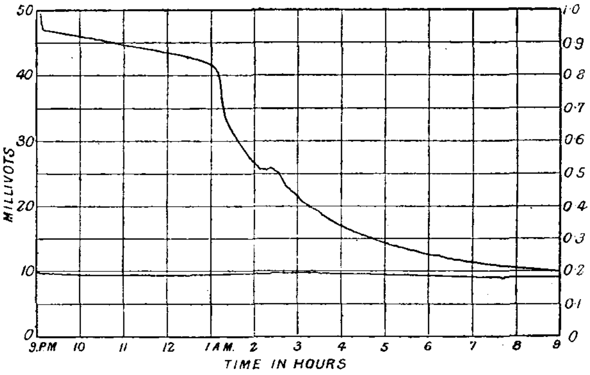
(*A.*)—ELECTRO-CHEMISTRY.

Cell; A Cadmium Standard ——. H. Tinsley. The Electrician, Oct. 18, 1901, **47**, [26], 991.

The author has designed a practical form of cadmium cell, based upon ideas advanced in a note by Prof. Callender, published in *The Electrician* of Sept. 10, 1897. The negative electrode of this cell is composed of an amalgam of cadmium and mercury suitably proportioned, above which is a layer of cadmium sulphate. Further above is the mixture of mercurous and cadmium sulphate crystals, in which is placed an amalgamed sheet of platinum. Connection to this sheet is made by a platinum wire, and the same metal is used to make electrical connection with the amalgam. A layer of glass wool comes above the layer of mixed crystals, and keeps the chemicals in place, while the cell is sealed with marine glue.

The advantages of this type of cell are, firstly, that the E.M.F. is invariable; secondly, that small currents can be taken from it without polarisation; and, finally, that if short-circuited by accident, recovery of the normal E.M.F. will occur without artificial aids. The constancy of the E.M.F. is shown by the following tests of six cells made on different dates, with chemicals from two different sources:—1·0191 v., 1·0192 v., 1·0191 v., 1·0192 v., 1·0190 v., 1·0191 v.

Below a typical discharge curve for this cell is shown. The internal resistance of the cell when passing a current of 1·8 milliampères, is seen from this curve to amount to only 40 ohms. If discharge be allowed to proceed at this rate for a longer period than four hours, a marked drop in the E.M.F. occurs, and complete recovery of the cell cannot be obtained without artificial aid.



The chief disadvantage of this type of standard cell is that the cadmium amalgam has a tendency to separate into its constituents when subjected to accidental jars. This drawback can be partially overcome by keeping the other constituents of the cell as solid as possible.—J. B. C. K.

Alkali and Chlorine; The Acker Cell, and Process for Electrolytic Production of ——. J. B. C. Kershaw. Electrician, Oct. 25, 1901, **48**, 20.

The principle of this process is not new, cells for the electrolysis of fused salt with molten lead as cathode material, having been patented in 1893 and 1894 by Vautin and Hulin respectively. A diagrammatic section of the Acker cell is shown in Fig. 1, and cross sections on the lines *a*—*b* and *c*—*d* in Figs. 2 and 3.

The bottom of the cell P is trough-shaped, and is constructed of cast iron. It serves as electrical connection between the main conductor, and the mass of fused lead L resting upon it. The upper portion of the cell Q is constructed of any refractory basic material, not attacked by chlorine gas or fused salt. The anode C is of carbon, and dips into the fused salt. A hollow rib runs along the centre of the cast-iron trough forming the base of the cell, and two rectangular channels run parallel with the centre opening for half the length of the cell. Figs. 2 and 3 show the position of these channels in the cell base. These channels connect with the side vessel K and with the interior of the cell by the openings O and B. At A, an injector supplies steam under pressure to the cell, and carries the lead-sodium alloy from the interior of the cell into the side vessel K. Here the steam and sodium react, and produce molten sodium hydrate and hydrogen, which rise to the surface of the lead and pass into the collecting vessel T by the overflow pipe Z.

Fig. 1.

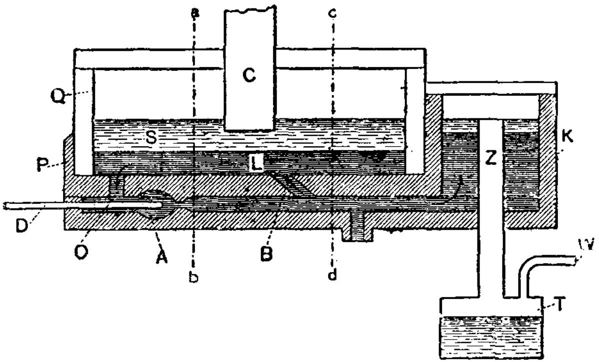


Fig. 2.

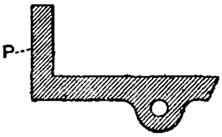
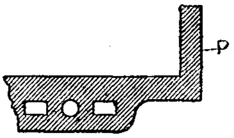


Fig. 3.



The purified lead returns to the cell by the side channels and openings B. The circulation and decomposition of the lead-sodium alloy is thus continuous, and sodium hydrate testing 79 per cent. (Liverpool test), Na_2O is said to be obtained without evaporation in the collecting vessel T. The author considers that this result will depend largely upon the care used in casting the aspirator neck at A, and in the position and size of the steam-supply pipe D.

The heat of the cell is partly maintained by the steam supply at A and by the heat liberated in the reaction $Na_2 + H_2O = Na_2O + H_2$.

The hydrogen escaping at W can also be collected and burned in a special apparatus to supplement this heat, or to fuse the salt used in feeding the cell when at work. No details have been published concerning the materials used in constructing the upper portion of the cell. Efficiency data are also lacking. The Niagara works were erected in 1900-1901, and the first unit of plant was started early in the present year.—J. B. C. K.

Ammonium Chloride dissolved in Liquid Ammonia; Electrolysis of ——. H. Moissan. Comptes Rend. **133**, [19], 713—714.

Pure liquid ammonia, as Frenzel has shown, has a very low conductivity; for the current of 115 volts, hardly 0·01 ampère passed between the platinum electrodes of the author's U-tube containing liquid ammonia; but on dissolving ammonium chloride in the ammonia, a great increase in the current took place, and gas was evolved at each electrode—chlorine at the anode (which at the low temperature of −60° or −80° C. did not react on the ammonia to form either nitrogen or nitrogen chloride), and pure hydrogen at the cathode. Ammonium iodide yields a similar result.—J. T. D.

Ozone; Formation of ——. A. Chassy. Comptes Rend. **133**, [20], 789—791.

The author has examined the rate at which oxygen becomes converted into ozone under the influence of the silent discharge caused by a constant current in the primary circuit of the Berthelot's ozoniser which he employed. The following figures express his results, where the unit of time is that required to convert into ozone the first 0·5 per cent. of the total oxygen. The figures are independent of the strength of the current, the unit of time, however, being longer the weaker the current. The temperature of experiment was 20° C.

Total Time.	Ozone Produced (Per Cent. of Total Oxygen).	Total Time.	Ozone Produced (Per Cent. of Total Oxygen).
1	0·5	10	3·15
2	0·91	12	3·45
3	1·27	14	3·74
4	1·61	20	4·40
5	1·92	36	5·40
6	2·22	60	6·45
7	2·50	90	7·05
8	2·72	120	7·30

Thus the quantity of ozone produced tends to a limit, not easy to fix precisely, depending on temperature but not on strength of current. It is clearly economical, if quantity only and not concentration of ozone is required, to ozonise the oxygen only to a slight extent.—J. T. D.

Strychnine and Brucine; Electrolytic Reduction of ——. J. Tafel and K. Naumann.

See under XX., *page* 1233.

PATENTS.

Electrical Insulating Materials; Manufacture of ——. E. A. Claremont, Knutsford, Cheshire. Eng. Pat. 21,804, Dec. 1, 1900.

Paper and the like materials are impregnated with the usual insulating substances, such as oils, first under considerably reduced pressure and then under the atmospheric pressure, whereby a better penetration of the material by the insulating compound is effected than is possible by the present methods.—H. I.

Alkaline Chlorides; Electrolytic Decomposition of ——, *and Apparatus therefor.* J. D. Gilmour, Glasgow. Eng. Pat. 18,397, Oct. 16, 1900.

The apparatus comprises inner and outer vessels in electric contact with the positive and negative poles of a source of electric current supply, and means are provided for permitting the circulation of the mercury between the vessels, and for preventing the chlorine compounds in the inner vessel from mixing with the caustic alkali formed in the outer one. The circulation of the mercury is effected by the pulsating action of a caustic liquor pump, which causes the circulating liquor to flow from one end of the outer vessel down under the bottom of the inner one, and after scrubbing the mercury, to pass out at a higher level on the opposite side of the inner vessel, and means are provided for causing a cycle of circulation of liquor from delivery to entering the end of the outer vessel.—G. H. R.

Alkaline Salts (Chlorine and Caustic Soda and Potash); Decomposition of —— *by Electrolysis, and Apparatus therefor.* [*Intermediate Electrode.*] J. Greenwood, London. Eng. Pat. 22,406, Dec. 8, 1900.

Two types of apparatus are described, one of which consists of two vessels, each divided into an even number of anode and cathode sections arranged alternately, by slate or porcelain-enamelled iron partitions dipping into mercury. The second type also consists of two vessels, one of which forms a single anode, and the other a single cathode section, the latter being divided into several compartments through which the mercury circulates in a reverse direction to the caustic lye, causing the latter to be highly concentrated. An intermediate electrode forms the base of each electrolytic vessel or section of either type, and is constructed preferably of a solid cast-iron plate, the internal surface of which is entirely coated with a suitable insulating substance, with the exception of lines or narrow grooves, about one or two inches apart, running in parallel lines across the plate. The bare metal becomes well amalgamated with the amalgam, and establishes perfect electrical contact equally at all points with the mercury flowing through the apparatus, and the intermediate electrode constitutes a practically perfect conductive material entirely independent of the mercury for the passage of the current at a high and uniform current density between the several anode and cathode sections respectively. —G. H. R.

Chromium Oxide Salts; Electrolytic Oxidation of Solutions of ——. C. Schneider, London. Eng. Pat. 19,029, Oct. 24, 1900.

This method of electrolysis with the employment of diaphragms is an improvement on that described in Eng. Pat. 15,724, 1898, and consists in compensating the alterations of concentration caused by the electrolytic process by mixing the fluids of both compartments of the electrolytic vessel by diffusion through the diaphragm and introducing a continuous or intermittent stream of new lye into the anode compartment, and drawing off the corresponding amount of old lye. The velocity of diffusion is increased with relation to the velocity of the migration of the sulphuric acid by regulating the density of the electric current, and the latter may be temporarily interrupted. The most rapid compensation of the alternations of concentration is effected by mixing the liquids of both compartments directly by filling in fresh lye, so that it overflows the brim of the diaphragm, allowing it to stand over for a time, and then drawing off again a sufficient amount; or the two lyes may be mixed by artificial means, as by a current of air, or by mechanical agitation.—G. H. R.

(*B.*)—ELECTRO-METALLURGY.

Anodes; Disintegration of ——. E. Wohlwill. Chem.-Zeit. 1901, **25**, [84], 917. (Paper read at the Hamburg Meeting of the Gesellschaft deutscher Naturforscher und Aerzte.)

The anode slime produced in electrolytic copper refining always contains copper, in addition to the insoluble constituents of the crude metal, so also silver is found in the anode residues in silver refining, and the slime produced in the electrolysis of gold may contain as much as 20 per cent. of that metal. The obvious hypothesis that the soluble matter is enclosed within a crust composed of insoluble constituents is untenable, inasmuch as a disintegration of the anode occurs even when the pure metals are under treatment. The author ascribes the phenomenon to the formation of a pulverulent metallic covering of lower solubility, which takes place directly the electric circuit is completed, and which is constantly renewed during the progress of electrolysis, with the result that the anode is unevenly attacked, becomes roughened, and finally disintegrates. In the case of gold, this film is due to the decomposition of the gold sub-chloride formed along with the chloride. Everything therefore points in the case of copper, to the formation of a certain amount of cuprous sulphate with the cupric salt, the cuprous compound at once breaking up into cupric sulphate and metallic copper. It was at first thought that the inferior solubility of the film was due to the physical condition of the metallic particles being different, but the author was afterwards led to the view that it was explained by the looseness of its contact with the anode plate. A negative proof of the correctness of the pulverulent-film hypothesis is that anodes of metals which do not form such a film do not disintegrate, for example, Cu, Ag, or Au, in potassium cyanide solutions. Disintegration, however, does not occur, even when a film is formed, if the deposit does not adhere to the anode metal, as is the case for example when a high current density is employed, and the flow of solution over the surface washes away the film as it is formed. Specimens were shown of identical bars of copper; one that had been used as anode with a current density of 0·3 amp. per sq. dm., and that appeared to be completely honeycombed; and the other, after use with a density of 20 amp. per sq. dm., retaining a perfectly smooth surface.—W. G. M.

Alloys; Cathode Polarisation and Formation of ——. Coehn. Zeits. angew. Chem. 1901, **14**, [42], 1074. (Paper read before the Versammlung deutscher Naturforscher und Aertze, Hamburg, 1901.)

When work is done by a secondary reaction during the electrolytic separation of a substance at the electrode, there must be a corresponding alteration in the potential difference required for the deposition. Such an alteration is to be expected when one metal is deposited upon another with which it is capable of alloying. Thus, when silver is deposited upon gold, there is a reduction of 0·05 volt in the pressure required; and when certain metals are deposited on mercury, the following reductions are observable, Zn, 0·15; Cd, 0·12; Ag, 0·03; Cu, 0·08; and Fe, 0·07 volt. Hydrogen deposited on palladium requires a pressure of 0·26 volt lower than when separated on platinum, whilst on all other metals, the pressure required is higher than on platinum. In determining the voltage required for the decomposition of caustic potash, two special points may be noted, the first probably corresponding to the separation of hydrogen ions, the other to the formation of a potassium-hydrogen alloy.—W. G. M.

PATENTS.

Minerals or Ores [*Electric*]; *Disintegration of* ——. E. L. Graham, Upper Warlingham, Surrey. Eng. Pat. 14,291, Aug. 9, 1900.

The ores are placed in a bath containing an acid solution with or without the addition of fluorspar. An electric current of 50 or more volts is then passed through the solution, when the ores are found to be disintegrated. —J. H. C.

Iron [*Electrolytic*]; *Manufacture of* ——. A. Simon, Bordeaux. Eng. Pat. 20,513, Nov. 14, 1900.

Oxide of iron, with or without an addition of carbon, is dissolved in calcium fluoride or other suitable electrolyte, and then treated electrolytically so as to deposit iron on the cathode. The voltage required varies under different circumstances from 7 or 8 up to 18 or 20.

About 500 kilos. of fluorspar are required per ton of iron produced.—J. H. C.

Heat-producing Devices for Smelting, &c. in [*Electric*] *Furnaces.* S. F. Pierce, St. Paul, U.S.A. Eng. Pat. 17,852, Sept. 6, 1901.

Cylindrical bars of carbon or other electrically conducting substance of special form and covered with fire-clay or other non-conducting refractory material, are made to extend through the furnace chamber. An electric current is then passed through the bars, so as to heat them to the required temperature.—J. H. C.

Iron and Steel; Process [*Electric*] *for Toughening, Hardening, or Annealing* ——. W. Holzer and W. F. L. Frith. Eng. Pat. 13,352, July 24, 1900.

See under X., *page* 1218.

XII.—FATS, OILS, AND SOAP.

Montan Wax. E. von Boyen. Zeits. angew. Chem. 1901, **14**, [44], 1110—1111.

The product known as "Montan wax" is derived from the lignites of Saxony and Thuringia, and comes into commerce as a white, high-melting candle-material. The raw material is a bitumen extracted from *pyropissite* by various solvents; this bitumen is a hard, odourless mass of the colour of ozokerite with a melting point between 80° and 90° C. By steam-distillation, a yellow, crystalline mass melting above

70° C. and of a fairly dry consistency was obtained. This was purified by a second steam-distillation, repeated treatment with brown-coal-tar spirit, and filter-pressing. Subsequent treatment with decolorising agents furnished at length a white crystalline substance melting above 70° C.

Montan wax is not a paraffin, but consists of two bodies in about equal proportions. One of these is a high molecular acid, montanic acid, having the formula $C_{29}H_{58}O_2$, m. pt. 83°—84° C. The sodium and potassium salts are readily soluble in hot water, less soluble in alcohol. The acid itself is with difficulty soluble in alcohol but readily soluble in brown-coal tar spirit. Concentrated sulphuric acid carbonises the acid, nitric acid having no apparent action even on boiling. Montanic acid when solidified after fusion, shows a radiating shining structure, similar to that of Chinese wax.

The other constituent of montan wax is apparently an alcohol; it is separated from the acid by hot-pressing with the benzine and melts at 60° C. Its behaviour towards solvents is similar to that of montanic acid; it is more readily decomposed by sulphuric acid than montanic acid is, and is oxidised by nitric acid to fatty acids of low melting points. The formula of this alcohol has not yet been determined; the bitumen is probably the non-crystallisable ester of these two bodies. They are always obtained by the steam distillation of the bitumen, the formation of mineral oils being due purely to a secondary decomposition.—J. F. B.

Lubricating Oils at High Temperatures; Friction of ——. S. Kapff. Zeits. Verein. deutsch. Ing. 1901, 343. Proc. Inst. Civil Eng. 1901, **146**, [4], 34.

Thurston, in 1899, stated that the coefficient of friction of lubricating oils decreases as the temperature rises to a limit of about 82° C. and concluded that heating of the bearings to this degree must not be regarded as harmful, and that a hot bearing need not be cooled unless its temperature exceeds 100° C. The author states that these views are fallacious, as they only take into account the frictional resistance, and that even if the latter were the principal factor to be considered, they would not hold true generally. The small saving of energy due to the low value of the coefficient of friction of the oil would be far more than counterbalanced by the amount of heat wasted in the maintenance of the bearing at a temperature of 82° C. If a bearing becomes heated to 80° it may be accepted that either the oil is bad, or too little of it is employed, or possibly the bearing is dirty or otherwise out of order. According to the author, a good lubricating oil should fulfil the following conditions:—(1) It must be free from acid and solid particles, and must not resinify or become acid on exposure; (2) it must have a suitable adhesion; and (3) at the temperature at which it is used, its frictional resistance must be as small as possible.—A. S.

Oils; Detection of Drying and Marine Animal ——. G. Halphen.

See under XXIII., *page* 1244.

Fats and Oils; Use of Iodine Monobromide in the Analysis of ——. J. Hanuš.

See under XXIII., *page* 1246.

Rosin Grease. L. Archbutt.

See page 1193.

Butter; Observations on the Composition of ——. A. Pagnoul.

See under XVIII. A., *page* 1228.

PATENTS.

Margarine; Production of ——. J. H. G. Neisse and J. H. Boll. Eng. Pat. 21,626, Nov. 29, 1900.

See under XVIII. A., *page* 1229.

Margarine; Manufacture of ——. A. Pellerin. Eng. Pat. 22,905, July 12, 1900.

See under XVIII. A., *page* 1229.

XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

(*A.*)—PIGMENTS, PAINTS.

PATENT.

Heating or Roasting Powdered Iron Salts [Production of Pigments] and similar Compounds; Method of, and Apparatus for ——. J. W. Hinchley. London. Eng. Pat. 18,669, Oct. 19, 1900.

The finely powdered compounds are allowed to fall down a vertical metal flue, so that they meet a slow current of hot gases passing up the tube which carry away the liberated acids and other volatile constituents down a second tube into a settling chamber, where the finest particles are filtered out of the gas stream by an asbestos screen. The gases are then freed from their acid vapours by suitable absorbents.—H. I.

(*B.*)—RESINS, VARNISHES.

Sandarac Resins; Constituents of the ——. T. A. Henry. Proc. Chem. Soc. **17**, [241], 187.

The sandarac of commerce is the naturally exuded resin of various species of *Callitris*, usually either *C. quadrivalvis* or *C. verrucosa.*

Both varieties of resin consist of a mixture of resin acids and terpenes separable by steam distillation. From the latter, *d-pinene* has been isolated and identified. The chief volatile constituent is a diterpene, boiling at 265° C., $\mu_D = 1{\cdot}5215$, $[\alpha]_D = +55°$, which behaves like a saturated substance, forming no additive compounds with bromine, hydrogen chloride, or nitrosyl chloride. Two resin acids have been isolated and examined. One of these has the composition, $C_{20}H_{30}O_2$, and closely resembles in behaviour the isomeric *d-pimaric acid* of Vesterberg (Ber. 1885, **18**, 3331), but is optically inactive, and is therefore named *inactive pimaric acid.* On reduction with hydriodic acid, it gives a diterpene, $C_{20}H_{32}$. On oxidation with permanganate, pimaric acid yields acetic acid and a crystalline substance melting at 206° C., which is probably *trimellitic acid.*

The second resin of sandarac has not been obtained in a crystalline form, but the highly deliquescent sodium salt and the characteristic lactone have been prepared; from analyses of these, the formula of the free acid is probably $C_{30}H_{48}O_5$. For this substance, the name *callitrolic acid* has been retained. The acid is remarkably resistant to the action of reagents, being unattacked, even by hot fuming nitric acid. When heated in a vacuum, it is decomposed with the formation of carbon dioxide and a diterpene dentical with that occurring naturally in the resin.

Shellac; Analysis of ——. E. J. Parry.

See under XXIII., *page* 1245.

Brass Lacquers; Action of Light on Coloured ——. H. Smith.

See page 1188.

(*C.*)—INDIA-RUBBER, &c.

India-rubber. Schneider. Chem.-Zeit. 1901, **25**, [84], 924. [Paper read at the Hamburg meeting of the Gesellschaft deutscher Naturforscher u. Aerzte (1901).]

The author exhibited several kilos. of caoutchouc milk which had been collected in 1899, and preserved with the aid of small quantities of ammonia and creosote. Precipitation with citric acid solution during the meeting produced an exceedingly tough and colourless caoutchouc.—W. G. M.

PATENT.

Fabric or Material (Leather and Rubber Substitute, Insulator, &c.), and Articles from Fibre and an Adhesive; Manufacture of ——. A. Oesterheld, Vöcklabruck, Austria. Eng. Pat. 10,007, May 14, 1901.

A new article of manufacture, produced from alternate layers of fibres (jute, cotton, silk, wool, asbestos, &c.) and an adhesive substance (india-rubber, gutta percha, spermaceti, artificial gum, resin, or other like material), the fibres lying in all directions, combined by pressure to form a homogeneous whole, is claimed.

The fibres are blown by hot air in a chamber on to an endless cloth covered with an adhesive layer, and when one layer has been deposited, the material is passed through a heated chamber to dry and then through calenders. It again receives a coat of adhesive and fibres, and is again dried and calendered, and so on, until the desired thickness is obtained. By spreading the layers on a model, cycle tyres, &c. may be made.

The material is said to be also useful for straps, belts, washers, tyre covers, floor cloths, wall coverings, insulating materials, and the like.—H. I.

XIV.—TANNING; LEATHER, GLUE, SIZE.

Tannoids; Contribution to the Study of the ——. H. Kunz-Krause. Chem. Centr. 1899, **1**, [9], 559.

The author treats of his subject under five headings:—(1) The so-called tannins and their significance in plant life. (2) Attempts to classify the so-called tannins. (3) Systematic classification of the tannoids. (4) The relation between the constitution of the tannoids and their behaviour to reagents. The reagents considered, comprise ferric chloride, gelatin (glue), albumin, alkaloids, and tartar emetic. The author found that the reagents named are not general reagents for tannin as is usually accepted, but are only specific group reagents for different subdivisions of his system, viz., for the tannoid anhydrides produced from two or more molecules of protocatechuic acid or gallic acid by abstraction of water. The reactions given by the above reagents and others with gallic, digallic, ellagitannic, and ellagic acids, tannin, trigallic acid, &c., are collected in a table. (5) The natural transformation products of the tannoids, and the relation of these to the phlobaphenes and rotene and to other groups of plant constituents. For the purpose of classifying the tannoids, besides the reactions and decompositions effected by chemical means, the author proceeds on the lines that use can be made also of the changes which occur naturally in the plants, especially the formation of "phlobaphene" and "reds" ("*Rote*"), *i.e.*, the red to reddish-brown amorphous colouring matters of barks, which, like gallotannic acid, may be considered as anhydrides of tannoid mother-substances (that is, as definite intermediate products of a process of dehydration of aromatic hydroxy-acids). The relation of phlobaphene and "reds" ("*Rote*") to gallotannoid anhydrides is also shown by the similar behaviour (precipitation) with gelatin. (See also this Journal, 1896, 831.)—A. S.

Leathers; Composition of Belgian ——, *and the Influence of the Nature of the Tannery Water on the same.* E. Nihoul, Bourse aux Cuirs de Liège, Sept. 1901.

A. *Composition of Belgian Leathers.*—The following table shows the composition (highest and lowest values to one decimal place) of 15 genuine leathers, tanned with waters of known composition, most of them different.

The samples had all been kept for some time in a warm atmosphere, so that the percentage of moisture is rather low (usually about 18 per cent. for Belgian leathers). The "total soluble" is high because at least three litres were used for extraction, instead of only one litre. (See page 1249. For the same reason "un-combined tannin" and "soluble non-tannin" are also high.

The "hide-substance" was determined by Kjeldahl's original method, completed by addition of permanganate, which gives rather high results, and has a consequent influence on the factors associated with this one.

For the full details of each leather, and the results when calculated as percentages of *dry* leather, the original paper should be consulted.

B. *Influence of the Nature of the Water used in Tanning.*—Each of the foregoing leathers (except in two cases) was tanned with a different water, the composition of which was completely determined. The analytical results (composition of each water and of each leather) are collected in a series of tables, and the author makes the following observations upon them:—

Constituents, per Cent. of Sample.	Sole Leather, Seven Samples.	Smooth Vache. Four Samples.	Light Leather. Four Samples.
	Per Cent.	Per Cent.	Per Cent.
Moisture........	14·3 — 15·7	13·6 — 15·6	11·3 — 14·1
Ash	0·6 — 1·0	0·7 — 1·1	0·7 — 1·0
Fatty matter....	0·1 — 0·5	0·2 — 4·5	0·2 — 6·5
Total soluble....	6·7 — 14·1	16·2 — 23·5	9·5 — 14·8
Ash from above .	0·6 — 1·4	0·8 — 1·6	0·7 — 1·1
Organic soluble .	6·0 — 13·1	15·4 — 21·9	8·6 — 13·9
Soluble non-tannin.	2·1 — 6·9	2·6 — 8·9	2·5 — 5·2
Ash from above .	0·5 — 1·3	0·9 — 1·2	0·5 — 1·3
Soluble non-tannin organic.	1·5 — 6·0	1·6 — 7·7	2·0 — 4·5
Uncombined tannin.	3·9 — 8·9	12·0 — 14·8	6·6 — 10·7
Leather substance.	69·8 — 78·6	62·8 — 77·2	69·5 — 76·4
Nitrogen........	8·3 — 10·5	7·8 — 8·9	9·4 — 10·1
Hide substance .	46·7 — 59·2	44·2 — 50·2	53·2 — 56·8
Combined tannin	16·6 — 24·7	14·1 — 22·3	16·4 — 19·6
Total tannin	21·2 — 30·2	28·9 — 34·3	23·7 — 28·1
Yield of leather.	163·9 —214·1	199·2 —226·2	176·0 —188·2
Index of tannage	28·0 — 49·5	28·1 — 50·2	30·8 — 34·5
Specific weight..	0·716— 0·899	0·734— 0·948	0·545— 0·784

Sole Leather.—(1) The amount of saline matter present in the water has a general relation to the ash of the leather, though, in one case at least, a soft water produced a leather with more ash than those tanned with hard waters.

(2) The percentage of fat depends chiefly on the nature of the hide, and on the care taken in fleshing it; in three instances a low percentage was associated with free ammonia in the water.

(3) It seems probable that very soft waters give a low "total soluble" number.

(4) With hard waters, the amount of uncombined tannin is nearly three times as great as the amount of soluble organic non-tannin, whilst with soft waters the two amounts are nearly equal. Excess of saline matters, therefore, appears to hinder absorption of tannin by the hide substance.

(5) The amount of "combined tannin" is greater when the water used is soft than when hard.

(6) As regards "percentage yields of leather," hard waters give extreme results in both directions; the average yield with soft waters is high and less variable. As a general conclusion, soft waters give better results than do hard waters, but the knowledge and skill of the tanner are the most important factors.

Vache Leather (South American Cowhides).—Waters containing a large amount of temporary hardness cause a great ash percentage, but this factor also depends largely on the extent to which the lime used in unhairing is removed. The "total soluble" figure also is more influenced by details of manufacture than by the chemical composition of the water. The relation of "soluble non-tannin" to "uncombined tannin," however, as in the case of sole-leather, is definitely connected with the amount of saline matter present in the water.

Light Leathers.—Here, the subsequent details of manufacture appear to cover up any differences that the nature of the tanning water may produce at the first stage.

In conclusion it seems impossible to prejudge the composition of leather from the nature of the water used. First quality leather can be made with hard or soft water, but the chief question of relative cost still remains to be investigated.—R. L. J.

Tannin; Report to the International Association of Leather Trades Chemists on the Methods in Use for Determining ——. H. R. Procter.

See under XXIII., *page* 1246.

Quebracho Extract; Analysis of ——. G. Klenk.

See under XXIII., *page* 1249.

Leather; Contribution to the Chemical Study of [*Analysis of*] ——. E. Nihoul.

See under XXIII., *page* 1249.

Chromed Hide Powder; Application of ——, *in Analysis of Tanning Materials.* J. Paessler and W. Appelius.

See under XXIII., *page* 1249.

PATENTS.

Raw Hide; Process for Treating ——. R. Croasdale Syracuse, State of New York, U.S.A. Eng. Pat. 19,542 Oct. 1, 1901.

The hide, after the removal of all hair, fleshings, &c., is soaked for 3—4 hours in a bath containing soft water (1,000 parts), salicylic acid (2 parts), picric acid (3 parts), and boric acid (25 parts). It is then stretched, dried in warm air, and sponged with a solution of potassium bichromate (2½ per cent.) in diffused daylight. Exposure to full sunlight completes the process.—R. L. J.

Artificial Sponges [*from Leather Fibres*]; *Manufacture of* ——. C. Paulitschky, Vienna. Eng. Pat. 18,808, Sept. 20, 1901.

Fine leather fibres, free from fat, are saturated with "sulphur milk," and, after drying, are soaked in liquid caoutchouc and ammonium carbonate, and the tough mass thus produced is heated gradually, preferably *in vacuo*, from 125° up to 140° C., whereby pores or channels are formed in the mass, by the escape of gas produced by the decomposition of the ammonium carbonate, and the caoutchouc is vulcanised by the sulphur.

The product thus obtained, may be dyed and used as a substitute for loofas and bath sponges, &c.—H. I.

Fabric or Material [*Leather Substitute*] *and Articles from Fibre and an Adhesive; Manufacture of* ——. A. Oesterheld. Eng. Pat. 10,007, May 14, 1901.

See under XIII. C., *page* 1222.

XV.—MANURES, Etc.

Nitrate Fermentation and its Significance for the Biological Processes of the Soil. J. Stoklasa. Zeits. angew. Chem. 1901, **14**, [41], 1029.

The author has made a series of experiments by inoculating aqueous solutions of nutrient salts, &c., containing sodium nitrate as the only source of nitrogen, with different species of bacteria, and determining the amounts of nitrogen in the form of ammonia, nitric and nitrous acids, and in the gaseous state, after incubation in the dark at 28°—30° C. From these experiments he concludes that there are two main groups of bacteria which effect the metamorphosis of nitrates. The first group comprises those which transform the nitrate nitrogen into elementary nitrogen, and includes *B. Harlebii, B. fluorescens liquefaciens, B. pyocyaneum B. Stutzeri, B. centropunctatum, B. filefaciens, B. denitrificans*, &c.

To the second group belong: *B. megaterium, B. mycoides, B. subtilis, B. mesentericus vulgatus, B. ramosus, B. proteus vulgaris* and *proteus Zenkeri, B. radicicola*, &c. These reduce the nitrogen in the nitric acid present to ammonia.

The course of the vital processes of both groups is naturally considerably influenced by changes in the composition of the culture media, whilst the introduction of air during the fermentation leads to the reduction of a much smaller amount of nitrate.

The author also points out that there is a connection between nitrate and alcoholic fermentations. He has identified alcohol in the culture medium in which certain denitrification bacteria were grown in the presence of an excess of certain kinds of sugar, and considers that this is due to intramolecular respiration. In fact, in every fermentation in which different alcohols are formed, elementary nitrogen is produced when nitric or nitrous salts are present. According to former observations, intramolecular respiration must be regarded as identical with alcoholic fermentation, and the author's opinion is that the alcohol here produced acts upon the nitrates or nitrites, yielding elementary nitrogen or ammonia, and being itself oxidised to carbon dioxide.

The *Bacillus radicicola* assimilates the nitrogen formed in the nitrate fermentation. If, however, nitrogen be first passed through red-hot tubes, it is no longer assimilated. The author suggests that this may be due to the nitrogen liberated in the fermentation being of an active form, or to the presence of certain nitrous oxides which may have a favourable influence on the assimilation.

Finally, the author deals with the problem of inoculating the soil with bacteria capable of assimilating the nitrogen formed in the nitrate fermentation, and thus reducing the loss of nitrogen which occurs in agriculture.—C. A. M.

XVI.—SUGAR, STARCH, GUM, Etc.

Cane Sugar; Specific Rotation of ——, *and its Alteration with Temperature and Wave Length.* H. Pellat. Ann. chim. phys. 1901, **23**, 289; Zeits. Vereins Deut. Zucker-Ind. 1901, [548], 815—836.

The author was commissioned by the French Government to determine, as accurately as possible and with all the refinements attainable, the following data:—

(1) The normal weight of cane sugar for the Laurent polarimeter, *i.e.*, the weight of sugar to be contained at 20° C. in 100 c.c.* of the solution so as to give, in the 20 cm. tube with sodium light, a rotation of 21·67° at 20° C.

(2) The alteration of the specific rotation of the normal solution of sugar with change of temperature with sodium light.

(3) The alteration of the specific rotation of the normal solution of sugar according to the wave length of the light employed.

The normal weight of sugar was also determined independently by Mascart and Bénard, whose results are practically identical with those of the author.

A special apparatus by Jobin, consisting of spectroscope and polarimeter, which took two years to construct, was employed by the author. This instrument is fully described in the paper.

The normal weight of pure sugar found by the author worked out at 16·275 grms., fully corrected. For practical work, where the weighings are not reduced to vacuum, the normal weight found was 16·28 grms., as against 16·29 grms. finally adopted by the French Commission. The specific rotatory power calculated from the author's observations was $[\alpha]_{D}20^\circ = 66\cdot536^\circ$.

The mean coefficient for change of temperature was found to be: $\beta = -0\cdot00036$ between 14° and 30° C. The value β includes the expansion of the metal tube; deducting this, the coefficient for the sugar solution alone is $\gamma = -0\cdot00038$. The value γ is itself composed of the difference between the coefficient of expansion of the sugar solution μ and the temperature coefficient for the change of specific rotary power k. Taking μ as equal to $-0\cdot00024$, the coefficient for the rotatory power $k = -0\cdot00014$. For practical work with glass tubes instead of metal, the mean temperature correction would be $-0\cdot00037$, and the corrected rotation $R\frac{D}{20} = R\frac{D}{t}\,[1 + 0\cdot00037\,(t - 20)]$ between the limits of 14° and 30° C.

As regards the variation with wave length, the author has determined the rotation of the normal sugar solution with eight different well defined rays, corresponding to chemical lines in the spectrum; the results are all referred to the mean of the D_1 and D_2 rays.

The law that the rotations are inversely proportional to the squares of the wave lengths was not confirmed by these

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* The French Commission, in spite of the resolution of the International Congress of Vienna and Paris to adopt metrical c.c.'s (*i.e.*, the volume of 1 grm. of water at 4° C. weighed *in vacuo* with brass weights), uses another standard such that 1 c.c. = the volume of 1 grm. of water at 4° C. weighed in air with brass weights. According to this standard (the c.c. in which, may be termed French c.c.'s) 100 c.c. = 100·106 metrical c.c.'s.

observations with sufficient closeness. The author has therefore drawn up an empirical formula which expresses his results within the limits of experimental error:—

$R\lambda = R_D \left(\frac{A}{\lambda^2} + \frac{B}{\lambda^4}\right)$, where A = 0·325483, and B = 0·00757003.

Critical Notes by Schönrock.—To the German translation of this paper certain critical notes are added by Schönrock. In the first place it is pointed out that the instruction given to Pellat to determine the concentration of the normal solution of sugar for "sodium light" is not sufficiently definite, since with modern polarimeters the results obtained with a sodium flame may vary, according to the intensity of the light and the method of purification of the salt, by as much as 1 per cent.

The experimental methods described by Pellat as "new" were completely worked out in 1885 by Lippich, and have been employed since by many other chemists. Certain details and remarks relating to the construction of the instruments used by Pellat are criticised. Further, it is remarked that the proportion of ash in the sugar employed, 0·06 per cent., is not "almost negligible" as claimed by the author; the sugar cannot be regarded as being especially pure and should have been purified further in the laboratory. The solutions to be polarised were filtered "three or four times," a procedure sufficient to alter their concentration by several hundredths of a per cent. The tube employed for polarising, measured 100·015 cm. at 20° C., a divergence from the metre which is left uncorrected by the author. The fact that the rotations diminished when the filled tube was allowed to remain in the water-bath of the instrument for some time can only be explained by leakage round the cover glasses. Schönrock regrets that the French Commission should have introduced yet another arbitrary definition of the cubic centimetre. His conclusion is that the normal weight of sugar, 16·29 grms. (or 16·28 grms. as found by Pellat), has only been established with an accuracy of ± 0·1 per cent. and that another more accurate determination will shortly be required. Other notes relate to divergences between the temperature coefficients found by Pellat and those of other authors, including Schönrock himself.—J. F. B.

Quotients of Purity and Saline Quotients of Sugar Products; Tables for Conversion of —— into Quotients of Impurity and Inorganic Quotients. F. Sachs. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1901, 19, [4], 419—421.

The author has shown that the method now used of giving the quotients of purity and the saline quotients of sugar products is unsatisfactory, and that it is preferable to reduce everything to the basis of 100 parts of sugar. Three tables are now given for converting results expressed in the old way to this new plan:—(1) This table is for transforming the quotient of apparent purity A into the quotient of impurity M, that is, the amount of impurity per 100 of sugar; the numbers are calculated from the formula, $M = 9000/A - 90$, which assumes a uniform average density for the non-sugar materials of 1·111 times that of the sugar. (2) This gives the quotient of impurity corresponding with the quotients of real purity R, the numbers being obtained from the equation, $M = 10000/R - 100$. (3) The third tables serves for transforming the saline quotient S into the inorganic quotient N, that is the amount of inorganic matter per 100 of sugar; the relation between these magnitudes is expressed by $N = 100/S$.—T. H. P.

Sugar Products; Nitrites in ——, and their Influence on the Frothing of Sugar Syrups. K. Andrlik and V. Stanek. Chem. Zeit. Rep. 1901, 25, [84], 303.

But little attention has hitherto been paid to the quantity or the practical effect of nitrites in sugar products. Molasses from Bohemian works usually has only 0·004—0·01 per cent. N present in the form of nitrites. Hungarian and Italian products usually have more than this, and they generally show higher acidity; and froth in boiling. Frothing has generally been ascribed to the escape of carbon dioxide in boiling; but the author has found not only this gas, but also nitrogen peroxide, in the escaping gases; this gas was recognisable by its smell and by its turning iodide starch papers blue; nitrous acid fumes were also observable. Three frothing syrups were examined and were found to give the following results:—

——	I.	II.	III.
Dry substance	93·46	87·72	87·42
Direct polarisation	71·20	54·40	48·00
Invert sugar, in mgrms. Cu.	0·45	0·51	0·40
Ash	8·23	9·33	13·37
Purity	76·2	62·0	59·0
Nitrogen (Jodlbauer)	1·49	1·85	2·22
„ as nitrates	0·074	0·059	0·49
„ as nitrites	0·032	0·041	(not estd.)
Acidity, in c.c. N/1 KOH per 100 grms.	6·0	10·7	10·0

From these and other experiments the author considers the characteristics of frothing syrups to be unusually high acidity and large proportion of nitrite nitrogen. He is in agreement with other observers that frothing chiefly occurs with syrups from abnormal beets, from rich nitrogenous soils, or by the alteration of the juices by the action of bacteria.—W. G. M.

Molasses Residues ["Schlempe"]; The Ether-soluble Acids of ——. A. Herzfeld. Zeits. Vereins deutsch. Zuckerind. 1901, 720; through Zeits. angew. Chem. 1901, 14, [44], 1115.

The molasses "Schlempe" examined had the following composition:—Apparent solids, 79 per cent. Brix; water, 22 per cent.; ash, 28 per cent.; organic non-sugar, 48 per cent., with 4 per cent. of nitrogen and 0·62 alkalinity to phenol phthalein; the sugar had been almost wholly removed by the strontia process. The sample, after acidification with sulphuric acid, was exhausted with ether; the residue, after evaporation of the ether, was distilled in the air and *in vacuo*, and the volatile portion was further separated by steam distillation. The acids were converted into esters which were fractionated, and crystalline salts of the acids were also prepared and separated.

The acids soluble in ether were found to contain about 5 per cent. of formic, 21 per cent. of acetic, and 21 per cent. of lactic acids; the remaining 53 per cent. could not be separated, but higher fatty acids, butyric and valeric acids, caramel bodies and succinic acid were present. Calculated to the original "Schlempe," 4·29 per cent. of acetic, 4·29 per cent. of lactic, and 1·02 per cent. of formic acids were found. A sample of "Schlempe" from another district gave practically the same results, so that the above may be taken as representing, on the whole, the proportions in the non-sugar of German molasses. The numbers obtained for acetic acid are important, since potassium acetate is the only really powerful, melassigenic substance known. The quantity of acetic acid found in the non-sugar is alone sufficient to account for the quantity of sugar always present in molasses in an uncrystallisable state.

The melassigenic action of lactic acid has not yet been properly studied.—J. F. B.

Saturation Mud; Analyses of the First ——, during the Season 1899–1900. K. Andrlik, K. Urban, and V. Staněk. Zeits. für Zuckerind. in Böhmen, 1901, 26, [1], 1—5.

The authors communicate the results of complete analyses of 14 samples of mud from the first saturation, taken during the season 1899–1900. The results are fully tabulated in the paper, and only the extreme values will be quoted here: CaO, 41·06—49·53 per cent.; $Ca(OH)_2$, 0·20—3·02 per cent.; $Mg(OH)_2$ 2·15—8·36 per cent; $Fe_2O_3 + Al_2O_3$, 0·35—1·53 per cent.; inorganic matter insoluble in HCl, 0·37—2·65 per cent.; P_2O_5, 1·02—1·83 per cent.; SO_3, 0·37—1·72 per cent.; CO_2, 25·11—34·79 per cent.; oxalic acid $C_2H_2O_4$ + 2 aq., 1·07—2·56 per cent.; citric acid, 0·16—1·21 per cent., total nitrogen, 0·23–0·44 per cent.; nitrogen calculated as albuminoids, 1·44—2·75 per cent.; sugar, 0·45—4·31 per cent.; *ethereal extract of the portion of the mud* insoluble in HCl, 0·21—0·90 per cent.; resin acids, 0·14—1·10; acidity of the organic acids extracted by boiling the lime salts with sodium carbonate, 38·3—94·7 cc. normal acid; acidity of the oxalic acid 17·0—40·6 cc. normal acid; acidity of the acids other than oxalic 9·1—59·0 cc. normal acid. All the above results refer to 100 parts of the dry substance of the mud. In six cases the acids extracted from the mud by means of ether after decomposing with HCl were titrated, and, after allowing for the oxalic acid, the values for the other acids soluble in ether varied between 7·0 and 38·1 cc. normal acid. Thus some muds are rich in extractable acids and others are poor. Of the nitrogenous bodies, which may legitimately be classed as albuminoids, from 30 to 60 per cent. were soluble in sodium carbonate solution. The analyses of saturation mud will assume greater importance when it is possible to refer the weight of mud to the weight of diffusion juice from which it is obtained.—J. F. B.

Beetroot Sugar Factory Effluent; Purification of——, by the Proskowetz Process. A. Pritzkow. Zeits. Vereins. deutsch. Zuckerind. 1901, **51,** [549], 402—425.

The Stössen sugar factory finding that its effluent water treated by Oppermann's process, still caused complaints from riparian inhabitants, adopted the Proskowetz process which this year was at work for the second season with satisfactory results. The water treated, included that used for condensing the waste from the diffusers, that rich in ammonia condensed from the vacuum pans, and the water from the pulp presses.

After partial subsidence, the overflow is returned for conveying and washing beetroots and for condensing; the rest is run into a fermentation or putrefaction reservoir, and thence on two successive irrigation fields. The drainage from the field is treated with lime, and after subsidence, again passed over a field. It is finally either run into the stream or returned to the factory for all purposes, diffusion, boilers, &c., so far without any evil consequences.

The oxidisability is diminished by 93 per cent., the organic nitrogen about 79 per cent. The purified water possesses only a faintly alkaline reaction, is colourless, without smell, and does not develop a putrid smell on standing. In the clarifying basins, besides the mechanical clarification, considerable chemical purification also occurs. In the fields the diminution in oxidisability is not great, but in the second field a considerable destruction of nitrogen compounds was noted. Putrefactive processes are working in both fields, but nitrification was not observed. The addition of lime occasions a further diminution of oxidisability by about 73 per cent., and this is not increased by subsequent earth filtration; merely the akalinity is removed. The quantity of lime added was very varying and might apparently be lessened. Automatic arrangements for adding lime and mechanical stirrers would alone render this possible.

The irrigation was not disturbed even by a prolonged severe frost. As only a part of the water of the Stössen factory was purified and the installation is very new, an exact estimate of the cost cannot be made.

It must also remain undecided whether the water has lost the power of favouring the development of the so-called "sugar algæ." Such algæ, particularly *Beggiatoa,* were observed in the river in quantity, but it is uncertain whether they were derived from the original water, the unpurified or the purified effluent.—L. J. de W.

Sucrose in Plants; Identification of——, by the aid of Invertase, and of Glucosides by the aid of Emulson. E. Bourquelot.

See under XXIII., *page* 1244.

Arabitol and Rhamnitol; Nitro-derivatives of——. Constitution of certain Nitric Esters. L. Vignon and F. Geren.

See under XXIV., *page* 1254.

Finishing Materials containing Starch and Starch Products. D. Saare.

See under V., *page* 1206.

XVII.—BREWING, WINES, SPIRITS, Etc.

Brewing Water; Influence of the Chemical Composition of ——. F. Bousquet. La Bière, **9,** [9], 129—130.

This question has been recently investigated anew by Raux, who concludes, from the results furnished by his experiments, that gypseous waters, containing up to 50 grms. per hectolitre, furnish very pale ales, but of high attenuation and dry. On the other hand, calcareous waters, containing up to 12 grms. of calcium carbonate per hectolitre, give beers slightly darker than gypseous water, but less so than soft water. The attenuation is medium, the beer fuller in flavour and with more condition, but leaving a bitter aftertaste. In the case of such water, the hopping should be reduced. Waters containing chlorine (up to 18 grms. of NaCl per hectolitre), give an excessive attenuation, beers extremely flat and dry, with little condition, but darker in colour than those from gypseous or calcareous waters. Furthermore, the beers are somewhat more bitter and contain about 20 per cent. more phosphates.—C. S.

Sucrase; Part played by ——. E. Delafond. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1901, **19,** [4] 421–423.

The author describes a case in which the fermentation of cane-sugar molasses, originally of 8° Baume, was arrested when the gravity reached the value $4\frac{1}{4}$° Baume. Nine-tenths of the liquor was removed and divided into three equal portions which were treated as follows:—(1) The first part was boiled, cooled, and returned to the original vat. (2) This portion was boiled, cooled, and pitched with pure yeast. (3) The third part was pitched, without treatment in any way, with the same yeast as was used for (2). Fermentation in (1) and (2) proceeded to exactly the same extent, the final gravity being $\frac{1}{2}$° Baume, whilst that of liquid No. (3) was $3\frac{3}{4}$°; in the last case a considerable quantity of non-inverted cane sugar remained. The author explains this inactivity of the sucrase as due to degeneration under the influence of certain substances present in the liquid.—T. H. P.

Pentosans of Brewers' Grains, Jute, and Loofah. A. Schöne and B. Tollens. J. Landw. 1901, **40,** 21—28, Zeits. Untersuch. Nahr. Genussm. 1901, **4,** [20], 938.

The above substances, as is known, are rich in pentosans, although only an approximately corresponding quantity of crystallised pentoses have been obtained from the same. The authors have endeavoured to obtain better results by altering the method of extraction. They found, however, that by heating with 1 per cent. sulphuric acid solution under pressure at a temperature of 125° to 128° C., worse results were obtained than by the ordinary process—heating with 5 per cent. sulphuric acid on the water bath. On digesting jute for some days with dilute caustic soda solution and precipitating the solution with alcohol, 4·6 per cent. of wood-gum was obtained, which yielded 1·2 per cent. of xylose (calculated on the jute) on hydrolysis. This wood-gum had a lævo rotation of $[\alpha]_D = 11°$.

Loofah on treatment with sulphuric acid under pressure gave only 0·63 per cent. of xylose. This showed $[\alpha]_D = +17·9°$ and a strong multi-rotation.

By heating brewers' grains with sulphuric acid on the water-bath, 24·77 per cent. of pentosan-extract was obtained, corresponding to 28·16 per cent. of pentose. Calculated as glucose the amount was 29·08 per cent. Although the hydrolysis was practically complete, the undissolved residue still gave a lignin re-action.—W. P. S.

Barley; Treatment of ——, *with Lime in the Steep.* W. Windisch. Woch. für Brau. 1901, **18,** [43], 546—547.

The author calls attention to the good results obtainable by treating barley with lime water in the first steep. This treatment prevents the grain from going mouldy on the malting floor, an occurrence which frequently forces the maltster to stop the germinating process before modification is properly complete. Mouldy malt presents a very unfavourable appearance, and the proteolytic enzyme present in the mould has the effect of breaking down the albuminoids in the mashing process. All tendency to mould is prevented by the use of lime water, and the green malt from the floor has a good colour and fresh pleasant aroma. The germinating power of new barley is often very inferior, owing, in some cases, to the formation of a film of mould-slime during the harvest which chokes the pores and prevents respiration. This slime is dissolved by lime water, and the germinating power is improved. Originally the author recommended the use of milk of lime, but the particles dried up during kilning and produced great irritation when inhaled by the workmen. The employment of clear lime water surmounts this difficulty. The lime water is prepared in a reservoir situated above the steeping vats; caustic lime is stirred with water in this reservoir and allowed to settle. The clear saturated liquor is drawn off from a cock placed about a foot from the bottom; after one batch is drawn off it is only necessary to add more water and stir up the lime-sludge at the bottom to obtain a further supply of saturated lime water. The lime should be renewed once or twice a week. According to the state of the barley, the first steep may be composed of lime water at 100, 75, or 50 per cent. saturation; the barley is steeped in this for 4—6 hours and the subsequent steepings are made with pure water in the ordinary way.

—J. F. B.

Malt for Aromatic Full-bodied dark Beers; The Kilning of ——. O. Winde. Woch. fur Brau. 1901, **18**, [42], 533-534.

Kilns intended for pale lager beer malts are constructed with high wide exhaust shafts provided with mechanically driven fans, the floors are made of wire gauze so as to be easily permeable, and the malt is spread in thin layers, the object being to remove all the water very rapidly at the lowest possible temperature, before the kilning proper. In kilns for Munich malt, high, wide exhaust shafts are also used, but the floors are made of perforated metal and are heavily charged with malt (up to 100 lb. of kilned malt per square metre). The object is not the rapid and intense removal of moisture, but a certain chemical process and incipient saccharification, the products of which, after roasting, give the full-bodied aromatic flavour of this type of malt. The whole character of Munich malt is determined in the earlier stages, in the first 10 hours when the malt is on the warm withering floor at a temperature increasing from 25° to 36° C., and subsequently when the malt comes on to the upper kilning floor and is subjected to a temperature of from 66° to 73° C., for a considerable time whilst it contains the relatively high proportion of 15—20 per cent. of water. It is at this stage that the saccharine constituents are produced which give the flavour to Munich beers. Many maltsters are under the impression that the roasting and colour are the only factors in preparing Munich malt. They dry off the malt rapidly at low temperatures as with pale malts, with the idea of avoiding gelatinisation. They then subject it to a very excessive, high-temperature kilning process which turns the whole endosperm yellow or brown; such malt can only yield poor beer of an unpleasant burnt flavour. The full bodied flavour of Munich malt depends on the qualitative composition of its extractive matter, brought about by the process described above. The malt is kilned off at a temperature of 100° to 105° C., and the end point must be very carefully watched for, by taking out samples from time to time. The starchy endosperm should never be attacked by the high temperature, and the section should only show the faintest tinge of yellowish colour.

High kilned malt must always be thoroughly cooled before packing into store, otherwise deterioration of its characteristic quality will occur.—J. F. B.

Malt; Influence of the Moisture in ——, on the Grist. E. Diehl. La Bière, **9**, [9], 130—131.

The mechanical texture of the grist depends largely on the percentage of moisture in the malt. If the latter be too dry (less than 3 per cent. of moisture), the grist will be so fine as to obstruct the filtration of the wort, and hence such a malt should be damped a few hours before crushing. On the other hand, when the moisture exceeds 6 per cent., it renders the malt tough, and a coarse grist is the result, the consequence of which is a diminution in the yield of extract, unless the rolls be set more than usually close. This question of the setting of the rolls has been investigated by the author, who found that when a dark malt, containing 8·3 per cent. of moisture, was crushed with rolls set so as to give a fine grist with a normal pale malt (4 per cent. of moisture), the grist came through very coarse, and the yield of extract was 4½ per cent. below that obtained in the laboratory; whereas, on setting the rolls closer, to produce a normal grist, the yield was increased by 1·6 per cent. He therefore advises that the rolls should be set in every case in such a manner as to furnish a grist of normal fineness, instead of being left at a fixed distance apart.

—C. S.

Yeast inured to Hydrofluoric Acid; Employment of ——, in Molasses Distilleries. M. Verbièse. Bull. Assoc. Chim. Suc. Dist. 1901, **18**, 383; through Zeits. Spiritus Ind. 1901, **24**, [43], 442—443.

In the fermentation of molasses there frequently occur irregularities, sluggishness or dying out of the fermentation before all the sugar is converted into alcohol and carbon dioxide. According to the practical experience of Effront, the cause of these difficulties lies in the action of the mineral constituents of the molasses on the sucrase (invertase) of the yeast, the activity of which thereby suffers.

In order, then, that the sucrase may resist these substances the yeast must be acclimatised to them; and to obtain a rapid and complete attenuation of the sugar, the following conditions must be fulfilled:—

(1) To the dilute molasses wort a sufficient quantity of an antiseptic (hydrofluoric acid) must be added, in order to suppress the development of bacteria.

(2) A filtration or decantation of the wort before fermentation is necessary.

(3) The wort must be pitched with a yeast acclimatised both to the presence of hydrofluoric acid and to a concentrated solution of molasses.—J. F. B.

Raisin Wine, Sweet; Constituents of ——. Aug. Schneegans. Archiv der Pharm. **239**, [8], 589—591.

In addition to the raisin and currant wines previously reported on (this Journal, 1901, 599), the constituents of three specimens of so called "concentrated" sweet raisin wines prepared from chopped fruits and water by simple extraction and subsequent fermentation of the strong must, without any addition, are described. These were found to have the following percentage composition:—

——	No. 1. From Currants.	No. 2. From Currants.	No. 3. From Thyra Raisins.
Specific gravity	1·0265	1·0249	1·0131
Alcohol per cent. by weight	11·34	12·03	12·19
„ „ volume	14·29	15·16	15·36
Extractive per cent.	11·55	11·36	8·35
„ „ „ (sugar free)	4·15	4·13	6·03
Inorganic matter	0·298	0·299	0·584
Free acids	0·96	0·79	0·73
Volatile acids	0·18	0·13	0·11
Non-volatile acids	0·73	0·63	0·59
Total tartaric acid	0·17	0·16	0·10
Free „ „	0·0	0·0	0·0
Potassium acid tartrate	0·11	0·15	0·13
Tartaric acid as tartrates of alkaline earths.	0·08	0·04	0·0
Glycerin	1·27	1·33	1·06
Invert sugar	7·40	7·23	2·32
Cane sugar	0·0	0·0	0·0
Sulphuric acid	0·034	0·031	0·025
Phosphoric acid	0·055	0·054	0·092
Tannin and colouring matter	0·09	0·11	0·19
Nitrogen	0·027	0·026	0·50

The ratio of alcohol to glycerin is in No. 1, 100: 11·20; in No. 2, 100: 10·9; in No. 3, 100: 8·69. Fermentation of concentrated must therefore produce glycerin in practically the same proportion to the alcohol formed, as in ordinary must. The proportion of volatile acids is low for sweet wines. The figures for sugar-free extractive and for phosphoric acid are satisfactory.—J. O. B.

Yeast; Autofermentation and Liquefaction of Pressed ——. A. Harden and S. Rowland. Proc. Chem. Soc. **17**, [241], 189.

The time required for the liquefaction of pressed yeast is greatly diminished by rise of temperature, whilst the rate of evolution of carbon dioxide is greatly increased. The evolution of gas ceases as soon as the yeast becomes liquid, and hence is small at 50°, owing to the rapid liquefaction of the mass (1—1½ hours), whilst the total volumes evolved at 39° and 26° do not differ greatly, although the times required for liquefaction are 5 and 53 hours respectively. At 14° the time required is 16 days, the rate of evolution being extremely slow, and the total volume evolved about 75 per cent. of that produced at 26°. Alcohol is produced simultaneously, the process being apparently a true alcoholic fermentation of the glycogen of the cell. Microscopic examination shows that as the evolution of gas proceeds, the glycogen disappears and the vacuole of the cell increases in size. Finally, the vacuolar contents are discharged and the cell appears shrunken and irregular in outline, whilst the protoplasmic contents are highly granular.

In the presence of oxygen, a process of oxidation accompanies the evolution of carbon dioxide, a considerable rise of temperature being produced and the total volume of carbon dioxide increased. When exposed to a continuous current of oxygen, a sample of yeast was found to lose 26 per cent. of its carbon.

PATENTS.

Mashing and Fermenting Vessels; Improvements in ——. A. Meyer, Paderborn, Germany. Eng. Pat. 377, Jan. 7, 1901.

The inventor claims the use of aluminium linings for mashing or fermenting purposes. The outer mantle is constructed of sheet-iron, steel, or copper plate, to which the inner shell of aluminium is fixed either by solder or rivets. Such vessels do not rust, they are more durable than those made of ordinary metal, they are easily kept clean, they do not spoil the beer, and they are not expensive.—J. F. B.

Alcoholometers; Improvement in ——. T. C. Manley, London. Eng. Pat. 20,165, Oct. 9, 1901.

The improvements introduced in connection with ebullition alcoholometers consist of a somewhat conical shaped boiler; a hemispherical bottom to the boiler; a dished plate having its convex surface attached to the hemispherical bottom in order to distribute the heat of the flame; a straight or uncoiled pipe surrounded by a water jacket, standing up from the cover of the boiler and the lower end of the pipe opening into the boiler; a thermometer, the lower end of which dips into the boiler, and a support and sliding scale for use with the thermometer.—J. F. B.

Liquid and Gas; Process and Apparatus for Treating Materials with [Treatment of Beet] ——. P. Naef, Eng. Pat. 17,054, Sept. 25, 1900.

See under I., *page* 1195.

Casks, Jars, Bottles, and Other Vessels, Mode of, and Apparatus for Purifying ——*; for the Purpose of Destroying Germs of Disease and Putrefaction.* H. Hill. Eng. Pat. 20,567, Nov. 14, 1900.

See under XVIII. C., *page* 1230.

XVIII.—FOODS; SANITATION; WATER PURIFICATION, & DISINFECTANTS.

(*A*.)—FOODS.

Report of the Departmental Committee Appointed to Enquire into the Use of Preservatives and Colouring Matters in the Preservation and Colouring of Food, together with Minutes of Evidence, Appendices, and Index. Parliamentary Blue Book. (Messrs. Eyre and Spottiswoode, East Harding Street, Fetter Lane, London, E.C. 1901. Price, 4*s.* 3*d.*)

It is recommended, in the first place, that the use of formaldehyde or formalin, or preparations thereof, in foods or drinks, be absolutely prohibited, and that salicylic acid be not allowed in a greater proportion than one grain per pint in liquid food, or one grain per lb. in solid food, its presence in all cases to be declared. The next recommendation is that the use of any preservative or colouring matter whatever in milk offered for sale in the United Kingdom be constituted an offence under the Sale of Food and Drugs Acts, while the only preservative which it shall be lawful to use in cream should be boric acid or mixtures of boric acid and borax, and in amount not exceeding 0·25 per cent. expressed as boric acid, the amount of such preservative being notified by a label upon the vessel. The only preservative which the Committee recommends should be permitted to be used in butter and margarine is boric acid or mixtures of boric acid and borax, to be used in proportions not exceeding 0·5 per cent. expressed as boric acid. In the case of all dietetic preparations intended for the use of invalids or infants, it is recommended that chemical preservatives of all kinds be prohibited, also that the use of copper salts in the so-called greening of preserved foods be prohibited. Finally, it is recommended that means should be provided, either by the establishment of a separate Court of Reference or by the imposition of more direct obligation on the Local Government Board, to exercise supervision over the use of preservatives and colouring matters in foods, and to prepare schedules of such as may be considered inimical to the public health.

Proteids or Albuminoids; Present Position of the Chemistry of ——. A. Kossel. Ber., **34**, [13], 3214—3245.

An important and exhaustive monograph with ample references.

Milk, Sterilisation of ——*, by Hydrogen Peroxide.* H. Chick. Centr. Bl. f. Bakter. u. Parasitenk. **7**, [2], 705—717. Chem. Centr. 1901, **2**, [17], 942.

The author states that hydrogen peroxide is a suitable agent for preserving milk for analytical purposes, as it does not alter the composition of the sample. The commercial "10 per cent. solution" can be used, an addition of 20 c.c. per litre being sufficient.

It was found that, for the complete sterilisation of milk, an addition of 2 parts per 1000 of H_2O_2 was necessary; the addition of 1 part per 1000, however, sufficed to keep the milk sweet and uncurdled for a week or even longer. The same results were obtained with milk in which propagation of bacteria had occurred, as with fresh milk, but with skimmed milk, a considerably smaller proportion of H_2O_2 was sufficient.—A. S.

Olive-Leaf Oil. Hænsel. Pharm.-Zeit. **46**, 582.

By steam distillation of dry olive leaves, 0·04 per cent. of an unctuous essential oil of a yellow colour and a peculiar very pleasant odour was obtained.—A. S.

Butters; Observations on the Composition of ——. A. Pagnoul. Bull. de l'Assoc. des Chim. de Sucr. et de Dist., 1901, **19**, [4], 414—418.

The author compares the percentage of volatile acids in a number of butters of French manufacture and known purity with the values obtained for Dutch butters, some of which were prepared by a Commission for the study of Dutch butters, whilst others were purchased from the manufacturers, and in the market. The determination is made as follows:—5 grms. of the melted and filtered butter are saponified with 2·5 c.c. of concentrated potash solution (120 grms. KOH per 100 c.c.). The soap is dissolved in water, a slight excess of phosphoric acid added, and the mixture steam-distilled. The distillate is neutralised with

—	French Butters	Dutch Butters.
Above 6 per cent.	58	0
Between 6 and 5 per cent.	41	57
„ 5 „ 4 „	1	41
Below 4 per cent.	0	2

N/5 sodium carbonate, the number of c.c. used, multiplied by 0·352, gives the percentage of volatile acids expressed as butyric acid. The percentage of the samples containing various amounts of volatile acids are given in the table on preceding page.

Thus, the Dutch butters contain far lower proportions of volatile acids than those of French production, a result possibly due in part to the fact that the former were prepared in October. The author points out that a permissible minimum of 5 per cent. of volatile acids would admit many butters admixed with margarine.—T. H. P.

Casein with Phosphoric Acid; Production of a Soluble Compound of ——. Chem. Fabrik, Rhenania, Aachen. Ger. Pat. 123,555, Aug. 11, 1900. Through Zeits. angew. Chem. **14**, [43], 1094.

The casein is dissolved in weak phosphoric acid, and precipitated by a soluble phosphate. The precipitate is washed free from acid with the phosphate solution, and is then soluble in water.—J. T. D.

Salicylic Acid; Occurrence of ——, *in Strawberries.* L. Portes and A. Desmoulières. J. Pharm. Chim. 1901. **14**, [8], 342—351.

According to the authors' experiments, fresh strawberries contain an appreciable amount of salicylic acid in the form of a salt. It was isolated by the following method:—Five kilos. of strawberries were macerated in a mixture of alcohol and water, and two litres of the extract freed from tannin by means of gelatin, and, after the addition of sodium hydroxide, boiled under a reflux condenser. The alcohol was next distilled off, and the residue acidified with dilute sulphuric acid, and shaken with benzene. The benzene extract gave a violet coloration with ferric chloride, and this liquid, after removal of the iron by means of sodium hydroxide, was acidified with sulphuric acid, and shaken with ether. The residue left on evaporation of the ethereal extract was taken up with absolute alcohol which, on concentration, deposited microscopic crystals of salicylic acid. The amount of salicylic acid determined colorimetrically was found to be about 1 mgrm. in a kilo. of Jugonda strawberries.

Its presence was also identified by various tests in nine other varieties of strawberries.—C. A. M.

PATENTS.

Preserving Food; Media for ——. F. C. Kullak, Berlin. Eng. Pat. 7236, April 6, 1901.

The preserving salt claimed is prepared by dissolving, evaporating, and re-crystallising a mixture of about 20 parts of borax, 30 parts of boric acid, 50 parts of sodium chloride, and 0·001 part of tartaric acid.—W. P. S.

Flour, Bread, and other Food Material; [Medicinal] ——. B. Hoffmann, Buda-Pesth, Hungary. Eng. Pat. 17,729 Sept. 4, 1901.

In making the bread claimed, 1 grm. of sodium bromide, instead of sodium chloride, is added to every 50 grms. of flour. Potato flour and carraway seeds or other flavouring material may be added.—W. P. S.

Margarine; Manufacture of ——. A. Pellerin, Paris, Eng. Pat. 22,905. Under Internat. Conv., July 12. 1900.

To render margarine more like butter in appearance, the patentee adds from 0·5 to 5 per cent. of animal or vegetable wax to the fats and oils used for the manufacture of the margarine. The method and the product are claimed.—W. P. S.

Margarine; Production of ——. J. H. G. Neisse, Hamburg and J. H. Boll, Hanover, Germany. Eng. Pat. 21,626, Nov. 29, 1900.

For producing a margarine which will "brown" easily on cooking, the patentees first mix yolk of egg with fresh milk or cream and sourish milk. This mixture is then churned, and the resulting egg-butter is incorporated with margarine.—W. P. S.

Eggs; Method of Preserving ——. K. E. Fryklind, Stockholm, Sweden. Eng. Pat. 9898, May 13, 1901.

The eggs are coated with linseed oil or other drying oil, and the latter is rapidly dried by means of an oxidising agent, such as potassium permanganate, chromic acid, ozone, &c. By quickly drying, the oil is prevented from penetrating to the interior of the eggs. The eggs may also be coated with dextrin before oiling.—W. P. S.

Fluids [Milk]; Apparatus for Equalising the Temperature of ——. T. T. Sabroe and H. J. T. Hansen. Eng. Pat. 13,128, June 27, 1901.

See under I., *page* 1194.

(*B.*)—SANITATION; WATER PURIFICATION.

Expired Air; Regeneration of —— *by Sodium Peroxide.* A. Desgrez and V. Balthazard. Comptes Rend. **133**, [20], 791—792.

The authors have made some improvements in their apparatus (this Journal, 1900, 920). The chief of these are, that the circulating fan is set to work, not electrically, but by the same clockwork which controls the supply, at regular intervals, of sodium peroxide into the regenerator; and that a receptacle of methyl chloride is provided to cool the air, and thus do away with the manipulation of a freezing mixture. The apparatus can thus be charged and kept ready for use: when required, one turn of a screw sets the clockwork in motion and opens a partition which separated the peroxide hopper from the regenerator, while the opening of a stopcock starts the evaporation of the methyl chloride.—J. T. D.

Water-borne Enteric Fever amongst Armies in the Field; Suggested Method of Preventing ——. L. Parkes and S. Rideal. Trans. Epidemiological Soc. of London, N.S. Vol. **20**, (1900–1901).

After pointing out the drawbacks to sterilisation by means of boiling or filtration, the authors proceed to inquire what substance added to water will destroy the virulence or inhibit the growth of the bacillus-typhosus, whilst not rendering the water unpalatable or injurious to health. After referring to previous investigations on this subject, they give the results of experiments showing that sodium bisulphate, tartaric and citric acids, dilute sulphuric acid, and dilute nitro-hydrochloric acid, when used in sufficient strength, and allowed to remain for a sufficient time in contact with infected water, are all capable of preventing any further growth of typhoid-bacilli. In the case of tartaric, citric, and nitro-hydrochloric acids, it was found that nothing less than the B.P. dose per pint of water sufficed to sterilise during 15 minutes' contact. Nitro-hydrochloric acid was more efficient than sulphuric, but no further experiments were made with these acids, as it was considered that liquids would be inconvenient in use, and that whatever substance was employed should be a solid. Sodium bisulphate seemed suitable, 1 grm. (15·5 grains) per pint of infected water, being sufficient to kill typhoid-bacilli during 5 minutes' contact, and to completely sterilise the infected water after 15 minutes' contact. The acid salt is probably not purgative, as the astringent properties of the free acid would tend to counteract the laxative action of the sodium sulphate, besides which the quantity necessary to be used is very much less than the minimum dose (¼ oz.) of the latter salt. Acid sodium phosphate and sodium sulpho-vinate were tried, but were found to be inefficient.

Further experiments showed that sodium bisulphate when used in the proportion above stated is effective after 15 minutes' contact in sterilising water containing as many as 50,000 colonies of *b. typhosus* per 1 c.c., but when the water is much more heavily infected, a longer contact is necessary.

After pointing out that only in about 1·25 per cent. of cases of enteric fever, the urine is so highly charged with the bacilli as to be turbid (in which case water infected with such urine might contain more than the above number of bacilli), and that it is very doubtful if the bowel excreta ever contain anything like the numbers met with in the urine of convalescing patients, the authors think they are justified in assuming that water naturally infected with the virus of the disease does not contain so very large a proportion of the bacilli, and, that, therefore, the use of such simple means of combating infection as they suggest has possibly a future before it, more especially under conditions where the means of sterilisation by boiling or filtration are not readily available. They recommend that the sodium bisulphate should be used in the form of tabloids, each containing 5 grains, and made so as to dissolve quickly. These should be contained in light metallic boxes holding about 350 tabloids, or ¼ lb., sufficient to sterilise over 100 pints of water. The acid tabloids have the additional advantage of rendering the water more effective in slaking thirst, and therefore they tend to reduce the quantity of water consumed.—L. A.

Sand Filters ; Note on the Working of ——. H. F. Rutter, Proc. Inst. Civil Engineers, **146**, (1900—1901), Part 4.

A FEW years ago it was believed that the function of the sand in ordinary sand filters was quite a secondary one, that the sand merely served as a convenient support for the surface film, on which the efficiency of the filter actually depended. It was stated that the integrity of this film must be maintained with the greatest care, as its rupture would be followed by immediate deterioration of the filtrate from a bacteriological point of view. In the practical working of open filters, however, the maintenance of the surface film is impossible. In the summer months gases, generated among the low forms of vegetable life which grow on the surface of the sand, float these matted growths to the surface of the water, thus frequently exposing clean patches of sand to the passage of the water. In mechanical sand filters, the supposed necessity of maintaining the surface film is entirely ignored.

In 1899, the chief chemist to the Massachusetts Board of Health, communicated to the author some results which seemed conclusively to prove that the rupture of the surface film has no prejudicial effect on the filtrate. In a filter plant with which the author is connected, great inconvenience was formerly caused in the spring of every year, and sometimes during the summer months, through the rapid clogging of the surface of the sand by the formation of a film which, though of extreme tenuity, was sufficient to entirely prevent the passage of water. Filters which, ordinarily, continued to work efficiently for 2—3 months, became useless after 5—6 days' working. Arrangements were therefore made to break up the film by passing a rake across the face of the filter-bed when in this condition, and after 18 months' experience this has been found to result in greatly increased facility of working, and material reduction of expense, while the high character of the filtrate has been fully maintained.

Immediately the filter shows signs of clogging, the outlet is closed and an interval of about six hours is allowed to elapse to ensure that the whole body of sand below the film is fully charged with water. The film is then disturbed by the rake, and after another resting period of six hours, it is put into use again. It has been frequently observed that a filter which has shown signs of becoming blocked up within five or six days of ordinary cleaning, will work for 3—4 weeks after raking the surface before it again requires raking. The filters in question can be thus raked two or three times before they require cleaning in the usual manner.—L. A.

PATENT.

Water ; Purification or Sterilization of ——. R. Orchard and C. E. Fox, London. Eng. Pat. 497, Jan. 8, 1901.

THE patentees claim the process of sterilising water by treating it for a period of at least 15 minutes with a small quantity (20—25 grains per pint of water) of sodium bisulphate. Tablets are prepared by mixing 25 grains of the bisulphate with four grains of sodium bicarbonate, and one or two grains of dextrin as a binding agent. When the tablets are placed in water, a portion of the bisulphate reacts with the bicarbonate, and the effervescence so caused promotes the solution of the remainder of the bisulphate and its distribution throughout the volume of water.—L. A.

(C.)—DISINFECTANTS.

Ammonium Ichthyol-Sulphonate ; Preparation of ——. A. C. McLaughlin. Amer. Pat. 681,568. Pharm. Zeit. **46**, [86], 858.

ASPHALT is submitted to distillation, the hydrocarbons collected in suitable vessels, and the sulphocarbonates shaken with strong sulphuric acid until reaction is complete. The liquid then separates into two layers, a dark heavy portion containing the sulpho-acids and a lighter supernatant fluid. The former is separated and neutralised with ammonium carbonate, when impure dark coloured ammonium ichthyol sulphonate is formed. This is washed with light petroleum spirit, dissolved in methyl alcohol and filtered. On distilling off the solvent, ammonium ichthyol-sulphonate is obtained.—J. O. B.

Creosote ; Determination of ——. B. Hafner and W. Kreissl.

See under XXIII., *page* 1251.

Creosote for Therapeutic Purposes ; Examination of ——.

See under XXIII., *page* 1245.

Formaldehyde ; Determination of ——. L. Vanino and E. Seitler.

See under XXIII., *page* 1251.

Mercury in Combination with Chlorine, Iodine, Cyanogen, Determination of ——, *in Antiseptic Solutions.* G. Meillère.

See under XXIII., *page* 1243.

PATENTS.

Casks, Jars, Bottles, and other Vessels for the purpose of Destroying Germs of Disease and Putrefaction : Mode of, and Apparatus for Purifying ——. H. Hill, London. Eng. Pat. 20,567, Nov. 14, 1900.

THE casks, &c. are placed in a closed chamber, where their interiors and exteriors are exposed to the action of sulphur dioxide, formaldehyde, or other suitable gas or vapour; or the casks are connected together in series by piping, and the gas or vapour passed through them successively. They are then rinsed out with hot water and dried.—L. A.

Disinfectant Compound ; An Improved ——. A. Strandh, Stockholm, Sweden. Eng. Pat. 508, Jan. 8, 1901.

THE disinfectant called "Lettubrin," for destroying all kinds of vermin, is "composed of 3—50 parts by weight of carbolic acid, 1—50 parts by weight of concentrated acetic acid, 1—10 parts by weight of benzene, and 72 parts by weight of 96 per cent. alcohol."—L. A.

Moths ; Improved Means for Exterminating ——, *and Process for the Producton of such Means.* A. Kornfeld and J. H. Zirner, Vienna. Eng. Pat. 18,560, Sept. 17, 1901.

FINE sawdust is mixed with powdered ammonium carbonate "prepared with oil of lavender," then added to diluted acetic acid, and stirred until effervescence ceases. Another portion of sawdust is mixed with diluted acetic acid, camphor dissolved in alcohol, and oil of turpentine. The two portions are then mixed together, and to the whole is added a further portion of ammonium carbonate "prepared with oil of lavender." The mass thus prepared "is allowed to dry in a tightly closed vessel." In use it is sprinkled about in rooms containing the articles to be protected from moths, or on the articles themselves.—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

Cellulose [Wood Pulp] ; Alkaline Process of Boiling ——. W. Schacht. Papier Zeit. 1901, **26**, [84], 3143-3144.

IT is well known that the alkaline processes for obtaining cellulose from wood on the industrial scale are attended with very large losses of fibre substance. For instance the soda and sulphite process only yields from 28 to 38 per cent. of unbleached pulp from wood as compared with a possible yield, according to Dammer, of 60 to 80 per cent. In the same way the yield of cellulose from straw varies from 36 to 48 per cent., whilst the theoretical proportion of cellulose and cellulosic matters is, in the author's opinion, from 56 to 70 per cent.

In order to restrict this loss of cellulose, the liquors should be far less alkaline or even neutral. Another drawback, which attends the processes in which alkaline sulphides are employed, is the very disagreeable and injurious odour evolved from the liquors owing to the production of mercaptans and similar bodies from the portions of the cellulose dissolved. Finally, cellulose produced by the alkaline processes is very prone to destructive fermentation and the attacks of fungi.

The author's new process for boiling wood, straw, &c., utilises the solvent action of sodium sulphite on the non-cellulose constituents of the material. In addition to avoiding the evil smell produced by the sulphide process, the new method has the advantage of increasing the yield of cellulose by 15 or 20 per cent., and of producing a cellulose of far higher quality, more comparable with the acid bisulphite product. The new liquor is prepared by nearly saturating the crude recovered soda solution with sulphurous acid, and then causticising the residual carbonate. The liquor consists, therefore, of a mixture of sodium sulphite, sodium thiosulphate and caustic soda. The last-named alkali is still necessary to a certain extent in order to deal with the silicates and alumina of the raw material, and it should not exceed the proportion required for this purpose. This process is being tried on the large scale, and is likely to be adopted by several factories in the near future.—J. F. B.

Silica; Removal from Alkaline Liquors of ——, especially from the Liquors of the "Sulphite" Cellulose Process. G. Lunge and W. Lohöfer. Zeits. angew. Chem. 1901, **14**, [44], 1102—1110.

In the boiling of wood, and especially of straw, by the alkaline process, a considerable proportion of the soda combines with the silica of the plant-ash forming silicate of soda, which is of no value for the boiling. This silicate has to be decomposed and the soda made available without at the same time destroying the valuable sodium sulphide. It is true that in the subsequent causticising of the recovered soda-ash the greater part of the silicate is precipitated as calcium silicate. But this makes the waste lime sludge so slimy that the removal of the caustic liquor by filtration and washing is very difficult and wasteful. The silicate must, therefore, be decomposed, and the silica may be removed from the solution of the recovered ash before causticising. The decomposition is effected by saturation with carbon dioxide, but experiments showed that under ordinary pressure at any temperature, the precipitated silica is so gelatinous and voluminous that the same difficulties as regards filtration and washing occurred here also. The problem was ultimately solved by saturating the dissolved ash with carbon dioxide whilst boiling under a pressure of about 7 lb. per sq. in., *i.e.* at temperatures from 103°—107° C. The silica was thus precipitated practically completely, and in a form in which it could be filtered off rapidly with a clear filtrate. If the saturation be stopped as soon as the whole of the silicate is decomposed, the sodium sulphide remains unaltered.

The sodium of the silicate forms a carbonate having a composition corresponding approximately with the formula $Na_2CO_3 + 2NaHCO_3$.

A 10 per cent. solution of sodium metasilicate Na_2SiO_3 after saturation in the above manner and filtered, required for a 96 per cent. causticisation 120 per cent. of the theoretical quantity of lime calculated for the carbonic acid; calculated for the soda present 170 per cent. of the theoretical quantity of lime was required because of the acid nature of the carbonate to be treated. For causticisation without previous filtration of the precipitated silica, the latter still combined with 75 per cent. of the quantity of lime required to produce $CaSiO_3$, probably with the formation of an acid silicate of the formula $4(SiO_3)Ca_3H_2$. Thus for the technical causticisation, without filtration, 250 per cent. of the theoretical quantity of lime calculated for the soda of the sodium silicate was required.

The authors invite experiments on a practical scale in order to determine whether the advantage of the removal of the silicate will compensate for the extra cost of the process.—J. F. B.

PATENTS.

Cellulose Solution; Manufacture of ——. J. Imray, London. From E. Bronnert, Niedermorschweiler, Alsace, and M. Frémery and J. Urban, both of Dreunmen, Aachen, Germany. Eng. Pat. 20,801, Nov. 17, 1900.

Ammonia solution (16—18 per cent.) is saturated in the cold with cupric carbonate, and cellulose is dissolved therein. No oxycellulose is formed even after a long period.
—R. L. J.

[Waterproof] Paper, Cardboard, and the like; Improved Manufacture of ——. L. Joseph, Cologne, Germany. Eng. Pat. 536, Jan. 8, 1901.

The ordinary process of waterproofing paper, &c. (*i.e.*, dressing with a solution of paraffin wax in hot linseed oil), is supplemented by a second treatment of paraffin and oil mixed with a solution of shellac, or any gum-lac, gum-resin, wax, or albumin, dissolved in spirit or other mineral solvent.

Talc or similar substance is then strewed on the paper and rolled in to produce a smooth glazed surface.
—R. L. J.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

Thorium; Existence of a New Element associated with ——. C. Baskerville. J. Amer. Chem. Soc. **23**, [10], 761—774.

The purest thorium salts obtainable were re-purified by converting them into sulphate; dissolving in cold water; adding solution of sodium sulphate, and allowing to stand 12—24 hours to separate any small amount of cerium compounds present, filtering, and precipitating the hydroxide by a large excess of sodium nitrite. After copious washing by decantation, the hydroxide was converted into chloride, reprecipitated by ammonia in slight excess, and washed thoroughly by decantation.

1. *Variation in the Specific Gravity of the Oxide.*—Muthmann and Böhm (this Journal, 1900, 267) have recommended the determination of the sp. gr. of the oxides as a means of judging the progress of the fractionation of the rare earths. The sp. gr. of ThO_2, as given by various observers, has varied from 9·077 to 10·2206; that of the oxide obtained by prolonged ignition of the hydroxide, purified as above, was 10·1 (corrected to 4° C., as in all cases throughout this paper). Part of the purified hydroxide was dissolved as chloride, and the solution, after neutralisation with ammonia, was saturated in the cold with SO_2. The basic sulphite which separated out was filtered off, the filtrate was boiled, filtered, and the earth remaining in solution was precipitated with ammonia. On igniting the two products to oxide, that obtained from the basic sulphite had a sp. gr. of 9·38, and that yielded by ammonia, 10·367.

Another portion of the purified hydroxide was added to a cold saturated solution of citric acid until no more was dissolved after 12 to 15 hours' stirring. On heating the diluted, filtered liquid at about 100° C., a white precipitate consisting of "an hydrated citrate of the real thorium" was obtained, which gave on ignition an oxide of sp. gr. 9·210—9·253. The filtrate, on evaporation to solidification, yielded on ignition an oxide of sp. gr. 10·50. A larger quantity of the saturated citrate solution, after removal of the precipitated citrate as just described, was evaporated till a crystalline scum began to form; on cooling over-night, a few crystals separated, which yielded an oxide of sp. gr. 8·47—8·77. On further concentration of the syrupy liquid, a white crystalline body, yielding an oxide of 10·14 sp. gr., separated out; and, after further concentration of the decanted solution, dilution with water, and boiling, a small precipitate was obtained, which yielded an oxide of 11·26 sp. gr. The filtrate from this was evaporated to dryness and ignited, giving an oxide of 10·46—10·53 sp. gr. The author considers that these results point to the presence of either an unknown element or a new oxide of greater density than the usual residue on ignition.

2. *Radio-activity of the Oxides.*—The oxide (sp. gr. 9·25) obtained from the insoluble citrate affected the sensitive plate but slightly, after an exposure of 72 hours, while the oxides of higher sp. gr. were quite active, the radioactivity increasing with increase of sp. gr. The 9·25 oxide is, therefore, probably not quite free from the oxide of higher sp. gr.

3. *The Atomic Weight of Thorium.*—Thorium dioxide, prepared from "the purest insoluble citrate" by ignition, was used for the determination, being converted into $ThCl_4$. The numbers 223·2 and 223·3 were obtained, and finally 222·13, after a more searching trial. These results lead the author to believe that ordinary thorium is not a simple substance, but contains an element which, if tetravalent, has an atomic weight lying between 260 and 280. If further investigation confirms this conclusion, he suggests naming the element *Carolinium*, with the symbol *Cn*.
—H. B.

Glycerophosphorous Acid and Glycerophosphites. A. and L. Lumière and F. Perrin. Comptes Rend. **133**, [17], 643—644.

Phosphorus trichloride and glycerin are allowed to act on each other in the cold; the product is dissolved in cold water, and the solution neutralised with calcium carbonate. The clear liquid is concentrated at a low temperature to a syrup, and the calcium glycerophosphite precipitated by alcohol. Glycerophosphorous acid has not been isolated: even dilute solutions decompose slowly in the cold, and very rapidly on heating. Most of its salts are soluble in water. The solution of the calcium salt can be boiled without decomposing, though it rapidly decomposes when a few drops of hydrochloric acid are added to the hot solution.—J. T. D.

Carbon Tetrachloride; Process for Manufacturing ——. Urbain. Rev. Prod. Chim. **4**, [15], 228.

Two parts by weight of aluminium chloride are boiled with 100 parts of carbon bisulphide, for about half-an-hour, the liquid being then cooled and treated with a current of pure dry chlorine until the weight has increased to 383 parts. To destroy the chlorosulpho products contaminating the carbon tetrachloride, the liquid is treated with about 0·05 per cent. of iron dust, or other analogous metal, and gently heated, the final products of the reaction being carbon tetrachloride and sulphur chloride. The former is separated by distilling at 60°—90° C., and is purified by washing with alkaline water and redistillation. The residual sulphur chloride is utilised for the furthur production of carbon tetrachloride by treating it with carbon bisulphide, in the proportion of 3 mols. of the former to 1 mol. of the latter, and 1 per cent. of iron dust. The ensuing brisk reaction is facilitated by gentle heat, applied until the liquid becomes quiescent, whereupon the product is distilled to recover the carbon tetrachloride. An almost theoretical yield is obtained, the extra molecule of sulphur chloride insuring complete conversion and maintaining the sulphur in solution during distillation.—C. S.

Quinine and Cinchonidine; Preparation of Carbonic Esters of ——. Vereinigte Chininfabriken Zimmer and Co., Frankfurt. Ger. Pat. 123,728 (Addition to Ger. Pat. 91,370 of 18th December 1895). (Through Zeits. angew. Chem. **14**, [43], 1094).

An alteration of the method described in the former patent, consisting in the treatment of the hydrated salts of the alkaloids by the esters of chloroformic (chlorocarbonic) acid in the presence of pyridine.—J. T. D.

Saloquinine and Rheumatine. Pharm. Zeit. **46**, [70], 694.

Two new preparations of quinine, which are claimed to have valuable therapeutic properties, have been introduced into commerce by Zimmer and Co.

Saloquinine is the quinine ester of salicylic acid $C_6H_4OH.COO.C_{20}H_{23}N_2O$; it occurs in crystals, melting at about 130° C., is insoluble in water, but soluble in alcohol and in ether. It is a powerful bactericide and prevents the growth of moulds, and also possesses marked analgesic properties when administered internally.

Rheumatine is the salicylate of saloquinine $C_6H_4OH.COO.C_{20}H_{23}N_2O.C_6H_4OH.COOH$. It occurs in white needles, very soluble in water, melting at 179° C.—J. O. B.

Cinchonine. E. Jungfleisch and E. Léger. Bull. Soc. Chim. 1901, **25**, [18], 880—884.

Having shown that hydrocinchonine is identical with cinchonifine, the authors recall the fact that when cinchonine is treated with aqueous sulphuric acid, a large proportion of hydrocinchonine is always obtained. They now show that ordinary cinchonine is in reality a mixture of cinchonine and hydrocinchonine; they have determined the proportion of the latter present and also give the properties of pure cinchonine. The basic sulphate of cinchonine employed, was obtained from wild cinchona. 200 grms. of the salt after treatment at 125° C. with sulphuric acid mixed with its own weight of water gave 28 grms. of hydrocinchonine corresponding to 34·28 grms. of the sulphate + $2H_2O$ or 17·19 per cent. Taking account of possible losses in the separation, and the amount of hydrocinchonine retained by the apocinchonine in spite of repeated crystallisations, it would appear that the original salt contained not less than 20 per cent. of hydrocinchonine sulphate. Oxidation with permanganate in ice of a cinchonine sulphate rich in hydrochonine gave only a very small quantity of the latter base. Hence the oxidation of the cinchonine is attended by destruction of the hydrocinchonine, even in the cold. After repeated crystallisations from water and 95 per cent. alcohol, a salt was obtained containing only about 1 per cent. of hydrocinchonine sulphate.

The following table gives the differences in solubility of one part of each of the various salts in water at different temperatures:—

—	Water at—		
	12° C.	36°·5 C.	101° C.
	Parts.	Parts.	Parts.
Purified cinchonine sulphate $+2H_2O$.	72·1	60·2	12·9
Ordinary cinchonine sulphate, containing about 20 per cent. of hydrocinchonine sulphate.	64·1	55·0	11·3
Hydrocinchonine sulphate....	37·6	34·8	10·7

The melting point of purified cinchonine crystallised in the cold from a mixture of chloroform and alcohol (2:1) was found to be 264°·3 C. The specific rotation of the same product in absolute alcohol (p = 0·1375, v = 30 cc. t = 17° C.) gave $[\alpha]_D$ = + 229°·6. Oudemans (Annalen, 1876, **182**, 44) gave $[\alpha]_D$ = + 223°·3, whilst a mixture of hydrocinchonine and purified cinchonine (1:4) gives $[\alpha]_D$ = + 223°·9. In a 1 per cent. aqueous solution with 4HCl for each molecule of cinchonine $[\alpha]_D$ at 17° C. = + 263°·4. Oudemans (*loc. cit.* 55) gives $[\alpha]_D$ = + 257°·7, whilst for the same mixture as above the authors find $[\alpha]_D$ = + 256°·3. These figures obtained for the mixtures have also been given by a sample of commercial cinchonine examined by the authors. They propose to investigate the question of purified cinchonine, and to determine how it is possible for cinchonine and its salts to crystallise with considerable quantities of hydrocinchonine. If the latter contains 2H more than cinchonine the law of isomorphism does not apply.—T. A. L.

Isoconiine. A. Ladenburg. Ber. 1901, **34**, [13], 3416.

Doubt has repeatedly been cast on the existence of isoconiine, the explanation of the observations made by the author being the presence of a mixture of *d*- and *l*-coniine. The author states that the reasons he gave in Ber. **29**, 2706, for the existence of isoconiine cannot be explained in any other way.—A. C. W.

Hyoscine and Atroscine. O. Hesse. J. prakt. Chem. 1901, **64**, [20], 353—386.

Hyoscine (active or normal scopolamine) has been found in the roots of *Scopolia japonica* and *S. atropoides*, the seeds of *Datura Stramonium*, the leaves of *Duboisia myoporoides*, the flowers of *Datura alba*, the mandragora root (this Journal, 1901, 605 and 1135), and in *Hyoscyamus niger*. When the solutions of the alkaloids, *e.g.*, from *Scopolia* roots, in dilute sulphuric acid, are made slightly alkaline with sodium bicarbonate and extracted with chloroform, the hyoscine is taken up by the solvent, and by a repetition of the process may be freed from hyoscyamine. After removal of the chloroform, the syrupy residue is neutralised with hydrobromic acid, and the hydrobromide purified by recrystallisation from water or alcohol. When the crude alkaloid consists mainly of hyoscine, the hydrobromide may be at once prepared. Hyoscine is decomposed by acids or bases. Hydrochloric acid in a sealed tube at 100° C. gives oscine and atropic acid, $C_{17}H_{21}NO_4 = C_8H_{13}NO_2 + C_9H_8O_2$. Some tropide is also produced. Excess of baryta at 60° C. gives oscine and tropic acid, $C_9H_{10}O_3$. Hyoscine is soluble in 9·5 parts of water at 15° C., more readily in boiling water, and easily in chloroform, alcohol, &c. The alkaloid cannot be crystallised; the solutions all yield a varnish which melts at about 50° C. Hyoscine is separated from solutions of its salts by caustic alkalis and normal carbonates. Bicarbonates give no precipitate from acid solutions, but the alkaloid is completely extracted by chloroform. The *hydrochloride*, $C_{17}H_{21}NO_4.HCl.2H_2O$, separates from strong aqueous solutions as thick glassy crystals; it separates from acetone as an anhydrous white crystalline powder, which melts at 197° C. The gold salt melts at 198° C. with effervescence. The higher melting point found by Schmidt appears to be due to the method of determination. The *hydrobromide* has been obtained in six forms, anhydrous and with $\frac{1}{2}$, 1, 2, and 3 mols. (needles or rhombs) of H_2O, according to the solvent. The *hydriodide*, $C_{17}H_{21}NO_4.HI.\frac{1}{2}H_2O$, melts at 197° C. *Acetylhyoscine*, $C_{17}H_{20}(C_2H_3O)NO_4$, is obtained by the action of acetic anhydride on anhydrous hyoscine hydrobromide; it is an uncrystallisable syrup, somewhat soluble in water and strongly basic. Boiling with excess of baryta water converts it into oscine and atropic and acetic acids.

Atroscine, $C_{17}H_{21}NO_4$, forms crystals which melt at 82°—83° C.; the hydrate, $C_{17}H_{21}NO_4.H_2O$, melts at 56°—57° C., and $C_{17}H_{21}NO_4.2H_2O$ melts at 36°—37° C. The latter hydrate was nearly always obtained by the author, though Gadamer regards it as the unstable form. Atroscine dissolves in 37 parts of water at 18° C.; it is optically inactive. Caustic potash and baryta water decompose it into oscine and tropic (or atropic) acid. The *hydrochloride*, $C_{17}H_{21}NO_4.HCl$, crystallises in long needles easily soluble in water. The gold salt is similar to the hyoscine gold salt; it melts at 201°—202° C. and effervesces at 204°—206° C. The *hydrobromide* separates from strong solutions in rhombic tables, $C_{17}H_{21}NO_4.HBr.\frac{1}{2}H_2O$, from weaker solutions with $3H_2O$, and from hot acetone as an anhydrous crystalline powder. *Acetylatroscine* is obtained by heating a solution of the base in acetic anhydride at 80°—90° C. for two hours. It is an uncrystallisable syrup, readily soluble in water. The solution has an alkaline reaction. *Atroscine methyl iodide*, $C_{17}H_{21}NO_4.CH_3I$, crystallises from chloroform in white leaflets, which melt at 202° C. The ethyl iodide compound is obtained by heating in a sealed tube at 80°—90° C.; it crystallises from hot water in fine octahedra, which melt at 170° C.

The optical rotation of anhydrous hyoscine hydrobromide was found to be $[\alpha]_D = -25{\cdot}9°$, which fell to −25·4° after keeping for 18 months, the original value being regained after recrystallisation from water. Wentzel, however, found −32·9°, falling to −25·79° after one year. The alkaloid was separated from the hydrobromide which gave −25·4°; when seeded with atroscine it formed crystals in quantity corresponding to 8·5 per cent. of atroscine hydrobromide. The hydrobromide from the mother liquor then gave $[\alpha]_D = -32{\cdot}9°$, but a second crop showed only $[\alpha]_D = -29{\cdot}3°$. An explanation of this difference is still to be found.

For the following experiments on the conversion of hyoscine into atroscine, the hydrobromide of $[\alpha]_D = -25{\cdot}9°$ was used, which the author considered to be pure. After fusion the rotation was −23·7°, and after keeping for 18 months in the dark, −25·4°. The activity of the pure alkaloid also slightly decreased on keeping in diffused light. Silver oxide decreases the activity by about one-eighth on standing in aqueous solution for five days. The conversion does not proceed further. Sodium and potassium carbonates in aqueous solution, barium and calcium hydroxides in aqueous and alcoholic solutions, are without action at 50° C. At 60° C. baryta decomposes the alkaloid on long-continued action. Caustic potash and soda rapidly decompose hyoscine in alcoholic solution, but are without action on an aqueous solution. Atroscine is best obtained by the action of small quantities of caustic soda on an alcoholic solution: a strong solution of 0·117 grm. of caustic soda is added to a solution in 60 c.c. of absolute alcohol of the alkaloid from 10 grms. of the hydrobromide. After standing 10 hours at the ordinary temperature, the solution is neutralised, alcohol removed, the alkaloid separated by soda and chloroform, the solvent distilled off, the syrupy residue stirred with water in a dish, and left to crystallise at a low temperature. Atroscine and hyoscine are similar in physiological action, but the former has certain advantages. Solutions of the alkaloids in castor oil are used in ophthalmic practice in England.

Oscine (Schmidt's scopoline), $C_8H_{13}NO_2$, crystallises in colourless prisms, sublimes at 90° C., distils at 241°—243° C., and when free from moisture melts at 106°—107° C. The base is tolerably hygroscopic. Oscine contains a hydroxyl group which can be readily esterified, but bases corresponding to atroscine cannot be obtained by evaporation with the proper acids and hydrochloric acid. It is not reduced by hydriodic acid and red phosphorus at 140° C.; also hydroxylamine and phenylhydrazine do not react with the base.—A. C. W.

Strychnine and Brucine; Electrolytic Reduction of——. J. Tafel and K. Naumann. Ber. 1901, **34**, [13], 3291—3299.

By the electrolytic reduction of strychnine in sulphuric acid solution with a lead cathode, tetrahydrostrychnine and strychnidine are produced (this Journal, 1898, 945). By using the closed apparatus (this Journal, 1899, 909) it is now found that tetrahydrostrychnine is the initial product of the reduction; it is formed in much the larger quantity at low temperatures, and the higher the temperature the more strychnidine is obtained.

Brucine is also reduced by electrolysis in sulphuric acid solution with a lead cathode; it is necessary to keep the temperature below 15° C., otherwise uncrystallisable compounds are produced, readily acted on by light. The filtered cathodic liquid gave with ammonia a reddish amorphous precipitate, which was dissolved in chloroform, the solution dried, the solvent removed, and the residual syrup dissolved in warm methyl alcohol. This solution was evaporated and the process repeated, in order to remove chloroform completely. On cooling, tetrahydrobrucine crystallised out. By rapid and repeated recrystallisation from hot methyl alcohol it was obtained colourless.

Tetrahydrobrucine, $C_{23}H_{30}O_4N_2$, begins to decompose in an evacuated capillary at 185° C., and melts at 200°—201° C. to a clear yellowish liquid. It is slightly soluble in water, the solution having an alkaline reaction. The base, similarly to tetrahydrostrychnine, forms two series of salts. Nitrous acid does not yield a nitrosamine; its action appears to be oxidising or condensing.

Brucidine, $C_{23}H_{28}O_3N_2$, is not found as a direct product of the electrolytic reduction. It is obtained by heating tetrahydrobrucine at 215°—220° C. in the oil-bath and recrystallising from warm acetic ester. Brucidine is perceptibly soluble in boiling water; the solution has an alkaline reaction.

Quinine, cinchonine, and cinchonidine are much more easily reduced electrolytically than strychnine and brucine. Four atoms of hydrogen are taken up by 1 mol. of alkaloid. No crystalline compound could be obtained from the products, which are exactly similar to the substance obtained by v. Norwall by reducing the alkaloids with sodium in amyl alcohol solution (Ber. **28**, 1637; **29**, 803). —A. C. W.

Thiopyrine and Selenopyrine. Michaelis. Zeits. angew. Chem. 1901, **14**, [41], 1027.

THESE compounds are obtained by the action of potassium hydrosulphide or potassium selenide (as the case may be) on the so-called antipyrine chloride. Thiopyrine readily combines with methyl iodide, and is characterised by its behaviour towards sulphurous acid. It is also converted by oxidation with chlorine in an aqueous solution into an inert dioxide. The author concludes that both compounds have the same constitution as antipyrine. He also gives particulars of their physiological action.—C. A. M.

Corydalis cava; The Alkaloids of ——. Gadamer and others. Chem.-Zeit. 1901, **25**, [80], 863.

THE alkaloids known to be present in the root-nodules of *Corydalis cava* are:—Corydaline, $C_{22}H_{27}NO_4$, m.p. 134·5° C.; corybulbine, $C_{21}H_{25}NO_4$, m.p. 238°—239° C.; corycavine, $C_{23}H_{23}NO_6$, m.p. 216°—217° C.; bulbocapnine, $C_{19}H_{19}NO_4$, m.p. 199° C. corytuberine, $C_{19}H_{25}NO_4$, m.p., 200° C. (Dobbie and Lauder); corydine, m.p. 65°—75° C. The last named is now found to be a mixture of the following crystalline bases:—

Isocorybulbine, $C_{21}H_{25}NO_3$, m p., 179°—180° C.
Corycavamine, $C_{21}H_{21}NO_5$, m.p., 149° C.
Corydine, $C_{21}H_{25}NO_4$, $C_{21}H_{23}NO_4$, m.p. 129°—130°.

Base, not identical with corydaline, m.p. 135° C., and, in addition, two amorphous bases, one of which forms a crystalline hydrochloride. The alkaloids (eleven in all) fall into three groups in order of basicity.

I. *Weak bases*, giving yellow, berberine-like derivatives (probably quarternary bases) with alcoholic iodine.
Corydaline, forming dehydrocorydalin, $C_{22}H_{23}NO_4$.
Corybulbine, forming dehydrocorybulbin, $C_{21}H_{21}NO_4$.
Isocorybulbine (not examined with iodine).

II. *Medium bases,* not acted on by alcoholic iodine, corycavin, and corycavamin.

III. *Strong bases*, bulbocapnine, corydine, corytuberine.

Group I.—The dehydro derivatives, on reduction, yield the inactive forms of the original bases. Attempts were made to split *i*-corydaline (m.p. 135°) into its active components but without success. A small quantity of *r*-corydaline, m.p. 158°—159°, was obtained in preparing the inactive variety. Iso-corybulbine is isomeric with corybulbine, and more readily soluble in alcohol. $[\alpha]_D^{20}$ for corybulbine = +303·3°; for iso-corybulbine = +299·8°.

Group II.—Corycavamine was separated by means of the sulphocyanide compound, which is insoluble in alcohol. It contains no methoxyl or hydroxyl groups, and forms an isomeride (*cf.*, narcotine and gnoscopine), m.p. 216°—217° when heated with acetic-anhydride. It melts at 149° C., is solid at 175° C., and again melts at 216°—217° C.

Group III.—These bases readily form yellow oxidation products in the air. Corydine has three methoxyl groups, and may be dimethyl-bulbocapnine. The optical characters are—

$[\alpha]^{D20}$ for bulbocapnine = 237°; for corydine = 770·6°.
Mol. rot. for bulbocapnine = 204·35°; for corydine = 721·4°.

Corytuberine, $C_{19}H_{23}NO_4 + 5\ H_2O$, extracted (finally) by chloroform from the ammoniacal extract, crystallises from hot water or hot dilute alcohol as slightly lustrous, white plates which turn grey in the light, melts at 240° C., contains two methoxyl and two hydroxyl groups, and has the value $[\alpha]_D^{20} = 282°·65$ in alcohol. Dobbie and Lauder give the formula $C_{21}H_{25}NO_4$.

Basicity of Berberine.—Berberine is a quarternary base, and reddens phenolphthaleïn, even in very dilute solution. The neutral sulphate is decomposed only by barium hydroxide, and the hydrochloride by silver oxide. —R. L. J.

Digitalinum germanicum; Process for obtaining all the Valuable Constituents of ——. H. Kiliani. Ber. 1901, **34**, [14], 3561—3562.

THE method here given enables the true digitalin and digitonin to be simultaneously obtained. 250 grms. of *digitalinum germanicum* are dissolved in 1 kilo. of 95 per cent. alcohol in a 5 litre flask by means of gentle warming and shaking. After cooling, 1 kilo. of ether (sp. gr. 0·720) is *gradually* added with shaking, the mixture is allowed to stand 24 hours, and the solution poured away from the precipitate. The solution is concentrated by distillation *in vacuo*, and then in a tared dish on the water-bath until a pellicle forms. The syrup is weighed, and washed into a flask with one-and-a-half times its weight of water. The flask is filled to the stopper with ether (0·720), closed, shaken round for a short time (violent shaking or partial filling of the flask gives rise to emulsions) and allowed to stand for several days. The ether abstracts resinous impurities; it is removed, and the treatment repeated until the ether remains colourless. Meanwhile the true digitalin has separated as a thick jelly, which is brought on to a perforated filter plate ("Nutsche") of relatively large surface, where it is allowed to drain thoroughly, it is then washed with small quantities of water containing 5 per cent. of alcohol, an operation which requires many days. The vacuum is applied only quite at the end, after which the precipitate is dried on a porous tile. The more thorough is the washing, the more easy is the subsequent "recrystallisation" of the digitalin from 95 per cent. alcohol (Arch. Pharm. **230**, 252), and the smaller is the loss. The filtrate from the digitalin contains the digitaleïn (Arch. Pharm. **237**, 458).

The original precipitate produced by ether is *at once* dissolved on the water-bath in 500 grms. of 85 per cent. alcohol, and the solution, without regard to any slight turbidity, allowed to stand in a covered dish for 48 hours. The crystalline digitonin is filtered off, washed with the least possible quantity of 85 per cent. alcohol, and spread out loosely to dry on porous tiles. The yield is 45–50 per cent. The crude digitonin may be recrystallised by the method given in Arch. Pharm. **231**, 460. The mother liquor of the digitonin is carefully saturated with ether and allowed to stand for eight days in a covered vessel; a crystalline crust of digitonin is generally obtained. Almost the whole of the glucoside in the original material is obtained in this manner.—A. C. W.

Iboga; Properties and Composition of ——, and the Alkaloid it contains. J. Dybowski and E. Landrin. Comptes Rend. **133**, [19], 748—750.

THE natives of the French Congo chew the woody portions, especially the roots, of a plant called *Iboga*; it is said to enable persons to withstand fatigue for long periods, and to lessen the need of sleep. It also acts as an aphrodisiac. The sensations it produces are said to resemble those produced by alcohol, but it does not cloud the reason. The authors have extracted from the roots of the plant, by treatment with milk of lime, evaporation to dryness, extraction by ether, and repeated conversion into sulphate and reprecipitation by soda, an alkaloid, which they call *ibogaïne*. From 1 kilo. of the root they prepared 6—10 grms. of the alkaloid, which is well crystallised, of a light amber colour, insoluble in water, but readily soluble in alcohol, and in most organic solvents. Its solutions turn the plane of polarisation to the left. The substance melts at 152° C. It oxidises readily in the air, and forms salts with great ease when acted on by acids; of these salts the hydrochloride is the only one that crystallises well. The solutions of the salts are precipitated by most of the usual reagents for alkaloids. The formula is $C_{52}H_{66}N_6O_2$; and the physiological action of the alkaloid is the same as that of *iboga*.—J. T. D.

Rhamnus Purshianus (Cascara Sagrada); Essential Oil of ——. H. Haensel's Report. Pharm. Zeit. **46**, [86], 858.

BY steam distillation, the bark of *Rhamnus purshianus* yields 0·055 per cent. of a concrete volatile oil of distinctly crystalline structure, having in a marked degree, the peculiar odour of the bark. It melts at 18·5° C. to a deep brown liquid, which gives an alcoholic solution of an intensely yellow colour, the reaction of which is slightly acid. The sp. gr. at 23° C. is 0·9475; it has an aromatic, pungent, slightly bitter taste. With the exception of *Rhamnus frangula*, no other member of the genus *Rhamnus* has hitherto been found to yield a volatile oil.—J. O. B.

Cordia Excelsa; Crystalline Body of ——. Thoms. Pharm. Post. **34**, [40], 634.

THE so-called cordianine, of Peckholt, isolated from the fresh bark, and leaves of *Cordia excelsa* by extraction with alcohol, is found to be allantoine $C_4H_6N_4O_3$. It occurs to the extent of 0·266 per cent. in the fresh leaves, and 0·78 per cent. in the fresh bark. By repeated recrystallisation it is obtained in columnar colourless crystals, which melt, with decomposition, at 224° C. On destructive distillation, ammonium cyanide is the chief product. Although first discovered in the amniotic liquor of animals and in other animal secretions, the base has also been found in vegetable tissue, by Schultze and Barbieri in the young shoots of *Platanus orientalis*, grown in water and by Schultze and Bosshart in the bark of the horse-chestnut. It is probably derived, in these cases, from vegetable albumin, and not from urea or uric acid, since it is doubtful if these bodies occur in the vegetable kingdom; urea has not been found to be present in the excreta of birds which feed solely on vegetable matter, although it is known to occur in that of those which are carnivorous.

—J. O. B.

Berberine; Gaze's pure Base. H. M. Gordin and C. G. Merrell. Archiv der Pharm. **239**, [8], 626—637.

Two methods of obtaining pure berberine have been hitherto employed, that of liberating the base by means of barium hydrate, and that suggested by Gaze of heating the insoluble acetone berberine compound for 12 hours under a reflux condenser, with chloroform, by which means it was claimed by the originator that the acetone was split off and the pure base only left. Berberine thus obtained is markedly different both in appearance and in physical and chemical characters from the base obtained by the baryta method, and has not that affinity for CO_2 which, according to E. Merck, renders it impossible to prepare pure berberine except in an atmosphere of hydrogen.

If, however, Gaze's berberine be treated with sulphuric acid, and then precipitated with barium hydrate, the base obtained is identical, in every respect, with that obtained by the other method. Gaze's berberine is also found to be abnormal in behaviour both towards iodine and towards acids; it shows no basic action towards the latter, and with the former, although a precipitate is obtained, no iodine appears to be used up. The authors find that this abnormal behaviour is readily explained, the so-called pure berberine is not the free base, but its hydrochloride $C_{20}H_{17}NO_4.HCl.2H_2O$ formed by the decomposition of the chloroform by berberine acetone according to the equation.

$$4C_{20}H_{17}NO_4.C_3H_6O + CHCl_3 + 4H_2O = 3C_{20}H_{17}NO_4HCl + 2H_2O + C_{20}H_{17}NO_4HCO_2H + C_3H_6O.$$

They find that this interaction between berberine and chloroform invariably takes place, and is quantitative, being analogous to the well known reaction between caustic potash and chloroform. The only other base which has been found to react with chloroform in a similar manner is piperidine.—J. O. B.

Artemisin. P. Bertolo. Atti. R. Accad. dei Lincei Roma, **10**, [5], 111—116. Chem. Centr. 1901, **2**, [17], 937.

MERCK has isolated *artemisin*, $C_{15}H_{18}O_4$, from the mother liquors obtained in the preparation of santonin from the seeds of *Artemisia maritima*. Artemisin forms colourless crystals, melting at 200° C., soluble in 3 parts of warm alcohol and 60 parts of boiling water; $[\alpha]_D = -84·3°$ (observed with a 10 per cent. solution in 99·2° alcohol). With chloroform it gives an addition compound, $C_{15}H_{18}O_4.CHCl_3$, which loses chloroform at 80° C. On boiling with soda solution, artemisin dissolves to a carmine red solution, which becomes decolorised on cooling. The sodium salt formed in this manner yields artemisin again on treatment with dilute sulphuric acid, but with the concentrated acid, gives a new crystalline compound, melting at 170°—171° C. With hydroxylamine, artemisin gives an oxime, $C_{15}H_{18}O_3NOH$, white needles from methyl alcohol, m. pt., 233°—234° C., slightly soluble in water, easily soluble in alcohol, ether and chloroform. With phenyl hydrazine, a semi-solid phenyl-hydrazone is obtained. Artemisin appears thus to be analogous to santonin, in that it contains a lactone group and a keto-group.—A. S.

Cantharides; valuation of ——. K. Dieterich. Pharm. Centralhalle, **42**, 674, Apoth. Zeit. 1901, 687.

FROM the amount of cantharidin obtainable from available commercial "Spanish flies," the author concludes that the requirement of the Ph. G. IV. for a cantharidin content of 0·8 per cent. is too high, and that an average of 0·6 per cent. obtained by the official method of Baudin, is sufficiently stringent. The official process should be further modified by pressing the powdered drug so as to obtain 52 grms. of chloroform extract, and the drying of the cantharidin obtained should be carefully conducted, since it is appreciably volatile at 100° C., at which temperature loss may be incurred by sublimation. In the following experiments the amount of free cantharidin was determined by merely omitting the addition of acid in the process of extraction. The brown beetles (*Mylabris*), were found to yield a well crystallised pure cantharidin in greater quantity than the green beetles (*Cantharis*); from the latter also, the cantharidin was less pure, and partly amorphous. Copper was detected in the ash of cantharides.

—	Green Cantharides (Cantharis vesicatoria).	Brown Cantharides (Mylabris cichorii).
	Per Cent,	Per Cent.
Ash of whole beetles	5·05 to 6·02	3·98 to 5·01
Ash of powdered beetles .	5·23 to 7·47	4·16 to 5·10
Free cantharidin	0·28 to 0·56	0·67 to 1·01
Combined cantharidin ...	0·03 to 0·3	0·136 to 0·95
Total cantharidin........	0·38 to 0·85	0·73 to 1·92
Water, in whole beetles ..	10·06 to 15·94	10·42 to 12·54
Water, in powdered beetles	7·06 to 15·05	7·53 to 11·64

It is recommended, therefore, that *Mylabris*, as well as *Cantharis*, should be recognised as the official source of the vesicant.—J. O. B.

Eucalyptus Oils (New). R. T. Baker. Proc. Linnean Soc. New South Wales, 1900, [2], 303, through Schimmel's Report, Oct. 1901, 27.

Eucalyptus delegatensis (white ash, silver-topped mountain ash), yielded 1·76 per cent. of pale lemon coloured oil, sp. gr. 0·8602; $[\alpha]_D = -68·12°$, consisting chiefly of lævophellandrene, and containing neither cineol nor endesmol.

Eucalyptus intertexta (spotted gum), gave varying quantities of oil, not exceeding 0·64 per cent., which was of a brownish yellow colour; sp. gr. 0·9078; $[\alpha]_D = +10·7°$ containing much dextropinene, 37 per cent. of cineol, but no phellandrene.

Eucalyptus morrisii (grey mallee), afforded 1·69 per cent. of faintly-coloured oil, with an odour of aldehydes; sp. gr. 0·9097; $[\alpha]_D = +6·7°$; it consists of dextropinene and more than 50—60 per cent. of cineol, but contains neither endesmol nor phellandrene.

Eucalyptus viridis (green, red, or brown mallee), gave 1·06 per cent. of pale brownish yellow oil with an odour recalling that of cuminaldehyde; sp. gr. 0·900 C.; $[\alpha]_D = -8·90°$. It yields less than ten per cent. of cineol and some lævopinene, but neither eudesmol nor phellandrene. An aldehyde (cumin aldehyde ?) is present in the higher boiling portion.

Eucalyptus vitrea (white top messmate), yielded 1·48 per cent. of colourless oil, sp. gr. 0·886; $[\alpha]_D = -33·92°$. It contains no phellandrene, but from 20 to 26 per cent. of cineol, and in the higher boiling fractions, some citral.

—J. O. B.

Eucalyptus Oil; Terpeneless ——. H. Haensel's Report. Pharm. Zeit. **46**, [86], 858.

AUSTRALIAN eucalyptus oil has been found to yield, on an average, 17 per cent. of terpene; the characters of the original oil, terpene, and terpeneless oil being shown in the following table:—

——	Original Oil.	Terpeneless Oil.	Terpene.
Sp. gr. at 15° C.	0·9122	0·9382	0·8677
Opt. Rot. at 20° C.	−4·46	−1·72	−13·07
Refraction index at 20° C.	1·4667	1·4679	1·4766
Zeiss-Wollny Refractometer number.	61·3	63·6	77·2

When cooled to −16° C. partial solidification took place in the terpeneless oil.—J. O. B.

Menthol; Halogen Derivations of——, and certain Hydrocarbons therefrom. N. Kursanoff. Annalen, **318**, [2, 3], 327—344.

MENTHYL chloride is a mixture of at least two isomers; one dextrorotatory and easily converted into menthene, the other lævorotatory and much more stable; in addition to these secondary chlorides there may also be a tertiary chloride. The author heated an ethereal solution of menthyl chloride, prepared by the action of phosphorus pentachloride on menthol, with sodium. The fraction of the product, boiling below 200° C., consisted of menthene, $C_{10}H_{18}$, and menthane, $C_{10}H_{20}$; the higher fraction distilled at 195°—197° C. under 30 mm. pressure. After purification, it consisted of two isomeric dimenthyls, $C_{20}H_{38}$, of which the crystalline isomeride melted at 105·5°—106° C. and boiled at 185°—186° C. under 21 mm. pressure, $[\alpha]_D = -51° 18'$. The second isomeride was an oil.

Menthyl iodide, obtained by heating menthol with hydriodic acid, when boiled with sodium in ethereal solution, gave the same products, but the crystalline dimenthyl in much smaller yield than the fluid. Menthyl chloride, similarly prepared by heating menthol with a large excess of hydrochloric acid in sealed tubes, had $[\alpha]_D = -24° 57'$. When boiled with ether it gave about equal yields of the solid and fluid dimenthyls.

Pure secondary menthyl chloride was prepared by heating the chloride, produced by the action of phosphorus pentachloride on menthol, with strong alcoholic potash until the menthyl chloride was no longer decomposed. The residual chloride boiled at 109°—110° C. under 30 mm. pressure; $[\alpha]_D = 50° 57'$. It was unaltered by heating with alcoholic potash in a sealed tube, or by boiling with aniline. The yield of stable chloride is about 25—30 per cent., a lower yield is obtained by boiling with aniline. The menthyl chloride obtained by heating menthol with hydrochloric acid also produced the stable chloride.

Stable menthyl chloride, when heated with sodium, gives the crystalline dimenthyl almost pure and without the oily isomeride. The optical activities of the stable menthyl chloride and of dimenthyl show that the former is a secondary chloride.

Attempts were made to obtain tertiary dimenthyl. Menthene was mixed at 0° C. with acetic acid saturated with hydrobromine acid gas, after standing 36 hours the menthyl bromide was separated and distilled. When boiled with sodium in ethereal solution, it gave a very small quantity of a dimenthyl, which boiled at 199°—202° C. under 21 mm. pressure and was not identical with the previous dimenthyl. Similar results were obtained with tertiary menthyl chloride.

Ethylmenthane was prepared from the stable menthyl chloride by the action of zinc ethyl; it boiled at 209°—210° C., $[\alpha]_D = -12° 15'$.—A. C. W.

Anethol. Schimmel's Report, Oct. 1901.

ALTHOUGH anethol now replaces anise oil in the German Pharmacopæia, [IV.], no reference, in the official test, is made to the optical behaviour; the point is of importance since pure anethol is optically inactive. The figures published by Pancoast and Kebler [Amer. Journ. Pharm. **75**, 356], show that impure anethol is still frequently met with in commerce. All the specimens reported on by them had an exceptionally low freezing point, a slight optical activity, and boiled between the temperatures 210°—235° C. These samples were evidently adulterated, or imperfectly purified. Attention has previously been called to the fact that prolonged storage under unsuitable conditions is detrimental to anethol, due to a process of oxidation, so that ultimately crystallisation becomes impossible. Care should be taken when applying the congealing point test, not to carry the temperature too low quickly. The melting and congealing point, when correctly obtained, are closely approximate. Freshly prepared pure anethol congeals always at 21° C., or a little above, and remelts between 22·5° and 22·7° C.—J. O. B.

Perfumes; New Method of Improving ——. Lavollay and Dourgoin. Rev. Prod. Chim. **3**, [23], 358.

THE essential oils to be improved are treated with calcium (or other alkaline earth) manganate, or other insoluble manganates, in presence of an electric current, the operation being conducted in the cold and in a closed vessel provided with an escape for the excess of gas produced. The amount of manganate employed is about 20—50 grms. per hectolitre of essential oil, and the strength of current ranges from 3 to 10 ampères per square metre of electrode surface. The operation should not exceed twenty minutes.

The action of the manganates under these conditions is claimed to be superior to ozone, besides being constant, easily regulated, and less expensive.—C. S.

Vanilla; Formation of the Perfume of ——. H. Lecomte. Comptes Rend. **133**, [119], 745—748.

THE author has discovered in many parts of the vanilla plant an oxidising ferment; and those varieties which are in highest esteem are precisely those which contain this oxydase most abundantly. At first it seemed that this ferment must be destroyed by the treatment of the pods, which are plunged for about 20 seconds into water at 80°—85° C.; but the author has found that the interior of the pod under these circumstances never reaches 50° C., so that the ferment, instead of being destroyed, is brought to a temperature very favourable to its activity.

A second ferment has also been found in the plant, which hydrolyses starch; and the author suggests that this ferment transforms coniferin, present in the pod, into glucose, and coniferyl alcohol, the latter of which is converted into vanillin by the action of the oxydase. He hopes to give experimental confirmation of this hypothesis when further material is available.—J. T. D.

Bergamot Oil; A new Crystalline Constituent of ——. H. V. Soden and W. Rojahn. Chem.-Zeit. 1901, **25**, [80], Rep. 282. From Pharm.-Zeit. 1901, **46**, **778**.

BERGAPTEN is already known as a crystalline constituent of bergamot oil. The distillation-residue contains *bergaptin*, which crystallises from light petroleum spirit as whitish leaflets, melts at 59·5° C., dissolves easily in ether, chloroform, and alcohol, and readily absorbs bromine. Phenolic and methoxy groups are absent.—R. L. J.

Lavender Oil; the Solubility Test for, of the German Pharmacopœia ——. Schimmel's Report, Oct. 1901, 35.

THE German Pharmacopæia [Editio IV.] requires that lavender oil should be soluble in three volumes of dilute alcohol (68 to 69 per cent. by volume). This requirement is found not to be met by certain oils of good quality. An oil having the prescribed ester content (29 to 30 per cent.) gives a clear solution with 3 to 3·5 volumes of 68 per cent. alcohol, and with 3 volumes of 69 per cent. alcohol. But oils having a higher ester content require 3·5 to 4 volumes of 68 per cent., and 3 to 3·5 volumes of 69 per cent. alcohol to give a clear solution. The solution in 68 per cent. alcohol soon becomes turbid, and remains so on adding more of that solvent. The solution in 69 per cent. alcohol remains clear, but shows opalescence on adding more solvent. Oils less rich in esters do not thus separate on further addition of alcohol. It is suggested that the official strength for diluted alcohol should be made less indefinite,

and that exactly 70 per cent. should be the standard strength. All good lavender oils give a perfectly clear solution with 2·5 to 3 volumes of spirit of this strength.
—J. O. B.

Geranium Oil. Schimmel's Report, Oct. 1901, 30—32.

In consequence of the high price prevailing for geranium oil, J. Grec advocates (Reveil agricole, May 26th, 1901) the more extended cultivation of the plant, since it is not only a remunerative crop, but also occupies the land only during six months of the year. It is estimated that an hectare of land will carry 40,000 kilos of herb, which will sell to the distillers for at least 2,000 francs. This profit may be very largely increased if the grower distils his own crop. The plants require much manure and plenty of water. The land under geranium crops in the Grasse district have become exhausted owing to the method of irrigation, and although the amount of manure used has been more than doubled, the crops continue to diminish each season. It is probable, therefore, that the cultivation of geraniums will cease to be conducted in the Grasse districts, and that, like that of violets, it will be moved to Venice and its neighbourhood. *Spanish* geranium oil undoubtedly occupies the highest position with regard to quality, and is in consequence of its fine aroma particularly suited for use in high-grade products, such as are required in the tobacco trade. *French* geranium oil differs so little in quality from that of Algeria that its higher price would seem to be hardly justified. The position of *East Indian* geranium oil is unsatisfactory. The production has not attained expectations. At the commencement of the season, genuine oil of high quality was only obtainable in small quantities, and shortly afterwards only second quality oil could be bought, and the supply of this was exhausted by last March. The exceptional drought which prevailed in the Kandesh district during the harvest is the cause of the failure of the crop. As a consequence, East Indian geranium oil is this season of poor quality, or adulterated with fractions of citronella oil.
—J. O. B.

Ylang-ylang Oil [Isoeugenol a Constituent of].
Schimmel's Report, Oct. 1901, 53.

Isoeugenol has been isolated from the constituents of ylang-ylang oil, this being the first instance of the phenol being recorded as occurring in a natural product. On fractionating the phenols removed from the oil by alkali, the portion distilling under 12 mm. pressure at 142—147° C. was found to have a marked odour of isoeugenol. It formed a benzoyl compound melting at 103° to 104° C., identical with that obtained with synthetic isoeugenol, a corresponding acetyl compound melting at 79°—80° C., and a dibromide of methyl-isoeugenol melting at 101°—102° C. The lower boiling fractions of ylang-ylang oil appear to contain creosol. The acids present are benzoic and salicylic acids in the form of methyl and benzyl esters. Dextro-pinene has been isolated from the terpenes present, and the oil also contains a body probably a ketone, with a peculiar odour, which boils at 145°—148° C.—J. O. B.

Mandarin Oil; Artificial ——. Schimmel's Report, Oct. 1901, 36.

It having been shown that the characteristic odour of mandarin orange oil is due to the presence of the methyl ester of methyl-anthranilic acid (this Journal, 1900, 848) together with dextrolimonene, and small quantities of dipentene, decylaldehyde, nonylaldehyde, linalool, and terpineol, the synthesis of the oil has now been effected, and artificial mandarin oil is a commercial article. Its odour and physical characters it is claimed that the synthetic oil closely resembles the natural product, while the cost is about one half.—J. O. B.

Rue Oil. Schimmel's Report, Oct. 1901, 46.

In addition to methyl-n-nonylketone (this Journal, 1901, 606) Thoms finds that a condensation product of that body with benzaldehyde, having the formula $C_{18}H_{26}O$, is also present. It occurs in small, greasy crystals melting at 44°—45°, which are readily soluble in alcohol, ether, and chloroform. There is also present in rue oil, a blue fluorescent basic body, which may be extracted by shaking out with acid and is almost certainly the methyl ester of methyl anthranilic acid, which has previously been isolated from mandarin oil (this Journal, 1900, 848).—J. O. B.

Citron Oil. H. E. Burgess. Analyst, 1901, **26**, 260—262.

The author obtained a genuine sample of this oil, and has examined it with the following results:—Sp. gr. at 15° C., 0·8513; refractive index $[N]_D$, 20° C., 1·4750; optical rotation $[\alpha]_D$, 100 mm. = +80°, 13′. On distillation a principal fraction boiling at 170° to 174° C., and having a rotation of +89° was obtained; it was identified as limonene. Another fraction representing about 6 per cent. of the oil gave all the reactions of citral. The original oil had a considerable deposit of resinous and crystalline matter which was filtered off and extracted with chloroform; on standing crystals were formed which when recrystallised from alcohol melted at 145° C., and had the formula $C_{18}H_{18}O_6$.—J. L. B.

Thyme; Some Essences of ——. P. Jeancard and C. Satie.
Bull. Soc. Chim. 1901, **25**, [18], 893—895.

Essence of thyme is valued according to the proportion of phenols it contains, and it is generally assumed that a pure essence ought to contain 25—30 per cent. Having occasion to examine several absolutely pure essences the authors found that the percentages of phenols varied from 5—60 per cent. They have distilled Algerian red thyme and essence of ajowan in a current of steam, and collected the distillate in separate portions. They conclude from their investigations that most of the phenols pass over towards the end of the distillation; the sp. gr. is a function of the phenolic contents, and rises about 0·0014 for each 1 per cent. of phenols, whilst the solubility in dilute alcohol, the surface tension, and the viscosity increase with the amount of phenols present. This behaviour of the essence of thyme on distillation corresponds with that which the authors have already found for essence of lavender; the ether contents of this essence largely depend upon the manner in which the distillation is conducted. Hence the 25—30 per cent. of phenols generally assumed obligatory in a pure essence of thyme is an arbitrary limit, and can at most be taken as a mean value.—T. A. L.

Calamus Oil; Constituents of ——. H. Thoms. Zeits. angew. Chem. 1901, **14**, [41], 1019—1020.

In a previous communication (this Journal, 1901, 606), Beckstroem, and the author stated that the fractions of highest boiling-point contained, in addition to asarone, a crystalline compound (calamus camphor) melting at 168° C., and having the composition, $C_{15}H_{26}O_2$. The same compound was also isolated by Soden and Rojahn (this Journal, 1901, 833). This substance was found to contain an alcohol related to the sesquiterpene alcohols. On boiling calamus camphor with sulphuric acid of 50 per cent. strength, 2 mols. of water are eliminated, and a hydrocarbon, $C_{15}H_{22}$ obtained. This boils at 144° C., under a pressure of 15·5 mm.; and has a sp. gr. of 0·9324 at 23° C., and a specific rotation of $[\alpha]_D$, (26°) = −11·31°. The reverse formation of the alcohol from the hydrocarbon could not be brought about.

After removing the asarone and calamus camphor by crystallisation from the fractions of higher boiling point, the residue was treated with 80 per cent. phosphoric acid, or better with 90 per cent. arsenic acid, and on heating the liquid, a white plastic mass separated out. This, on extraction with petroleum spirit, yielded an oil boiling at 180° C., under 30 mm. pressure, and having the composition $C_{15}H_{24}O$. The white mass rapidly became coloured on exposure to the air. On treatment with water, the whole of the combined arsenic acid was gradually dissolved out, leaving a white amorphous body, which was readily soluble in acetone. On the addition of water to this solution microscopic crystalline needles (m. pt. 173°—184° C.) were deposited. Its formula agreed with that of asarone ($C_{12}H_{16}O_3$), whilst its molecular weight, determined by Beckmann's method, was three times as great. The author therefore concluded that it was a polymer of asarone and gave it the name of parasarone.

That asarone yields a polymeric modication on treatment with 90 per cent. arsenic acid was shown by treating a solution of asarone in pinene, with arsenic acid of that strength, when a white plastic mass was obtained, which yielded a compound with the same properties as the parasarone described above.

On oxidation asarone yields asaryl aldehyde, but this was not formed by the oxidation of parasarone. The latter is for the most part reconverted into asarone, when distilled under reduced pressure.

The substances which have now been found in the higher-boiling fractions of calamus oil are as follows:—Acetic acid, œnanthic acid, palmitic acid, eugenol, calamus camphor, asarone, and an alcohol of the composition, $C_{15}H_{24}O$, which probably stands in a close relationship to the hydrocarbon, $C_{15}H_{22}$, which can be obtained from calamus camphor.—C. A. M.

Copaiba Balsams; Resins of ——. E. Keto. Archiv der Pharm. **239**, [7 and 8], 548—580.

In their general chemical characters the Copaiba oleo-resins are closely allied to the coniferous secretions, consisting mainly of amorphous acid resins, volatile oil, with varying amounts of indifferent resene resins, and small quantities of crystalline acid resins. When the ethereal solutions of these oleo-resins are shaken out with different alkaline solutions according to the method of Tschirch, the various constituents may be isolated.

Pará Copaiba "Balsam."—In addition to volatile oil, resene, and amorphous acid resins a crystalline acid, pará-copaivic acid, $C_{20}H_{32}O_3$ melting at 145°—148° C., soluble in ammonium carbonate solution, was isolated. It occurs in pointed and quadratic leaflets. Another crystalline acid, insoluble in ammonium carbonate solution is also present; this is homo-pará-copaivic acid, $C_{18}H_{28}O_3$, melting at 111°—112° C., and crystallising in pointed needles.

Maracaibo "Balsam."—This contains β-metacopaivic acid, $C_{11}H_{16}O_2$ (or possibly $C_{16}H_{24}O_3$ or $C_{22}H_{34}O_2$) melting at 89°—90° C. separating in pointed prisms. From another sample of balsam, illurinic acid, $C_{20}H_{28}O_3$, was isolated, as hexagonal pyramids melting at 128°—129° C.

Illurin (African) Copaiba Balsam.—African copaiba balsam contains from 2 to 3 per cent. of a strongly lævorotatory, dimorphous, crystalline acid, illurinic acid, $C_{20}H_{28}O_3$ separating in fine hexagonal hemihedral or hexagonal hemihedral crystals, having in alcoholic solution, the specific rotation $[\alpha]_D = -54° 89'$; melting when quite pure at 128°—129° C. It is a powerful acid, and forms crystalline salts with alkalis, with barium and with lead. Its silver salt is amorphous. The crystalline barium salt $(C_{20}H_{27}O_3)_2Ba + (4H_2O?)$ is characteristic. When an ethereal solution of the acid is shaken with baryta water a few needles appear in a few minutes, and then, suddenly, the whole ethereal layer is covered with a network of fine needles. From the iodine and bromine number the presence of two ethylene groups in the molecule is indicated. This acid is probably identical with the crystalline acid of African copaiba balsam previously described by Umney and by Peinemann, and also closely resembles the oxy-copaivic acid of Fehling. Schweitzer and Rose's copaivic acid and the meta-copaivic acid of Strauss have not been isolated by the author.—J. O. B.

Asarum Canadense; Constituents of the essential oil of ——. F. B. Power and F. H. Lees. Proc. Chem. Soc., 1901, **17**, [242], 210.

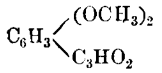
In a previous investigation of this oil by one of the authors (Power, Inaug. Diss. Strassburg, 1880; Proc. Amer. Pharm. Assoc., 1880, **28**, 464) it was found to contain a terpene, $C_{10}H_{16}$; two fragrant alcohols, b. p. 196°—199° and 222°—226°, both having the composition $C_{10}H_{18}O$; a nearly inodorous body, b. p. 254°—257°, which, on oxidation with chromic acid, afforded a crystalline acid, $C_9H_{10}O_4$, the latter having since been shown to be veratric acid by Petersen (Ber., 1888, **21**, 1062), who has also identified the substance affording it as methyleugenol; a deep blue oil collected at 275°—350°; a large amount of acetic acid combined with the alcohols of the oil in the form of esters, and a very small amount of a less soluble oily acid which was believed to contain valeric acid.

The present investigation has shown the oil to have a much more complex composition than was at first supposed. The authors have now identified the following compounds, most of them by well-defined crystalline derivatives. (1) A phenol, $C_9H_{12}O_2$, having a creosote-like odour; (2) pinene (nitrosochloride, m. p. 103°—104°, and nitrolpiperidine, m. p. 118°—119°); (3) *d*-linalool (citral and citryl-β-naphtho-cinchoninic acid, m. p. 195°—198°); (4) *l*-borneol (camphor, m. p. 175°, $[\alpha]_D = -40{\cdot}3°$, and oxime, m. p. 115°—116°); (5) *l*-terpineol (a ketolactone, $C_{10}H_{16}O_3$, m. p. 62°—63°; terebic acid, $C_7H_{10}O_4$, m. p. 173°—174°; and dipentene dihydriodide, $C_{10}H_{18}I_2$, m. p. 80°); (6) geraniol (diphenylurethane, m. p. 81°—82°, and citral); (7) methyleugenol (bromomethyleugenol dibromide, $C_6H_2Br(OCH_3)_2{\cdot}C_3H_5Br_2$, m. p. 78°—79°; it is also shown that methyl*iso*-eugenol does not exist in the oil); (8) a blue oil, boiling above 260°, and consisting of oxygenated compounds of undetermined composition but of alcoholic nature; (9) a lactone, $C_{14}H_{20}O_2$, having a very aromatic odour, but present in the oil in very small amount; (10) palmitic acid; (11) acetic acid; (12) a mixture of fatty acids, $C_6H_{12}O_2$ to $C_{12}H_{24}O_2$. The acetic acid is contained in the oil in the form of esters, whilst the higher fatty acids are in a free state.

A quantitative determination of the principal constituents was made. The amount of methyleugenol, determined by Zeisel's method, was found to be 36·9 per cent. The amount of esters, calculated as $C_{10}H_{17}{\cdot}C_2H_3O_2$, is 27·5 per cent.; the free alcohols, $C_{10}H_{18}O$, 13·3 per cent. The amount of pinene obtained by direct fractionation of the oil was about 2 per cent., and the amount of high boiling constituents, such as the blue oil, is therefore about 20 per cent.

Limettin; Constitution of ——. W. A. Tilden and H. Burrows. Proc. Chem. Soc. 1901, **17**, [242], 216.

In a previous communication (this Journal, 1892, 264), it was shown that limettin, $C_{11}H_{10}O_4$, had the following constitution:



$$C_6H_3\begin{cases}(OCH_3)_2\\ C_3HO_2\end{cases}$$

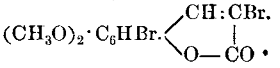
Further experiments have been undertaken, with the object of determining the structure of the group C_3HO_2.

In the meantime, the publication of a communication (Citroptene) by E. Schmidt (this Journal, 1901, 1017) on a substance which he has isolated from oil of lemon and has identified with limettin, and the appearance of a recent note by Burgess (this Journal, 1901, 1745 and 844), render it necessary to place on record the following results, although at present incomplete.

The dibromo compound previously described by Tilden (*loc. cit.*) melts at 297° with decomposition, and not at 257°.

On treatment with 10 per cent. solution of potash, it yields an acid of the composition $C_{11}H_9O_5Br$. Continued treatment with potash does not remove the second atom of bromine.

It is therefore highly probable that the structure of the group C_3HO_2 is similar to that of the ring in coumarin, dibromocoumarin behaving in exactly the same way. Dibromolimettin may therefore be represented as—



$$(CH_3O)_2{\cdot}C_6HBr.\begin{cases}CH{:}CBr.\\ \quad\quad\;\,|\\ O—CO\end{cases}\cdot$$

Alcohol in Ether; Determination of ——. F. Freyer.

See under XXIII., *page* 1250.

Lemon Oil Industry; The ——. H. Burgess and J. F. Child.

See page 1176.

Clove Oil; Determination of Eugenol in ——. A. Verley and T. Bölsing.

See under XXIII., *page* 1250.

Coca Leaves; Assay of ——. W. R. Lamar.
See under XXIII., *page* 1250.

Guaiacol; Approximate Determination of ——. Adrian.
See under XXIII., *page* 1251.

XXI.—PHOTOGRAPHY.

Sodium Perborate [*Photography*]. J. M. Eder and E. Valenta. Chem. Ind. 1901, **24**, [20], 560.

Sodium perborate, $NaBO_3 + 4H_2O$, is prepared by the electrolysis of a cold concentrated solution of sodium borate. Fontar has recommended it for the removal of residual thiosulphate in insufficiently washed prints. The solution of the salt is stable, and has little action on the silver image. Although alkali persulphates have been used with advantage as photographic reducers, sodium perborate, like the permolybdates, pervanadates, perchlorates, &c., is not suited for this purpose.—A. S.

Developer; New Photographic ——; *and a new Method for the production of Aromatic Oxy-Alcohols.* A. Eichengrün. Zeits. angew. Chem. 1901, **14**, [42], 1070—1071.

The reaction of formaldehyde and salicylic aldehyde in the presence of strong hydrochloric acid, yielding a chlormethyl derivative having the composition $C_6H_3.CHO.OH.CH_2Cl$, has been extended to other oxy-compounds. Thus salicylic acid was made to yield $C_6H_3.COOH.OH.CH_2Cl$, which, by the action of water is converted into the corresponding alcohol. This, on oxidation yielded first a *p*-aldehydocarbonic acid (which with sodium amalgam gave a product identical with that produced by the action of the amalgam on Reimer's *p*-aldehydosalicylic acid), and then 4-oxyisophthalic acid $C_6H_3.COOH.(OH).CO.OH$. The *p*-oxymethylsalicylic acid now produced for the first time, melts at 140° C. The chlorine atom of the chlormethylsalicylic acid and its analogues is very readily displaced, so that esters, ethers, &c. are readily prepared. Phenols may be made to yield such products as β-oxynaphthyl-*o*-oxy-*m*-toluyl acid ($C_6H_3.COOH.OH.CH_2.C_{10}H_7.OH$), which is now found on the market under the name "Epikarin," as a remedy for itch and other skin-diseases. The chlormethyl derivatives of nitrophenol (and the alcohols yielded by them) can be reduced to amido compounds. Of these the ortho derivative is a weak, the para-derivative ($C_6H_3.OH.CH_2OH.NH_2 = 1:3:4$), an exceedingly powerful photographic developer. The *o*-amidometaoxybenzyl alcohol will shortly be placed on the market under the name *Paramol*. As a developer, this substance stands between those of the phenol class (pyrogallol, hydroquinone, &c.) and those of the amidophenol class, the so-called rapid developers, rodinal and metol. In its action it is nearly equal to the rapid developers. It is, however, more like the phenol developers in developing through more slowly, and in being readily weakened by the addition of sodium bicarbonate. Hence with paramol developers errors of exposure may be rectified, as in the case of pyrogallol, whilst there is less tendency to "fogging," and to staining of the hands and plates, than when pyrogallol is used. In respect to solubility also, paramol stands between the two classes, its solubility being 1:12, whilst that of amidophenol, for instance, is only ½ per cent. Thus, whilst working solutions of the rapid developers must be made with the aid of caustic alkalis, concentrated solutions of paramol may be prepared with alkali carbonates.—W. G. M.

[*Photographic*] *Chrome Pictures; a New Method of intensifying* ——. G. Hauberisser. Phot. Rundsch. 1901, **15**, 200. (Through Chem. Zeit. Rep. 1901, **25**, [83], 304.)

Bichromate of potash mixed with organic matter (*e.g.*, gum arabic) applied to paper, and exposed to light, gives up a portion of its oxygen and becomes the unstable, dark brown, amorphous chromium chromate. By the further action of light, sulphides, photographic developers, or other reducing agents, this compound is still further reduced to Cr_2O_3 or $Cr(OH)_3$. Hydrogen sulphide is the best reducing agent for the purpose (especially if the paper is moist, or is laid in a solution of the gas, yielding very rapidly a clear green, instead of a brown, image where the paper had been exposed to light, thus—

$$4Cr_2O_3.CrO_3 + 6H_2S + 12H_2O = 12Cr(OH)_3 + 3S_2.$$

Eder has shown that potassium bichromate reconverts the hydroxide into chromium chromate, thus—

$$Cr_2O_3 + K_2Cr_2O_7 = Cr_2O_3.CrO_3 + K_2CrO_4.$$

The intensification process is based on these two reactions. From 2 mols. of chromium chromate are produced 3 mols. of chromium hydroxide, which can then be reconverted into 3 mols. of the chromium chromate. By alternations of this treatment, a weak image may be intensified as desired. Further intensification may then be effected in another way, viz., by the addition of a metallic salt, such as $MnSO_4$, or $CuSO_4$, which gives a precipitate with chromate, but not with bichromate of potash, the necessary chromate being produced *in situ* by the action of the bichromate on the hydroxide. The practical use of this method is somewhat restricted, but it possesses great theoretical interest.
—W. G. M.

XXII.—EXPLOSIVES, MATCHES, Etc.

Chlorate Explosives less Susceptible to the Action of Heat; Manufacture of ——. J. Bonnet, Paris. Ger. Pat. 124,237, Sept. 1900. Zeits. angew. Chem. 1901, **14**, [44], 1120.

In the manufacture of explosives, consisting of a solution of aromatic nitro- or azo-compounds in a vegetable oil or fat to which chlorates or perchlorates are added, an advantage is derived by employing the free fat-acids in place of part or the whole of the fat generally used. This advantage lies in the relatively higher melting points of the fat-acids, and consequently the explosive mixtures so prepared resist the action of hot climates without softening.
—J. F. B.

Pyroxylin; Instructions for Making ——. Brit. J. Phot. 1901, **48**, [2162], 643.

Hardwick's instructions of 40 years ago are repeated with modifications; Sulphuric acid (ordinary commercial), 33 oz.; nitric acid of sp. gr. 1·42, 7 oz.; water, 4¼ oz., are weighed out and placed in a porcelain jar; after mixture the temperature is maintained at 150° F., and half an ounce of cotton wool (absorbent) is immersed in pellets of about 30 grains each, the mixture being stirred after each addition; after exactly 10 minutes, the cotton is squeezed into a lump, taken out, violently stirred about in a large vessel of water, washed until free from acid, and spread out to dry without heat. The product should have increased in weight not less than 20 and not more than 30 per cent.; outside these limits the collodion, ultimately made, will be difficult to use; too much water or heat will cause the cotton to be short and broken, and the resulting collodion will be opaque and papery looking; opposite conditions will give a collodion which will set in streaks or ridges.—J. W. H.

Phosphorus Suboxides; Non-existence of the so called ——. *Part II.* C. H. Burgess and D. L. Chapman. Proc. Chem. Soc. **17**, [241], 189.

Substances obtained according to the methods described by Michaelis and Pitsch (Annalen 1899, **310**, 45) and Michaelis and v. Arend (Annalen 1901, **314**, 259) have been prepared and analysed, and shown to contain such a large amount of hydrogen that they cannot be regarded as a suboxide, but as red phosphorus containing hydrogen compounds. The authors show that some of the analytical results of Michaelis and Pitsch indicate that the substances prepared by them contained water, and that the proof of the absence of hydrogen in the substance, described by them as a pure suboxide, is inconclusive.

As red phosphorus is easily soluble in aqueous alcoholic potash, with formation of a red solution, red phosphorus resembles in this respect the so-called suboxide.

The authors conclude that both the properties and the analytical results support the view that the substances hitherto described as suboxides of phosphorus are only impure forms of amorphous phosphorus.

PATENTS.

Gunpowder; Improvements in the Manufacture of ——. C. H. Curtis, London, and G. G. André, Argyllshire. Eng. Pat. 19,773, Nov. 3, 1900.

Nitrocellulose powders tend to give varying ballistic results owing to their porous nature. It is claimed that the introduction of nitroglycerin in no greater proportion than is required for the complete filling of the pores produces regularity in ballistic results, a high velocity, moderate pressures, and a minimum erosive effect. In general from 12 to 20 per cent. of nitroglycerin is required, the exact proportion being determined with reference to the tendency of the nitrocellulose employed to produce a porous powder. The nitrocellulose used depends on whether ether-alcohol or acetone is the solvent employed. In the former case a nitrocellulose of 80 per cent. solubility is taken, in the latter one of 20 per cent. solubility is employed. For flake powders the following composition is given:—Nitrocellulose (80 parts), nitroglycerin (20 parts), vaseline (5 parts), while for cords or tubes the proportions are, nitrocellulose (76 parts), nitroglycerin (24 parts), vaseline (5 parts).—G. W. McD.

Priming; Improved ——, for Detonating and Percussion Caps of various Descriptions. M. Bielefeldt, Berlin. Eng. Pat. 20,133, Nov. 8, 1900.

It is proposed that such compounds as picric acid, nitronapthalene, nitrocellulose, &c., should be substituted wholly or in part for the fulminate of mercury in detonating or percussion caps. For use with safety explosives, aluminium nitrate is also added to the detonating composition. No proportions are given in the specification.—G. W. McD.

Explosive resembling Dynamite; Process for the Manufacture of a Safety ——. R. Kändler, Dresden. Eng. Pat. 20,799, Nov. 17, 1900.

Nitrate of potash (76 parts) is intimately mixed with a nitrocarbohydrate, nitrocellulose, nitrostarch, &c. (4 parts), and to this is added a nitrohydrocarbon, nitrobenzene, &c. (20 parts). The explosive is said to be safe for use in fiery mines, and when the proportion of saltpetre is increased to 85 per cent., it is specially adapted for coal getting.—G. W. McD.

Explosive Compounds; Improvements in ——. N. Ceipek, Vienna. Eng. Pat. 21,627, Nov. 29, 1900.

The addition of trinitrophenol to the ammonium nitrate class of explosives is said to increase the shattering effect without lowering the standard of safety. The proportions given are:—Ammonium nitrate (80 per cent.), aniline nitrate (10·9 per cent.), trinitrophenol (9·1 per cent.). The dry finely pulverised ammonium nitrate is mixed in a drum with the aniline nitrate and the trinitrophenol then added. —G. W. McD.

Explosives; Improvements in ——. C. H. Curtis, C. L. W. Smith, D. J. Metcalfe, A. C. Pearcy, London, and A. F. Hargreaves, Roslin. Eng. Pat. 984, Jan. 15, 1901.

The claim is for the use of carbonate of magnesium or carbonate of manganese, preferably the former, for reducing temperature, extinguishing flame, and yet not materially affecting the dynamical power of explosives of the gunpowder type, when used in fiery mines. In ordinary gunpowder in which sulphur is present, there is little or no original safety, judged by the standard required to enable such explosives to be safely used in gaseous mines, and preference is therefore given, for the purpose in view, to explosives containing nitrate of potassium in excess of the amount required to fully oxidise the carbon present. Where saltpetre is present in such proportion that an excess is available for absorbing heat, or other cooling agent is present, it is considered that the proportion of either of the materials which can be effectively used will not be less than 1 per cent. or more than 10 per cent. The best results are obtained with the following mixture:—Nitrate of potassium (84·5 parts), charcoal (12·5 parts), and magnesium carbonate (3 parts).—G. W. McD.

Explosives; New or Improved ——. A. A. da Silva, Rio de Janiero. Eng. Pat. 12,617, June 20, 1901.

The claim is for the combination of guanidine with acid elements of common explosives, examples given being picrate of guanidine which can be incorporated with nitrates or chlorates to form an explosive mixture, and nitrate of guanidine which can be dissolved in alcohol or ether and then incorporated with nitrocellulose to produce a powder. No proportions are stated in the specification. —G. W. McD.

PATENT.

Protecting the Barrels of Fire-arms from Rust or Corrosion, and for Cleaning the same. Preparations for ——. A. Beck, Graz, Austria. Eng. Pat. 15,078, July 24, 1901.

The preparation claimed consists of an aqueous solution of caustic potash, together with soft soap in Phœnix lye (crude soda, potash, water and partly saponified caustic alkalies, with 60 per cent. total alkalinity and 2¼ per cent. fatty acid content.), cleared by "liquid ammonia" or spirits of wine or common salt.—H. I.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

INORGANIC CHEMISTRY.—QUALITATIVE.

Radiations; Coloration of Salts by ——. Goldstein. Chem.-Zeit. 1901, **25**, [84], 918. (Paper read at the Hamburg Meeting of the Ges. deutscher Naturforscher und Aerzte.)

The author has observed in other than haloid salts, when melted or strongly heated under the influence of cathode rays, a coloration, similar to that undergone by the haloid salts of the alkali-metals when treated in a cathode-ray tube. The salts become coloured under the influence of ultra-violet light. The grass green coloration of K_2SO_4, the grey of Na_2SO_4, and the rose-red of Na_2CO_3, were especially noteworthy. Exposed for some time to daylight, the salts lose their colours again. Ignited fluor-spar, exposed to cathode-rays, rapidly becomes grey, and, while coloured thus, phosphoresces brilliantly in the dark. The glow gradually dies out but returns strongly when the specimen is placed in hot water. The colour produced with two mixed salts is not merely that produced by a mixture of those of the components. Mere traces of impurities in the salts markedly affect the colours even $\frac{1}{10000}$ NaCl modifies the colour whilst $\frac{6}{10000}$ changes it completely. Absolutely pure oxy-salts show no coloration, but the presence of $\frac{1}{25000}$ of impurity gives rise to an unmistakable colour; hence a purity-test of great sensitiveness is now available. The presence of several impurities in small quantity causes the coloration to vary in course of time, and after exposure, the remanent tints gradually change, since the colours of different substances have different degrees of permanence on exposure to day light. Hence the presence of several impurities may be observed successively. Since oxy-salts show remanent coloration only after fusion, a ready test is at hand to prove whether a mineral has been produced by plutonic or sedimentary agencies. It is probable that, on heating, solid solutions of one salt in another are produced, and that ionisation commences and traces of impurities are dissociated. But, since the haloids of the alkali metals exhibit colours without previous fusion, and also when perfectly pure, it appears that, in the case of these salts, there is always a partial dissociation of the molecule. In course of the discussion it was elicited that aqueous solutions of the coloured salts are not distinguishable from those of the normal salts.—W. G. M.

Pyrite and Marcasite, and Methods of Distinguishing them. H. N. Stokes. Bull. U.S. Geological Survey, 1901, [186], 11—48.

The author sought to devise a chemical method by which the native forms of iron disulphide may be distinguished with certainty, and by which their relative amounts in mixtures of the two may be quantitatively determined. Pyrite and marcasite are usually distinguished by their differences of crystalline form, colour, density and ease of oxidation, but the results are very uncertain. After a large number of experiments, the author arrived at the following conclusions:—There is no well-established evidence of the existence of true paramorphs of pyrite after marcasite or of marcasite after pyrite; and the hypothesis that most natural specimens, even when well crystallised, are intimate mixtures of the two forms, is without foundation. Specimens crystallising in the regular system consist of true pyrite, and those forming rhomic crystals, of true marcasite. When the two are mingled or intergrown, it is generally possible to distinguish each by the colour after cleaning with acid. The density does not afford a trustworthy means of determining pyrite or marcasite in mixtures of the two.

When pyrite or marcasite is boiled with an excess of a solution of a ferric salt to complete reduction of the latter, the ratio of sulphur oxidised to mineral decomposed is perfectly definite and characteristic of each mineral, provided certain standard conditions are observed. Under the conditions described in the paper, the percentage of sulphur oxidised in pyrite is about 60·4 per cent. and in marcasite about 18 per cent. of the total sulphur. These figures are the characteristic "oxidation coefficients." An empirical curve for the oxidation coefficients of mixtures of pyrite and marcasite can be constructed, by aid of which the composition of naturally occurring mixtures can be quantitatively determined.

In a sample of pyrite containing 3 per cent. or less of copper, it is possible to determine by the oxidation method, whether the copper is present as chalcopyrite or as chalcocite or bornite. Chalcopyrite mixed with pyrite or marcasite can be readily detected, if not in too small amount or too finely divided, by exposing the sample to bromine vapour for half a minute, and then to hydrogen sulphide gas; the chalcopyrite is blackened whilst the iron sulphides remain bright.

It is stated that the principle of the oxidation method, with appropriate modifications, is probably capable of wider application in distinguishing dimorphous minerals, in determining whether a given complex mineral is a mixture or compound of its simpler constituents, and in determining the nature of small amounts of impurities.—A. S.

Chromic Acid in presence of Vanadic Acid; Detection of ——. C. Reichard. Zeits. anal. Chem. 1901, **40**, [9], 577—586.

The formation of the blue coloration, due to perchromic acid, produced by hydrogen peroxide in dilute solutions of chromic acid, is prevented by the presence of vanadic acid, or the coloration when obtained is rapidly destroyed, according to the amount of vanadic acid present. This action, which appears to be catalytic, is perceptible when one part of ammonium vanadate is present to 10 parts of potassium bichromate. Sodium phosphate and arsenate prevent the vanadic acid from destroying the coloration, other salts (sodium nitrate and nitrite, ammonium persulphate, potassium iodate and uranium nitrate) are without action. Molybdic and tungstic acids behave in a similar manner to vanadic acid.—A. C. W.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Sulphur in Fuel, Determination of the Total ——. R. Dubois. Bull. Assoc. Belge des Chim. **15**, (6), 225—227.

Superior results to those furnished by the Eschka method are yielded by the following modification:—One grm. of the powdered fuel is intimately mixed with manganese dioxide, 4 grms., potassium permanganate, 1 grm., and pure dry sodium carbonate, 2 grms., the whole being placed in a platinum crucible and covered with a thin layer of the same oxidising mixture. After heating gently for half an hour, the cover is removed and the crucible heated to dull redness, thus burning off all the combustible matter in the fuel. All the sulphur is transformed into alkali sulphate. After cooling, the contents of the crucible are washed into a glass with 40—50 cc. of boiling water; the solution is acidified with HCl, filtered, and the sulphuric acid in the filtrate determined by means of barium chloride. The reagents must be free from sulphur, or else the content determined by a blank experiment.—C. S.

Coal and Coke, Arsenic in ——. A. C. Chapman. Analyst, 1901, **26**, 253—256.

The author criticises the existing processes for the estimation of arsenic in coals and cokes and suggests the following method:—From 0·5 grm. to 2 grm. of the finely divided fuel is intimately mixed with about 2 grms. of pure calcined magnesia and $\frac{1}{2}$ grm. of dry sodium carbonate in a deep platinum or silver crucible. This is placed in a slanting position and heated by a flame which is just capable of keeping the bottom of the crucible at a dull red heat, the contents being stirred by means of a platinum wire from time to time. At the end of an hour the oxidation will be complete, and $\frac{1}{2}$ grm. of pure ammonium nitrate is then added, and the crucible ignited more strongly for five minutes. The latter treatment is intended to oxidise any traces of sulphides which may have been formed, and which would tend subsequently to form insoluble arsenious sulphide, and so to remove it from the sphere of action in the Marsh apparatus. The powdery mass is washed into a beaker with dilute sulphuric acid and the solution is then concentrated so as to decompose any nitrate that may have remained after the above ignition. The solution is submitted to the modified Marsh-Berzelius method, and the mirror so obtained compared with the standard.

The following results will show that this method is accurate with larger amounts of arsenic than would ever occur in coal or coke. Weighed quantities of arsenical pyrites of known composition were mixed with coal in which the amount of arsenic present was negligible in comparison with that which was added, and the mixture treated as described above, save that the arsenic was determined gravimetrically as sulphide:—

Arsenic.

Taken.	Found.
Grm.	Grm.
0·0050	0·0042
0·0092	0·0096
0·0096	0·0085
0·0063	0·0055

In the subjoined table numbers are given showing the amounts of arsenic present in six samples of coal as well as in the coke made on a large scale from these samples, and in the ash, whilst the percentages of ash, iron and of calcium are also given.

Sample of Coal.	Arsenic in Coal.	Arsenic in Coke.	Ash of Coal.	Arsenic in Ash, calculated on Coal.	Iron in Ash, calculated on Coal.	Calcium in Ash, calculated on Coal.	Alkalinity of Ash.
	Grains. per lb.	Grains. per lb.	Per Cent.	Grains. per lb.	Per Cent.	Per Cent.	
1	1·4	1·7	6·4	0·6	2·27	Trace	Distinct
2	0·5	0·7	16·2	0·3	1·71	,,	Slight
3	0·7	1·0	6·7	0·3	2·62	None	Neutral
4	0·7	0·6	2·1	0·6	0·73	,,	,,
5	0·9	1·1	12·5	0·7	1·96	,,	,,
6	0·8	0·6	12·2	0·3	3·50	1·8	Very distin

There appears to be no definite relation between the amount of arsenic present in the coal and that left in the ash on ignition, the proportion being apparently dependent upon the composition of the ash, as well as upon the manner in which the ignition has been performed. The author considers that arsenic in coal exists almost entirely as a constituent of the pyrites present.—J. L. B.

Potassium Permanganate; Determination of ——, by means of Sodium Thiosulphate. A. Åŀander. Zeits. anal. Chem. 1901, **40,** [9], 574—577.

The author has quantitatively examined the reaction which occurs when sodium thiosulphate is added to potassium permanganate. The thiosulphate is mainly oxidised to sulphate — $8KMnO_4 + 3Na_2S_2O_3.5H_2O = 3Na_2SO_4 + 3K_2SO_4 + 2KOH + 8MnO_2 + 14H_2O$. A certain small quantity of tetrathionate also appears to be produced.
—A. C. W.

Potassium in Mixtures of Salts; Separation and Determination of Small Quantities of ——. F. H. van Leent. Zeits. anal. Chem. 1901, **40,** [9], 569—573.

Potassium may be quantitatively separated from solutions containing much sodium, calcium and magnesium by means of sodium cobaltinitrite (see this Journal, 1900, 852); the precipitate is, however, not of constant composition, but can be used as an intermediate stage in the determination. The author has employed this method to determine the potassium in sea water, taken at Scheveningen at ebb-tide. The sp. gr. of the sample was 1·0224 at 15° C.; it contained 16·522 grms. Cl in the litre, and was thus not greatly diluted by river water. The filtered water (300 c.c.) was evaporated until sodium chloride began to separate, it was then filtered and the residue washed, when gypsum was left. From another portion of 500 c.c., calcium and magnesium were separated partially by sodium carbonate and the filtrate acidified with acetic acid.

The cobalt reagent contained 9·58 grms. of crystallised cobalt chloride and 25 c.c. of glacial acetic acid in 500 c.c. The nitrite solution contained 90 grms. of sodium nitrite in 500 c.c. Immediately before use equal volumes of these solutions were mixed and 80 or 130 c.c. added to the liquids prepared as above. The mixture was kept at 40—50° C. for 6—7 hours, and then allowed to stand overnight in the cold, the precipitate was several times washed with the reagent, and then with 80 per cent. alcohol until the washings were colourless. The filtrate never gave a further precipitate on treatment with more reagent. The potassium in the dried precipitate is best weighed as perchlorate; for this purpose it was decomposed with hydrochloric acid; the filter was incinerated in a platinum crucible just at a red heat, the ash extracted with water and the extract added to the main liquid, which was evaporated to dryness and again evaporated with dilute hydrochloric acid. Water and 7—10 c.c. of perchloric acid (18 per cent.) were added, and the liquid evaporated until white fumes of perchloric acid was evolved. The disappearance of the hydrochloric acid was recognised by the conversion of the blue cobalt chloride into the red perchlorate. The latter was gently ground with 10 c.c. of 96 per cent. alcohol containing 0·2 per cent. of perchloric acid, the solution of cobalt and sodium perchlorates filtered off, the potassium salt again treated with the alcohol containing perchloric acid and finally washed on the filter with a mixture of equal parts of alcohol and ether to disappearance of acid reaction. The filter was dried at 120°—130° C. to constant weight.

When the platinum chloride method was employed, the precipitate was gently heated in a platinum crucible, and the paper incinerated. In extracting the residue, it was found necessary to add a little sodium chloride to the water, which prevented the cobalt oxide from going through the filter paper. After two evaporations with hydrochloric acid, the residue was taken up with water and precipitated by platinum chloride.

The average result was 0·3947 grm. K_2O per litre of sea water, or 2·39 parts K_2O to 100 parts Cl, which compares with the results of Ehrmann, Dittmar, &c., of 2·42—2·57 parts per 100 parts Cl. Control experiments on a mixture of potassium and sodium chlorides, and these chlorides with magnesium sulphate, gave satisfactory results.
—A. C. W.

Iron, Separation of ——. P. Nicolardot. Comptes Rend. **133,** [18], 686—688.

When to a boiling and not too acid solution of ferric choride an alkali sulphate is added, the iron is precipitated as basic sulphate, and can be thus separated from all the other metals of the group, but the conditions requisite to make the precipitation complete are so difficult of attainment that the method is not a practicable one. If the ferric chloride be heated to 125° C., however, it undergoes a change which greatly simplifies the process. To carry out the author's plan, a quantity of the substance (alloy) containing about 1 grm. of iron is dissolved in *aqua regia*, the solution evaporated to dryness with sufficient hydrochloric acid to expel the nitric acid, and heated to 125° C. for four hours. The residue is dissolved in about 500 c.c. of water, heated to boiling, 1 grm. of ammonium sulphate is added, and the boiling continued for quarter of an hour. As soon as the precipitate has settled a little, the whole is filtered through a close filter. If mercury or cadmium be present, the operation of heating to 125° C. cannot be carried out; in this case the author neutralises the solution with ammonia, adds ammonium sulphate, boils, and filters as above; he then adds ammonia to the filtrate till a slight precipitate of iron appears, boils again, and filters; the separation of the iron is now complete.

Other salts (selenates, phosphates, arsenates, vanadates, molybdates, &c.) will also carry down iron in this manner; and the method thus promises to afford a means of separating selenium, phosphorus, &c., from all metals other than iron.—J. T. D.

Platinum and Iridium; Determination of ——, in Platinum Ores. Leidié and Quennessen. J. Pharm. Chim. 1901. **14,** [8], 351—355.

When only the amounts of platinum and iridium in an ore are required, the following modification of Leidié's method (this Journal, 1901, 45), is recommended:—Five grms. of the ore are repeatedly extracted with hot *aqua regia*, composed of one part of nitric acid (sp. gr. 1·32), and three parts of hydrochloric acid (sp. gr. 1·18) so long as anything is dissolved. The united extracts are evaporated to dryness first on a sand-bath, and then in an oven at 105°—110° C., and the residue taken up with a little water and filtered.

The filtrate is heated to about 70° C., and sodium nitrite added until the liquid is neutral to turmeric, after which sodium carbonate is introduced until the precipitate no longer increases. The liquid is then heated to the boiling point, and filtered to separate foreign metals from metals of the platinum group.

The filtrate is rendered alkaline with sodium hydroxide, and a current of chlorine passed through at a temperature of 70°—80° C., in order to expel the osmium and ruthenium in the form of volatile peroxides. The liquid is now neutralised with hydrochloric acid, and more sodium nitrite added to reconvert into nitrites any chlorides of the other metals. In addition to the alkali salts there will now only be present the double nitrites of sodium with platinum, palladium, iridium, and rhodium.

Determination of the Platinum.—At this point, instead of saturating the liquid with ammonium choride, as in the original method, from 30 to 35 per cent. of potassium chloride are added. The rhodium and iridium are precipitated as double nitrites of the metals with potassium, which are insoluble in solutions of alkali chlorides. The liquid is filtered, and the nitrites of platinum and palladium converted into chlorides by means of hydrochloric acid, the saline mass being finally taken up with boiling water. The solution is boiled with a slight excess of formaldehyde, which precipitates the two metals in the metallic form. The precipitate is collected, ignited, reduced in a current of hydrogen, and dissolved in *aqua regia*. This solution is evaporated, the residue taken up with water, the solution treated with a reducing agent to convert palladic chloride into palladous chloride, and the platinum precipitated with ammonium chloride, and determined by any of the usual methods.

Determination of Iridium.—The liquid from which the osmium and ruthenium have been expelled is divided into two parts, of which one can be used for the determination of the platinum as described above. In the other the double nitrites of rhodium and iridium are converted into chlorides by means of *aqua regia* containing but little nitric acid. The iridium is precipitated by ammonium chloride in the form of a chloro-iridate, the precipitate washed with a solution of ammonium chloride, ignited, reduced in hydrogen, and weighed as metallic iridium. It would also be possible to precipitate the iridium with potassium chloride in the form of the double chloride, $IrCl_4.2KCl$, the addition of a little chlorine water accelerating the precipitation. Finally the reduced product is washed with water to remove the potassium chloride, and dried at 115° C. The authors remark in conclusion that this method only gives the available iridium, or, in other words, that which is in combination with the platinum or other metals, and so dissolves in *aqua regia*. Any iridium in the free state or combined with the osmium is insoluble, and remains undetermined in the extracted residue.—C. A. M.

Mercury, Copper, and Zinc; Volumetric and Gravimetric Determination of ——. R. Cohn. Ber. 1901, **34**, [14], 3502—3508.

The methods brought forward by the author depend on the fact that mercury sulphocyanide is almost insoluble in water and undergoes no electrolytic dissociation.

Mercury.—When a dilute aqueous solution of a mercury salt, mixed with ferric alum, is made faintly acid with nitric acid and small quantities of ammonium sulphocyanide are added, the blood-red colour of ferric sulphocyanide does not appear until all the mercury has been converted into the sulphocyanide $Hg(SCN)_2$. The colour change is, however, not sharp so that the best way to carry out the determination is to run into the mercury solution first a measured volume in excess of N/10 ammonium sulphocyanide, then more than sufficient N/10 silver nitrate solution to decolorise the liquid, the excess of silver being afterwards titrated by means of N/10 ammonium sulphocyanide, which is added until the light brown colour persists on shaking the solution for a few minutes. The conditions of the titration are those required by Volhard's silver method.

Copper and Zinc.—When added to solutions of salts of these metals, ammonium mercury sulphocyanide forms insoluble compounds of the formula, $MHg(SCN)_4$. For the precipitation, a solution containing about 27 grms. of mercuric chloride and 32 grms. of ammonium sulphocyanide is prepared, and its exact strength determined by titration with N/10 silver nitrate solution, the reaction being expressed by the equation:—

$$HgCl_2 + 4NH_4.SCN + 4AgNO_3 = Hg(SCN)_2 + 2AgSCN + 2AgCl + 4NH_4NO_3.$$

The copper or zinc is determined as follows: to a definite volume of the copper or zinc solution placed in a dry flask is added a measured excess of the above liquid, the solution being then well shaken, filtered through a dry filter into a dry vessel and an aliquot part of the filtrate titrated with N/10 silver nitrate solution, the reaction being—

$$HgCl_2 + 4NH_4.SCN + CuSO_4 + 2AgNO_3 = Hg(SCN)_2Cu(SCN)_2 + 2AgCl + (NH_4)_2SO_4 + 2NH_4.NO_3.$$

The greatest accuracy is necessary in the titration, since an error of 0·1 c.c. may lead to great inaccuracy in the results. The mean of several determinations should hence be taken, and the individual results controlled by running in excess of silver nitrate and titrating back with ammonium sulphocyanide. A complete determination can, however, be carried out in 10 minutes.

Gravimetric Determination of the Three Metals.—Copper or zinc may be determined by adding to the acidified solution an excess of the liquid containing mercuric chloride and ammonium sulphocyanide described above, the precipitate, after standing for two hours in the cold, being filtered off and washed with cold water. In the case of copper, the filter and precipitate are burnt in a porcelain crucible; if the precipitate is large in quantity, the residue is converted into cuprous sulphide by adding sulphur and reducing in a stream of hydrogen, but smaller quantities are best oxidised by ignition with pure mercuric oxide, and weighed as cupric oxide. The latter method is also applicable to zinc but the filter paper must be previously removed or any reduced zinc may be volatilised. For the gravimetric determination of mercury, the liquid is mixed with a solution containing zinc sulphate (1 mol.) and alkali sulphocyanide (4 mols.) and the precipitate formed treated exactly as in the determination of zinc.

All these methods give good results.—T. H. P.

Mercury in Combination with Chlorine, Iodine, or Cyanogen; Determination of ——, *in Antiseptic Solutions.* G. Meillère. J. Pharm. Chim. 1901, **14**, [8], 356—359

In the case of the chloride and iodide, the aqueous liquid can be shaken with an excess of ether or ethyl acetate, and the ethereal extract evaporated, leaving the salt in a pure state. In a test experiment 5 c.c. of a solution containing 0·0343 grm. of mercuric chloride left 0·0338 grm. of salt on evaporation of the extract. For the determination of the mercury in the salt thus obtained, precipitation of the free metal by one of the following reagents is recommended, *viz.*, stannous chloride, alkaline hypophosphite, magnesium and hydrochloric acid, or sodium and hydrogen peroxide. The precipitation, washing, and drying are best carried out in the same tube with the aid of centrifugal force.

Cyanide and Oxycyanides.—Mercuric cyanide is usually met with in a sufficient state of purity, and its solution presents little difficulty in analysis. The pure oxycyanide is much less soluble, and the commercial salt is often a mixture in varying proportions of the cyanide and true oxycyanide. The alkalinity of the solution is assured by having a small quantity of mercuric oxide in excess of that required by the formula of the cyanide; or the same result is sometimes attained by the addition of potassium cyanide or another salt until the reaction is alkaline. The mercury in mercuric cyanide can be determined by titration, after being converted into the chloride. The pure salt should contain 79·3 per cent. of mercury, whilst theoretically the oxycyanide contains 85·04 per cent.

Four samples of commercial oxycyanide examined by the author contained from 76·8 to 81 per cent. of mercury.

The cyanogen can be determined by means of standard iodine solution in the presence of an excess of alkali bicarbonate. A direct titration invariably gives incorrect results, and it is therefore necessary to use a large excess of iodine solution, and to leave it in contact with the salt for several minutes before titrating with standard thiosulphate. Owing to the difficulty of dissolving a salt containing the theoretical quantity of oxycyanide, the author advocates the use of a solution of ordinary mercuric cyanide with the addition of borax.

The stock solution should contain:—mercuric cyanide, 500; borax, 100; Orange Poirrier, 1; and water, 6,000 parts. Of this solution 2 parts per 1,000 are used for the sterilisation of instruments. Steel needles can be preserved for several months in such a solution without altering to the slightest degree in length as they do in solutions containing only mercuric cyanide.—C. A. M.

Arsenic Sulphide; The Action of Ammonium Carbonate on ——. L. Vanino and C. Griebel. Zeits. anal. Chem. 1901, **40**, [9], 589—591.

In the ordinary course of analysis the sulphides of arsenic are dissolved in ammonium carbonate, forming arsenite and thio-arsenite, or arsenate and thio-arsenate respectively. Theoretically, the whole of the arsenic should be reprecipitated as sulphides on acidifying, without further addition of sulphuretted hydrogen, but the directions in the text-books vary as to this point. The authors find that hydrochloric acid does not reprecipitate the arsenic quantitatively from saturated solutions of As_2S_3 and As_2S_5 in ammonium carbonate, but that the reprecipitation is complete in the presence of much water in closed flasks.

In separating arsenic from antimony and tin, by treatment with strong hydrochloric acid, vigorous heating is not permissible since Piloty and Stock have shown that arsenic sulphide is volatilised when boiled with strong hydrochloric acid (this Journal, 1897, 764).—A. C. W.

Antimony; Detection and Determination of Traces of ——, in presence of large Amounts of Arsenic. G. Denigès. Comptes Rend. **133,** [18], 688—689.

The antimonial, or mixed antimonial and arsenical, stain or mirror, is dissolved in nitric acid, evaporated to dryness, redissolved in a drop of hydrochloric acid (1:3), and treated in one or other of the following ways:—1. The drop is placed in a platinum capsule, and a pointed rod of tin made to touch the capsule through the drop; a brown stain of antimony forms, the depth of tint and rapidity of formation depending on the amount present. With 0·002 mgrm. per 0·05 c.c., the stain is very distinct after half an hour. The amount of antimony can be estimated by comparison of the stain with similarly produced stains from known amounts of the metal. Arsenic, under these circumstances, unless the amount present exceeds 0·25 mgrm. per 0·05 c.c., gives no stain. 2. The drop is allowed to come in contact, on a microscope slide, with a drop of reagent prepared by dissolving 1 grm. of potassium iodide and 3 grms. of cæsium chloride in 10 c.c. of water. The formation of yellow or garnet red groups of hexagonal plates of the double cæsium and antimony iodide is easily visible under the microscope. This reaction is not interfered with by arsenic, unless the concentration of the latter be higher than 5 mgrms. per 0·1 c.c.—J. T. D.

Boric Acid and Lactic Acid; Determination of ——. Partheil. Zeits. angew. Chem. 1901, **14,** [41], 1020—1021.

In the method proposed, the boric acid is extracted from its solution in dilute hydrochloric acid by means of ether, the extraction (which requires about 18 hours) being carried out in a special apparatus. The boric acid left on evaporation of the ether is dried *in vacuo* over sulphuric acid, and weighed as H_3BO_3.

Lactic Acid.—Equally good results are obtained with this apparatus in the determination of lactic acid, the error noted by Kunz being reduced to a tenth, and thus only amounting to 0·002 to 0·003 grm., when a quantity of 0·5 to 0·6 grm. is taken for the analysis.—C. A. M.

Boric Acid; Direct Gravimetric Determination of ——. A. Partheil and J. A. Bose. Ber. 1901, **34,** [14], 3611—3612.

This process, which makes use of the apparatus shown in the figure, is carried out as follows:—The boric acid solution, made acid with hydrochloric acid, is placed in the spiral tube which it should fill no higher than the spherical bulb; recently rectified ether is then carefully added, until the bulb is nearly filled. About 20 c.c. of the same ether is added to the small weighed flask, which is then heated, either on the water-bath, or on wire gauze, by means of a small burner, until the ether boils vigorously, and separate drops of the liquid no longer fall from the condenser. The extraction is continued for 18 hours, at the end of which time the weighed flask is replaced by a second, the heating being carried on for two hours longer, to make certain that the extraction is complete. The flask is placed in a vacuum desiccator over sulphuric acid to remove the ether, and the residual boric acid weighed. Two determinations of aqueous solutions of 0·31015 grm. of boric acid gave the values, 0·3105 and 0·31025 grm. respectively, whilst from a solution of borax containing 0·12408 grm. of boric acid, 0·12425 grms. of the acid were extracted. The method has been applied to the analysis of minerals containing boric acid and can also be used for determining the acid in food materials. The boric acid solution should contain neither sulphuric acid, nitric acid, phosphoric acid, nor large quantities of iron; zinc chloride also passes over with ether, and the same is the case with aqueous solutions of arsenious acid. The purity of the acid weighed may be tested by evaporating repeatedly with pure methyl alcohol, the non-volatile residue being weighed, and the weight subtracted from that of the boric acid.—T. H. P.

ORGANIC CHEMISTRY.—QUALITATIVE.

Oils, Drying and Marine Animal; Detection of ——. G. Halphen. J. Pharm. Chim. 1901, **14,** [8], 359—365.

The test here described is a modification of that of Hehner and Mitchell (this Journal, 1899, 77), which is based upon the fact that most of these oils yield insoluble derivatives on treatment with bromine suitably diluted.

The author's reagent is prepared by mixing together 28 parts by volume of glacial acetic acid, 4 parts of nitrobenzene, and 1 part of bromine. In testing an oil, 0·5 c.c. is shaken in a stoppered tube with 10 c.c. of the reagent, and a note taken whether any precipitate is formed.

The following oils gave no precipitate, and the liquid remained clear after standing for an hour:—Olive, almond, castor, earthnut, cotton-seed, poppy, lard, and neatsfoot oils.

With Jaffa sesamé oil and with certain samples of horsefoot oil there was a slight turbidity.

Heavy precipitates were given by nut oil (after 5 to 10 minutes), hempseed and linseed oil of different origin; also by whale oil, seal oil, cod-liver oil, and Japanese fish oil; whilst colza oil produced a turbidity which, on standing, separated, leaving two layers of liquid. In testing for drying oils in colza oil it is advisable to add 10 c.c. of sulphuric ether when a turbidity is produced.

The author describes test experiments to show that it is possible to detect in this way the presence of 10 per cent. and less of these drying oils (with the exception of nut oil) or of marine animal oils in non-drying vegetable oils.

—C. A. M.

Nitromannitol and Nitrocellulose; Distinguishing ——. L. Vignon and F. Gerin. Comptes Rend. **133,** [14], 515—517.

Like nitrocellulose (this Journal, 1898, 794, and 1900, 1039), both penta- and hexanitromannitol reduce Fehling's solution, their reducing power being more than double that of glucose. Unlike nitrocellulose, however, they do not redden Schiff's reagent; so that their reducing action is not attributable to the formation of mannose.

Ferrous chloride reduces nitrocellulose to oxycellulose, which still exerts a reducing action on Fehling's solution; but nitromannitol, treated with ferrous chloride, is completely reduced to mannitol, destitute of any reducing power.—J. T. D.

*Sucrose in Plants; Identification of —— **by the Aid of** Invertase, and of Glucosides **by the Aid of Emulsin.*** E. Bourquelot. Comptes Rend. **133,** [18], 690—692.

The alcoholic extract of the plant under investigation is evaporated to dryness, the residue taken up with water saturated with thymol, and filtered. Equal portions of the solution are taken; to one is added a solution of invertase in thymolised water, and to the other an equal bulk of thymolised water alone. After three days the rotatory power and the Fehling reducing power of each are determined. In the case of the rhizome of *Scrophularia nodosa*, the angle of deviation diminished from 1° 43′ to 1° 13′, while the reducing sugar increased from 0·140 to 0·460 per cent. The inversion of 0·320 per cent. of cane sugar exactly agrees with the diminution of dextro-rotatory power; and the amount of cane sugar in the original rhizome is calculated (from the quantities taken) to be 4·054 grms. per kilo. Invertase hydrolyses only gentianose and raffinose besides sucrose, and these two sugars are rare and the products of their inversion easily distinguished from those of the inversion of sucrose.

After the treatment above, if the solution be boiled to destroy the invertase, and emulsin be then added, the dextro-rotatory power and the reducing sugar both increase from the hydrolysis of lævo-rotatory glucosides.

The author is pursuing investigations on these lines.

—J. T. D.

Dehydromucic Acid; Reactions of ——, and a New Method of Preparation. P. A. Yoder and B. Tollens. Ber. 1901, **34**, [13], 3446—3462.

The authors prepare dehydromucic acid (furfurane dicarboxylic acid) by heating mucic acid with twice its weight of concentrated sulphuric acid for 40 minutes, at a temperature of 133°—137° C. The product is purified by dilution and conversion into barium salt. About one-third of the mucic acid is converted into dehydromucic acid.

The authors have prepared series of salts and esters of dehydromucic acid, the latter bodies all being crystalline solids.

The authors have also investigated the colour reactions which dehydromucic acid and allied bodies yield when heated with isatin and concentrated sulphuric acid.

In 2 c.c. of pure sulphuric acid, 1—5 mgrms. of the substance, and about an equal quantity of isatin, were dissolved. The mixture was then slowly heated, and the temperature at which a coloration was produced was observed:—

——	Temperature.	Colour Reaction.
	° C.	
Pyromucic acid	80— 95	Violet-blue.
Ethyl pyromucate	125—140	"
Dehydromucic acid	145—155	"
All the esters of dehydromucic acid.	145—155	"
Mucic acid	130—140	Green.
Saccharic acid	140—150	"
Isosaccharic acid	130—140	"
Metasaccharic acid (double lactone).	150—160	"
Calcium arabonate	150—160	Violet.
Rhamnohexonic acid lactone	115—130	Wine-red.
Starch, milk-sugar, calcium *d*-mannonate, galactonic acid lactone, calcium gluconate, and rhamnonic acid.	..	No colour reaction; solution charred at 170°—190°.

The colour reactions were studied spectroscopically.

Unsuccessful attempts were made to reduce pyromucic acid with sodium amalgam, and to introduce a second carboxyl group into pyromucic acid, by treating ethyl pyromucate with sodium and carbon dioxide, and pyromucic acid with caustic soda and carbon dioxide.—J. F. B.

Starch; Iodolactic Acid as a Reagent for ——. G. Lagerheim. Svensk. Farm. Tidskr. 1901, [67]; through Pharm. Zeit. **46**, [28], 283.

By the addition of a few crystals of iodine to hot syrupy lactic acid, a reagent is obtained which is useful for the examination, *in situ*, of the starch grains of foods and drugs. The lactic acid renders the tissues transparent, while the iodine, tinting the starch grains blue, makes their form more evident. The author states that it is possible, with this reagent, to differentiate between exhausted and genuine tea-leaves. In the former, the starch grains in the cells are seen to be swollen and altered in form, while in genuine leaves they are in their normal condition.—J. O. B.

Creosote for Therapeutic Purposes; Examination of ——. M. E. Merklen. Zeits. anal. Chem. 1901, **40**, [9], 621—623.

Merklen detects water in creosote by heating 10 c.c. with 2 grms. of crystallised calcium chloride; when the salt melts, the liquids are mixed and allowed to cool. In the presence of water the calcium chloride does not again solidify. Phenol is detected by heating 4 c.c. of creosote with 1 c.c. of ammonia to 60° C., spreading the mixture over the surface of a large basin, and subjecting it to the action of bromine vapour. If phenol be present, a blue coloration is obtained; pure creosote gives a brown or green colour. Guaiacol may be approximately estimated in creosote as follows:—5 c.c. are mixed with 50 c.c. of alcoholic potash (1 part caustic potash in 5 parts of absolute alcohol). According to the amount of guaiacol, the mixture solidifies in 10—30 or 40 minutes; the crystalline mass is pressed between filter paper, decomposed by warming with 5 c.c. of sulphuric acid (1:10), water added until the mixture of creosol and guaiacol sinks, the aqueous solution poured off, and 4 c.c. of ammonia added. Guaiacol then gives a hard crystalline compound, whilst creosol gives only semi-fluid scales after some time. If the mixture be treated with petroleum spirit, the guaiacol compound remains. According to Merklen, pure creosote should have a specific gravity of 1·080, mix with dilute caustic soda without turbidity, and not act on blue litmus paper.

Béhal and Choay prepared pure guaiacol from pyrocatechol; it was a white substance, melting at 28 5° C. and boiling at 205·1° C. The crystals were hard prisms of the rhombohedric system; they exhibited the phenomenon of superfusion. Strong sulphuric acid gave no red coloration.

Marfori has obtained the following colour reactions with guaiacol prepared from pure potassium guaiacolate:—A solution in ammonia gives a blue coloration on warming with a little sodium hypochlorite; bromine water gives an orange-red precipitate, soon changing to coffee-colour. On heating with equal volumes of caustic potash and chloroform, guaiacol gives a fine red to brownish-red coloration; several other phenols give the same reaction. Guaiacol was found to dissolve in strong sulphuric acid, with a stable purple-red coloration; if traces of creosote be present the solution is a dirty greyish-green. Marfori found that 1 c.c. of guaiacol required 60 c.c. of water for solution.

Fischer determined the solubility of guaiacol in water to be 1 in 200.

Thoms has also examined synthetic guaiacol, which agreed in melting and boiling points with the preparation of Béhal and Choay. The solubility was 1:50. It dissolved in cold strong sulphuric acid to a colourless solution, which went from yellow to green to reddish-brown on warming (compare above). 10 c.c. of a 1 per cent. alcoholic solution of guaiacol at once gave an emerald green coloration with 1 drop of ferric chloride solution; but if the latter was diluted with nine parts of water, a blue coloration was first obtained, which quickly turned green. 10 c.c. of a 0·5 per cent. aqueous solution of guaiacol with one drop of the ferric chloride solution gave a blue coloration, which quickly disappeared and became brownish-red; on further addition of ferric chloride, the liquid became dark brown. A cherry-red coloration is obtained on adding a little permanganate solution to an aqueous solution of guaiacol acidified by hydrochloric acid; the colour slowly changes to brownish-red. Bromine water gives a brown precipitate in the aqueous solution.—A. C. W.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Acetylene and Ethylene; Solubilities of ——. S. A. Tucker and H. R. Moody. J. Amer. Chem. Soc. 1901, **23**, 671—674.

In order to devise a convenient method for separating acetylene and ethylene, the authors have examined the solubilities of the two gases in various solvents. Ammoniacal cuprous chloride solution, which is generally supposed to have no solvent action on ethylene, is found to dissolve this gas very readily, both in the pure state and when mixed with acetylene. Also Nordhausen sulphuric acid absorbs both the gases. Ammoniacal silver chloride, however, only dissolves ethylene to a slight extent, 26 c.c. of the gas, when treated with excess of the reagent, diminishing by 0·2 c.c. For the exact determination of acetylene and ethylene in a mixture of the two gases, recourse must be had to absorption by ammoniacal cuprous chloride, with which acetylene forms an insoluble red precipitate of the composition $(Cu_2C_2H)_2O$; this is collected, washed, dissolved in hydrochloric acid, converted into sulphate, and the copper determined electrolytically.

—T. H. P.

Shellac; Analysis of ——. E. J. Parry. Chem. and Druggist, 1901, **59**, [1135], 689.

From the results of the examination of numerous samples, the author concludes that by far the greater part of the shellac appearing on the London market is more or less adulterated with rosin. This frequent adulteration is probably the cause of the divergent figures published relating to the analytical constants of shellac. The author has examined a number of specimens of pure shellac of known origin, and gives the results obtained. Oberdörffer has recommended the determination of the specific gravity and the solubility in light petroleum spirit as tests for indicating the presence of rosin, and has suggested the application of these tests for quantitative purposes. The author considers both these tests to be unreliable. The limits of the specific gravity of both rosin and shellac are too wide to allow of any reliable deductions being drawn. The solubility of the two substances in light petroleum spirit certainly differs considerably, but with a mixture of shellac and rosin which have been melted together, the particles, even after reduction to a very fine powder, are so well coated with shellac, they are to a great extent protected from the solvent action of the petroleum spirit. The solubility is useful from a qualitative point of view, as the resinous matter extracted can be examined.

For judging as to the purity of a sample of shellac, the author states that the only determinations upon which reliance can be placed are those of the iodine absorption, free acid, and ester values. The following are the author's results :—

—	Pure Shellac.	Rosin.
Iodine absorption value.........	4—10 average 6	105—120 average 110
Acid value......................	55—65 average 60	150—170 average 162
Ester „	155—175 average 168	Usually less than 20, average 10

—A. S.

Fats and Oils; Use of Iodine Monobromide in the Analysis of ——. J. Hanuš. Zeits. Untersuch. Nahr. Genussm. 1901, **4**, [20], 913—920.

Iodine monobromide is recommended instead of iodine monochloride for the determination of the iodine absorption of fats and oils. The monobromide is prepared by slowly adding 13 grms. of bromine from a tapped funnel to 20 grms. of finely powdered iodine contained in a beaker. During the addition the mixture is well stirred and kept cool (from 5° to 8° C.). At the end of the operation, which should take about 10 minutes, a strong stream of carbon dioxide is passed through the beaker to drive off excess of bromine. Iodine monobromide is a grey crystalline substance, with a metallic lustre. It is soluble in ethyl alcohol and glacial acetic acid, and keeps well in closed vessels. The reagent is prepared by dissolving 10 grms. of the monobromide in 500 c.c. of glacial acetic acid. The exact strength of this solution is determined by titrating 10 c.c. with standard thiosulphate solution after the addition of 15 c.c. of a 10 per cent. solution of potassium iodide. Starch solution need not be added.

For the estimation of the iodine number, from 0·1 to 0·7 grm. of oil is placed in a stoppered flask and dissolved in 10 c.c. of chloroform. 25 c.c. of iodine monobromide solution are then added, and the mixture allowed to stand for 15 minutes. 15 c.c. of potassium iodide solution (10 per cent.) are then added, and the titration carried out with standard thiosulphate solution. With oils having an iodine absorption below 100 per cent., the reaction is complete in 10 minutes. Figures given in the original paper show that the results obtained by this method agree closely with those of the Hübl process.—W. P. S.

Tannin; Report to the International Association of Leather Trades' Chemists on the Methods in use for Determining ——. H. R. Procter. Leather Trades Rev., July 31 and Aug. 7, 1901.

There are at present two standard methods in use for the commercial estimation of tanning matters, *viz.*, that of the International Association of Leather Trades' Chemists, which is almost universally employed in Europe, and that of the Association of Official Agricultural Chemists of the United States, which holds a somewhat similar position in America. With a view to the adoption of a unified standard method, which is desired by both bodies, a careful comparison of the two methods has been made in the Leather Industries Laboratory of the Yorkshire College, of which results are hereinafter summarised.

In the present state of scientific knowledge, it is impossible in some cases to draw a definite line between actual tannins and other matters which are more or less absorbed by hide, and which are, therefore, included under the general head of "tanning matters." The amount of these estimated is to some extent dependent on the method of analysis employed, and, for commercial purposes, the most important point is that the method employed should be uniform, and capable of giving concordant results in the hands of different chemists, the question of whether a method gives high or low results being of secondary importance so long as it is universally adopted.

To ascertain the amount of uniformity which might be expected of the different methods, samples of four selected extracts (*viz.*, a Slavonian oakwood, "Mimosa D," which is a mixed extract, chemically decolorised, a hemlock extract, and a solid quebracho extract), together with hide-powder of batch No. 5 as supplied to English members of the International Association, and the necessary filter papers, were sent to all the English chemists who were willing to undertake the work. Of these, three only are regularly engaged in commercial analysis, the rest being careful and reliable workers, but without an equal amount of special experience. In order to obtain a judgment free from bias on the concordance of the different methods, the "mean error of experiment" was calculated from each series by the method generally used by chemists and physicists. What this really gives is an estimate of the concordance of individual experiments with the average of the series, and it is obvious that where the total number of experiments is small, a single widely divergent result may raise the mean error largely, not only by its own divergence, but by altering the mean of the whole. In all cases in the report, the mean error of the whole series of experiments has been given, but in cases where one or more of the results were widely divergent from the general average, showing the occurrence of some special source of error, a recalculation has been made excluding these results. In this direction some special allowance should be made for the results with the "shake" method of the American Association, which was comparatively new to several chemists, and with which more experience would certainly bring more concordant results.

Passing over the very important question of sampling, on which the American Association give no instructions, the first point of difference from the International is the prescribed strength of solution to be employed, which the A.O.A.C. prescribes as 0·8 grm. of "soluble" matter per 100 c.c., while the I.A.L.T.C. last year adopted 0·35 to 0·45 grm. of "tanning matters" per 100 c.c. In the case of oakwood extracts and many other materials, these two rules practically mean the same thing, but with quebracho and some other materials containing little soluble non-tanning matter, the former gives a solution too strong for convenient use. The change has proved very advantageous.

If the first half of the extract be obtained below 50°, Boegh has shown that the remainder may be extracted in most cases at steam heat without sensible loss.

In the solution of extracts for analysis, the A.O.A.C. prescribes heating to 50°, shaking well, and allowing to cool before weighing, dissolving at a temperature of 80° C., and allowing to cool slowly overnight; the I.A.L.T.C. only prescribes heating the extract when it is too thick to be otherwise effectively mixed, but in other cases it is merely carefully mixed by stirring. The required quantity of extract is then dissolved in boiling water, cooled rapidly under the tap, and made accurately to the required volume. This method possesses the advantage of rapidity, and is somewhat less troublesome; but it has been asserted in America that, in some cases, the insoluble matter of the extract was increased by the rapid cooling, which, if true, is an important objection. After cooling, in both cases the extract is well mixed, and a portion measured off and evaporated, to determine "total solids" and water in the original extract, an alternative method being to dry a small weighed portion of the original extract. The prescribed method of drying slightly differs in the I.A.L.T.C. and A.O.A.C. methods.

The extract solution is next filtered, the I.A.L.T.C. prescribing paper 605 S. and S., and the A.O.A.C. paper 590 S. and S., and permitting an addition of washed barium sulphate in cases where the extract will not filter clear without it. Paper 605 is a dense paper hardened by acid, which in almost all cases gives a clear filtrate, but filters with extreme slowness. Paper 590 is a somewhat expensive porous paper, purified with hydrochloric and hydrofluoric acids, and filters rapidly, but will not alone give a clear filtrate with hemlock and quebracho liquors. As both papers absorb traces of soluble matters, the I.A.L.T.C. prescribes that 200 c.c. must be filtered before taking 50 c.c. for determination of "total soluble," while the A.O.A.C. only requires 150 c.c. The prescribed addition of barium sulphate is little used in America, kaolin being found much more effective and less liable to come through the filter.

It is obvious that in comparing the two methods several points have to be taken into consideration :—

1. Does the heating of the extract to 50° produce any effect in cases where it can be adequately mixed without ?

2. Does rapid or slow cooling affect the solubility ?

3. By which method and paper can a clear filtrate be most readily obtained, and which produces the least absorption of tanning matters ?

4. How far are the results affected by the modes of evaporation and drying ?

To answer these questions, each chemist was asked to make the following experiments :—Each of the four extracts was to be dissolved in both ways, the solutions being marked A and B. Separate portions of each of these solutions were to be filtered through 605 paper (A1 and B1), and through 590 paper (A2 and B2) ; and, in addition, experiments were to be made with barytes and kaolin, 50 c.c. of the filtrates being evaporated and dried in each case according to the rules prescribed by each method.

The following table represents the results of evaporation of the unfiltered solutions, A representing the I.A.L.T.C. method, and B the A.O.A.C.

TABLE I.

——	No. of Determinations.	Average.	Mean Error.	Maximum Difference.
Oakwood :		Per Cent.	Per Cent.	Per Cent.
A	6	41·57	0·62	1·6
B	5	41·58	0·59	1·3
A & B......	11	41·57	0·58	1·6
Mimosa D. :				
A	6	49·90	0·46	1·4
B	5	50·04	0·45	1·1
A & B......	11	49·97	0·45	1·4
Hemlock :				
A	7	40·53	0·43	1·0
B	5	40·92	0·62	1·3
A & B......	12	40·69	0·50	1·4
Sol. quebracho :				
A	8	87·4	0·93	2·3
B	7	88·0	1·11	3·1
A & B......	15	87·67	1·11	3·7

The first point to be noticed is the large mean error of all determinations by both methods, averaging over $\frac{1}{2}$ per cent. This cannot occur in weighing out or dissolving the original sample, but is due either to differences in drying or in weighing the small dried residue. The rapidity with which residues absorb water on the balance is a formidable difficulty when it is reflected that on instance 1 mgrm. on the residue equals 0·2 per cent.

As regards different methods of drying, Dr. A. Turnbull made the following experiments, which are confirmed in regular work. The A.O.A.C. prescribes the use of flat porcelain basins of not less than 6 cm. in diameter. After evaporation to dryness on the water-bath, the basins are dried either for 24 hours in a water-oven, 8 hours in an air-oven at 105°—110° C., or till constant *in vacuo* at 70° C. The I.A.L.T.C. prescribes no definite size or shape of basin, and it became evident that the form of basin is of slight importance, the flat evaporating rather quicker, and the round being less rapidly oxidised. The vacuum oven, where it is available, is decidedly the most satisfactory, but accurate results can be got in the air oven at 105°. At temperatures below 100° the drying of solutions containing catechin is too slow, and probably involves much oxidation.

Returning to the comparison of methods, it will be noted that in every case the average result is slightly higher by the B than by the A method, and most so in the case of the solid quebracho, where heating before weighing is not employed. In the case of quebracho and other very strong extracts, it is probable that a more accurate determination of water and total solids would be obtained by drying, say, 0·5 grm. of the original substance without solution, or at most after the addition of a little alcohol.

Results of experiments on solution and filtration are next given. For each extract there are four sets of experiments, *viz.*, the I.A.L.T.C. method of rapid cooling under tap with 605 paper, the same method with 590 paper, and the A.O.A.C. method of slow cooling with 605 and 590 papers respectively. The following table gives the results :—

TABLE III.

Total Soluble.

Extract.	I.A.L.T.C.			
	Determination.	Average.	Mean Error.	Maximum Difference.
Oakwood :		Per Cent.	Per Cent.	Per Cent.
Paper 605	5*	40·46	0·195	0·5
„ 590	4*	41·08	0·515	1·1
Mimosa D :				
Paper 605	7	49·34	0·48	1·6
„ 590	6	49·50	0·64	1·8
Hemlock :				
Paper 605.....	6	37·65	0·48	1·5
„ 590.....	6	39·55‡	0·24	0·7
Quebracho :				
Paper 605.....	7	79·45†	1·94	5·5
„ 590.....	5	85·90‡	0·29	2·4

Extract.	A.O.A.C.			
	Determination.	Average.	Mean Error.	Maximum Difference.
Oakwood :		Per Cent.	Per Cent.	Per Cent.
Paper 605.....	3*	40·3	0·52	0·9
„ 590.....	4*	40·8	0·51	1·2
Mimosa D :				
Paper 605.....	6	49·55	0·57	1·8
„ 590.....	6	49·70	0·63	1·2
Hemlock :				
Paper 605.....	5	37·50	0·48	1·2
„ 590.....	5	38·82‡	0·76	1·2
Quebracho :				
Paper 605.....	4	79·75	0·88	2·0
„ 590.....	5	84·62‡	1·15	2·7

* One chemist omitted on account of very divergent results.
† Solution somewhat turbid in two out of seven determinations.
‡ Solution turbid.

Discussing the results of these determinations, it is noted that the opinion of the American chemists, that slow cooling gives higher results in soluble matters with the same filter paper, is not justified. On the whole, the results are somewhat higher with rapid cooling. It may also be noted that, as far as there is any noticeable difference, rapid cooling gives the smaller mean error, so that the advantage lies decidedly with the I.A.L.T.C. method of cooling, which has at the same time the merit of rapidity and convenience.

Comparing the filter papers, results so far do not indicate a clear course, and experiments are now in progress to establish a more scientific means of correction.

The addition of 3 grms. of purified kaolin to 150 c.c. of the extract solution, which was run through a 590 filter, and a further 50 c.c. filtered for analysis was found to give in every case a clear filtrate, and the time was somewhat shorter than with paper 605 alone, while the absorption by the filter was less, except in the case of mimosa D., which obviously contains some matter easily removed by absorption. The following figures may be given for comparison with Table III., but it must be remembered that they represent single determinations, and have not an equal claim to accuracy with the averages of the table.

Total Solubles, Kaolin Filtration.—Oakwood, 41·5 per cent.; mimosa D., 49·0 per cent.; hemlock, 38·7 per cent.; solid quebracho, 80·2 per cent.

There is, therefore, no obivous reason for the rejection of kaolin filtration by the Conference, and it is probably the best method known of obtaining a filtrate free from finely divided suspended matter. It is also evident that absolute clearness (absence even of opalescence) is necessary to concordant results in the case of turbid extracts, with solid quebracho the extreme difference between clear filtrates with paper 605, and with kaolin is only about half per cent., and in favour of the latter; while the turbid filtrates with 590 alone show suspended matter to the extent of 5 or 6 per cent.

There seems no reason for the compulsory adoption of an expensive quantitative paper like 590 for the filtration of extracts—the removal of silicates by hydrofluoric acid is only important where the paper is to be ignited, and it has been shown by Dr. Klenk (Ledermarkt) that paper in an acid condition absorbs more tanning matter than after it has been neutralised, a fact confirmed by experiment by the author.

Detannisation. — The solution in which the "total soluble" has been determined, is deprived of tannin by treatment with hide powder, and the remaining soluble matter again determined by evaporation of a measured quantity, which gives the "soluble non-tanning matter," and by subtraction from the "total soluble" gives the "tanning matters absorbed by hide." Though in all cases the principle is the same, the detailed methods differ considerably. The principle was first announced by Berthold Weiss, of the K.K. Vienna Research Institute. The "filter method" was devised by the writer, and improved in detail by Weiss, and is now the standard method of the I.A.L.T.C.; and the "shake method" of the A.O.A.C. was, I believe, the invention of Dr. Yocum. In addition to these, there is the "Palmer method," invented by Mr. A. N. Palmer, and now official with the I.A.L.T.C. for the analysis of used liquors. In the filter method very finely powdered and absorbent hide powder is placed in a small glass bell fitted with a siphon, and after this has been gradually soaked with the tanning liquor, the siphon is set in action, and after rejection of the first portion, which contains traces of dissolved hide substance, 50 c.c. is evaporated for analysis. In the shake method, the powder is thoroughly washed with water immediately before use, and a weighed portion of the squeezed, but still wet powder, is shaken mechanically for 5 or 10 minutes with a measured portion of the liquor, filtered through a funnel plugged with cotton, and 50 or 100 c.c. evaporated, allowance being made for the water introduced with the wet powder. The Palmer method is a sort of compromise between the two, the powder being washed with a small quantity of the liquor itself, and shaken by hand in a burette with a further portion, which is afterwards passed through the powder in the burette as a filter.

Table IV. gives the average results of the three methods, the liquors used being obtained by mixing the liquors produced by the two methods of solution, so as to obtain a sufficient quantity and a fair average. In certain cases widely divergent results have been rejected, and these figures are marked with a *.

TABLE IV.

Non-Tannins.

—	Filter Method.			
	Determination.	Average.	Mean E.	Max. D.
Oakwood	7	13·95	0·33	1·0
Mimosa........	8	11·09	1·25	3·9
	7*	10·73	0·79	2·3
Hemlock.......	9	12·88	0·05	1·9
	8*	12·72	0·33	1·1
Quebracho.....	8	11·36	2·11	7·2
	5*	11·56	0·24	0·5

—	Shake Method.			
	Determination.	Average.	Mean E.	Max. D.
Oakwood	5	15·54	1·04	2·4
	4*	15·08	0·10	0·2
Mimosa........	6	13·10	1·02	3·0
	5*	12·84	0·56	2·2
Hemlock.......	6	14·43	1·11	2·8
	4*	13·77	0·21	0·4
Quebracho.....	7	15·25	1·59	4·1
	5*	14·46	1·00	2·6

—	Palmer Method.			
	Determination.	Average.	Mean E.	Max. D.
Oakwood	4	17·3	0·72	1·7
Mimosa D.......	6	17·2	1·83	4·2
Hemlock........	6	16·3	1·01	2·3
Quebracho......	5	17·9	1·51	3·9

It may be remarked that, in the case of each of these typical extracts, the filter method gives the lowest result in non-tannins, and, consequently, the highest in tanning matters, the shake method averaging 2—3 per cent., and the Palmer method 4—5 per cent. lower. While this is not, in itself, any proof of accuracy or the reverse, it seems probable that the method which shows the largest absorption tends nearest to some fixed maximum which cannot be passed. It is known, however, that this maximum includes other matters than true tannins, so this argument is not of much account, though it carries with it the commercial difficulty that it is always easier to raise than to lower a commercial result, the sellers, on the whole, being more alive to its importance than the buyers! Turning to the point of mean error, the Palmer method shows a decided inferiority to the other two. Between the filter and the shake methods there is not much to choose. To gain some ideal of the accuracy which may be expected under favourable conditions, a series of six parallel estimations of non-tannins were made by Mr. Blockey with Freiberg powder on the oakwood extract by each method.

The results were as follows :—

Filter Method.—Average, 13·61; mean error, 0·059; maximum difference, 0·16 per cent.

Shake Method.—Average, 16·02; mean error, 0·135; maximum difference, 0·38 per cent.

The accuracy is somewhat greater by the filter method, though in both cases the mean error may be neglected. The result in the case of the filter method approaches very nearly to the average obtained with the mixed powder sent out, but by the shake method is about 1 per cent. higher than that of the starred or most probable average of the powder sent out, which agrees exactly with the mean of five determinations made with five different batches of the powder (principally Vienna) supplied by J. G. Parker. The presence of a trace of acid seems essential to the best results in shaking.

In order to test the degree to which the two methods were affected by differences in the powder, five determinations of the non-tannins of the oakwood extract were made by each method by Mr. Blockey, using the five different batches of powder sent out to English chemists by Dr. Parker, with the following results :—

Filter Method.—Average, 13·56; mean error, 0·53; maximum difference, 1·2 per cent.

Shake Method.—Average, 15·08; mean error, 0·22; maximum difference, 0·5 per cent.

The shake method was, therefore, less affected by differences in the (Vienna) powder. In further test of the possibility of working the shake method with very varied powders, an oakwood extract (not that analysed above, but which gave 14·2 per cent. of non-tannins by the filter method), was treated by the shake method with a powder made from the sawdust of raw buffalo hide pickers (a very cheap material) by simply deliming with sulphurous acid, washing repeatedly with water, and drying, first, in cool air, and then in the vacuum oven, and afterwards regrinding and allowing to absorb moisture. This powder was coarse, granular, and so unabsorbent (probably from the presence of a trace of fatty acids) that it was quite impossible to use it in the filter, but quite free from lime and acid. Shaken for 10 minutes it gave a non-tannin result of 22·7, and for 20 minutes of 18·4 per cent., and in neither case could the filtrate be got clear or free from colour. A fresh experiment was now tried, in which 10 c.c. of N/1 acetic acid was added to the last litre of wash water, and the powder pressed out as usual. A brilliantly clear filtrate was at once obtained, quite free from colour, and two duplicates, one shaken for 10 and the other for 20 minutes; each gave a result of 15·1 per cent.

The filter and the shake method each claim to be the less troublesome, and this is probably true in both cases, when the latter is carried out as is practically done in America, the shake method being the more rapid where large numbers of simultaneous analyses are made, and the filter where only a few determinations are required. In proof of this Mr. Alsop states that he has made 120 non-tannin determinations in a day with only one laboratory assistant. For this purpose the whole quantity of hide-powder required is washed and pressed at once. With some experience it is easy to decide sufficiently approximately what quantity of the wet hide powder is required for each analysis; the required amount is weighed out and added in each case, the bottles are shaken for five minutes, and in the meantime 10 grms. of the wet powder is dried to determine the correction for water, which is to be applied to all the determinations. On the other hand, with the excellent Freiberg powder, containing cellulose, no special skill is required in packing the filters; the apparatus is cheap, and as many can be set up as required, all can be wetted up and siphoned at once, and no moisture correction is required.

The author states that there is no evidence to justify giving up the filter method, but that more experiments should be made on the effect of small additions of acid either in washing the powder, or direct to the liquor, and this question must be considered carefully in the light of Dr. Paessler's experiments on the analysis of extracts rendered soluble with sulphide or alkalis, which showed that in these cases only a portion of the tannin present could be estimated with a neutral hide powder. For the analysis of used liquors, he is inclined to prefer the shake method very decidedly to the Palmer method, and to propose that it should be accepted as official for this purpose.

The use of chromed-hide powder, the author thinks, does not seem to have reached a completeness which would justify it as an official method.

Quebracho Extract; Analysis of ——. G. Klenk. Chem.-Zeit. 1901, **25**, [80], Rep. 283; from Wissenschaft-techn. Beil. Ledermarkt, 1901, **2**, 60.

The author states that in the analysis of weakly acid or nearly neutral quebracho extracts (soluble in cold water) discrepancies arise, owing to the presence of free hydrochloric acid in the filter paper employed.—R. L. J.

Chromed Hide-powder; Application of —— *in Analysis of Tanning Materials.* J. Paessler and W. Appelius. Chem.-Zeit. 1901, **25**, [80], Rep. 283; from D. Gerber-Zeit. 1901, **44**, [94], 96.

The results obtained by analysing each of certain materials with different hide-powders, *viz.*, ordinary Freiberg, Vienna, and chromed hide are given in the following table. Thus chromed hide-powder may give nearly the same results as are obtained with Freiberg powder (except in the case of sumach, gambier, and acid liquors), but no cause is indicated for abandoning the use of Freiberg powder.

Tanning Material.	Freiberg.	Vienna.	Chromed Hide.			
			Filter Method.		Shake Method.	
			Dry.	Moist.	Dry.	Moist.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Oakwood extract I. …	23·6	23·0	23·5	24·4	22·1	21·6
„ „ II. …	23·4	21·0	24·1	23·7	21·8	19·2
Mimosa extract …….	27·5	..	28·6	27·6	27·0	26·0
„ bark ………	31·4	..	31·3	31·0	..	31·4
Sumach……………	21·9	20·7	19·7	23·4	20·2	23·3
Gambier……………	48·1	33·2	52·3	44·1	41·0	39·8
Tan-liquors:	Results in Grms. per 100 cm.					
(*a*) 0·39 per cent. acid	1·72	1·45	1·35	1·68	1·64	1·46
(*b*) 0·34 per cent. acid	1·34	1·34	1·30	1·49	1·23	1·19

—R. L. J.

Leather [*Analysis of* ——]; ***Contributions to the Chemical Study of*** ——. E. Nihoul. Bourse aux Cuirs de Liège, 1901, September.

The author offers the following remarks upon the analysis of leather, without prejudice to the methods at present officially recognised.

Estimation of Moisture.—Crust leathers are completely desiccated at the temperature of 100°—105° C. only after the expiry of about 11 hours. If, however, after 1½ to 2 hours at this temperature, the oven is raised to 120°—122° C., desiccation is completed in about half an hour without damage to the leather.

Estimation of Fat.—The apparatus now used fails to remove all the fat even after three or four hours' extraction, as the Soxhlet tube never completely empties itself, and often the whole of the leather is not submerged in the liquid used. Better results are obtained if a tap separating-funnel is used, the stem being adjusted to a broad, squat flask which holds the extracting fluid. Some pieces of glass are put in the funnel, and upon them a layer of washed asbestos in a flakey condition; the leather rests upon this filter bed. The funnel is closed at the top by a stopper carrying (*a*) the end of a condenser, (*b*) a tube coiled several times and passing downwards into the distillation flask. The tap of the funnel is closed, and the fluid distilled upwards until the leather is quite covered; it is then completely drained off by opening the tap, and the operation repeated.

Estimation of "Total Soluble."—One litre of water is insufficient to remove all the soluble matter from leather. Appreciable quantities of tannin and non-tannin are dissolved out by a second and a third litre. After this point, however, the extract only contains tannin, indicating that all the uncombined tan-liquor, properly speaking, has been removed; the soluble matter obtained in this fourth and in successive litres is due to a physical dissociation of the leather. The author has therefore extracted with 3 litres in his examination of Belgian leather. Chestnut and pine-bark and sumac contain small quantities of soluble organic nitrogen.

Estimation of Tannin and Non-Tannin in the Leather Extract ("Total Soluble").—If the liquor, during concentration to a strength suitable for analysis by hide-powder, is covered by a funnel, the time of evaporation is so much extended that (*a*) part of the tannin (about 0·2 to 0·5 per cent.) is converted into non-tannin; (*b*) the glass vessel is attacked, alkali is liberated, the ash and non-tannin become higher, and organic matter is decomposed. Rapid evaporation is therefore desirable.

Estimation of Hide Substance.—All the well-known modifications of Kjeldahl's process were examined. The following conditions are recommended as most efficient and most economical. 0·6 to 0·7 grm. of the leather (free of fat and soluble matter) is boiled for half an hour with 10 c.c. of concentrated sulphuric acid. The flask is allowed to cool, and a dozen large crystals of potassium permanganate are added. The flask is shaken, heated till the liquid is decolorised, and then allowed to cool. The liquid is made up with water (including washings) to 250 c.c., 150 c.c. of caustic soda solution (30 per cent.) are added and a known quantity of zinc dust, and the ammonia is distilled off for three-quarters of an hour, the speed being such that about 200 c.c. of distillate are obtained. After this time, any traces of ammonia that might be left, affect the result only in the second place of decimals. The use of mercury or copper compounds is not recommended. Nor, under these conditions, more than 10 c.c. of sulphuric acid.

The ammonia distils into excess of N/5 sulphuric acid, and is titrated with equivalent soda or potash. Any indicator (except phenolphthalein) one is most accustomed to, may be used.—R. L. J.

Esters ; Quantitative Production of ——, and the Determination of Alcohols and Phenols. A. Verley and F. Bölsing. Ber. 1901, **34**, [13], 3354—3358.

A MIXTURE of alcohols or phenols with an acid anhydride only reacts slowly in the cold ; the authors have found that an addition of pyridine at once causes a vigorous action. $R.OH + (R'.CO)_2O + Py. = RO.CO.R' + R'.COOH.Py.$ The free acid produced at once forms a neutral salt with the pyridine, so that the ester cannot be again saponified. This reaction furnishes a very convenient quantitative method for the estimation of alcohols and phenols. A mixture is made of 120 grms. of acetic anhydride with 880 grms. of pyridine ; when water is added, pyridine acetate is formed, which is decomposed by alkalis into pyridine and alkali acetate, both of which substances are neutral to phenolphthaleïn ; the acid may therefore be titrated. In a flask of 200 c.c. capacity, 1—2 grms. of the alcohol are mixed with 25 c.c. of the pyridine mixture ; the flask is then heated for 15 minutes on the water-bath without condenser (acetic anhydride is not lost under these conditions). After cooling, about 25 c.c. of water are added, and the free acid titrated by normal or (better) seminormal caustic soda, using phenolphthaleïn as indicator. It is stated to be important to bring the pyridine mixture and standard soda, before measurement of the requisite volumes, to the temperature at which their comparative strength was determined. The following alcohols and phenols have given satisfactory results :—Ethyl and cinnamyl alcohols, phenylglycol, glycerin, phenol, β-naphthol, guaiacol, saligenin, thymol, eugenol, carvacrol, santalol. It is necessary to use a considerable excess of the acetic anhydride mixture with amyl alcohol and menthol, *i.e.*, not to take more than 1 grm. to 25 c.c. of the mixture. The process was applied to a sample of geraniol which the authors imagined to be pure, but not more than 90 per cent. could be esterified by any variation of the conditions ; the results were concordant, and the conclusion is that the geraniol contained 10 per cent. of impurities. Terpineol is only slightly esterified under these conditions. Vanillin and salicyl aldehyde react at once, but the ester is decomposed during the titration. It appeared that benzyl alcohol could be esterified only to the extent of 92 per cent. Linalol also gave much too low results.—A. C. W.

Clove Oil ; Determination of Eugenol in ——. A. Verley and F. Bölsing. Ber. 1901, **34**, [13], 3359—3362.

THE eugenol in oil of cloves is estimated by Umney by shaking with 10 per cent. alkali solution and determining the volume of the undissolved non-phenols. This method gives too high results ; it is better to use caustic soda of 3—4 per cent. strength, but the modified process gives 95 per cent. of eugenol in oils which only contained 80—85 per cent. as shown by the methods of Thoms and the authors.

Thoms' method consists in converting the eugenol into the crystalline benzoate, which is weighed. The results, however, vary with the operator, and the solvent action of the alcohol used to remove the terpenes varies with the temperature.

The authors have applied their process for the estimation of phenols (preceding abstract) to this determination. Synthetic oils containing 85, 90, and 95 per cent. of eugenol gave practically correct results by this process and that of Umney ; the results obtained by Thoms' method were 3—4 per cent. too low. The same agreement between the esterification and Umney's method was observed with certain natural oils, which were to be regarded as normal. Other oils, also doubtless genuine, gave 94 and 95 per cent. of eugenol by Umney's method, but only about 80 per cent. by the processes of Thoms and the authors. An oil evidently adulterated with clove oil terpenes gave similar results by Umney's and the authors' processes, but much too low results by Thoms' method. The authors conclude that the quantitative esterification process is suitable if no other alcohol or phenol be present, which may be ascertained by examination of the physical properties.

—A. C. W.

Alcohol in Ether ; Determination of ——. F. Freyer. Zeits. landw. Vers.-Wes. Öst. **4**, 955—959. Chem. Centr. 1901, **2**, [16], 900.

THE author has obtained satisfactory results by the application of Adam's method (Oesterr. Chem.-Zeit. **2**, 241), which is based upon the action of acetyl chloride on alcohol. Previous to the actual test, 20 c.c. of the sample of ether are shaken with a saturated solution of calcium chloride, the diminution in volume of the ether giving the amount of alcohol + water present. For the actual test, the quantity of ether taken must not contain more than 1 grm. of alcohol + water. 25 c.c. of the ether, or a smaller quantity diluted with anhydrous ether to this volume are placed in a strong-walled Erlenmeyer flask of ¼ litre capacity with 50 c.c. of a 10 per cent. chloroform solution of acetyl chloride. The flask is closed with a rubber stopper carrying a separating funnel of ¼ litre capacity containing 100 c.c. of water. After 1 hour, the stopcock of the funnel is opened and some water allowed to enter the flask. The mixture is shaken and when decomposition is complete, the free acid is titrated with twice normal alkali and phenolphthalein. A blank test is made with 50 c.c. of acetyl chloride solution, and the amounts of anhydrous ether and water used in the actual determination, and from the difference in the quantities of alkali used, the amount of alcohol can be easily calculated as acetyl chloride forms 1 molecule of free acid with alcohol and 2 molecules with water.—A. S.

Coca Leaves ; Assay of ——. W. R. Lamar. Amer. J. Pharm. 1901, **73**, 125 ; through J. Pharm. Chim. 1901, **14**, [8], 365—366.

THE author attributes the divergent results obtained by different chemists to a want of the recognition of the instability of the different alkaloids which are present. Cocaine, cinnamyl-cocaine, and isatropyl-cocaine are all methyl esters of ecgonine, and the methyl group attached to the carboxyl is readily split off, yielding, in the case of cocaine, benzoyl-ecgonine which has alkaloidal properties, but is insoluble in the liquids ordinarily employed for the separation of alkaloids from an alkaline solution. To obviate this it is essential to add only sufficient alkali to just liberate the alkaloids.

The author considers petroleum spirit as the most suitable solvent for dissolving the alkaloid without taking up much of the foreign substances. He recommends the following modification of Squibb's method : 25 grms. of the powdered coca leaves are treated with 25 c.c. of a 2 per cent. solution of ammonium hydroxide, and allowed to stand for 30 minutes, with occasional agitation, care being taken that the odour of ammonia is still perceptible. After the addition of 75 c.c. of petroleum spirit the mixture is again left for an hour or more, and shaken every 10 to 15 minutes during the interval.

The contents of the flask are now transferred to a percolator, and the mass continually washed with petroleum spirit until 450 c.c. of liquid in all have been collected. This extract is shaken in a separating funnel with 25 c.c. of N/10 hydrochloric acid, the acid layer withdrawn, and the extraction twice repeated with the same quantities of acid. The acid extracts are united, and shaken first with 20 c.c., and then with 15 c.c. of ether, in order to remove the last traces of petroleum spirit and colouring matter.

The united ethereal extracts are shaken with two portions (5 c.c.) of water, and the aqueous layers added to the original acid liquid. The latter is now rendered very slightly alkaline by means of ammonium hydroxide, and the alkaloids extracted with successive portions of 100 c.c. of ether. The ethereal extract is evaporated at 30°—35° C., and the residue dried at 60° C. until constant in weight.

As a check on this determination, the solution may be titrated by adding an excess of N/20 sulphuric acid, and titrating the uncombined acid with N/20 alkali, turmeric being used as indicator. Each c.c. of the N/20 acid corresponds to 0·0154 grm. of coca alkaloids. According to the author, coca leaves of good quality yield by this method not less than 0·7 per cent. of total alkaloids.
—C. A. M.

Guaiacol; Approximate Determination of ——. Adrian. Zeits. anal. Chem. 1901, **40**, [9], 624—625.

The permissible limits to the solubility of guaiacol are 1·2—1·5 per cent.; 1·602 grm. of pure guaiacol gives a clear solution in 100 grms. of water. Guaiacol may be approximately estimated by conversion into pyrocatechol; 100 grms. are heated with 10 c.c. of water for one hour in a current of hydrobromic acid gas, the residue, which contains the pyrocatechol and homopyrocatechol, is taken up in ether, the solvent removed, and the residue treated with benzene, in which only the pyrocatechol dissolves. From the weight of the pyrocatechol after recrystallisation, the percentage of guaiacol may be calculated to within 5—6 per cent.

The colorimetric process is as follows: 0·5 grm. of the guaiacol is dissolved in water and 10 c.c. of alcohol, and the solution made up to 1,000 c.c., 20 c.c. of this solution are mixed in a test-tube with 1 c.c. of sodium nitrite solution (1 : 100) and 1 c.c. of dilute nitric acid (1:200). A characteristic reddish-brown coloration is obtained, which is compared within 10 minutes with the colorations given by suitable standard solutions (see Creosote, page 1245).
—A. C. W.

Carbolic Acid; The Determination of Crude ——. G. Schacherl. Zeits. anal. Chem. 1901, **40**, [9], 615—616; and Zeits. d. allgem. österr. Apotheker-Ver. **46**, 794.

The following process is proposed:—100 c.c. of a crude carbolic acid, which is little soluble in caustic soda, or 50 c.c. of a better quality, are shaken with 100 c.c. of caustic soda (sp. gr., 1·1). The alkaline layer is run into a litre flask, the oil again shaken with 100 c.c. of caustic soda, and a similar treatment continued with 50 c.c. until no drops of oil separate on acidifying with hydrochloric acid. The united alkaline liquid, which contains all the phenol, small quantities of hydrocarbons and varying amounts of empyreumatic resins, is diluted with an equal volume of water and distilled until the distillate runs off quite clear. The residue is acidified with strong hydrochloric acid and again distilled; when 200 c.c. have come over, the water is returned to the distilling flask and the phenol transferred to a measuring cylinder. This operation is repeated until drops of oil are no longer seen in the distillate. The last distillate, which must not exceed 60—70 c.c., is also transferred to the measuring cylinder, a slight excess of common salt added, the cylinder well shaken and allowed to repose until the volume of phenol can be read off. The resins entirely remain behind in the distilling flask.

In the case of crude carbolic acid of high strength, completely soluble in caustic soda, the proper quantity is dissolved in 300 c.c. of caustic soda (sp. gr. 1·1) and treated as above.—A. C. W.

Carbolic Acid; The Determination of Crude ——. F. Seiler. Zeits. anal. Chem. 1901, **40**, [9], 618.

In this process 100 grms. of the crude substance are heated for 1 hour in a beaker on the water-bath, with frequent stirring with 100 grms. of milk of lime (1 part quicklime, 5 parts water). An equal volume of water is then added; the resins separate as insoluble soaps, whilst the hydrocarbons evaporate, so that on the addition of water an almost pure solution of calcium cresylate is obtained. The cresols are separated by acidifying and adding common salt; they are then weighed without further purification. The author has found in so-called crude carbolic acids of 25—30 per cent, 2—3 per cent. of cresols; 40—60 per cent., 3—5 per cent. of cresols; 80 per cent., 50 per cent., of cresols and in 90—100 per cent., 80 per cent. of cresols.
—A. C. W.

Creosote; Determination of ——. B. Hafner and W. Kreissl. Zeits. anal. Chem. 1901, **40**, [9], 625—626.

The active constituents of creosote all contain methoxyl groups, whilst the less valuable monatomic phenols do not. The authors recommend an estimation of the methoxyl group by means of Zeisel's method, using potassium arsenite as the washing liquid (Glücksmann, Monatsh. 1898, 142) and determining the silver iodide in the manner given by Gregor (this Journal, 1898, 609). The following are to be regarded as minimum limits:—sp. gr. at 15° C., 1·07; 75 per cent. to boil between 200° and 220° C.; methoxyl 12 per cent., corresponding to 48 per cent. of guaiacol.
—A. C. W.

Formaldehyde; Determination of ——. L. Vanino and E. Seitter. Zeits. anal. Chem. 1901, **40**, [9], 587—589.

Formaldehyde is determined by H. M. Smith (Analyst, **21**, 148) by means of potassium permangnate in alkaline solution; the end-point is difficult to recognise. The estimation is readily performed in strong sulphuric acid solution, when the aldehyde is oxidised to carbon dioxide and water. The process is as follows:—35 c.c. of N/5 permanganate are mixed in a stoppered flask of 250 c.c. capacity with a cooled mixture of 30 grms. of sulphuric acid and 50 grms. of water, and 5 c.c. of formaline solution (10 c.c. of formaline diluted to 400 c.c.) are slowly dropped in with constant shaking. After standing 10 minutes with occasional shakings, the excess of permanganate is titrated by N/10 hydrogen peroxide. The method gave 37·3 per cent. of formaldehyde as a mean of 3 concordant analyses, compared with 37·08 per cent. by Romijn's method, which is: 30 c.c. of N caustic soda are mixed in a stoppered 500 c.c. flask with 10 c.c. of dilute formaline solution (12·5 c.c. of formaline in 500 c.c.); 45 c.c. of N/5 iodine are slowly added from a burette with constant shaking, until the liquid is deep yellow. After well shaking for 1 minute, 40 c.c. of N hydrochloric acid are added and the excess of iodine titrated by thiosulphate after standing some time.
—A. C. W.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Patent Laws in Chemical Industry; The Question of ——. J. Ephraim. Zeits. angew. Chem. 1901, **14**, [36], 897—904; [37], 918—922.

The subject is fully discussed under the following headings:—

(1) Previous publication for chemical patents; (2) patentability of analytical processes; (3) methods for the preparation of intermediate products.—A. S.

Radium; Chemical Effects produced by Radiation from ——. H. Becquerel. Comptes Rend. **133**, [19], 709—712.

Berthelot has recently indicated some endothermic reactions produced by radium. These serve as a means of roughly measuring the energy of the radiation, which is of the order of magnitude of 5·1 ergs per second per sq. cm. But exothermic reactions of radium have also been recorded, such as its action on a gelatin bromide plate, the colours produced by it on glass, porcelain, and paper, rock salt and sylvine. The author has found that if phosphorus be melted under water, and a tube containing radium in an inner sealed tube wrapped in aluminium be immersed so as to be level with the phosphorus, a slow conversion of yellow to red phosphorus occurs, which ceases after the tube of radium is removed. A repetition of this experiment without the aluminium screen showed practically no difference in the intensity of the effect. A similar tube of radium immersed in a solution containing mercuric chloride and oxalic acid produced, in the dark, a gradual precipitation of calomel. The action of radium on organic tissues was examined by exposing to its radiation seeds of cress and of white mustard; 24 hours' exposure produced no sensible effect, but those seeds which had been exposed for a week or more failed in all cases to germinate when planted, though 8 out of every 10 of those not so exposed germinated.—J. T. D.

Dithionic Acid; Formation of ——. J. Meyer. Ber. 1901, **34**, [14], 3606—3610.

The author shows that the action of sulphur dioxide on manganese dioxide suspended in water, resulting in the formation of manganese dithionate, proceeds according to the equations—

$$2MnO_2 + 3H_2SO_3 = Mn_2(SO_3)_3 + 3H_2O + O\,;$$
$$Mn_2(SO_3)_3 = MnSO_3 + MnS_2O_6;\ MnSO_3 + O = MnSO_4.$$

Ferric hydroxide and sulphuric acid also yield a dithionate, the ferric sulphite first formed decomposing into ferrous sulphite and dithionate: $Fe_2(SO_3)_3 = FeSO_3 + FeS_2O_6$; cobalt and nickel hydroxides behave in a similar manner. Sulphurous acid yields sulphuric acid and barium sulphate with hydrogen and barium peroxides respectively, while the peroxides of lead, sodium and magnesium and mercuric oxide are unaltered. Barium and sodium dithionates give the normal molecular weights in freezing water. Measurements were made of the molecular conductivity of the barium salt.—T. H. P.

Liquefied Gases; New Method of manipulating —— *in Sealed Tubes.* H. Moissan. Comptes Rend. **133**, [20], 768—771.

For cooling the apparatus, a bath of solid carbon dioxide, and acetone is used, in a Dewar's vacuum vessel. When the evaporation of the dioxide is hastened by a current of air at the ordinary temperature of the laboratory, the temperature of the bath is about −98° C.; and if the air be previously cooled to about −80° C., by passage through a spiral tube immersed in a similar bath, the mixture may be further cooled to −110° C. For still lower temperatures, liquid air, or liquid oxygen, may be employed. The investigation of the action of a liquefied gas on a solid is carried out by bringing the liquefied gas into a tube, cooling it till it has solidified, adding the solid, exhausting the tube by the Sprengel pump, and then carefully sealing it. On gradually allowing the temperature to rise, the gas liquefies, and (provided the temperature reached is below its critical point) remains liquid under the pressure of its vapour. The author has kept ammonia, chlorine, and hydrogen sulphide in such tubes, of 10 mm. external and 6 mm. internal diameter; acetylene and hydriodic acid in tubes of 7 mm. external and 3 mm. internal diameter; and, in cases where the pressure may reach 300 atmospheres, used tubes of 6 mm. external and 1·5 mm. internal diameter. When the action is over, the tubes are cooled again so as to solidify the contents, connected with the mercury pump, the sealed ends carefully broken, and the temperature is allowed gradually to rise. Great care must be taken that all the materials used are free from moisture; and actions evolving hydrogen should not be carried on in sealed tubes. It is to be remembered, too, that glass which has been cooled to such low temperatures is apt to become brittle and untrustworthy, even when very gradually warmed again. Under the most favourable circumstances, there is considerable danger in these experiments, and all suitable precautions against accident should be taken.—J. T. D.

Calcium-Ammonium and Lithium-Ammonium; Decomposition of ——, *by Ammonium Chloride.* H. Moissan. Comptes Rend. **133**, [19], 715—717.

Ammonia was condensed at −40° C. into a U-tube containing calcium, when the calcium-ammonium formed dissolved in the excess of liquid ammonia. On cooling to −80° C., and then introducing some ammonium chloride, immediate evolution of gas occurred. Measurement and examination of this gas and analysis of the remaining liquid showed that the reaction was—

$$2NH_4Cl + Ca(NH_3)_4 = CaCl_2 + 4NH_3 + (2NH_3 + H_2).$$

No indication of the production of ammonium, NH_4, was given. Lithium-ammonium behaved in exactly the same way.—J. T. D.

Borimide, $B_2(NH)_3$. A. Stock and M. Blix. Ber. 1901, **34**, [12], 3039—3047.

The reaction between boron bromide and hydrogen sulphide, which results in the formation of a hydrosulphide of boron sulphide, B_2S_3, H_2S, proceeds briskly at first, but afterwards slackens. The authors find that this is due to the formation of a crystalline compound of the composition B_2S_3, BBr_3, which can be more readily obtained by dissolving the boron sulphide hydrosulphide in excess of boron bromide, and evaporating the solution to dryness in a vacuum. It forms colourless crystals, melting above 100°, and decomposing into its components on strong heating or on boiling its carbon bisulphide solution. A similar compound of the composition B_2S_3, BCl_3, has also been prepared.

When ammonia is liquefied in a specially constructed tube containing boron sulphide hydrosulphide, and the tube sealed off and shaken at the ordinary temperature for several hours, the hydrosulphide is completely dissolved, yielding a deep yellow liquid. After opening the tube at a low temperature, and allowing the ammonia to evaporate at 0°, a yellow liquid remains, which, at the ordinary temperature of the air, gives off large quantities of ammonia and hydrogen sulphide, and deposits at the same time intensely yellow twin crystals of the composition B_2S_3, $6NH_3$. This compound, probably identical with one obtained by Moissan from boron trisulphide and ammonia, is decomposed by water, while on heating it splits up, yielding ammonium hydrosulphide and borimide, according to the equation $B_2S_3, 6NH_3 = 3NH_4.SH + B_2(NH)_3$. The borimide thus obtained contains appreciable quantities of sulphur, a purer product being obtained by passing ammonia in a slow stream over boron sulphide hydrosulphide heated to 75°. As thus prepared, borimide is a light white powder which is decomposed by water with evolution of much heat and formation of boric acid and ammonia. When heated, borimide begins to evolve ammonia at 125°—130°, and at higher temperatures is decomposed quantitatively according to the equation—

$$B_2(NH)_3 = 2BN + NH_3.$$

—T. H. P.

Ferric Oxide and its Hydrates. O. Ruff. Ber. 1901, **34**, [13], 3417—3430.

The author has examined the formation of hydrated ferric oxide, with respect to the influence of (1) the pressure of the water vapour; (2) time; (3) pressure; (4) saturated magnesium chloride solution; and (5) crystalline form. His results lead to the following conclusions:—(1) The red colloidal ferric hydroxide, when subjected to a high pressure under water, is transformed in a relatively short space of time into a true hydrated oxide; at about 42·5° C. it passes into a form practically identical with that of brown ironstone, $Fe_2O_3 + 1\frac{1}{2}H_2O$, at 42·5—62·5° into Göthite ($+ H_2O$) and at higher temperatures into hydrohæmatite ($+ \frac{1}{3}H_2O$). The nature of the hydrate is hence dependent only on the magnitude of the tension of the water vapour. (2) The yellow modification of ferric hydroxide, obtained by the oxidation of ferrous oxide or its hydrate or of ferrous carbonate, is not a true colloid, since under high pressure, its water content scarcely varies with a change of temperature of 40°—70°. (3) Under ordinary pressure and temperature, red colloidal ferric hydroxide passes very slowly into anhydrous oxide, which at the same time gradually absorbs water and is transformed into the hydrate containing $1\frac{1}{3}H_2O$, and this latter is the only form stable under ordinary conditions. (4) The form of ferric oxide contained in pure red iron ore can only be produced from its hydrate at high temperatures, as, for instance, when in contact with granite, but it can be produced under favourable circumstances by the dehydration of the colloid.—T. H. P.

Copper Oxide; Simultaneous Reaction of Carbonic Acid and Salts of the Alkali Metals on ——. O. Kühling. Ber. **34**, [12], 2849—2852.

By leading a stream of carbon dioxide through a solution of sodium chloride in which is suspended copper oxide (freshly precipitated and dehydrated at as low a temperature as possible), there is obtained after 2—3 days a gray-green amorphous insoluble powder of the formula—

$$Cu_7Cl_3O_{11}H_{11}(CuCl_2.CuClOH.5Cu(OH)_2).$$

At 240° C. this substance gives off water and oxygen, leaving a blackish-brown residue of formula $Cu_7Cl_3O_5$. Even prolonged boiling with water does not remove the whole of its chlorine (as hydrochloric acid) from the green substance.

If for sodium chloride be substituted, in the above process, sodium nitrate, a bright green amorphous basic cupric carbonate, $CO(OCu.OH)_2$, is obtained; and there is no formation as there is in the former case, of sodium bicarbonate.

Neither in the differing solubilities of the two alkali-salts, nor in the dissociation-constants of their solutions, can there be found an explanation of their very different behaviour; and the facts that sodium bromide, though reacting similarly to the chloride, does so very much more slowly, and that the corresponding potassium salts react very much more slowly than the sodium salts, show that the explanation of the reactions is yet to be sought.—J. T. D.

Antimony; Localisation and Dissemination of ——, *in the Organism.* G. Pouchet. Comptes Rend. **133**, [14], 526—527.

After death from antimony poisoning, the metal in notable quantity was found only in the intestines, traces were found in the bones, kidneys, liver, skin and fur, and muscles; doubtful indications in the heart, lungs, and blood, and none whatever in the brain.

The addition of a little arsenic does not modify the distribution of the antimony in the tissues (though the toxic action of the arsenic seems to be increased by its association with the antimony). After death in these cases, arsenic, but no antimony, was found in the brain and spinal cord, muscles, and liver; arsenic with a trace of antimony in the bones; arsenic and antimony in more notable quantity in the skin and fur, and much more antimony, with but little arsenic, in the digestive organs.

The administration of potassium bromide simultaneously with the antimony seems to modify both the symptoms of its action and its distribution in the body.—J. T. D.

Mercury: its Alteration in Volume on Melting, and its Thermal Expansion when Solid. L. Grunmach. Chem.-Zeit. 1901, **25**, [84], 919—920. Paper read at the Hamburg Meeting of the Gesellschaft deutscher Naturforscher und Aerzte.

The research was conducted by testing an ordinary alcohol thermometer with an arbitrary scale at various temperatures against a standard alcohol thermometer, and determining thus the value of each degree of the (arbitrary) scale. This thermometer was then half filled with mercury and half with alcohol, and the new values of the degrees were determined and compared with the first. The results obtained with the instrument employed were:—

Condition of Hg.	Range of Temperature.	Value of 1°.	
		Alcohol alone.	Half Alcohol. Half Mercury.
	° C. ° C.		
I. Solid.....	−78·2 to −38·5	1·9321	1·0527
II. Melting..	−38·5 „ −33·7	1·9160	8·4375
III. Liquid...	−33·7 „ 0	1·8998	1·0777

Deducting from the alcohol-mercury value of the several degrees half the corresponding alcohol values, the numbers relating to the expansion of the mercury alone are:—I., 0·0867; II., 7·4795; III., 0·1278. Hence, during the melting of the mercury, its expansion per degree is 58·68 times as great as it is when liquid, and 86·5 times as great as when solid; and the coefficient of expansion of solid mercury is approximately two-thirds (actually 0·6784) that of liquid mercury. So 1 c.c. of solid mercury occupies a volume of 1·05098 c.c. when melted.

—W. G. M.

Silver Oxide; Action of Hydrogen Peroxide on ——. Berthelot. Comptes Rend. **133**, [16], 555—569.

A new series of calorimetric experiments. When silver nitrate and sodium hydroxide in dilute solutions are mixed in molecular proportions, the reaction is complete in a very few seconds, and the heat-evolution is 17·3 cal. for the precipitation of a molecule of Ag_2O. When dilute nitric acid is now added in molecular proportion, and the solution is agitated, the whole of the precipitate is dissolved in less than three minutes, with a heat-evolution of 10·32 cal. Sulphuric and lactic acids effect the solution of the silver oxide with almost equal rapidity; the heat-evolutions being respectively 18·75 and 8·4 cal.

When, after the precipitation of the silver oxide as above, hydrogen peroxide is added in quantity sufficient to supply 1 atom of oxygen for each molecule of Ag_2O, the precipitate becomes perfectly black, and oxygen is evolved. The amount of this oxygen, as also the accompanying heat-evolution, correspond exactly with the decomposition of one molecule of peroxide into water and oxygen; and, since no undecomposed peroxide remains in the solution, it would appear as though the silver oxide had played no part in the action. On now adding nitric, sulphuric, or lactic acid, however, there is practically no heat-evolution, or only a very slow and gradual one, the substance behaving qutie differently from ordinary silver oxide; while if the precipitate be heated with dilute sulphuric acid, it partly dissolves, leaving behind metallic silver equivalent to exactly half that taken into solution as sulphate.

The author considers that these experiments confirm his previously expressed views (this Journal, 1901, 625) of the reactions involved, which may be summarised in the equations:—

$$6Ag_2O + 6H_2O_2 = 6Ag_2O_2 + 6H_2O;$$
$$6Ag_2O_2 = 2Ag_2 + 3O_2 + 2Ag_4O_3.$$

—J. T. D.

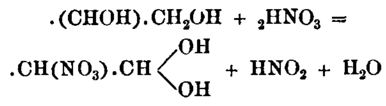
Unsaturated Hydrocarbons; Conversion of Alcohols into —— *by the Action of Oxalic Acid.* N. Zelinsky and J. Zelikow. Ber. 1901, **34**, [13], 3249—3256.

The authors have made use of the dehydrating action of oxalic acid for the preparation of a number of unsaturated hydrocarbons from alcohols. The oxalic acid was employed in the anhydrous state, but the authors have found that the hydrated acid also acts as a dehydrator, although in some cases it gives somewhat different products. The hydrocarbons obtained from the various alcohols examined are as follows:—Pinacoline alcohol yields tetramethylethylene; cyclohexanol (synthetical), tetrahydrobenzene (naphthylene); β-methylcyclohexanol, methylcyclohexene; menthol, menthene; dextro-borneol with hydrated oxalic acid gives an optically active terpene, melting at 3°—4°, and having an $[\alpha]_D$ cf 8·12°, whilst with the anhydrous acid two different terpenes are obtained; lævo-borneol gives an optically active terpene; 1:3-dimethyl-3-cyclohexanol yields a hydrocarbon of the formula C_8H_{14}, which boils at 126°—127°, and has an $[\alpha]_D$, + 95°; 1-methyl-3-ethyl-3-cyclohexanol, a compound, C_9H_{14}, boiling at 148°—149°, and having $[\alpha]_D = + 56·8°$; 1:3-dimethyl-4-isopropyl-3-cyclohexanol, a hydrocarbon boiling at 180°—182°, and having $[\alpha]_D = 88·53$; and methylfenchylalcohol gives a compound which boils at 172°—173° C., has an $[\alpha]_D$, + 19·68°, and may be regarded as the first known methyl-terpene of the formula $C_{11}H_{18}$ (methylfenchene).—T. H. P.

Arabitol and Rhamnitol; Nitro-derivatives of ——. Constitution of certain Nitric Esters. L. Vignon and F. Gerin. Comptes Rend. **133**, [17], 641—643.

On reducing *d*-arabinose and rhamnose, the arabitol and rhamnitol formed were found to have no reducing action on Fehling's solution, but on completely nitrating these products, the nitro-derivatives had a strong reducing action.

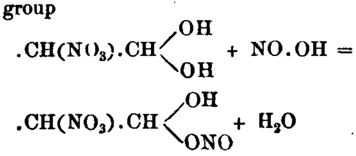
The authors suggest the following explanation of the fact that Fehling solution is reduced by the nitro-derivatives of *n*-hydric alcohols, where *n* is not less than 4. It is known that aldehydic hydrates are readily formed where the rest of the molecule is acidic or electro-negative in its character —thus $CCl_3.CH(OH)_2$ (chloral hydrate) is stable, while $CH_3.CH(OH)_2$ is non-existent. The action of nitric acid on these alcohols, then, is in the first place to nitrate the CHOH groups, and to oxidise the terminal CH_2OH group :—



$$.(CHOH).CH_2OH + {}_2HNO_3 =$$

$$.CH(NO_3).CH\left\langle\begin{matrix}OH\\OH\end{matrix}\right. + HNO_2 + H_2O$$

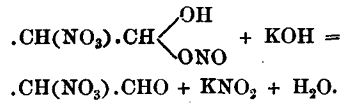
the nitrous acid so formed immediately reacts on the $CH(OH)_2$ group



$$.CH(NO_3).CH\left\langle\begin{matrix}OH\\OH\end{matrix}\right. + NO.OH =$$

$$.CH(NO_3).CH\left\langle\begin{matrix}OH\\ONO\end{matrix}\right. + H_2O$$

and the potash of the Fehling's solution converts this into an aldehydic group, which then reduces the Fehling—



$$.CH(NO_3).CH\left\langle\begin{matrix}OH\\ONO\end{matrix}\right. + KOH =$$

$$.CH(NO_3).CHO + KNO_2 + H_2O.$$

Unless there be at least two internal $.CHONO_2$ groups, the acidity of the molecule is insufficient to allow of the formation of the terminal $.CH(OH)_2$ group. Tollens' pentaerythritol, which contains only CH_2OH groups, and the nitro-derivative of which, $C(CH_2ONO_2)_4$, though containing four ONO_2 groups, does not reduce Fehling, lends support to the authors' view.—J. T. D.

Albumin; Formation of an Isatin-Derivative of ——. J. Gnezda. Comptes Rend. **133**, [14], 517—518.

By reacting on albumin with hypochlorous acid, reducing the product with zinc and hydrochloric acid, distilling with steam, and extracting the distillate with ether, a whitish crystalline product is obtained, which after long desiccation over sulphuric acid, gives brown needle-shaped crystals of a neutral substance, insoluble in water, but soluble in alcohol or ether. This substance gives the distinctive reactions of chlorisatin. The yield is very small.—J. T. D.

Oxide of Hydrogen higher than the Dioxide; Supposed Formation of an ——. W. Ramsay. Proc. Chem. Soc. **17**, [241], 197.

Bach (this Journal, 1900, 863) has stated that evidence can be obtained of the existence of a peroxide of hydrogen higher than the dioxide, by comparing the amount of oxygen evolved from peroxide on treatment with permanganate with the weight of the peroxide present in solution, as determined by titration with permanganate. It was suggested by Armstrong (Proc. Chem. Soc. 1900, **16**, 154) that the discrepancy should be attributed to the formation of persulphuric acid and Caro's acid, due to the action of the peroxide on the sulphuric acid present, during the titration. The author now shows that if peroxide be added to a mixture of sulphuric acid with permanganate, all the oxygen is evolved, but that if the permanganate be added to a mixture of peroxide with sulphuric acid, only a partial evolution of oxygen takes place, for neither persulphuric acid nor Caro's acid is readily attacked by permanganate. On the other hand, if acetic acid be substituted for sulphuric acid, the amount of oxygen evolved corresponds exactly with the amount of permanganate added, whether the latter be added to a mixture of peroxide and acetic acid, or whether the peroxide be added to a mixture of permanganate and acetic acid.

Trinitrobenzene and Trinitrotoluene; Reduction of —— with Hydrogen Sulphide. J. B. Cohen and H. D. Dakin. Proc. Chem. Soc. 1901, **17**, [242], 214.

By the reduction of 1:3:5-trinitrobenzene and 2:4:6-trinitrotoluene in alcoholic solution with hydrogen sulphide in presence of a trace of ammonia, the corresponding dinitrohydroxylamine compounds are formed—

$$C_6H_3(NO_2)_3 + 2H_2 = C_6H_3(NO_2)_2NHOH + H_2O.$$

Dinitrophenylhydroxylamine is an orange crystalline substance, m. pt. 114°—116°, soluble in alcohol, benzene, and in hot dilute hydrochloric acid; *dinitrotolylhydroxylamine* crystallises from benzene in yellow rhombohedrol crystals and from dilute hydrochloric acid in needles, m. pt. 143°—145°. Both compounds reduce alcoholic silver nitrate and Fehling's solution; they are slowly decomposed on boiling with dilute, more rapidly with concentrated hydrochloric acid, yielding the amino-compounds and losing oxygen—

$$C_6H_3(NO_2)_2NHOH = C_6H_3(NO_2)_2NH_2 + O.$$

In the case of trinitrotoluene, the nitro-group in the ortho-position undergoes reduction, a curious fact, seeing that ammonium sulphide effects the reduction of the para-nitro-group. The 2:4-dinitro-6-toluidine, obtained by the action of strong hydrochloric acid on dinitrotolylhydroxylamine, is a colourless substance crystallising in needles and melting at 212°—213°.

Research Scholarship.

Iron and Steel Institute.

A research scholarship or scholarships, of such value as may appear expedient to the Council of the Iron and Steel Institute, from time to time, founded by Mr. Andrew Carnegie (Vice-President), who has presented to the Iron and Steel Institute 64, 1,000 dol. Pittsburg, Bessemer, and Lake Erie Railroad Company 5 per cent. debenture bonds for the purpose, will be awarded annually, irrespective of sex or nationality, on the recommendation of the Council of the Institute. Candidates, who must be under 35 years of age, must apply, on a special form, before the end of March, to the Secretary of the Institute.

The object of this scheme of scholarships is not to facilitate ordinary collegiate studies, but to enable students who have passed through a college curriculum, or have been trained in industrial establishments, to conduct researches in the metallurgy of iron and steel and allied subjects, with the view of aiding its advance or its application to industry. There is no restriction as to the place of research which may be selected, whether university, technical school, or works, provided it be properly equipped for the prosecution of metallurgical investigations.

The appointment to a scholarship shall be for one year, but the Council may at their discretion renew the scholarship for a further period, instead of proceeding to a new election. The results of the research shall be communicated to the Iron and Steel Institute in the form of a paper to be submitted to the annual general meeting of members, and if the Council consider the paper to be of sufficient merit, the Andrew Carnegie Gold Medal shall be awarded to its author. Should the paper in any year not be of sufficient merit, the medal will not be awarded in that year.

By Order of the Council,
Bennett H. Brough,
Secretary.

Prizes.

INSTITUTION OF MINING AND METALLURGY.

THE Council of the Institution of Mining and Metallurgy have pleasure in announcing that a gold medal and premium of the value of fifty guineas, presented by the Consolidated Gold Fields of South Africa, Ltd., will be awarded annually by the Institution on the following conditions, namely :—

1. The award will be made by a committee of three gentlemen who will be nominated by the Council of the Institution.
2. The first award will be made in June 1902, and succeeding awards in June in each year.
3. The gold medal and premium will be awarded to the author of the paper of highest merit contributed to the Transactions by any member, associate, or student of the Institution, during the preceding session, upon the mining, treatment, or reduction of gold ores.

Whilst the Council do not deem it expedient to specify any exact subject within the limits above stated, a list of suggestions of suitable subjects is sent herewith for the assistance of those intending to submit papers.

By Order of the Council,
C. McDERMID,
Secretary.

The following subjects are suggested as suitable for papers in connection with the Annual Gold Medal and Premium, of the value of Fifty Guineas, to be awarded by the Institution of Mining and Metallurgy :—

The comparative merits of Wet and Dry Crushing.
Crushing Machinery.
Ore Sampling Apparatus and Methods.
Design, construction, and erection of Stamp Batteries.
Milling practice and amalgamation.
The Relative Value of Diamond and Electric Rock Drills.
Boring.
The testing, upkeep, and life of Winding Ropes for deep level Shafts.
Shaft-sinking; Timbering; Plant; sizes for deep level Shafts, &c.
Hoisting Engines for deep level Mines.
Ventilation of deep level Mines.
Explosives.
Lighting.
Pumping: electric, steam, hydraulic, and air.
The Geology of the Rand, with special reference to the extension of the Main Reef Series east and west of present known boundaries.
Methods of prevention of over-winding.
The merits of Compressed Air, as compared with Electricity, for transmission of power.
The transmission of energy, by electricity, in Gold-milling.
The best method of Laying-out a Mine and of Stoping the Reefs when dipping at a slight angle.
Mine Sampling and Estimation of Ore Reserves.
Sorting Appliances, and advantages of sorting.
The influence of Crushing on the Rand Conglomerates.
Recent developments in Cyaniding and in Chlorination.
Methods of treatment of Slimes.

New Books.

THE LABORATORY COMPANION TO FATS AND OILS INDUSTRIES. By Dr. J. LEWKOWITSCH, Consulting and Analytical Chemist and Chemical Engineer, &c. Macmillan and Co., Ltd., London. 1901. Price 6s. net. The Macmillan Company, New York.

8vo volume, containing preface, table of contents, and 141 pages of text, with, finally, the alphabetical index. The work is divided into three parts :—PART I. SYSTEM AND EXAMINATION OF FATS AND WAXES. This again is subdivided into : A. System of Fats and Waxes. B. Saponification of Fats and Waxes ; and C. Examination of Fats and Waxes. Following this, are tables of the Glycerides, Waxes, Fatty Acids, &c., with their various constants, values, and chief properties, and finally their occurrences. PART II. is devoted to the FATS, OILS, and WAXES, and the COMMERCIAL PRODUCTS DERIVED THEREFROM. It comprises a series of tables wherein at a glance is seen any specific value, property, use, or occurrence. A. Fats, Oils, Waxes, and their Constants and Variables. B. Commercial Products of the Fats and Oils Industries. Under these we have 1. Lubricants. 2. Wool Oils, Cloth Oils. 3. Sod Oils—Dégras. 4. Oxidised Oils. 5. Vulcanised Oils. 6. Candle Making. 7. Soap Manufacture. 8. Glycerin. PART III. A series of General Tables, principally referring to Thermometric Scales, Hydrometer Degrees, and Specific Gravities of Acids, Common Salt Solutions, and Alcohol.

FERMENTS AND THEIR ACTIONS. By CARL OPPENHEIMER, M.D., Ph.D. Translated by C. A. MITCHELL, B.A. Chas. Griffin and Co., Ltd., Exeter Street, Strand. 1901. Price 7*s*. 6*d*. net.

THIS work, commencing with author's and translator's prefaces, and a table of contents, fills 304 pages. Then follows a systematic bibliography of works, treatises, and papers bearing upon the general subject. These are classified as follows :—I. Text-books. II. Ferments and Fermentations in general. Nature of Ferments, &c. III. Mode of Action of Ferments, &c. IV. Physiological Action. V. Distribution. VI. Digestion and Proteolytic Ferments generally. VII. Pepsin. VIII. Trypsin. IX. Decomposition of Proteïds. X. Papaïn. XI. Rennet. XII. Diastase. XIII. Cytase, &c. XIV. Ferments of Disaccharides. XV. Ferments decomposing Glucosides. XVI. Other Hydrolytic Ferments. XVII. Lactic Acid Fermentation. XVIII. Alcoholic Fermentation. XIX. Oxydases. XX. Oxidising Fermentations. XXI. Miscellaneous References.

After this follows a list of abbreviations and the alphabetical indexes of subjects and authors. The work itself is subdivided into General and Special Parts. THE GENERAL PART, after its introduction, runs the following course. I. Definition of a Ferment. II. Chemical Nature. III. Influence of External Factors on Ferments. IV. Mode of Action of Ferments. V. Physiological Action. VI. Secretion of Ferments. VII. Importance of Ferments to the Vital Process. SPECIAL PART. A. THE HYDROLYTIC FERMENTS. B. The Oxidising Ferments.

DIE BRENNSTOFFE DEUTSCHLANDS UND DER ÜBRIGEN LÄNDER DER ERDE, UND DIE KOHLENNOTH. Von Dr. FERD. FISCHER, Professor an der Universität Göttingen. Friedrich Vieweg und Sohn, Braunschweig. 1901. Price M. 3.00.

8vo volume, containing preface, table of contents, and subject-matter filling 107 pages. The subject of this work is treated under three principal heads. I. The Fuels of Germany. II. Demand for Coal in the remaining Countries of the World. III. The Coal Famine. Germany is described as the richest country of Europe in coal, the demand during the last year, 1900, representing a money value of over one milliard of marks. England, it is said, in fifty years, will be threatened with a partial exhaustion of coal supply. As fuel, Germany at present, it is said, counts upon wood and peat as well as coal, and the author strongly advocates scientific economy, in order that these indispensable treasures may be extended as far as possible to future generations.

DIE NORMALELEMENTE UND IHRE ANWENDUNG IN DER ELEKTRISCHEN MESSTECHNIK. Von Dr. W. JAEGER, Kaiserl. Professor, Mitglied der Phys.-Tech. Reichsanst. in Charlottenburg. William Knapp, Halle a. S. 1902. Price M. 6.

FOLLOWING the preface and table of contents are 117 pages of text, illustrated with 38 wood engravings. Then follow an appendix with tables for the Electromotive Power of the Clark's and the Cadmium Element in int. Volt, between 10° and 30° C., a bibliographic list, and finally the alphabetical indexes of authors and subject-matter. The work is divided into four parts. I. Definition and

Arrangement of the Normal Elements. II. Theory of the Normal Elements. III. Description, &c. of the Elements individually. IV. Appropriation as to Standard and Employment of the Normal Elements.

Prospecting for Gold. A Handbook of Practical Information and Hints for Prospectors, based on Personal Experience. By Daniel J. Rankin, formerly Manager of The Central African Company and Leader of African Gold Prospecting Expeditions. Crosby Lockwood and Son, 7, Stationers' Hall Court, Ludgate Hill, London. 1901. Price 7*s.* 6*d.*

Small volume arranged as a pocket-book. A frontispiece illustrates the Prospectors' Camp Cabinet, designed by the author. The aim of the compiler, it is stated, is to furnish the prospector, in a portable form, with information, all-important in his quest for the gold, but hitherto spread through a number of volumes, the bulk of which makes their carriage in such expeditions all but impracticable. After the preface and table of contents follow 178 pages of subject-matter and the alphabetical index. The subject-matter itself is subdivided as follows:—I. Kinds of Rocks. II. Condition, Structure, and Arrangement of Rock Masses. III. Minerals associated with Gold. IV. Gold in Africa, Banket Formation. V. Methods for the Preliminary Determination of Gold in Quartz and Ores. VI. Prospecting for Alluvial Gold or Placer Deposits. VII. Prospecting for Auriferous Veins and Rocks. VIII. Tables of Characteristics of Mineral Ores, also of Blowpipe Analyses of Ores. Addenda. Darton's Test for Gold. Gold Assay by the Touchstone. Estimation of Gold in Alloys.

Chemisch-Technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 40. Jahrgang. 1901. Erstes Halbjahr. Erste Hälfte. R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder, Schönebergerstr. 26, Berlin, S.W. 1902.

This is the number of the "Repertorium" for the first half of the first half-year of 1901, and it treats of the following subjects of chemical technology:—I. Building Materials. II. Dyestuffs, Dyeing, and Printing. III. Fats, Oils and Illuminants, and Fuels. IV. Fermented Liquors. V. Tanning, Leather, and Glue. VI. Textiles. VII. Glass and Earthenware. VIII. Wood and Horn. IX. India-rubber and Gutta-percha. X. Cements and Adhesives, &c. XI. Spirit and Oil Varnishes, and Paints. XII. Metals.

Subject List of Works on certain Chemical Industries, including Destructive Distillation, Mineral Oils and Waxes, Gas Lighting, Acetylene; Oils, Fats, Soaps, Candles, and Perfumery; Paints, Varnishes, Gums, Resins; Paper, and Leather Industries,—in the Library of the Patent Office. Patent Office Library Series No. 7. Bibliographical Series No. 4. Printed for His Majesty's Stationery Office, by Darling and Son, Ltd., 34–40, Bacon Street, London, E. Published at the Patent Office, 25, Southampton Buildings, Chancery Lane, London, W.C. 1901. Price 6*d.*

Introduction to the Study of Chemical Philosophy. The Principles of Theoretical and Systematic Chemistry. By William A. Tilden, D.Sc., F.R.S., Professor of Chemistry in the Royal College of Science, London. 10th edition, completely revised and re-arranged, with answers to problems. Longmans, Green, and Co., 39, Paternoster Row, London. Also New York and Bombay. 1901. Price, 5*s.* 6*d.*, or without answers, 5*s.*

Small 8vo volume containing prefaces, table of contents, and 359 pages of subject-matter, 3 pages of exercises, index of names and subjects, and 24 pages of answers to problems. The work is subdivided into three sections:—I. Chemical Combination and Chemical Compounds. II. Classification (Elements, Metalloids, Metals, The Periodic System, and Classification of Compounds). III. Chemical Kinetics (Conditions of Chemical Change, Thermal Effects of Chemical Action, Heat of Combination and Combustion, Electro-Chemical Decomposition, &c., Solution).

Trade Report.

I.—GENERAL.

Patent Law Amendment.

Standard, Dec. 14, 1901.

Sir Herbert Jekyll, replying to the Memorial of the Association of Chambers of Commerce on the subject of the Amendment of the Patent Laws, says:—

"I am directed by the Board of Trade to advert to your letter of the 16th October, addressed to the President, transmitting a copy of resolutions adopted by the Association of Chambers of Commerce of the United Kingdom, at its recent conference at Nottingham, in favour of the Amendment of the Patent Law in certain particulars, and in reply, I am to say that Mr. Gerald Balfour has given instructions for the drafting of a Bill to give effect to the recommendations of Sir Edward Fry's Committee, and that His Majesty's Government will consider in due course whether the Bill shall be introduced next Session."

The Council of the Association have resolved to apply to the President of the Board of Trade to receive a deputation in support of the resolution adopted at the autumnal meeting.

Patent Law Reform.

Chem. Trade J., Dec. 14, 1901.

At a meeting ot the Board of Directors of the Manchester Chamber of Commerce, on the 11th inst., it was intimated that the Council of the Association of Chambers of Commerce had decided to arrange for a deputation to the President of the Board of Trade, for the purpose of urging the importance of introducing, in the next session of Parliament, a Bill for the reform of the Patents Act, 1883, on the lines of the resolution passed by the representative conference held in Manchester last June, and afterwards adopted by a general meeting of the Association. It is expected that the deputation will include delegates, not only of various Chambers of Commerce, but also of several powerful bodies specially representing the leading industries of the country.

Mineral Production of India.

Imp. Inst. J., Dec. 1901.

The production of petroleum in Burma has rapidly advanced, being nearly twice as great, in 1899 and 1900, as in 1897 and 1898. In 1900, Burma yielded 36,974,000 galls., and Assam, 753,000 galls. In spite of the large increase in output, the supply is insufficient for the needs of the Indian market, and two-thirds of the requirements have to be imported. Saltpetre is largely produced for export, especially in Behar, though it is less in demand than formerly, owing to a decline in its use for gunpowder, food preservation, and manure. The shipments from Calcutta, in the last five years, have averaged about 20,000 tons. Madras exports, mainly to England, a considerable quantity of manganese ore. The extraction of mica has gone on for many years in Bengal, and this mineral has recently been obtained in increasing quantities from Madras. Tin mining is carried on by Chinese in Lower Burma.

II.—FUEL, Etc.

Petroleum in Canada.

Chem. Trade J., Nov. 23, 1901.

According to a report of the Ontario Bureau of Mines, there was a slight decrease in the output of crude petroleum in 1900, as compared with 1899, the figures being 23,381,783 imperial galls., in 1900, as against 23,615,967 galls., in 1899. The total value of the substances refined from the oil was considerably larger, being 1,869,045 dols., as compared with 1,747,352 dols. The following table gives details :—

——	1899.	1900.
	Dols.	Dols.
Illuminating oil	1,059,485	1,076,242
Lubricating oil	189,294	232,805
Benzine and naphtha............	148,963	174,346
Gas and fuel oils and tar.........	213,544	200,934
Paraffin wax and candles	136,066	184,718
Total	1,747,352	1,869,045

There was a diminution in the quantity of gas and fuel oils, as compared with 1899, and an increase in the production of paraffin wax and candles. The falling off in gas oils is due to the fact that crude oil is now used instead of gas oil in the making of gas. For a long time crude oil could not be employed to advantage for this purpose, because too much free carbon was generated in the retorts, ultimately choking up the mains, and blackening the ceilings and walls of rooms. This difficulty has now been overcome, and gas producers, on equal terms, now prefer crude, to the product of crude, gas oil.

Gas and Oil Industry in Germany.

J. f. Gasbeleucht. 1901, **44**, [41], 751—755.

The German Association of Gas and Water Managers addressed a memorial, on Dec. 31, 1900, to the Federal Council of Germany, praying for the importation of duty-free mineral oil for the manufacture of carburetted water-gas. The memorial drew attention to the fact that the Council was empowered, under par. 1 of No. 29 of the Customs laws, to allow mineral oil, destined for industrial uses, other than the manufacture of lubricating or burning oil, to pass free of import duty. It then referred to the recent increase in the cost of gas coal, and the consequent desirability of processes for the manufacture of illuminating gas, other than the coal-gas process, being available for adoption in gasworks. Among such processes, that of carburetted water-gas manufacture has been adopted in America, England, and Belgium, where the mineral oil residues required are obtainable duty-free, and at a low price. In Germany the existing scale of Customs duties imposes an import duty of 6 or 10 marks per 100 kilos., according to the specific gravity, on such oil residues, and as there is no good native substitute for them, the manufacture of carburetted water-gas is thereby precluded. The residues, or oils suitable for that manufacture, are by-products in the production of burning oil from crude petroleum, and hence are produced chiefly in America and Russia, though also in Galicia and Roumania. Of late years, large quantities have passed through Germany from Galicia, for use in carburetted water-gas manufacture in Belgium. The high Customs duty, however, prevents their use in Germany. The percentage of residues or heavy oils obtained from crude petroleum varies with its origin, and is, for the chief varieties, as follows :—Pennsylvania, 10—20; Ohio, 35—50; Baku, 36—60; Galician, 30—45; and Roumanian, 25—35. Great quantities are consumed as fuel, and some is worked up in the manufacture of lubricating oil and paraffin. In America, however, so much is used in making carburetted water-gas, that many towns are supplied entirely with that gas instead of coal-gas. In Europe, especially England, great quantities of American and Russian oil residues are now used in carburetted water-gas manufacture, but the residues from the Galician and Roumanian distilleries, which could more readily be brought to Germany, are at present used as fuel at the place of production. There is an annual output of about 26,000 metric tons of petroleum from Alsace and Hanover, but there seems no prospect of this being increased, or of there being other supplies of native German petroleum. Residues, or gas oils, amounting to about 12,000 metric tons annually, are produced in the manufacture of burning oil, &c., from lignite, in Saxony-Thuringia and at Messel, but these are almost wholly used for making oil-gas for carriage lighting. Notwithstanding such unfavourable conditions for the production of carburetted water-gas, the process has been adopted recently at Hamburg, Bremen, and Posen, but its further adoption in Germany is precluded by the inadequate supply of native oils, and the high rate of duty on imported oils. At Königsberg and Erfurt, owing to the difficulty of obtaining oil residues, water-gas has been carburetted by the addition of vapour of benzol, but such gas can only be used in limited proportion, in admixture with coal-gas, owing to the tendency of the benzol to condense, and cause stoppages in the supply pipes in the cold season, which is the time when there is the greatest demand for gas.

The memorial then points out that the interests of the lamp-oil traders and of the home petroleum industry would not be prejudicially affected by the removal of the Customs duty on oil residues destined for use in the manufacture of carburetted water-gas, and that any other use of the residues exempted from duty could readily be prevented. The residues might be defined, in order to avoid confusion with burning and lubricating oils, as oils having a specific gravity at 15° C. exceeding 0·830, and a flashing point above 70° C. The memorial prayed that such oils might be allowed to pass duty-free, when destined for use in public gas works in Germany.

Oil gas is applied universally for lighting railway carriages, lighthouses, beacons, and buoys, but a more extended use of it in Germany is hindered by the Customs duty on imported mineral oil. This duty, which ostensibly protects the small group of oil-producers in Germany, amounts to 6 marks on all mineral oil except lubricating oil, on which it is 10 m. per 100 kilos., including the weight of the barrels. If imported in bulk, 25 per cent. is added to the duty, calculated, according to the above scale, on the net weight of oil, as an allowance for the weight of barrels. Oil of higher specific gravity than 0·830 is classed as lubricating oil, and as the specific gravity of the cheap gas oils exceeds this limit, they are subjected to the higher duty. Hence, whereas petroleum residues suitable for gas making could be bought for about 3½ m. per 100 kilos. in 1898, the import duty amounted to 12½ m., or three to four times their value. In 1898, the German gas oils cost 8 m., but they now sell for 12 to 14 m., and are inferior for gas making to the foreign petroleum residues, which are now worth 4 to 5 m. per 100 kilos. exclusive of duty.

The German State railways use about 12,000 metric tons of gas oil per annum for making oil gas, and there is no doubt that they now pay for it 7 to 8 m. per 100 kilos. more than they would if the foreign petroleum residues were admitted duty-free, and so competed on equal terms with the home products. The recent rise in the price of oil has almost ruined German oil-gasworks, which supply gas under contract, as, though the foreign oils have risen only 1½ m. in value, the German oils, owing to the protective duty, have risen at least 8 m. per 100 kilos.

Actually, burning oil, paying the lower import duty of 7½ m. per 100 kilos. *net*, is obtainable in Germany for 16 m., at which price it comes into competition, for gas making, with the intrinsically less valuable gas oils of home production, and the foreign petroleum residues, which pay the higher rate of duty. A moderate duty of 2 to 3 m. on these residues might be justified, as a protection of the home petroleum industry, but as the other products of that industry fetch good prices, it seems unnecessary to protect the gas oil by any duty on imported residues. The German oil works are earning good dividends, while the oil-gas works, in which fully as much capital is invested and more men are employed, are hampered by the duty on imported gas oil, and are falling behind similar works in other countries. Moreover, cheap oil is urgently required for the manufacture of carburetted water-gas (*c.f.* preceding abstract). Practically none of the oil, classed as lubricating oil, at present imported into Germany, is used for gas making, consequently the removal of the import duty on oils imported for that purpose would not prejudicially affect the Customs returns under that heading.—J. A. B.

III.—TAR PRODUCTS, PETROLEUM, Etc.

Coal-Tar Pitch in Germany.

U.S. Cons. Reps., Dec. 4, 1901.

The James Stevenson Aktien Gesellschaft, of Stettin, wish to buy coal-tar pitch. The article must be almost pure, not to exceed one-half of 1 per cent. of ash. The present supplies of the firm come from Great Britain, and the cost is about 40*s.* (9·72 dols.) per ton, c.i.f. Stettin.

IV.—COLOURING MATTERS, Etc.

United States Customs Decision.

Bd. of Trade J., Nov. 28, 1901.

The following dyes are free of duty, under par. 469 of the Tariff, as " alizarin (natural or artificial) and dyes derived from alizarin or from anthracene " :—

Alizarin grey
,, ,, F F
,, blue B R 3 G
,, ,, H R
,, ,, S A P
,, ,, S A C
,, ,, S A E

Alizarin blue S A F
,, fast black
,, ,, S P
,, black B
,, ,, 1 A
,, ,, 3 B

The following are dutiable at the rate of 20 per cent. *ad val.*, under par. 15 of the Tariff, as products of coal tar, not colours or dyes :—" Binitrotoluol, reduced," and " sodium salt of amido naphthol sulpho acid."

The following are dutiable at the rate of 30 per cent. *ad val.*, under par. 15 of the Tariff, as " coal-tar dyes or colours, not specially provided for :—

Alizarin brown G
,, ,, W
,, ,, R
,, blue G C
,, ,, S G R
,, ,, S 5 R
,, ,, 5 R
,, ,, B A R
,, ,, S 3 R
,, ,, C B B
,, black (Bayer) 1 B
,, ,, S G
,, ,, 2 B

Alizarin black V B
,, ,, N V B L
,, ,, F
,, ,, G A
,, yellow
,, ,, A
,, ,, C D
,, ,, C R X
,, ,, 3 G P
,, green
,, ,, S S
,, ,, P extra

VII.—ACIDS, ALKALIS, AND SALTS.

Carbonate of Baryta, precipitated.— U.S. Customs Decision.

Bd. of Trade J., Dec. 12, 1901.

The exemption of carbonate of baryta from duty, under par. 489 of the Tariff, extends only to the natural mineral product known as " witherite." The artificial product, known as "carbonate of baryta, precipitated," is dutiable as a chemical salt or compound, under par. 3 of the Tariff, at the rate of 25 per cent. *ad val.*

Sulphur in New Zealand.

Eng. and Mining J., Dec. 7, 1901.

The New Zealand Mines Department reports the output of sulphur in the colony, in 1900, at 1,692 tons, an increase of 465 tons over the previous year.

VIII.—GLASS, POTTERY, AND ENAMELS.

Lead Poisoning in the Potteries: Home Office Arbitration.

Chem. Trade J., Nov. 23, 1901.

The arbitration between the Home Office and the manufacturers on the proposed new special rules for the pottery trade, which was opened on Nov. 7, at Stoke, was concluded on Nov. 12. The substance of the decision of the umpire (Lord James of Hereford) is as follows:—

Lord James said that the State had taken upon itself the duty of interference with trades that were dangerous to health and destructive of life, and it was in the performance of that very important duty that these rules had been framed. On the other hand, the Home Office recognised that interference with a trade which was a trade of the country, and which affected the community as a whole, should not take place if it could be avoided. Bearing these two principles in view, as this arbitration had proceeded, it seemed to him that the decision, if not well considered, might effect an injury which possibly could be avoided.

The witnesses for the Home Office had admitted that the most important rules—Nos. 1 and 2—which dealt with fritting and the 2 per cent. of soluble lead, would require considerable modification, and that their terms would have to be remodelled before they could be accepted. The employers admitted that they had undertaken, in regard to the use of frit, a burden which they now said they could not bear. Endeavouring to protect the interests of all parties, the practical course he suggested was, that the substance of the rules should come into effect in a reasonable time, with the exception of Nos. 1, 2, and 6. With respect to these, dealing respectively with the exclusive use of fritted lead, with the maximum of 2 per cent. of soluble lead, and with the monthly medical examination of adult males, since the rules of 1898 came into force, great progress had been made in diminishing the evil. The percentage of lead-poisoning cases had been reduced from 12 to 3·5. Experiments had been made by the employers in order to see how they could lessen the amount of lead, and the evil effects of it, in their manufactures, and those experiments were still proceeding. If nothing were done, the diminution of the evil would be certain and progressive, and when the new rules, as now revised, came into existence, that natural progress would continue. What he now proposed was, not to let that arbitration go on at present. He proposed to adjourn it, as regarded rules 1, 2, and 6, for 18 months, with power then, if necessary, still further to adjourn it. The result would be that those three rules would not come into operation for over 18 months at least. He appealed to the employers first, in the interests of themselves—who must look forward to the dealing with these rules in 18 months' time—to do all that they could in the matter. They could do a great deal more by extending the system of fans, making more general those water cisterns into which dust dropped; and some of the older factories, by better flooring and better cleansing of the floors; the use of overalls, &c., should be general, and they should always be kept in a cleanly state. He appealed to the manufacturers also to see whether they could not reduce the quantity of soluble lead used. He congratulated them that they could make goods of such a quality that they could compete with a 60 per cent. tariff, but he asked them also to look to their foreign brethren, and see if they could not learn something from them in regard to lessening the dangers of the trade. He would recommend the Home Office to consider whether a rule could not be made, which should provide that, upon application made by any employer, showing that the lead used in his factory had been reduced, say, to 2 per cent. of solubility, the Home Office should exempt such manufacturer from other rules which seemed to press hardly upon him. If, by any system they could reduce the lead used to a low test of solubility, then the necessity of the prohibitive rules would be obviated.

With regard to the operatives, he also appealed to them. There were two classes of them, the adult males, and those who could not protect themselves so much—the women and young people. He asked the former not to think so much of themselves, but to think of others in the factory, and to do all that they could in every way to see that a state of things existed that was conducive to long life. It would be a very great advantage if some arrangements could be come to between the two, by which the employer would take upon himself the burden of the Compensation Act, in regard to sickness brought on by poisonous ingredients used in the trade. That must be a matter of agreement. Every employer would then desire that his factory should be in such a condition, that no compensation would have to be paid. He hoped that the course he had taken would, first, in the cause of humanity, be sufficient; and, secondly, would not impose the least burden upon a trade which, he thought, was conducted with credit to the country. (See also this Journal, 1901, 475.)

China Clay in the Province of Florence (Italy).

Foreign Office Miscellaneous Series, No. 570.

In the china factories of this province, the principal material employed is china clay, imported from the United Kingdom, and then Italian kaolin, pegmatites, felspars, quartzes, and other silicious minerals and earths. The Italian clays have hitherto proved inferior to the French (Bollène and St. Yrieix), and especially to the British kaolins (Cornwall). In Sardinia, numerous lodes of china clay are said to exist, but the clay obtained near the surface is impure. If the lodes were dug to some depth, better results might be obtained.

IX.—BUILDING MATERIALS, Etc.

Cement: Valuation Tariff in Egypt.

Bd. of Trade J., Nov. 20, 1901.

The Egyptian *Journal Officiel* for the 16th November, contains a Valuation Tariff* in use by the Egyptian Customs Authorities, for the purpose of assessing duties on cement. This Tariff, which was to come into operation on the 13th inst., and to remain in force for 12 months, or until denunciation, is as follows:—

Description.		Valuation † per 1,000 kilos.
		£ E‡
Artificial Portland cement, English ...	in barrels	2·250
	in bags ..	2·000
Artificial Portland cement, all other kinds..............................	in barrels	1·870
	in bags ..	1·620
Natural cement of all descriptions....	in barrels	1·625
	in bags ..	1·375
"Valentin Roquefort" and "Gris Bedoule" cement	in barrels	1·350
	in bags ..	1·100

* For particulars as to the nature of thes Egyptian Valuation Tariffs, see *Board of Trade Journal* for 28th February last, p. 517.

† Duty is levied on these valuations at the rate of 8 per cent. *ad val.*

‡ £E = 1*l.* 0*s.* 6*d.*

Mica Scrap: U.S. Customs Decision.

Eng. and Mining J., Dec. 7, 1901.

Small pieces of mica, which fall off in the process of thumb-trimming mica, do not constitute waste, but being still mica, used as such, and not having lost their character as merchantable mica, are dutiable under par. 184, Act of July 24, 1897. Such merchandise is not known as mica waste, nor is there any trade understanding or meaning of the term "mica waste," and it is, therefore, not dutiable, under provisions of par. 463, for waste not otherwise provided for.

X.—METALLURGY.

Lead Mining in England.

Chem. Trade J., 23 *Nov.* 1901.

At a meeting of the Manchester Geological Society, Mr. J. Dickinson read a paper, in which were given particulars of the lead mining districts of the North of England and Derbyshire. To show the change that has come over lead mining, the output of lead ore in Derbyshire, in 1858, was 6,277 tons. In 1878, it diminished to 2,090 tons, from which figures it had somewhat revived (mainly by the output of one mine), the output, in 1900, being 4,395 tons. In the four northern counties, in 1900, the production of lead ore was 7,176 tons. Of this, Cumberland produced 1,321, Durham 3,849, Northumberland 520, and Westmoreland 1,486, tons. All these figures indicate a great falling off, as compared with the output in olden times. In the three years ended 1768, the average annual output of the Alston Moor mines alone was 8,244 tons, being more than six times the present production of the whole of Cumberland, and considerably more than the present production of the whole of the four counties. The diminution had been general throughout the whole of the United Kingdom. According to the reports of His Majesty's inspectors of mines, the total production of lead ore, in 1900, was only 32,010 tons, whilst, during the ten years ended 1882, it averaged yearly 73,357 tons. Had the diminished production been confined to isolated places, it might be attributed to local mismanagement, but being national, except at a few favoured mines, it must apparently be set down to causes over which this country had no control. One of the main causes seemed to be the import at low prices from foreign silver and other mines, where lead was a residual product.

Platinum in Canada.

Eng. and Mining J., Nov. 30, 1901.

The *Bulletin* of the Ontario Government Assay Office says: "The commercial use of metallic platinum is being so widely extended at present, and the consumption is increasing at such a rate, that ingot platinum is now quoted at 21 dols. per ounce, platinum in the ore being worth about 17 dols. per ounce, according to assay value. The value of platinum is not likely soon to decrease; hence any ore carrying platinum capable of being extracted commercially is in constant demand. The rise in value of platinum is caused by increased consumption in photographical work, electric lighting, incandescent lighting mantles heated by burning gas, instrument-making, and various apparatus made entirely or in part of platinum, and other uses in the arts.

"Platinum is principally found in the native form, occurring generally in alluvial sands, associated with garnets, magnetite, topaz, corundum, zircons, native gold, and other heavy minerals. Platinum metals are found as small grains or scales of a tin-white colour when cleaned with nitric acid. The scales are generally too fine to catch the eye, and collect with a heavy fine sand, as streaks of brown or lead-coloured particles, which are very heavy, and can be concentrated by washing with running water or by panning. The ordinary riffles in a gold-saving sluiceway will not save all of the platinum metals to be found in alluvial sand, as some of the values creep over and are lost. Nor will the platinum metals amalgamate with mercury without special treatment. There are several satisfactory methods of concentrating alluvial sands so as to save the platinum metals.

"Platinum occurs in the Sudbury mining district as sperrylite, which is a combination of platinum and arsenic found in the gossan of the Vermilion nickel mine. The matte formed by smelting the nickel-copper bearing pyrrhotites of the Sudbury district contains platinum, which is saved in the process of refining the metallic contents. It is possible that closer search of placer sands will result in finding more of this valuable metal in paying quantities for working by hydraulic methods."

Lead and Zinc in Austria.

Eng. and Mining J., Nov. 16, 1901.

In 1900, the Austrian mines produced 14,314 metric tons of lead ore, of which 10,904 tons were from the mines of Carinthia. As compared with 1899, there was an increase of 735 tons. The output of metallic lead was 10,650 tons, an increase of 914 tons over 1899. In addition to this, a production of 1,288 tons of litharge is reported, being 239 tons less than in 1899.

The production of zinc ore, during 1900, amounted to 38,243 metric tons, an increase of 1,142 tons over 1899. Of this ore, 21,421 tons were mined in Carinthia. The production of metallic zinc was 6,742 tons, or 450 tons less than in 1899. Of the zinc produced in 1900, Galicia turned out the largest proportion, 3,365 tons; whilst the works in Steiermark turned out 2,958 tons.

Chrome, Coal, Silver-Lead, and Antimony Mines in Salonica and Kossova (Turkey).

Bd. of Trade J., Nov. 28, 1901.

The British Vice-Consul at Uskub states that the chrome and coal mines at Elleshan, the first station on the Uskub

Mitrovitza line, and the silver-lead and antimony mines, close to Uskub, are in the market.

The coal mine at Elleshan, if really as rich as it is believed to be, would prove a valuable investment, with no greater attendant risks in Turkey than in any other country.

The chrome mine of Elleshan, and Junuz Bey's silver-lead and antimony mines are also considered of great value, owing to their comparative proximity to Uskub and Elleshan, and their situation in a more or less civilised part of the country. The chrome is said to exist in enormous blocks. Some years ago, the mine was energetically worked and the output was very large, but only a small percentage of the ore which was discovered could be extracted owing to the sudden inrush of a stream which would need diverting from its actual channel, and the cost, though insignificant, was more than the owner could bear.

In the case of both these mines, capital is, according to report, alone required to open up a great store of wealth.

MANGANESE ORE IN ITALY.

Eng. and Mining J., Nov. 16, 1901.

In 1900, the production of manganese ore in Italy was 6,014 tons, against 4,356 tons, in 1899, showing an increase of 1,658 tons last year.

TIN IN ALASKA.

Bd. of Trade J., May 12, 1901.

According to the *British Columbia Review* of 30th Nov., a deposit of oxide of tin, containing $78\frac{1}{4}$ per cent. of metallic tin, has been discovered near York, in Alaska. It is an alluvial deposit and looks like magnetic iron, except that it is whiter. Miners can easily detect it, as the iron minerals make a red powder, whilst the tin mineral makes a white powder.

The discoverers report that they got 150 to 200 lbs. of the ore to the cubic yard. It can be saved by very little extra trouble, as it exists in auriferous gravel, and accumulates in the sluice boxes.

XII.—FATS, OILS, Etc.

EARTH-NUTS, GUM, AND RUBBER IN SENEGAL (W. AFRICA).

Foreign Office Annual Series, No. 2725.

The exports for 1900 are valued at 1,317,285*l.*, being 375,428*l.* in excess of the previous year. The increase is mainly due to the large output of earth-nuts, of which 138,708 tons, valued at 969,612*l.* were shipped to Europe.

The exports were distributed among the various countries as follows:—France received earth-nuts, gum, rubber, &c.; Holland took earth-nuts, 85,318*l.*; Germany, earth-nuts, 55,218*l.*; Belgium, earth-nuts, 24,134*l.*; gum, 373*l.*; rubber, 4,365*l.*; the United Kingdom, earth-nuts, 1,212*l.*; gum, 385*l.*; rubber, 6,476*l.*

Earth-nuts, the chief product of the country, are grown largely between Dakar and St. Louis. Their cultivation is simple. At the commencement of the rainy season, about June, the seed is planted, and by November or December the crop is fully matured. Rubber, growing in three varieties, viz., the *Ficus Vogelii*, the *Landolphia Fomentosa*, and the *Ceara* tree, is receiving the attention of the Government, as it is regarded as the future resource of the colony in the event of the failure of the earth-nut trade. With this view many plantations have been laid out at Casamance, and in the region of Mayes, near the coast, between Dakar and St. Louis, where the soil is most favourable. Producers are encouraged to prepare only the best qualities for the market.

SOAP IN THE PROVINCE OF FLORENCE (ITALY).

Foreign Office Miscel. Series, No. 570.

The Soap and Perfumery Company (*Industria dei Saponi e Profumi*) has, in the commune of Sesto Fiorentino, an important factory, employing 87 hands. The motive-power is supplied by two steam engines of 20 h.-p.

Another factory of scented soaps, employing eight workmen, is situated at Castello, in the commune of Sesto Fiorentino. There are also five small factories of common soap there, and two employing 64 hands, in the commune of Florence.

XIII. A.—PIGMENTS, PAINTS, Etc.

CRUDE COLOUR, UNWROUGHT EARTH: U.S. CUSTOMS DECISION.

Bd. of Trade J., Nov. 28, 1901.

An unwrought earth which is used as a colour is dutiable at the rate of 30 per cent. *ad val.*, under par. 58 of the Tariff, as a "colour not specially provided for," and not under par. 93, as an unwrought and unmanufactured earth.

XIII. B.—RESINS, VARNISHES, Etc.

XANTHORRHŒA RESIN IN GERMANY.

Färber-Zeit.; through Imp. Inst. J., Dec. 1901.

The resin occurs in two forms, the yellow and red varieties; both are employed in Germany in the preparation of spirit lacquers for coating metals, and their potash and soda solutions for sizing paper.

The red xanthorrhœa resin is used as a substitute for dragon's blood, and the yellow variety, instead of gamboge, for colouring shellac varnishes. Solutions of the resin in alcohol have also replaced the ordinary gold lacquer used for painting the brass parts of scientific instruments, and possess the advantage of not bleaching, even after long exposure to light. A concentrated xanthorrhœa varnish, mixed with a little castor oil or copaiba balsam, to prevent cracking, when painted on glass, leaves a perfectly transparent, slightly yellow coating; by appropriate staining this can be changed to a fine ruby colour, and glass so tinted has been employed for the manufacture of cheap photographic red-light lanterns. In combination with copal and shellac, these resins form excellent wood varnishes, which leave a lustrous transparent coating. Xanthorrhœa has the further advantage of dissolving easily in spirits, and such solutions clear themselves rapidly by deposition; further, they can be applied to metals without previous warming of the surface to be varnished. (See also this Journal, 1898, 163).

SCAMMONY RESIN: U.S. CUSTOMS DECISION.

Bd. of Trade J., Nov. 28, 1901.

Scammony resin, an article prepared from gum scammony or scammony root, and used principally in the compounding of medicinal preparations, and not as a medicine, is dutiable as a drug advanced in value or condition, under par. 20 of the Tariff, at the rate of $\frac{1}{4}$ cent per lb., and 10 per cent. *ad val.*

GUM AND RUBBER IN SENEGAL (W. AFRICA).

Foreign Office Annual Series, No. 2725.

See under Class XII. above.

XIV.—TANNING; LEATHER; GLUE, Etc.

TANNERIES IN THE PROVINCE OF FLORENCE (ITALY).

Foreign Office Miscellaneous Series, No. 570.

The tanneries number 86, seven of which are furnished with machinery. The good quality of the water, the large quantities of skins on the spot, the abundance of tanning material, and the good quality of the leather obtained, have been the cause of the increase in the number of tanneries of late years. The production of leather is therefore considerable, and as it chiefly consists of strong sole leather from oxen, calves, and cows' skins for which a short preparation is required, even tanners of small means may yield a considerable production. The total number of hands employed in the tanneries of the province of Florence is 1,026.

XVI.—SUGAR, STARCH, Etc.

Dutch Sugars: Countervailing Duties on:—U.S. Judicial Decision.

Bd. of Trade J., Nov. 28, 1901.

With reference to the notice which appeared on p. 24 of the *Board of Trade Journal* for the 4th April last, relative to the countervailing duties leviable in the United States on bounty-fed sugars produced in the Netherlands, the Board of Trade are now in receipt of information to the effect that the payment of these duties has been disputed by certain importers, on the ground that Holland does not grant bounties on sugar. This question having been brought before the Circuit Court of Appeals, it has now been judicially decided as follows:—

"A so-called 'deduction,' which concededly is a bounty or grant, or premium, is paid" (in the Netherlands) "for the production of raw sugar, of 2·50 florins per 100 kilogs., for the year in question, and for the production of refined sugar therefrom, a further bounty of 0·34 florins per 100 kilogs., a total of 2·84 florins per 100 kilogs. The amount of such premium or bounty is placed to the credit of the person to whom it is due, in his excise account; and it is provided that if it 'should cause the credit to exceed the debit, the difference shall be paid to the manufacturer or refiner from the revenue from the excise of the year of which the deduction takes place.' In view of the great disparity between the bounty and the excise tax (there is a similar disparity for all kinds and grades of sugar), it is quite evident that there can be no excess 'paid' from the Government's revenues to any one who is liable for the excise tax. Finally, it is provided that sugar withdrawn for exportation to a foreign country, and actually exported, shall be exempt from the excise tax. That tax being thus eliminated from the debit side of the account, the manufacturer or refiner receives from the Government the excess of credit over debit, which is the precise amount of the bounty. Undoubtedly, this premium or 'deduction' is called a bounty on production, and is a bounty on production; but the other provisions of the law have the practical effect of making it, from the standpoint of other countries, a bounty on exportation. The result of the whole Act is no different, so far as the foreign country is concerned, from what it would be had it provided: All sugar producers shall receive a bounty paid in cash from the revenues of the Government of so much per 100 kilogs. Those who export their sugar may keep this bounty, those who do not export it must forthwith return it to the Government."

Countervailing duties will consequently continue to be levied on Dutch sugars, in accordance with the provisions of section 5 of the United States Tariff Act of 1897.

XVII.—BREWING, Etc.

Sucramine in Beer: Official Prohibition.

London Gazette, Dec. 7, 1901.

Prohibition, under Section 5 of "The Customs and Inland Revenue Act, 1898," of the use in Beer of a certain substance.

Whereas it appears to the satisfaction of the Lords Commissioners of His Majesty's Treasury that the chemical or artificial product, commonly called or known by the name of "Sucramine," is capable of being used in the manufacture and preparation for sale of beer, and that the use of the said product is calculated to affect prejudicially the interests of the Revenue.

Now, the said Lords Commissioners, under the power conferred upon them by Section 5 of "The Customs and Inland Revenue Act, 1888." do hereby prohibit the use of "Sucramine" in the manufacture and preparation for sale of beer.

Dated this 7th day of December, 1901.

N.B.—A penalty of 50*l.* is imposed by the said Section for any breach of this prohibition.

W. H. Fisher.
H. T. Anstruther.

(See also this Journal 1901, 1046.)

Herbs in Alcohol: U.S. Customs Decision.

Bd. of Trade J., Dec. 12, 1901.

Herbs in alcohol, being articles manufactured by uniting or combining two elements or ingredients, are essentially compounds, and, as they contain alcohol, are dutiable at the rate of 60 cents per lb., and 45 per cent. *ad val.*, under par. 2 of the Tariff. Alcoholic extracts of herbs are dutiable at the same rate.

XIX.—PAPER, PASTEBOARD, Etc.

Paper Industry of the Province of Florence (Italy).

Foreign Office Miscellaneous Series, No. 570.

There are seven paper mills at present working in the province of Florence, five being situated in the commune of Pistoria, and one in each of the communes of Reggello and S. Marcello.

A mill, at Gello, is fitted with a steam boiler of 30 h.p., two hydraulic and one steam engine, of a total of 60 h.p. and 122 hands.

The extensive mills, known as the "Cartiere della Lima" are situated in the valley of the Lima. By using the water of the torrent of the Lima, these mills have a constant motive power of 400 hydraulic horse-power. They are furnished with three boilers giving 85 h.p. for heating, drying, and boiling the rags, and with 13 hydraulic motors giving 300 h.p. and three machines "sans fin." The rags are obtained locally. The quantity used per annum is about 394 tons, worth 2,000*l.* The kaolin is procured from Carnovaghi. The cellulose employed is 492 tons per annum, and the wood pulp, 1,476 tons per annum. The number of hands varies from 230 to 240. The wages paid to the men is 2 to 3 lire, females 1 lire, per diem. Printing and writing paper of various qualities, as well as ordinary paper, amounting to 2,460 tons per annum, and valued at 40,000*l.*, are produced. Of the three machines "sans fin," two are of British manufacture and one is Italian.

XX.—FINE CHEMICALS, Etc.

Camphor in Formosa (Japan).

Foreign Office Annual Series, No. 2728.

The value of camphor exported, in 1900, was 253,574*l.*, an increase of nearly 40,000*l.* over 1899, whilst there was a decrease of nearly 20,000*l.* in the value of the oil exported during the same period. Nearly half of the camphor export went to Japan, as did the whole of the camphor oil.

The amount and value of the camphor and camphor oil bought by the Government, during 1900, were as follows:—

——	Quantity.	Value.
	*Catties.	£
Camphor, 1st class	2,608,983	77,089
" 2nd class	701,158	18,696
" 3rd class	168,038	4,073
Total	3,478,179	99,858
Camphor oil	2,362,108	34,591
Grand total	5,840,287	134,449

The amount and value of prepared camphor and oil sold by the Government, during 1900, were as follows:—

——	Quantity.	Value.
	Catties.	£
Camphor, Class A	1,007,800	101,725
" Class B	2,677,700	241,830
" oil	1,227,694	48,103
Total	4,913,194	391,658

* 1 catty = 1·33⅓ lb. (avoir.).

The figures for refined camphor were not obtainable, but the amount was not large.

The Government has shown great energy in establishing plantations, the first being made in 1896. When the camphor monopoly was established, in 1899, fresh plantations were made. The cultivation of camphor trees has lately been attended with very successful results, the number of young trees suitable for transplantation reaching more than 1,000,000.

The camphor refinery at Taihoku is capable of turning out 1,800 lb. of refined camphor, and 5,800 catties of Class A camphor per day.

There is a branch office of the camphor bureau at Kobe, to which a refinery is also attached. This factory is capable of turning out 1,000 lb. of refined camphor and 2,000 catties of Class A camphor per day.

ICHTHYOL: UNITED STATES CUSTOMS DECISION.

Bd. of Trade J., Nov. 28, 1901.

Ichthyol, in par. 626 (free list) of the Tariff, means only ichthyol *oil*. All other ichthyol preparations or salts are dutiable at the rate of 25 per cent. *ad val.*, under par. 3 of the Tariff, as chemical compounds and salts not otherwise provided for.

OIL OF RHODIUM MANUFACTURE IN FRENCH GUIANA.

Chem. and Druggist, Nov. 30, 1901.

According to *La Quinzaine Coloniale*, this oil is distilled in three small factories--two at Cayenne and one at Sinnarmary. These factories exported, in 1900, essential oil to the value of 52,000 frs., against 19,000 frs., in 1899. Besides this, 243 tons of rosewood (valued at 19,000 frs.) were shipped to France. This industry is capable of considerable development, and should attract capital from France. Rosewood is found everywhere in the interior of the colony, and it can easily be transported in small vessels to the factories situated on the mouth of the rivers. One ton of rosewood yields about 10 kilos. of essential oil; the price of the wood is about 80 frs. per ton, and that of the oil about 28 frs. per kilo. The product obtained from 1 ton of the crude material has, therefore, a value of about 280 frs. Fuel of great caloric value is obtainable in unlimited quantity, and there is no difficulty in securing, in the villages, free grants of land near the rivers for the erection of factories. Sinnamary, Montsinery, and Tonnégrande, situated on deep rivers, in districts where rosewood is abundant, are said to be excellent positions for establishing this industry.

PERFUMES OF THE PROVINCE OF FLORENCE (ITALY).

Foreign Office Miscellaneous Series, No. 570.

In the commune of Florence, the Officina Profumo Farmaceutica di S. M. Novella produces various specialities of scents and odoriferous essences, employing a gas motor and 11 workmen. Another small factory of the same kind gives work to five hands. Before 1842, orris root was thrown away as a useless plant. For the last 20 years, its cultivation has been greatly extended, especially in the Chianti, Val di Pesa, and Piano di San Paolo districts. At present, this industry is a source of moderately good profits, the production of orris root being more than 5,905 cwts. per annum.

XXI.—PHOTOGRAPHY.

PHOTOGRAPHIC SUPPLIES IN BRAZIL.

U.S. Cons. Reps., Dec. 4, 1901.

Plates.—The plates used are all imported, the Lumière, which has the largest sale, coming from France, and the Ilford and Marion from Great Britain. U.S. plates are unknown. A plate of metric measurement is wanted, 9 by 12, 13 by 18, 18 by 24, and 24 by 30 centimetres, which can stand climate similar to that of the Gulf States, with a temperature seldom above 90° F., but with more than 85° humidity. The plates should be capable of being worked without ice in the developer, without frilling or softening after being washed with water of 65° to 75° F., and should be of good speed and rich in silver.

Papers.—The papers in use are gelatin printing-out paper, both plain and matte, and a quick-printing developing paper. These papers come from England and France, in bundles of 1 and 2 doz. sheets of cut sizes. They are first wrapped in several sheets of paper impervious to moisture, and finally placed in non-actinic envelopes and sealed.

Much albumin paper and a little platinum paper is employed by professionals, who import the paper and sensitise as needed. A little ferro-prussiate paper in cut sizes has come from the United States, packed in sealed tin tubes, and also another water-development paper, which has met with fair success.

Good gelatin and collodion papers are needed, which will keep fresh for a few months, do not need complicated formulas for toning and fixing, and can with safety be washed in water of 65° to 75° F. The paper should make prints which will not soon fade, provided they have been properly manipulated. There should be a good market for development papers which give platinum and carbon effects. Water-development platinum and other papers should take well with amateurs.

Paper and plate makers entering this market should have their formulas printed in Portuguese, or, in lieu of that, Spanish or French; directions in English can be read by very few.

Developers.—Professional photographers have been using iron developer, but now they prefer metol and hydroquinone. Two are relying chiefly on pyrogallol. The amateur uses metol and hydroquinone, either separate or combined, whilst a few are using glycin.

The developing agents are almost exclusively of German manufacture. But with such chemicals as hyposulphite, the various forms of potash and soda, gold, platinum, sugar of lead, &c., the United States might compete with England and France, who now enjoy the trade.

In chemicals, the anhydrous preparations of sodium carbonate, sodium sulphite, potassium carbonate, &c., are imported, because the duty charge is by weight. Such preparations do not require much care, and do not deteriorate in this climate, whilst the crystalline preparations soon spoil. (See also this Journal, 1901, 1049.)

BOARD OF TRADE RETURNS.

SUMMARY OF IMPORTS.

Articles.	Month ending 30th Nov.	
	1900.	1901.
	£	£
Metals	2,972,915	2,745,563
Chemicals and dyestuffs	400,357	423,356
Oils	1,102,777	972,097
Raw materials for non-textile industries.	10,170,831	9,649,379
Total value of all imports	14,616,880	13,790,395

SUMMARY OF EXPORTS.

Articles.	Month ending 30th Nov.	
	1900.	1901.
	£	£
Metals (other than machinery)	3,659,401	3,408,346
Chemicals and medicines	707,079	774,299
Miscellaneous articles	3,227,428	3,279,568
Total value of all exports	7,593,908	7,462,213

IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Bleaching materials Cwt.	27,724	25,026	13,362	8,319
Soda compounds... ,,		18,387		5,078
Borax, &c. .... ,,	37,871	40,830	18,706	17,000
Brimstone .... ,,	29,951	11,059	7,980	2,942
Nitrate of potash.. ,,	18,368	18,537	15,900	16,334
Chemicals, other. Value £	..	..	131,281	135,678
Cutch and gambier. Tons	1,526	2,096	30,031	35,209
Dyes:—				
Alizarin.......Value £	..	..	13,297	17,529
Aniline and other ,,	..	..	53,923	56,587
Indigo .... Cwt.	67	1,649	2,268	28,884
Bark.............. ,,	25,828	25,815	7,474	9,122
Valonia........... Tons	2,490	1,388	24,433	15,741

IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Bark, Peruvian .. Cwt.	1,511	3,787	3,938	12,558
Caoutchouc ...... ,,	39,505	41,402	481,281	479,888
Gum:—				
Arabic......... ,,	10,471	8,521	18,043	15,148
Lac, &c. .... ,,	4,406	2,828	14,220	18,667
Gutta-percha .... ,,	8,533	7,964	154,181	143,653
Hides, raw:—				
Dry............ ,,	24,304	23,862	64,186	66,745
Wet .... ,,	70,922	77,656	164,189	182,075
Ivory .... ,,	603	554	19,626	24,370
Manure:—				
Guano .... Tons	279	945	1,054	6,845
Bones.......... ,,	4,556	8,103	18,069	26,997
Paraffin.......... Cwt.	91,140	138,203	133,965	140,875
Linen rags .... Tons	1,241	1,307	12,602	12,719
Esparto .... ,,	12,989	14,700	58,301	59,772
Pulp of wood..... ,,	39,418	33,305	248,338	187,754
Rosin............ Cwt.	234,417	172,195	60,876	42,123
Tallow and stearin ,,	4,616	4,235	181,340	144,886
Skins:—				
Goat .... No.	518.981	1,167,679	55,219	105,188
Sheep.......... ,,	999,576	1,189,423	111,766	135,892
Nitrate of soda... Tons	20,201	2,215	171,536	23,169
Phosphate of lime ,,	25,120	20,490	39,198	27,925

IMPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Cement.......... Tons	12,178	18,191	22,948	29,595
China and Earthenware. Cwt.	32,737	37,336	79,942	94,337
Drugs .... Value £	..	..	99,634	122,595
Glass:—				
Sheet .... Cwt.	44,163	136,647	27,796	76,464
Plate .... ,,	16,799	39,844	18,435	44,325
Flint .... ,,	42,719	59,452	80,880	104,654
Bottles .... Gross	128,323	133,509	58,912	58,635
Other.......... Cwt.	26,305	25,476	47,322	50,765
Glue and Gelatin ,,	17,447	20,464	34,964	40,735
Leather, unmanufactured. ,,	124,245	104,926	696,617	673,874
Oil-seed cake .... Tons	35 053	26,639	243,148	177,805
Paints and Pigments. Value £	..	..	113,620	99,714
Paper, Pasteboard Cwt.	515,837	531,793	365,616	372,552
Scientific instruments. Value £	..	..	54,425	58,674
Soap and Soap powder. Cwt.	14,713	25,031	18,913	27,372
Zinc manufactures. ,,	44,449	32,482	53,856	37,490

IMPORTS OF METALS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Copper:—			£	£
Ore............. Tons	8,739	6,789	119,530	87,540
Regulus .... ,,	7,229	7,858	276,355	309,668
Unwrought ..... ,,	5,575	6,801	418,193	467,423
Lead, pig and sheet ,,	17,249	21,751	297,308	252,226
Pyrites .... ,,	58,336	41,906	99,495	65,836
Quicksilver .... Lb.	416,500	50,865	50,890	6,350
Silver ore....... Value £	..	..	90,938	90,434
Tin .... Cwt.	62.201	70,379	408,260	401,968
Zinc.............. Tons	5,270	5,781	102,161	97,191

IMPORTS OF OILS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Cocoa-nut....... Cwt.	54,154	32,570	64,319	42,767
Olive............ Tuns	340	458	13,270	18,124
Palm .... Cwt.	70,326	76,636	78,999	87,950
Petroleum:—				
Illuminating... Gall.	27,540,261	24,153,238	526,732	437,217
Lubricating... ,,	3,267,915	3,268,137	103,019	100,363
Seed............ Tons	4,134	4,410	113,595	118,929
Train, &c. ...... Tuns	1,879	1,790	36,921	34,276
Turpentine ..... Cwt.	58,269	48,863	89,370	63,713

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Brass............ Cwt.	9,680	10,898	52,786	53,936
Copper .... ,,	70,886	93,850	276,707	341,998
Lead............. Tons	2,418	2,696	47,507	39,506
Plated wares... Value £	..	..	56,565	54,465
Tin .... Cwt.	10,229	8,319	68,192	49,909
Zinc.............. ,,	11,040	14,955	10,567	11,726

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Gunpowder..... Lb.	479,000	Cwt.6,733	12,651	17,956
Candles......... ,,	2,391,200	2,474,800	40,300	41,490
Caoutchouc..... Value £	..	..	120,524	92,776
Cement......... Tons	21,329	20,751	40,322	37,861
Products of Coal. Value £	..	..	167,107	104,554
Earthenware ... ,,	..	..	163,510	150,560
Stoneware ...... ,,	..	..	14,267	15,293
Glass:—				
Plate .... Sq. Ft.	179,412	Cwt.7,272	11,626	11,528
Flint .... Cwt.	10,072	8,193	25.518	20,482
Bottles .... ,,	73,031	71,455	35,053	34,714
Other kinds ... ,,	18,615	27,825	19,723	30,569
Leather:—				
Unwrought.... ,,	12,556	11,078	130,323	117,630
Wrought......Value £	..	..	48,246	49,896
Seed oil .... Tons	4,021	4,195	105,341	108,255
Floorcloth.......Sq. Yds.	1,924,300	2,025,700	92,685	98,796
Painters' materials Val. £	..	..	163,043	165,352
Paper............. Cwt.	96,937	92,563	150,988	138,721
Rags.............. Tons	4,362	5,435	23.891	27,698
Soap .... Cwt.	74,759	63,929	77,762	71,748

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
			£	£
Alkali .... Cwt.	293,527	..	91,927	..
Bleaching materials „	110,764	109,386	34,415	36,314
Copper sulphate .. Tons	758	339	17,816	7,193
Chemical manures „	31,815	34,535	184,836	202,283
Medicines....... Value £	..	..	107,959	123,811
Soda compounds:—				
Ash............. Cwt.	..	127,186	..	27,576
Caustic .... „	..	113,062	..	56,340
Bicarbonate .... „	..	27,067	..	9,425
Crystals .... „	..	20,269	..	3,282
Sulphate........ „	..	68,995	..	4,945
Other sorts ..... „	..	33,323	..	13,081

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.

23,277. H. Koppers. Improvements in wall constructions for coke ovens by means of a particularly shaped stone. Complete Specification. Nov. 18.

23,533. G. Hohoff.—From D. Hoffsümmer, Germany. An improved photometer. Nov. 20.

23,614. W. R. Lysaght and F. G. Treharne. Improvements relating to the manufacture of annealing pots and similar apparatus. Nov. 21.

24,150. A. F. Spooner.—From J. B. F. Loubriat, Russia. Apparatus for charging and discharging furnaces, ovens, and the like. Nov. 27.

24,399. J. A. Elsner and G. F. Knox. Improvements in apparatus employed for refrigerating by compression. Complete Specification. Nov. 30.

24,568. H. C. Owens. A method of separating damp from air, and apparatus for that purpose. Dec. 3.

24,699. J. G. Accles and F. H. de Veulle. Improvements in and relating to carburetters. Dec. 4.

24,832. G. Wilton. Improvements in apparatus for the distillation of ammoniacal liquors. Dec. 5.

24,840. H. H. Lake.—From the firm of C. A. Schuppmann, Germany. Improvements in and relating to kilns or furnaces. Dec. 5.

24,928. O. Lindemann.—From E. Körting, Germany. Improvements in and relating to liquid fuel furnaces. Complete Specification. Dec. 6.

25,376. F. A. Woolley. Improvements in and relating to hydro-extractors and like centrifugal machines. Dec. 12.

25,457. J. Hargreaves. Improvements in the condensation of vapour, and in the concentration of solutions, and in the means and apparatus employed therein. Dec. 13.

COMPLETE SPECIFICATIONS ACCEPTED.*

1900.

22,836. H. A. Vachell. Water-distilling apparatus. Dec. 18.

23,044A. P. Naef. Apparatus for stirring or injecting fluid into fuel in gas producers or into other solid material. Dec. 4.

1901.

1393. G. C. Marks.—From A. Hannetelle-Chapius, France. Concentrating apparatus, applicable to acids, saline solutions, and in general to other liquids capable of concentration. Nov. 27.

2109. H. Ruelle and E. Montagne. Smoke-consuming apparatus for furnaces and the like. Dec. 18.

2775. H. C. Anstey. Evaporators. Dec. 18.

5568. J. I. Friedrich. Centrifugal separators. Dec. 11.

6216. J. Birkett and J. Thornton. *See* Class XVII.

17,555. O. Francke. Non-conducting material for coating steam pipes or the like. Dec. 4.

17,884. V. Schwaninger. Apparatus for raising fluids. Nov. 27.

19,071. W. F. Singer. Compressors. Nov. 27.

21,727. A. F. Kécheur and W. A. B. Heerey. Carburetting apparatus. Dec. 4.

II.—FUEL, GAS, AND LIGHT.

APPLICATIONS.

23,260. H. Schild. A new and improved process of manufacturing coal or the like fuel cakes. Nov. 18.

23,277. H. Koppers. *See* Class I.

23,278. H. Koppers. Improvements in regenerative coke ovens with separate distribution of gas and air to the heating flues. Complete Specification. Nov. 18.

23,478. C. S. Ball. Acetylene headlight. Nov. 20.

23,480. D. Boyde. Preventing escape of gas and noxious fumes from the incandescent gas light when the by-pass is burning. Nov. 20.

23,543. D. Losfeld. Improvements in apparatus for generating acetylene gas. Complete Specification. Nov. 20.

23,733. W. T. Sugg. Improvements in the production of surfaces for use in incandescent gas lighting. Nov. 22.

23,764. W. B. Hartridge. Improvements in and relating to combustion of fuel. Nov. 22.

23,895. H. J. Dowsing. Improvements in apparatus for electrically producing radiant heat. Nov. 25.

23,904. O. G. Blunden, J. W. Malden, and A. Malden. Improvements connected with the manufacture of fuel briquettes. Nov. 25.

23,908. E. W. Sprott. Improvements in apparatus for gas purification. Nov. 25.

23,910. A. McLean. Improvements in the mode of and apparatus for treating peat. Nov. 25.

23,945. The Star Patent Fuel Company, Ltd., and T. E. Heath. Improvements in apparatus for use in the manufacture of artificial fuel. Nov. 25.

23,960. H. J. McBride and H. W. McBride. An improved smoke-preventing composition to be applied to coals. Nov. 26.

24,030. J. Heaton and N. T. Worthley. Improvements in acetylene gas generating apparatus. Complete Specification. Nov. 26.

24,085. R. Gilmour. Improvements in and relating to incandescent gas burners for lighting purposes. Nov. 27.

24,218. J. L. Muller and J. Bonnet. Improvements relating to machines for the manufacture of mantles employed in incandescent lighting. Complete Specification. Nov. 28.

24,287. W. B. Cowell. Water or producer gas. Improvements in their manufacture for motive lighting and heating purposes. Nov. 29.

24,304. W. P. Houze. Carbureter. Nov. 29.

24,386. A. E. Sutton. Improvements in and relating to incandescence gas lighting. Nov. 30.

24,484. W. T. Sugg. Improvements in gas lamps for incandescent gas lighting. Dec. 2.

24,491. A. Dobson. Improvements in method of drying peat. Complete Specification. Dec. 2.

24,514. R. Thomson. Improvements in and relating to the combustion of fuel. Dec. 2.

24,532. T. Dunne. An improved method for the manufacture of fire-lighters from peat. Dec. 3.

24,570. A. McIntosh. Improvements in and relating to apparatus for generating and storing acetylene gas. Dec. 3.

24,577. M. Toltz and A. Sipschutz. Acetylene gas lighting system. Complete Specification. Dec. 3.

24,691. J. S. Kemp-Welch. Improvements relating to producer gas apparatus. Dec. 4.

24,837. S. T. Wellman and C. H. Wellman. Improvements in coke ovens. Complete Specification. Filed Dec. 5. Date applied for May 22, 1901, being date of application in United States.

24,903. O. H. Bayldon and H. E. Morriss. An improved generator of gas from paraffin oil for heating purposes. Dec. 6.

24,960. W. J. Crossley and J. Atkinson. Improvements in gas cleaning and cooling apparatus. Dec. 7.

25,092. A. J. Boult. — From F. M. Hille, Germany. Improved method of regulating gas generators, and apparatus employed in connection therewith. Dec. 9.

25,157. G. Davidson and The Fietz Preliminary Syndicate, Ltd.—From C. Fietz, Italy. Improvements in the manufacture of illuminants. Dec. 10.

25,173. H. H. Lake.—From Koale Sava Manufacturing Company, United States. An improved composition for use with coal to increase its efficiency. Complete Specification. Dec. 10.

25,180. E. W. Phelps and T. Nadeau. Improvements in incandescent gas burners. Complete Specification. Dec. 10.

25,226. C. Rainey. Improvements in or relating to carburettors. Dec. 10.

25,281. F. B. Passmore. Improvements in atmospheric gas burners. Dec. 11.

25,493. E. D. Holmes.—From O. N. Guldin, of The Western Gas Construction Company, United States. *See* Class III.

25,494. E. D. Holmes.—From O. N. Guldin, of The Western Gas Construction Company, United States. A duplex valve for gas purifiers and for other purposes. Dec. 13.

25,495. E. D. Holmes.—From O. N. Guldin, of The Western Gas Construction Company, United States. Improvements in the mouthpieces and lids of gas retorts, also applicable to the lids of other receptacles. Complete Specification. Dec. 13.

25,516. W. P. Warren. Improvements in and relating to processes and apparatus for the generation of acetylene. Dec. 13.

25,590. A. J. Boult.—From J. Braunstein, Germany. Improvements in or relating to incandescent gas burners. Dec. 14.

25,604. G. J. Snelus. *See* Class X.

25,616. E. Helbing. An improved process of manufacture of peat briquettes. Complete Specification. Dec. 14.

Complete Specifications Accepted.

1900.

15,108. W. H. Dargue. Apparatus for the production of acetylene gas. Nov. 27.

21,511. T. Rathmell. Apparatus for generating acetylene gas for household lighting purposes. Nov. 27.

22,618. W. Harris. Shock-proof fitting for use in conjunction with incandescent gas lighting in particular. Dec. 4.

22,782. W. W. Clark and G. Bone. Apparatus for generating acetylene gas. Dec. 18.

22,960. J. Shackleton and A. Ross. Acetylene generators. Dec. 18.

23,044. P. Naef. Apparatus for recovering products from fuel. Dec. 4.

1901.

240. J. A. Burgess. Acetylene gas generators. Dec. 4.

1525. J. W. Ward. Burners for incandescent gas lighting. Dec. 4.

2225. W. J. Crossley and J. Atkinson. Heat interchanger for use in connection with gas producers. Dec. 11.

15,005. J. Y. Johnson.—From The Wellmann-Seaver Engineering Company, United States. Apparatus for charging and discharging coke ovens or retorts. Dec. 4.

15,846. J. J. Hendler. Apparatus for the manufacture of acetylene and other gases. Dec. 4.

17,292. T. H. Oswald and F. E. Bowman. Gas-producing plant. Nov. 27.

19,857. V. Powers and The Bevois Patent Lighting Company, Ltd. Apparatus for the generation, storage, and purification of acetylene and like gases. Nov. 27.

20,001. L. Stöckmann. Process for increasing the heating value of fuel. Nov. 27.

20,760. H. Praetorius. Process and apparatus for mixing liquid fuels of different specific gravities.

21,376. J. Radcliffe. Manufacture or production of combustible gas. Dec. 4.

21,377. J. Radcliffe. Apparatus suitable for the manufacture or production of combustible gas for calcining or roasting ores and other substances, and for other purposes. Dec. 4.

21,534. C. Killing. Incandescent gas burners. Dec. 4.

22,486. J. F. Henderson and C. W. Clayton. Automatic carbonic acid gas generator and holder. Dec. 11.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, PETROLEUM.

Applications.

23,296. H. J. Spooner. A new and improved vacuum still for the distillation of petroleum, and liquid fuel furnaces for the same. Nov. 18.

25,493. E. D. Holmes.—From O. N. Guldin, of The Western Gas Construction Company, United States. Improvements in apparatus for extracting tar from illuminating gas. Dec. 13.

Complete Specification Accepted.

1901.

2105. J. Meikle. Distillation of tar, and apparatus therefor. Dec. 4.

IV.—COLOURING MATTERS and DYESTUFFS.

Applications.

23,944. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new colouring matter, of colouring matter lakes, and of intermediate products for use therein. Nov. 25.

24,354. 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 the employment of colouring matters of the anthracene series in dyeing. Nov. 29.

24,400. O. Imray.—From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of new acridinium compounds. Nov. 30.

25,009. O. Imray.—From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of yellow to orange-yellow dyestuffs of the acridine series. Dec. 7.

25,089. O. Imray.—From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of new substituted auramines. Dec. 9.

25,100. G. W. Johnson.—From C. F. Boehringer und Soehne, Germany. Improvements in the reduction of aromatic nitro-compounds to the corresponding amines. Complete Specification. Dec. 9.

25,393. C. D. Abel. — From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of new colouring matters belonging to the triphenylmethane series. Dec. 12.

25,608. P. Jochum. Improvements in the preparation and use of colours for chemical printing on fabrics and other textile materials. Dec. 14.

Complete Specifications Accepted.

1900.

22,297. H. H. Lake.—From K. Oehler, Germany. Manufacture and production of new sulphonic acids and of colouring matters therefrom. Dec. 11.

22,397. H. H. Lake.—From K. Oehler, Germany. Manufacture and production of new colouring matters and of intermediate products for use therein. Nov. 27.

22,989. B. Willcox. — From Badische Anilin und Soda Fabrik, Germany. Manufacture and production of new products for use in the manufacture of colouring matters, and of colouring matters therefrom. Nov. 27.

23,419. R. B. Ransford. — From L. Cassella and Co., Germany. Manufacture of hydroxylated acridines. Nov. 27.

23,887. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of naphthalene compounds and their employment in dyeing and printing. Dec. 18.

1901.

330. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of a black colouring matter directly dyeing cotton. Dec. 11.

999. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of substantive black colouring matters. Dec. 4.

1357. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of colouring matters of the anthraquinone series. Dec. 18.

1358. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of colouring matters of the anthracene series. Dec. 11.

1766. F. Ullmann. Manufacture of diphenyl derivatives. Dec. 4.

2188. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of colouring matters directly dyeing cotton. Dec. 4.

2339. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of blue dyestuffs. Dec. 11.

V.—PREPARING, BLEACHING, DYEING, PRINTING, AND FINISHING TEXTILES, YARNS, AND FIBRES.

Applications.

23,434. H. H. Lake.—From Farbwerke Mühlheim vorm. A. Leonhardt and Co., Germany. Improvements in and relating to the dyeing and printing of vegetable fibre. Nov. 19.

23,556. W. H. Perkin, jun., and Whipp Bros. and Tod, Ltd. Improvements in the treatment of raw cotton and cotton goods, to reduce the inflammability thereof. Nov. 20.

23,557. W. H. Perkin, jun., and Whipp Bros. and Tod, Ltd. Improvements in the treatment of raw cotton and cotton goods to reduce the inflammability thereof. Nov. 20.

23,695. R. J. Urquhart.—From Chemische Fabriken vorm. Weiler ter Meer, Germany. Improvements in the process of dyeing piece goods with sulphur dyes. Complete Specification. Nov. 22.

24,323. E. Bottelli. Improvements in and relating to the dressing and finishing of fabrics. Filed Nov. 29. Date applied for April 30, 1901, being date of application in Italy.

24,697. R. B. Ransford.—From L. Cassella and Co., Germany. Improvements in dyeing and printing with sulphur colours. Dec. 4.

24,856. A. Born. An improved process and apparatus for cleansing, scouring, and removing oil from wool, hair, bristles, and other materials. Complete Specification. Dec. 5.

24,902. W. T. Forbes and G. P. J. Leclercq. A process and apparatus for ungumming, cleansing, and bleaching vegetable fibre. Complete Specification. Dec. 6.

25,162. F. J. Hill. Improvements in or relating to the treatment of fabrics. Dec. 10.

25,248. E. Knecht and P. Spence and Sons, Ltd. Improvements in the method of and means for preventing or resisting the local production of colour in the printing of textile fabrics. Dec. 11.

25,338. J. W. W. Shaw. Improvements in the process of and apparatus for bleaching certain textile fabrics and fibrous material. Dec. 12.

25,365. D. R. Choksey. Improved means for extracting aloe and other fibres. Complete Specification. Dec. 12.

25,404. J. W. Peet and J. H. Peet. Improvements in dyeing vessels or kiers. Dec. 12.

Complete Specifications Accepted.

1900.

21,073. F. V. M. Raabe. Treatment of certain textile fibres preparatory to their conversion into yarn. Nov. 27.

22,033. H. W. Church. Dyeing machines. Dec. 11.

1901.

1042. J. E. Prestwich. Method of and apparatus for treating textile yarns and threads with liquids. Dec. 11.

1140. O. Imray.—From Farbwerke vormals Meister, Lucius und Brüning, Germany. Process for dyeing paper fabrics. Dec. 4.

1160. J. E. Bousfield, of the firm of G. F. Redfern and Co.—From F. Cantù, G. Miglioretti and G. Maffei, Italy. Manufacture of sheets, waterproof fabrics, imitation linen goods, and the like from casein. Dec. 4.

1834. J. Skoupil. Process and apparatus for dyeing, bleaching, and finishing textiles and fibres. Nov. 27.

2927. R. B. Ransford.—From L. Cassella and Co., Germany. Preventing the deterioration of dyed cotton. Dec. 18.

11,942. J. Wezel. Preparation of art, chromo, and other colour printing papers. Dec. 18.

18,562. H. Ryder. Machines for making manilla rope. Dec. 11.

VI.—COLOURING WOOD, PAPER, LEATHER, Etc.

Complete Specification Accepted.

1900.

20,940. C. L. Burdick. Apparatus for facilitating the application of colours or liquids to surfaces such as paper, porcelain, cloth, wood, and the like. Nov. 27.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

23,747. A. S. Plews. Process for the manufacture, direct from the ore, of white oxide of antimony and compounds thereof. Nov. 22.

23,817. J. Boulton and B. A. Spaull. Improvements in and relating to the manufacture of potassium cyanide and sodium cyanide. Nov. 23.

24,456. G. Craig. Improvements in obtaining hydrocyanic acid from ferrocyanides, and in apparatus therefor. Dec. 2.

24,684. G. F. Berry and J. B. Lawes and Co., Ltd. A new or improved method or process for the recovery, collection, and utilisation of waste carbonic acid gas given off in the manufacture of certain acids and acid salts. Complete Specification. Dec. 4.

24,832. G. Wilton. *See* Class I.

24,904. W. Feld. Improvements relating to the production of hydrocyanic acid. Dec. 6.

24,920. W. Feld. Improvements relating to the production of hydrocyanic acid. Dec. 6.

25,046. H. R. Angel. Improvements in the manufacture of caustic soda as a by-product from the reduction of refractory and other ores. Dec. 9.

25,299. J. Jeffreys. Improvements in the utilisation of chloride of calcium and the production of valuable products thereby. Dec. 11.

25,444. J. E. Riley and J. Barnes. Improvements in the recovery of arsenic and sulphur from precipitated sulphide of arsenic. Dec. 13.

Complete Specifications Accepted.

1900.

22,121. G. Craig and R. M. Paterson. Production or purification of alkaline cyanides. Dec. 4.

1901.

601. P. L. Martin. Manufacture of anhydrous caustic baryta. Dec. 4.

754. R. Wolffenstein. Manufacture of peroxide preparations. Nov. 27.

1904. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture of sulphuric acid. Dec. 4.

2342. H. Precht Process for the production of potassium magnesium carbonate. Dec. 4.

2368. O. Imray.—From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of sulphuric anhydride by the contact process. Dec. 18.

VIII.—GLASS, POTTERY, AND ENAMELS.

Applications.

23,563. W. W. Pilkington. Improvements in glass manufacture, and apparatus therefor. Nov. 20.

24,319. C. M. White. — From C. W. Foster, United States. Improvements in apparatus for delivering melted glass. Complete Specification. Nov. 29.

24,324. H. Brooke. Improvements relating to apparatus for dividing molten material, chiefly designed for use with molten glass. Complete Specification. Nov. 29.

24,328. G. W. Laybourn. Improvements in or relating to the deposition of metals on earthenware bodies. Complete Specification. Nov. 29.

24,333. H. Brooke. Improvements relating to receptacles for containing molten material, chiefly designed for use in connection with glass furnaces. Complete Specification. Nov. 29.

24,468. O. Imray. — From J. A. Chambers, United States. Improvements in the manufacture of glass articles, and apparatus therefor. Complete Specification. Dec. 2.

24,780. A. J. Boult.—From O. Leprouz, France. Improvements in or relating to the decoration of earthenware, china, and the like. Dec. 5.

24,927. C. Boucher. Improvements in or relating to machines for making bottles and other articles of blown glass. Dec. 6.

Complete Specification Accepted.

1900.

20,940. C. L. Burdick. *See* Class VI.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Applications.

23,313. W. P. Thompson. Improvements in or relating to mastic or other cement compositions, concrete, artificial asphalt, waterproofing material, and the like, and in the process of manufacturing the same, and articles manufactured therefrom or therewith. Nov. 18.

23,443. C. von Forell. Improvements in the manufacture of cement. Complete Specification. Nov. 19.

23,962. C. A. Burghardt, C. F. Hall, and W. A. Jones. Improvements in tiles, plates, or slabs for walls, ceilings, and for use in the construction or decoration of buildings generally. Nov. 26.

24,072. J. Burn. An improved treatment of impure clay for use in the manufacture of bricks, and articles of a like nature, made or manufactured from clay. Nov. 27.

24,092. J. Tuckwell. Improvements in the manufacture of artificial marble and the like. Nov. 27.

24,393. J. B. Sangwin. Improvements in or in connection with the manufacture of artificial stone. Nov. 30.

24,580. W. S. Wilkinson. Improvements in the manufacture of materials for pavements. Dec. 3.

24,683. G. F. Berry and J. B. Lawes and Company, Ltd. Improvements in or relating to cements or plasters and in the manufacture of bricks, tiles, slabs, artificial stone, and the like. Complete Specification. Dec. 4.

24,841. F. Oberschulte. Improvements relating to the manufacture of artificial sandstone. Dec. 5.

24,923. G. C. F. Boivie. Improvements in the manufacture of artificial stone. Complete Specification. Dec. 6.

24,998. A. Vickery. Improvements in the manufacture of cements, waterproof coatings, artificial stone, and the like. Dec. 7.

25,239. C. Heap and T. Oddy. Improvements in the manufacture of Portland slag cement and hydraulic lime. Dec. 11.

25,512. E. Rott. Improvements in the manufacture of bricks or blocks of artificial stone. Dec. 13.

Complete Specifications Accepted.

1900.

22,139. L. Hatschek. Process for the manufacture of slabs, plates, and other suitable articles of artificial stone. Dec. 4.

23,718. W. W. Hewitt. Cement kilns and drying chambers. Dec. 4.

1901.

801. J. S. Rigby. Kiln for the manufacture of Portland cement or lime. Dec. 11.

2409. H. E. Rüsager. Manufacture of Portland cement. Dec. 18.

2499. J. Wilkinson. Manufacture of slabs or plates suitable for use as building material and for other purposes. Dec. 11.

20,891. S. Höyer-Ellefsen. Fire-proof floors and the like. Dec. 4.

X.—METALLURGY.

Applications.

7084a. D. Bates and G. W. Peard. Improvements in and connected with the annealing of metals. Filed Dec. 4. Date claimed April 4, 1901.

23,422. W. H. Martin and The Thames Mining Machinery Company, Ltd. An improved process and means for the extraction of gold from sea water. Nov. 19.

23,531. J. T. Pagan and W. Buckley. Improvements in the production of metal castings. Complete Specification. Nov. 20.

23,654. W. F. L. Frith. An improved process for toughening or annealing steel and other metals. Nov. 21.

23,790. A. Lamberton. Improvements in and relating to crushing and amalgamating machinery for the reduction of quartz and the recovery therefrom of the precious metals and the grinding or pulverising of other substances. Nov. 23.

23,840. W. L. Wise.—From C. H. Homan, Norway. Improvements in the treatment of titaniferous ores. Nov. 23.

23,929. W. S. Lockhart and The Automatic Gem and Gold-Separator Syndicate, Ltd. An improved apparatus for the concentration of ores, substances held in slimes, and the like. Nov. 25.

23,939. W. F. L. Frith. A new process for toughening or annealing steel, iron, and other metals. Nov. 25.

24,026. F. T. Mumford. Improvements in the method of and apparatus for the recovery of precious metals from ores and slimes. Nov. 26.

24,234. C. A. Keller. Improvements in the obtainment of metals and alloys, and in furnaces to be employed therein. Complete Specification. Filed Nov. 28. Date applied for July 6, 1901, being date of application in France.

24,235. C. A. Keller. Improvements in the manufacture and treatment of alloys. Complete Specification. Filed Nov. 28. Date applied for July 6, 1901, being date of application in France.

24,361. S. Cowper-Coles and Co., Ltd., and S. Cowper-Coles. Improvements in and relating to aluminium welding. Nov. 30.

24,427. C. Billington, J. Newton, and C. Billington, jun. Improved metallic alloys. Dec. 2.

24,524. C. Billington, J. Newton, and C. Billington, jun. Improved metallic alloys. Dec. 3.

24,734. T. C. Hamilton. An improvement in coating metals. Dec. 4.

25,046. H. R. Angel. *See* Class VII.

25,187. A. G. Brookes.—From C. Casper and F. Oertel, Germany. Improvements in the manufacture of chromic silicon steel or steel alloyed with silicon and chrome. Dec. 10.

25,476. W. B. Johnson.—From N. McConnell, United States. Improvements in the manufacture of steel. Dec. 13.

25,604. G. J. Snelus. An improved method of and apparatus for purifying and enriching blast-furnace gas. Dec. 14.

Complete Specifications Accepted.

1900.

21,388. G. G. M. Hardingham.—From R. Davey, Italy. Rotary furnace applicable for use in roasting or calcining ores. Nov. 27.

22,570. H. F. Kirkpatrick-Picard. Recovery of zinc from sulphide ores. Dec. 18.

1901.

331. J. C. Butterfield. Obtaining antimony and separating gold from antimony ores. Dec. 11.

430. R. Langhans. Process of metallising with precious metals. Dec. 11.

510. W. J. Foster. Method of keeping cool the tuyères of blast and like furnaces and forges, and of heating the air or blast used. Nov. 27.

1738. W. Pethybridge. Treatment of telluride gold ores. Dec. 4.

2756. H. Leyendecker. Process for diminishing the solubility of lead in acids and other chemical compounds. Dec. 18.

18,149. D. Laird. Furnace for the cupellation of precious metals, and for oxidising and reducing ores. Dec. 11.

20,160. J. Tennett. Casting of chill rolls. Nov. 27.

20,566. E. Knudsen. Process and apparatus for smelting and concentrating unroasted sulphide ores. Dec. 11.

21,097. C. Rogers and A. M. Oswald. Process for the extraction and recovery of zinc from sulphide ores or tailings. Nov. 27.

22,753. H. J. Haddan.—From the Meteorit Gesellschaft, G. m. b. H., Germany. Process for improving the quality of aluminium. Dec. 18.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

Applications.

24,012. R. W. James.—From The Mica Insulator Company, United States. Improvements in or relating to compound insulating material for electrical purposes. Nov. 26.

24,277. C. S. Kaufmann. Improvements in and relating to plates for electric storage batteries. Nov. 29.

24,511. A. Bainville. Improvements in or relating to electrodes for electric accumulators or batteries. Complete Specification. Dec. 2.

24,627. L. M. G. Rosée d'Infreville. Improvements in electric batteries. Complete Specification. Dec. 3.

24,834. R. Pearson. An improvement in primary batteries. Dec. 5.

24,992. A. P. T. Tully. Improvements relating to electric primary cells or batteries. Dec. 7.

25,182. G. Brewer.—From E. R. Taylor, United States. Improvements in electric furnaces, and in the production of chemicals in such furnaces. Complete Specification. Dec. 10.

25,282. F. E. Elmore.—From J. O. S. Elmore, India. Improvements in apparatus for the generation and electrolytic application of electric currents. Dec. 11.

25,395. E. Geisenberger. Improvements in or relating to the manufacture of soda and other substances by the aid of electrolysis. Filed Dec. 12. Date applied for May 25, 1901, being date of application in Belgium.

Complete Specifications Accepted.

1900.

21,566. C. A. von Welsbach. Accumulators or secondary batteries. Dec. 4.

21,886. L. E. Lacroix, jun. Electrical storage or accumulators. Dec. 11.

22,019. A. Meygret. Storage batteries. Dec. 4.

22,902. E. Edser and M. Wilderman. Electrolytic methods and apparatus. Dec. 18.

23,543. W. E. Ayrton and A. W. Fithian. Plates for secondary or storage cells. Nov. 27.

1901.

1890. J. T. Niblett. Electric storage batteries or accumulators. Dec. 4.

1981. J. T. Niblett. Electric storage batteries or accumulators. Dec. 4.

2587. G. Rosset. Electric batteries. Dec. 11.

17,480. Siemens Bros. and Co., Ltd.—From Siemens and Halske Actiengesellschaft, Germany. Aluminium electrolytic cells. Dec. 4.

20,072. T. A. Edison. Accumulators. Dec. 4.

21,294. A. Tribelhorn. Electric accumulator batteries. Nov. 27.

XII.—FATS, OILS, AND SOAP.

Applications.

24,603. H. J. Haddan.—From E. R. Edson, United States. A new or improved process of reducing or rendering fish-waste or fish and other oil-yieldable material to obtain oil. Complete Specification. Dec. 3.

24,821. W. P. Thompson.—From A. N. Nielsen, Newfoundland. Improvements in the bleaching of whale and seal oil. Complete Specification. Dec. 5.

24,850. A. E. Iveson. Apparatus for pressing animal fat, especially applicable for use in preparing fat in a "granular form" for cooking purposes. Dec. 5.

25,425. F. V. Speltie. A new or improved process and apparatus for extracting oil from fish and obtaining dried residues serving as "guano." Complete Specification. Dec. 12.

Complete Specifications Accepted.

1901.

759. W. F. Haywood. Means or apparatus to be employed in the manufacture of soap. Dec. 18.

19,029. F. C. Thiele, J. M. Parker, and J. F. Finke. Refining of crude mineral oils and their distillates. Dec. 4.

XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

23,470. J. Deborde. The manufacture of an elastic insolubilised gelatinous compound. Nov. 20.

23,718. R. Walker. Improvements in the manufacture of whiting, and accessory plant therefor. Nov. 22.

24,370. J. F. Allen and R. G. Brooke. An improved anti-corrosion paint. Nov. 30.

24,590. A. Gentzsch. Improved manufacture of a substitute for gutta-percha. Complete Specification. Dec. 3.

25,608. P. Jochum. *See* Class IV.

Complete Specifications Accepted.

1901.

10,696. C. Wallis and R. E. M. Lagerwall. Roller mills applicable for the manufacture of paint, white lead, pigments, printing ink, confectionery, or the like. Dec. 18.

11,533. A. Thilmany. *See* Class IV.

17,555. O. Francke. *See* Class I.

22,201. H. H. Lake.—From Vereinigte-Gummiwaaren Fabrik Harburg-wein vormals Meiner J. N. Reithoffer, Germany. Manufacture of rubber-sponge. Dec. 11.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Applications.

24,342. R. W. Barker.—The International Lactayne Company, United States. Artificial glue or size. Nov. 29.

24,604. H. J. Haddan.—From E. R. Edson, United States. Improvements in obtaining a congealable glue-yieldable or gelatine-yieldable material from fish-waste or fish and other material. Complete Specification. Dec. 3.

25,063. G. Klenk. Improvements in the manufacture or treatment of tannin extracts to increase their utility of tanning leather. Dec. 9.

25,071. A. Jeffrey. Improvements in apparatus for melting and applying marine glue and other convenient substances. Complete Specification. Dec. 9.

XV.—MANURES, Etc.

Applications.

23,959. W. Milligan. Improvements in and connected with the manufacture of manure, the same being also applicable for the destruction of worms, slugs, and such like. Nov. 26.

25,425. F. V. Speltie. *See* Class XII.

Complete Specifications Accepted.

1901.

2035. C. Beyer. Machine for the distribution of artificial manure. Nov. 27.

3178. J. Hughes. Treatment of superphosphate for fertilising purposes. Dec. 18.

XVI.—SUGAR, STARCH, and GUM, Etc.

Applications.

23,911. J. R. Hatmaker.—From J. A. Just, United States. Casein adhesive. Nov. 25.

24,569. C. A. Spreckels and C. A. Kern. Improvements in the purification of sugar-bearing materials, and cleansing compositions for that purpose. Dec. 3.

24,847. F. Scheibler. Improvements connected with the manufacture of cube-sugar or analogous forms of sugar. Dec. 5.

Complete Specifications Accepted.

1900.

21,256. R. Harvey. Continuous defecators for the clarification of sugar juice and other liquids. Nov. 27.

1901.

2294. G. W. Johnson.—From M. Leconte and Compagnie Electro-Sucrière, France. Manufacture of rice-starch and other amylaceous compounds. Dec. 4.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

24,146. Spiritine, Ltd.—From Jules Drapier and P. Dubois, France. Improvements in or relating to solidified spirit. Nov. 27.

24,243. W. Ackroyd and F. Buckley. Improvements in and relating to malt kilns. Nov. 29.

24,477. E. H. Josephi. A new or improved beverage. Dec. 2.

Complete Specifications Accepted.

1901.

2501. A. de Schepper. Machines for malting purposes. Dec. 18.

6216. I. Birkett and J. Thornton. Kilns for malt or the like. Nov. 27.

10,015. H. J. Haddan.—From J. C. Riley, United States. Process for ageing liquors. Dec. 18.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

25,037. C. Ollendorff. Improvements in pasteurising apparatus. Complete Specification. Dec. 9.

25,295. A. F. S. Kent and W. G. Barnfield. An improved process for the sterilisation and preservation of eggs. Dec. 11.

25,599. J. Meinl. An improved process of manufacturing cocoa substitutes. Dec. 14.

B.—Sanitation; Water Purification.

23,469. J. C. Haller and R. H. Machell. Improvements in the treatment of sewage, and apparatus therefor. Nov. 20.

24,602. H. J. Haddan.—From E. R. Edson, United States. Improvements in apparatus for reducing or rendering fish-waste, fish, and other material. Complete Specification. Dec. 3.

Complete Specifications Accepted.

A.—Foods.

1900.

19,343. A. Büttner and C. Meyer. Process and apparatus for drying fruit and other vegetable matter. Dec. 4.

23,377. P. Mongiraud and H. Labranche. Apparatus for the sterilisation and aseptic transfer or storage of milk. Dec. 11.

B.—Sanitation; Water Purification.

1901.

517. F. Candy. Treatment of sewage and other polluted liquids, and apparatus for use in connection therewith. Dec. 18.

16,547. J. Howden and J. H. Hume. Refuse destructors. Nov. 27.

C.—*Disinfectants.*

1901.

5492. B. J. B. Mills.—From E. D. Taylor Company, United States. Apparatus for producing formaldehyde gas for fumigating, disinfecting, and sterilising purposes. Dec. 18.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

23,540. C. Smith and The Globe Manufacturing Company, Ltd. Improvements in the manufacture of celluloid. Nov. 20.

23,841. B. Guerrin and G. G. Leflère. Improvements in and relating to the production of antiseptic paper. Nov. 23.

24,381. J. White. Improvements in apparatus for straining paper pulp and like material. Complete Specification. Nov. 30.

Complete Specification Accepted.

1900.

22,418. The Westfield Paper Co., Ltd., and G. Stewart. Surfacing of paper. Dec. 4.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

23,585. W. E. Corrigall. Improvements in or relating to menthol cones. Nov. 21.

23,645. J. E. Kistruck. Improvements in or connected with devices for extracting the juice of lemons and other fruit. Nov. 21.

24,321. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of pharmaceutical compounds. Nov. 29.

25,444. J. E. Riley and J. Barnes. *See* Class VII.

Complete Specifications Accepted.

1900.

23,418. R. B. Ransford.—From L. Cassella and Co., Germany. *See* Class IV.

1901.

18,733. R. H. Page. Production of organic peroxides. Dec. 4.

XXI.—PHOTOGRAPHY.

Applications.

23,292. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of flash lights for photography. Nov. 18.

23,299. C. D. Abel. — From Actiengesellschaft für Anilinfabrikation, Germany. Improvements in photographic roll films. Nov. 18.

23,551. Société Anonyme des Produits Photographiques M.-Y. Improvements relating to the manufacture of photographic films. Filed Nov. 20. Date applied for May 3, 1901, being date of application in France.

24,025. K. A. Pallin. Improvements in and relating to photographic films. Nov. 26.

24,255. L. C. Login. Improvements in stereoscopic photography. Nov. 29.

24,469. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Improvements in coating photographic films, and in apparatus therefor. Dec. 2.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

23,284. M. Fiedler. Improvements in explosives. Complete Specification. Nov. 18.

23,740. F. L. Nathan and R. Robertson. An improvement in the manufacture of explosives. Complete Specification. Nov. 22.

23,889. M. Bielefeldt. Improvement in priming compositions or charges for percussion caps and detonators. Nov. 25.

24,372. L. Davies. Improvement in explosives. Nov. 30.

Complete Specification Accepted.

1900.

23,064. J. Ramsden and W. R. Hirst. Matches. Dec. 4.

XXIII.—GENERAL ANALYTICAL CHEMISTRY.

Applications.

25,037. C. Ollendorff. *See* Class A.

25,469. J. H. Müller. Improvements in the process of and in the apparatus for fractional distillation. Complete Specification. Dec. 13.

PATENTS UNCLASSIFIABLE.

Complete Specifications Accepted.

1901.

10,926. K. Koch and A. Stelling. Article of manufacture consisting of felt manufactured from metal. Dec. 11.

15,153. A. E. Cummer. Process of treating hydrous compounds. Nov. 27.

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