

# THE JOURNAL OF THE Society of Chemical Industry.

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 11.—VOL. XX.]

NOVEMBER 30, 1901.

[Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6, all prepaid.]

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**SESSION 1901—1902.**

Monday, Dec. 2nd, 1901:—

Messrs. Herbert E. Burgess and J. F. Child. "The Lemon Oil Industry."

Mr. J. W. Hinchley. "The Separation of Materials of Different Specific Gravity."

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**SESSION 1901—1902.**

Thursday, Feb. 27th, 1902:—

Mr. Wilton P. Rix. "Preparation and Behaviour of Insoluble Frits for Pottery Glazes."

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## NOTICES.

## 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.

Foreign and Colonial Members are reminded that the subscription of 25s. 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.

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### Newcastle Section.

*Meeting held on Thursday, October 31st, 1901.*

MR. W. L. RENNOLDSON IN THE CHAIR.

#### CHAIRMAN'S ADDRESS.

THE Chairman's address consisted of an interesting review or criticism of many popular beliefs, which he considered would not exist if a knowledge of chemistry, even slight, were more general.

### Liverpool Section.

*Meeting held on Wednesday, October 30th, 1901.*

MR. C. LONGUET HIGGINS IN THE CHAIR.

#### THE SECOND HURTER MEMORIAL LECTURE.

THE CHAIRMAN, in opening the meeting, said no doubt those present would remember that in March 1898 the Section lost its brightest ornament by the death of Dr. Ferdinand Hurter. In response to the generally expressed wish that something should be done to keep his memory alive, it was after much thought and deliberation determined that a memorial lecture should be instituted, to be delivered at the opening meeting of the Liverpool Section every second year.

Accordingly the first lecture was given in October 1899, by one whose name had long been famous in connection with chemical industry, Prof. George Lunge; and now after a lapse of two years they were to hear the second. Dr. Hurter, as was well remembered, had devoted a large amount of time and energy to the study of one of the most charming of all subjects, photography, and some of the results of his work were contained in the pages of the Society's Journal. The present lecture was to be given by one who had studied deeply and successfully the science of photography, and who scarcely needed introduction. He then called on Sir William Abney to deliver his lecture.

#### THE PHOTOGRAPHY OF COLOUR.

BY SIR WM. DE W. ABNEY, K.C.B., D.C.L., D.Sc., F.R.S.

WHEN I had the honour of an invitation to deliver the Hurter Memorial Lecture, I hesitated to accept it, for although I was an original member of your Society and one of the first members of its Council, I have for so many years ceased to belong to it that I could not convince myself I could find a suitable subject for it consistent with the objects of the Society. I remembered, however, an epoch-



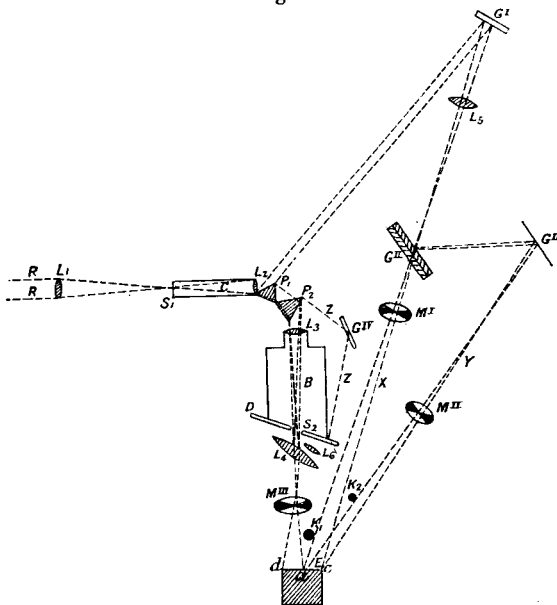
making paper by Dr. Hurter and Mr. Driffield in the Journal of 1891, and it struck me that photography, with which it dealt, was not excluded as an appropriate subject for the Hurter Memorial Lecture. This being so, I accepted the invitation, and all the more readily, as I believe I am right in saying that it was owing to some remarks of mine made on a public occasion at Widnes that Dr. Hurter was induced to turn his attention to certain scientific problems in photography then waiting solution. I cannot hope to give you a lecture at all of the same calibre as the first memorial lecture, but I shall endeavour to make clear to you the scientific principles which underlie the reproduction of coloured objects as coloured pictures by means of photography, or in short the photography of colour. I shall not touch on photography in natural colours, that is, the production of a photograph which by the direct action of light takes the hues of nature. At present any such photographs are either evanescent by exposure to light, or have to be viewed in such a manner that they are rather of scientific than industrial value.

I propose to treat the methods of colour photography, not historically, but from the scientific principles which underlie it, and which must be followed to obtain success in working it.

An instrument will be used in the lecture to-night which I devised for studying colour, and this must be briefly explained. Its object is to obtain pure monochromatic colours which will fill a fairly large space, and which space can also be filled by a mixture of any or all of the spectrum colours. The diagram shows the arrangement.

The rays R R, coming from the positive pole of the electric light, are collected by a lens, L, and an image of the crater thrown on the slit S. After passing through the collimator C, the rays emerged as parallel rays; part passed through the prisms P<sub>1</sub> and P<sub>2</sub>, were collected by a lens, L<sub>3</sub>, and a spectrum was formed on a slide, D (which will be more fully described), in which slits could be placed, and an image of the surface of the first prism was formed on the white surface of a cube, E, by means of the lens L<sub>4</sub>, so arranged that the image of one edge of the prism fell at a, the other edge falling outside d. The other beam which

Fig. 1.



passed through the collimator was reflected from the surface of the first prism to a mirror, G<sub>1</sub>, and passed through a lens, L<sub>5</sub>, then through a bundle of glass, G<sub>11</sub>, placed at an angle to the beam, and on to the surface d c of the cube, a rod, K<sub>1</sub>, being placed in its path, to secure that this white beam did not fall on a d, on which the colour mixture fell. The portion of the beam which was reflected from G<sub>11</sub> was again

reflected by G<sub>11</sub>, a silvered mirror, on to a c, a rod, K<sub>2</sub>, being placed in its path to prevent it falling on a d. In all three beams, sectors, M<sub>1</sub>, M<sub>11</sub>, and M<sub>111</sub>, were placed, to allow any or all to be reduced in intensity at pleasure. A small ray of light, Z, was allowed to pass beyond P<sub>2</sub>, and fell on a small mirror, G<sub>12</sub>, which reflected it on to the back of D, casting a shadow of a needle, N, fixed to B, the camera, on S, a scale at the back of D. L<sub>6</sub> is a lens of short focus which could be moved into a fixed position behind L<sub>4</sub> to throw an enlarged image of the slit on a scale placed below d c. By the insertion of a supplementary lens the spectrum can be thrown on the screen instead of a white patch being formed by the recombination of all the spectrum rays.

I must remind you that in the spectrum we have the only pure colours, that each coloured ray has a different wave length, and that from the limit of the red to the limit of the violet, not an octave of vibrations is embraced. Even with this limitation it might well be conceived that the retina which receives shocks from waves of such varying lengths must be a very complex piece of apparatus to distinguish one hue from the other. It would be so if there was an apparatus in it for receiving and sorting out each series of vibrations, but Young emphatically showed that in order to receive the sensation of any colour, only three pieces of visual apparatus were necessary, each of the three being devoted to one single sensation. In other words, he announced that any colour in nature would be imitated by mixing in varying proportions a red, a green, and a blue light. In 1861 Clerk Maxwell followed up Young's hypothesis and published a paper in which he gave curves of necessary intensities of the three different standard spectrum colours, which he chose, to match each colour in the spectrum. His method consisted of choosing three rays, one in the red, another in the green, and a third in the blue as standards and placing two slits in the spectrum in position of two of those rays, say, in the red and blue. The third slit he primarily placed in the third standard ray, and by opening or closing the slits he obtained a match with a white light both in colour and in intensity. He then altered the position of the green slit, and again by similar methods matched the same white. After the positions in the green had been exhausted, he again placed his slits in the standard positions as a check, matched the white and then altered the position of the red slit, and so on, always forming white and noting the width of the three slits on each occasion. He really observed through the slit of the spectroscope and illuminated the three slits in the spectrum with white light, and made the matches with the colour seen on the prism face. The principle is obvious for a spectroscope is reversible. From these results he obtained equations in terms of the width of the slits such as are given below. The numbers in brackets denote the scale number which applied to known colours in the spectrum. The standard colours he chose were at scale numbers, (24), (44), and (68).

$$\begin{aligned} 16 \cdot 1(28) + 25 \cdot 6(44) + 30 \cdot 6(68) &= \text{White} \\ 22 \cdot 0(32) + 12 \cdot 1(44) + 30 \cdot 6(68) &= \text{ } \\ 21 \cdot 7(24) + 10 \cdot 4(44) + 61 \cdot 7(56) &= \text{ } \\ 18 \cdot 0(24) + 31 \cdot 2(44) + 32 \cdot 3(72) &= \text{ } \end{aligned}$$

Eliminating the white from the equations he got the following results:—

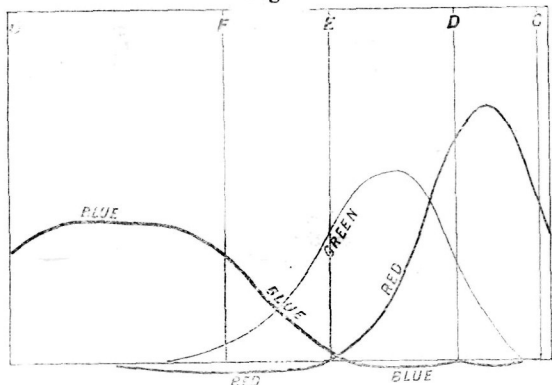
$$\begin{aligned} 44 \cdot 3(20) &= 18 \cdot 6(24) + 0 \cdot 4(44) + 2 \cdot 8(68) \\ 16 \cdot 1(28) &= 18 \cdot 6(24) + 5 \cdot 8(44) - 0 \cdot 1(68) \\ 22 \cdot 0(32) &= 18 \cdot 6(24) + 19 \cdot 3(44) - 0 \cdot 1(68) \\ 25 \cdot 2(36) &= 12 \cdot 2(24) + 31 \cdot 4(44) - 0 \cdot 8(68) \\ 41 \cdot 4(48) &= -2 \cdot 6(24) + 31 \cdot 4(44) + 3 \cdot 5(68) \\ 62 \cdot 0(52) &= -3 \cdot 4(24) + 31 \cdot 4(44) + 17 \cdot 4(68) \end{aligned}$$

(It may be remarked that on his scale C. was about 21½, D. 29½, E. 44 (a standard colour) F. 56, and G. 80. The standard red was a little yellower than the red lithium line and the blue about the same as the blue lithium line.)

The annexed diagram shows the results graphically. The equations which have in them minus quantities of standard colours are puzzling at first, but the difficulty will vanish after seeing a subsequent diagram. It should be

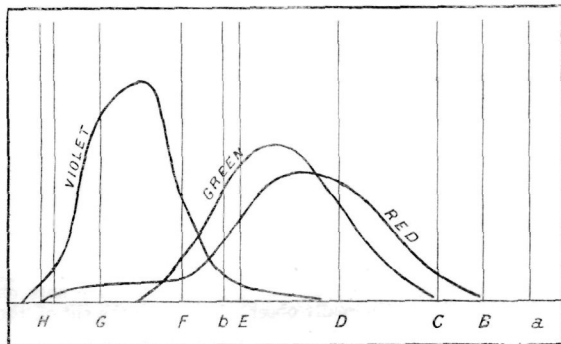
remembered that Maxwell's curves are given in terms of the width of the slit, and have only individually, and not collectively, any physical meaning.

Fig. 2.



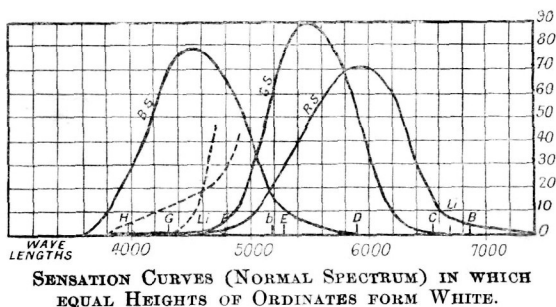
Koenig was the next observer to whom allusion need be made, and from his curves of the sensations we gather that the fundamental three sensations so overlapped that it was impossible to reproduce, with even pure spectrum colours, the intermediate colours except tinged with white.

Fig. 3.



Your lecturer was, I believe, the next investigator of the colour sensations, and whilst in details his results differ from Koenig's, in the main features they are identical. The curves which he obtained (given below) show the sensations when it is supposed that equal stimuli applied to each of the three make white light. Parenthetically it may be remarked that, on this supposition, the areas of the three curves must be equal, since the combination of the whole spectrum gives white light. It must be remembered that the curves show fundamental colour sensations, and are not colours.

Fig. 4.

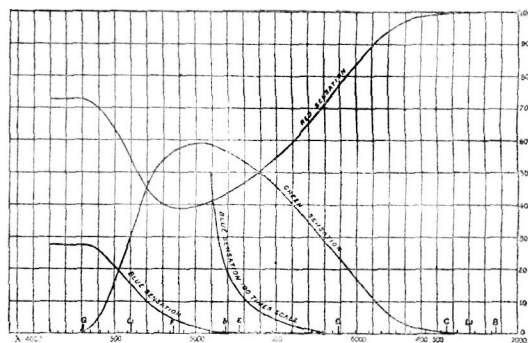


It will be noticed that the only rays which stimulate one sensation are in the red of the spectrum, and the red lithium line may be taken as typical of them. The colours

most nearly approaching the colours that would be given by stimulation of the green and blue sensations alone are rays close to the green magnesium line *b* and the blue lithium line of the spectrum. At these points they are the sensation colours to which a little white light has been added. These positions in the spectrum are not far from those empirically selected by Clerk Maxwell, and a study of the figure will show that his *minus* quantities are fully accounted for. Broadly, it may be stated that, in order to match an intermediate spectrum colour from the yellow to the blue by a mixture of any three colours, white light must be added to it. With the apparatus before you this can be effected and a match made in the laboratory, but it is beyond a lecture experiment possibility.

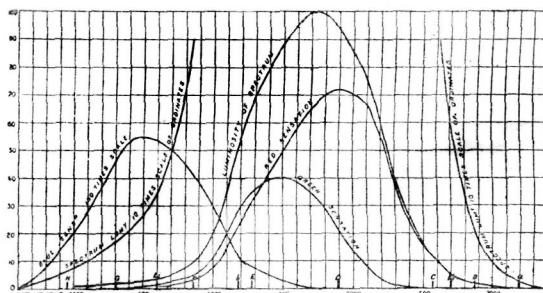
The next two diagrams show the percentage of sensations in the different colours and the actual luminosity (or brightness) of the spectrum divided up into sensation luminosities. It will be seen that the luminosities of the red and green sensations are very large compared with that of the blue.

Fig. 5.



PERCENTAGE COMPOSITION IN SENSATIONS OF THE SPECTRUM COLOURS, NORMAL SPECTRUM.

Fig. 6.



NORMAL LUMINOSITY CURVES IN WAVE-LENGTHS (ELECTRIC LIGHT) DIVIDED INTO SENSATION LUMINOSITIES.

Table I. shows the actual measures from which the Figs. 4, 5, and 6 were constructed.

Column I. shows the wave-lengths; Columns II., III., and IV. the percentage luminosity of the sensations; Column V. the luminosity of the spectrum; Columns VI., VII., and VIII. the luminosity of the sensations in the normal spectrum. The approximate areas of the three curves shown in Columns VI., VII., and VIII. are 747.6, 344.3, and 5.3 for the red, green, and blue sensations respectively, and the equation for white is—

$$RS \quad GS \quad BS \quad W \\ 68.14 + 31.38 + 0.48 = 100.$$

Columns VII. and VIII. have to be multiplied by  $\frac{68.14}{0.48}$ , i.e., by 2.17 and 142 respectively, to give equal areas with the red sensation curve in Column VI.



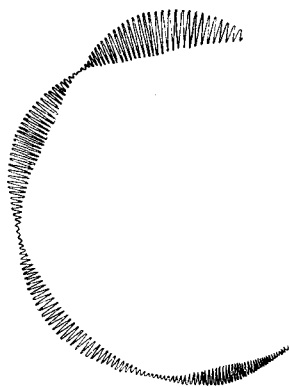
TABLE I.

I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.
λ.	Percentage Luminosity.			Luminosity of Spectrum.	Sub-Division of Spectrum Luminosity into Luminosities of			Equal Ordinates to give White.		
	RS.	GS.	BS.		RS.	GS.	BS.	RS.	GS.	BS.
7100	100	0	0	0.5	0.5	..	..	0.5	..	..
7000	100	0	0	0.9	10.9	..	..	0.9	..	..
6900	100	0	0	1.6	1.6	..	..	1.6	..	..
6800	100	0	0	3.2	3.2	..	..	3.2	..	..
6700	100	0	0	6.0	6.0	..	..	6.0	..	..
6600	99.8	0.2	0	10.0	9.9	0.1	..	9.9	0.22	..
6500	99	1.0	0	17.0	16.8	0.2	..	16.8	0.44	..
6400	98	2.0	0	26.0	25.48	0.52	..	25.48	1.13	..
6300	97	3.0	0	41	39.77	1.23	..	39.77	2.07	..
6200	94	6.0	0	59	55.46	3.54	..	55.46	7.68	..
6100	89.5	10.5	0	75	67.13	7.87	..	67.13	17.08	..
6000	83.3	16.7	0	85	70.81	14.19	..	70.81	30.79	..
5900	77.5	22.5	0	93	72.08	20.92	..	72.08	45.30	..
5800	71.3	28.7	0.005	99	70.58	28.42	0.005	70.58	61.67	0.7
5700	65.3	35.0	0.01	100	65.00	35.00	0.01	65.00	75.95	1.4
5600	59.3	40.5	0.023	95	56.52	38.46	0.02	56.52	83.45	2.9
5500	54.3	45.7	0.040	89	48.33	40.64	0.035	48.33	90.10	4.9
5400	49.7	50.2	0.065	80	39.78	40.16	0.052	39.78	87.15	7.38
5300	47.2	52.7	0.11	70	33.04	36.58	0.077	33.04	79.37	10.93
5200	33.3	56.2	0.18	56	24.00	29.90	0.099	24.00	64.88	14.10
5100	41.2	58.4	0.42	35	14.42	20.43	0.147	14.42	44.33	20.87
5000	39.3	59.0	1.6	18	7.09	10.61	0.300	7.09	23.02	40.89
4900	39.0	57.5	3.5	11	4.48	6.50	0.385	4.48	14.10	54.60
4800	40.0	54.0	6.0	7.5	3.00	4.05	0.456	3.00	8.79	64.75
4700	45.0	44.8	10.2	5.0	2.25	2.24	0.510	2.25	4.88	71.00
4600	54.5	30.0	15.5	3.5	1.90	1.06	0.542	1.90	2.30	78.5
4500	63.5	16.0	20.5	2.7	1.71	0.44	0.553	1.71	0.95	78.5
4400	69.8	4.5	25.7	2.1	1.47	0.09	0.540	1.47	0.20	78.68
4300	72.5	0	27.5	1.7	1.23	0	0.470	1.23	0	69.74
4200	72.5	0	27.5	1.3	0.94	0	0.357	0.94	0	50.69
4100	72.5	0	27.5	1.0	0.72	0	0.275	0.72	0	39.05
4000	72.5	0	27.5	0.75	0.64	0	0.206	0.64	0	29.25
3900	72.5	0	27.5	0.50	0.36	0	0.137	0.36	0	19.45
3800	72.5	0	27.5	0.25	0.18	0	0.068	0.18	0	9.22

Columns IX., X., and XI. shows the ordinates of these areas, which will then be equal. As the areas, when equal, give white, the ordinates at any point where equal will give white.

It is well that we should pause and consider how it arises that a variety of different vibrations can affect one piece of apparatus which can be set in oscillating motion. We may connect two pendulums, or, rather, cause them to react one upon another. We may have one of them vibrating with a set swing which remains unalterable in amplitude, whilst the other may be capable of increasing or diminishing its length of swing. When the two pendulums are of different length and both are vibrating, if that which has a fixed swing is so connected with the other that it gives it a blow during each swing, the blow will tend to increase the swing, and gradually the phase of swing will alter, so that when one begins to descend the arc described the other will be ascending. The swing of the latter pendulum will then diminish, and finally cease altogether, when it will again begin to move, and once more reach its maximum

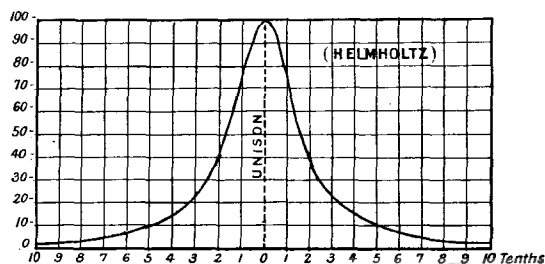
Fig. 7.



amplitude. Such a motion is to be seen in Fig. 7, which is one drawn by a modification of the apparatus used for Lissajou's figures. The increase and decrease of the swings of the second pendulum are well seen.

The same kind of motion is found in a resonator. Helmholtz calculated the loudness of a sound which would be given out from it when set in motion by notes in tune and out of tune with it, and he obtained a curve which is the same as that which would be got by lengthening or shortening the pendulum which was influenced by the oscillations of the pendulum having a fixed length and swing. The maximum ordinate is taken as 100, and represents the loudness of the note given out by the resonator when a note in tune with it was struck. The following table and figures give his results. They show that it is a symmetrical figure on each side of the maximum when the supposition is made that the notes struck were all equally loud. Where the notes struck are of gradually diminishing loudness the figure would be altered, and if for "sound" you read "light," and for "resonator" read "sensation apparatus," the curve would be altered as shown in Fig. 9, since the energy of the spectral rays increases from the violet to the red.

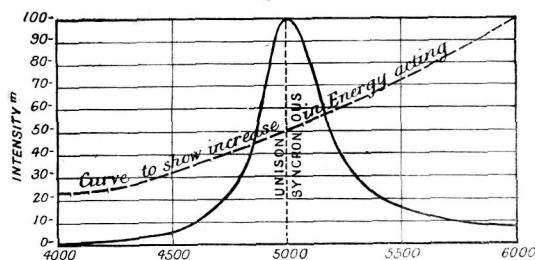
Fig. 8.



INTENSITY OF VIBRATION OF RESONATOR TO NOTES IN TUNE AND OUT OF TUNE WITH THE MEMBRANE.



Fig. 9.



AMPLITUDE OF VIBRATION OF AN ATOM ACTED UPON BY WAVES OF INCREASING LENGTH AND INCREASING ENERGY (CALCULATED).

The following table applies to the curve shown in Fig. 8.

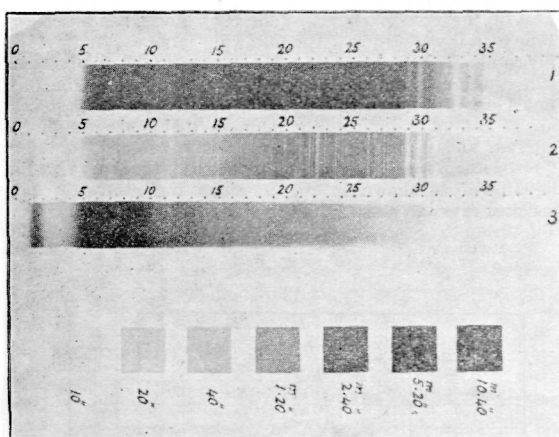
TABLE II.

Difference of Pitch.	Intensity of Vibration in Resonator.
0.0	100
0.1	74
0.2	41
0.3	24
0.4	15
0.5	10
0.6	7.2
0.7	5.4
0.8	4.2
0.9	3.3
1.0	2.7

Now I have been somewhat particular in bringing this by-subject before you because what applies to the sound waves impinging on a resonator applies not only to light waves when they act on the visual apparatus, but also when they act on the atoms of the molecules of the silver salt which form the sensitive substance in a photographic film.

Adopting as unchallenged the chemical theory of the formation of the photographic image, we can show how the proportion of the number of molecules that are decomposed in a given time by the "swinging out" of the sphere of molecular influence of the halogen atoms varies through the different average amplitudes which are caused by the blows of the ether waves of different oscillation frequency.

Fig. 10.

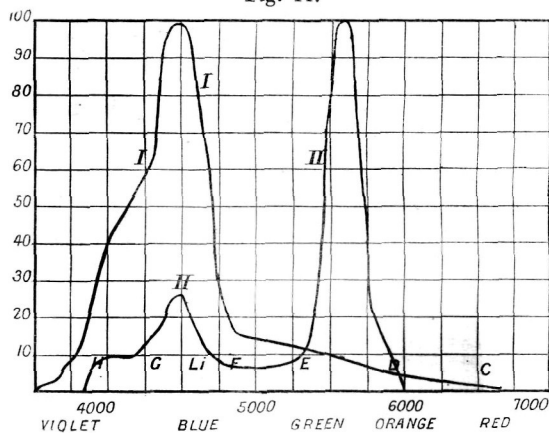


If we take pure bromide of silver as an example, and expose it on a photographic plate to the spectrum for a given time, each ray of a different wave length will decompose a different number of the molecules. The number of molecules so decomposed can be estimated by the amount of silver reduced on development, and the amount of silver in its turn can be estimated by the amount of obstruction

which it offers to the passage of light through it, supposing always that some known scale of obstruction can be applied.

Such a scale of obstruction can be made by exposing small portions of the same plate to a light of fixed intensity for varying known times. Fig. 10 shows the negative of the solar spectrum on a Cadett spectrum plate. When the spectrum and this scale are developed together, and the obstruction to light measured in the latter, we can apply the obstruction offered by different parts of the developed spectrum image to the obstruction curve given by the scale, and obtain an estimate of the relative number of molecules decomposed by each separate ray. Curve I. in the diagram (Fig. 11), taken from a similarly exposed bromide plate, shows that, allowing for the increase in spectrum energy from the red to the blue, the curve obtained is not unlike that which Helmholtz calculated for the resonator.

Fig. 11.



We see from the figure that the maximum number of molecules is decomposed by the blue ray near the lithium line and that it extends beyond the place where the blue visual sensation starts near the green. If an exposure of a plate be made to a view through a cell of sulphate of quinine (which cuts off the ultra violet rays) it may be taken that the print from the negative will closely represent what such view would appear to a person possessed only of the blue colour-sensation, and it may be said that such persons do exist.

Up to this point I have been endeavouring to clear the ground for a further look into the question of the photography of colour.

There are two methods of representing the pictures of coloured objects to the eye. The first is of course as prints, and the second as images thrown from a lantern on the screen. The second is the easier to deal with first, for reasons which will be apparent as we proceed. This method, again, is capable of subdivision (a) where the colours are produced by mixed lights, and (b) where produced by absorption of colours in a film. I propose to deal with (a) first.

We have already seen that to produce any colours only three sensations are necessary, and if we could have the three pure colours stimulated by the three sensations these colours would be theoretically the perfect ones with which the mixed lights ought to be made. This is impossible, but with this limitation I propose first of all to consider what is the theoretically perfect plan of producing a coloured picture on the screen by the superposition of three coloured images.

We must choose the three spectrum colours which are theoretically best, and which, when combined, will give tints which are as nearly as possible unmixed with white. These three colours are the colours which nearest approach to the three sensations, and, as before said, are best represented by the red lithium line, the green (b) magnesium line, and the blue lithium line of the spectrum. Taking these for our starting point, we have to consider what should be the intensity of these three colours. It may be taken for



granted that, if we have three transparencies whose images are to be combined on the screen, at one part they will have to represent a white, and that the white will be represented in each transparency (positive) as bare glass. The intensity of the three colours must therefore be such as will when mixed, without any obstruction to any of the three rays, give white light.

The colours and their intensities are thus fixed.

The next question to deal with is the theoretically perfect negative from which the positives are to be produced. Now for the purposes of this lecture I have taken the above-named three colours of the spectrum and matched the intermediate spectrum colours by their mixture and obtained the percentage composition of all the spectrum colours in the terms of these three colours, omitting the white added. The table and Figs. 12, 13, and 14 show the results.

TABLE III.

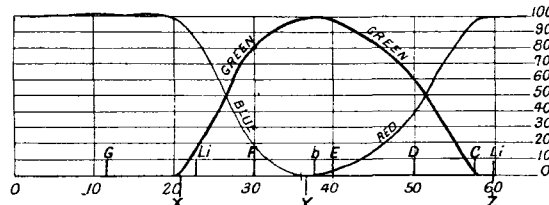
Table of Percentages and Luminosities of Colours in the Prismatic Spectrum.

Scale No.	Wave Length.	Percentage Composition.			Spectrum Luminosity.	Luminosity.			Equal Amounts of Colours make White.		
		Red.	Green.	Blue.		Red.	Green.	Blue.	Red.	Green.	Blue.
64	7,217	100	..	..	0.5	0.5	..	..	1.05	..	..
62	6,937	100	..	..	2.0	2.0	..	..	4.2	..	..
60	6,728	100	..	..	7.0	7.0	..	..	14.7	..	..
58	6,520	100	..	..	21	21	0	..	48.2	0	..
56	6,330	85	15	..	50	42.5	7.5	..	99.2	7.5	..
54	6,152	69	31	..	80	55.2	24.8	..	116	24.8	..
52	5,996	50	50	..	96	48	48	..	101	48	..
50	5,850	36	64	..	100	36	64	..	75.5	64	..
48	5,720	25	75	..	97	24.2	72.8	..	50.8	72.8	..
46	5,596	17	83	..	87	14.8	72.2	..	31.1	72.2	..
44	5,481	11	89	..	75	8.25	66.75	..	17.3	66.75	..
42	5,378	6	94	..	62.5	3.75	58.8	..	7.9	58.8	..
40	5,270	2.5	97.5	..	50	1.25	48.6	..	2.6	48.6	..
38	5,172	0.5	99.5	..	36	0.2	35.8	..	0.5	35.8	0
36	5,085	..	99	1	24	..	23.76	0.24	..	23.76	10.4
34	5,002	..	96	4	14.2	..	13.63	0.57	..	13.63	21.7
32	4,924	..	91	9	8.5	..	7.785	0.765	..	7.785	33.1
30	4,848	..	82	18	5.7	..	4.674	1.26	..	4.674	44.4
28	4,776	..	68	32	4.0	..	2.72	1.28	..	2.72	55.4
26	4,707	..	45	55	2.9	..	1.26	1.64	..	1.26	68.1
24	4,639	..	25	75	1.9	..	0.48	1.42	..	0.48	81.4
22	4,578	..	11	89	1.4	..	0.16	1.25	..	0.16	94.1
20	4,517	..	0	100	1.1	..	0	1.10	..	0	97.3
18	4,459	..	..	100	0.86	..	..	0.86	..	..	37.3
16	4,404	..	..	100	0.70	..	..	0.70	..	..	30.3
14	4,349	..	..	100	0.56	..	..	0.56	..	..	24.2
12	4,296	..	..	100	0.45	..	..	0.45	..	..	19.5
10	4,245	..	..	100	0.34	..	..	0.34	..	..	14.7
8	4,197	..	..	100	0.26	..	..	0.26	..	..	11.3
6	4,151	..	..	100	0.18	..	..	0.18	..	..	7.8
4	4,106	..	..	100	0.14	..	..	0.14	..	..	6.1
2	4,060	..	..	100	0.10	..	..	0.10	..	..	4.3
0	4,018	..	..	100	0.6	..	..	0.6	..	..	3.8

If we were to translate the three colours into their sensation equivalents, Fig. 5 and Fig. 12 would be identical.

Fig. 12.

PERCENTAGE COMPOSITION OF SPECTRUM COLOURS IN LUMINOSITIES OF RED, GREEN, AND BLUE.



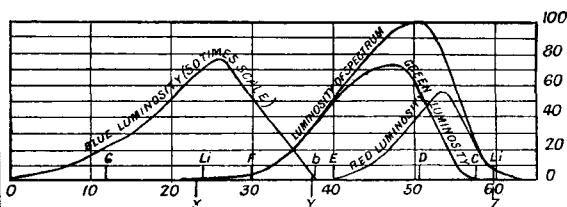
Scale of prismatic spectrum.  
(X, Y, Z = position of slits.)

Knowing the luminosity of the different rays, the luminosity curve of the spectrum can be converted into the luminosity of the three colours (Fig. 13), and from these we can calculate the scale of ordinates to make the areas of the three luminosity curves equal (Fig. 14), which is diagrammatically saying that the three naked colours will make white. The ratio of the areas of the colour luminosities into which the total spectrum luminosity was divided, of course, would be equally well found (and indeed was found) by measuring the luminosity of the colours coming through the three slits in the spectrum which formed white light. The ratio of

the areas to one another was found to be identical with the ratio of the luminosities coming through the three slits.

Fig. 13.

LUMINOSITY OF SPECTRUM DIVIDED INTO RED, GREEN, AND BLUE LUMINOSITIES.



Scale of prismatic spectrum.  
(X, Y, Z = position of slits.)

From the figure it will be seen that the spectrum is divided into two parts, the point of separation being the spot from which the green light is selected. One of the portions is occupied by the red luminosity and the other by the blue, whilst a curve of green luminosity overlaps both the others, finishing at the red and blue lithium lines, which are the colours selected to represent the red and blue lights. The opacities of the negatives at every point must be proportional to the luminosity of the three sensations, and from such negatives, positives should be taken in which the transparency of deposit is similarly proportional. In

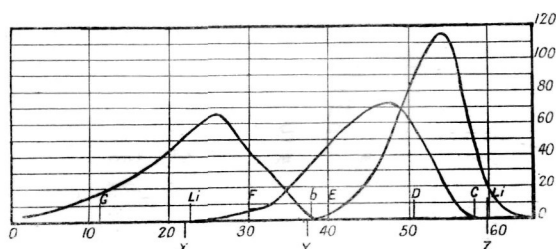




practice, it is here where Hurter and Driffeld's work gives aid, and their researches have much simplified this problem.

Fig. 14.

CURVES OF COLOURS WHEN EQUAL WIDTH OF SLITS FORMS WHITE.

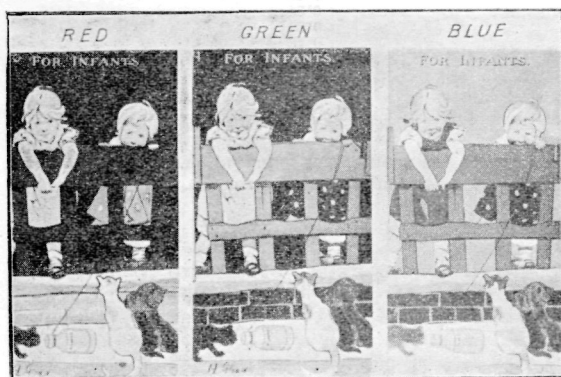


Scale of prismatic spectrum.

(X, Y, Z = place of slits in spectrum.)

Now I will try and represent what I have so far endeavoured to explain in a simple manner, so that it may impress itself on you. First of all, the three slits are placed in the spectrum in the theoretical positions, and a patch of white light is made by their mixture. I then place in front of the slits three lenses of equal foci, instead of the single lens  $L_4$  (Fig. 1), and form three patches of red, green, and blue. In contact with the face of the prism (see Fig. 1) I place a coloured transparency, and we have three images of the picture in different colours (Fig. 15). I

Fig. 15.

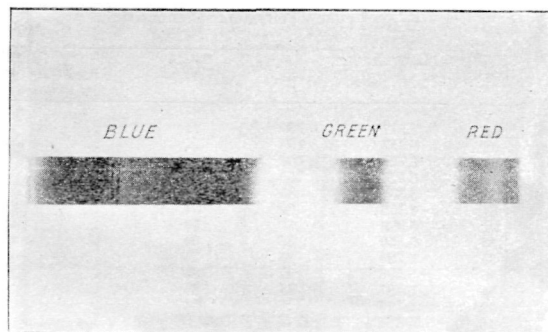


withdraw the three lenses and substitute the one for them, and we see the coloured image. I withdraw the slits, and we have the image in white light, more brilliant than before, but still the same in colour. I repeat this with another coloured image, with the same results. Thus, for three-colour photography we have to see that the three unobstructed colours sent through the three positives make white, and then the three images formed of the same colours and superposed will give a correct representation, or nearly so, of the object.

The subject, so far, has been treated from a theoretical standpoint, and not from a practical or practicable basis. The spectrum colours are not available for ordinary commercial work, and means have to be taken to get colours from other sources than that of the spectrum. Ives, by his intuitive genius, and by his study of Maxwell's curves, produced three colours for illuminating his three positives closely approximating to the colours which present theory indicates as correct. They are produced by transmitting white light through coloured screens or filters. The red is as pure a red as can be obtained—not quite as red,

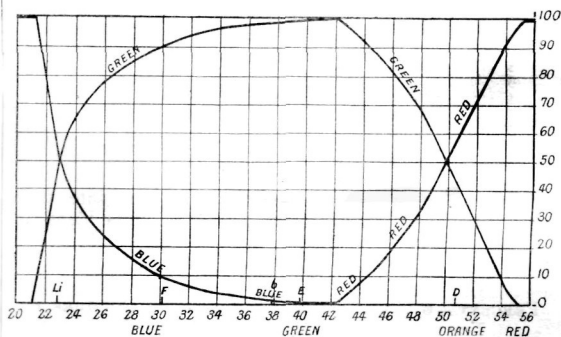
perhaps, as the lithium true red, but still not far off. His green is a green which matches a spectrum colour a little nearer the red than that given by the study of the colour sensations. By this choice the reds, oranges, and yellows are less mixed with white than they would have been, but the blues and blue-greens suffer proportionally. His blue matches one close to the blue lithium line, and allows no red to pass, which is most frequently a fault of blue screens. Their spectra (Fig. 16) show that the two

Fig. 16.



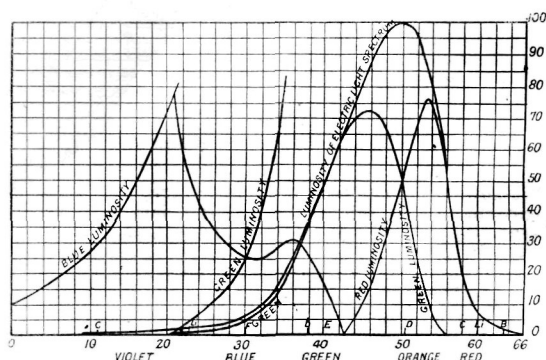
first occupy but small spaces in the spectrum. Their luminosities are chosen so that when the light passes through each, unobstructed, to the screen, the resulting mixture is white. The curves in Fig. 17 show the colour percentage

Fig. 17.



PERCENTAGE COMPOSITION OF SPECTRUM COLOURS IN LUMINOSITIES (IVES' VIEWING SCREENS).

Fig. 18.



LUMINOSITY OF SPECTRUM IN THE LUMINOSITIES OF IVES' VIEWING SCREENS.





of each which goes to make up the different colours of the spectrum, and Fig. 18 shows the spectrum luminosity divided into the luminosity of the three colours.

The following tables apply to Figs. 17 and 18 :—

TABLE IV.

"Colour at Scale.	Red. Green.		Luminosity of Spectrum.	Luminosity of Components.	
				Red.	Green.
55.4 matches	100.0 + 0.0		58.0	58.0 + 0.0	
55.0 "	98.0 + 2.0		65.0	63.7 + 1.3	
54.0 "	93.0 + 7.0		80.0	74.4 + 5.6	
53.0 "	83.0 + 17.0		90.0	74.7 + 15.3	
52.0 "	72.0 + 28.0		96.0	69.1 + 26.9	
51.0 "	62.0 + 38.0		99.0	61.4 + 37.6	
50.0 "	52.0 + 48.0		100.0	5.2 + 48.0	
49.0 "	42.0 + 58.0		99.0	41.6 + 57.4	
48.0 "	32.0 + 68.0		97.0	31.0 + 66.0	
47.0 "	24.0 + 76.0		92.5	22.2 + 70.3	
46.0 "	18.0 + 82.0		87.0	15.7 + 71.0	
45.0 "	12.5 + 87.5		82.0	10.2 + 71.8	
44.0 "	8.0 + 92.0		75.0	6.0 + 69.0	
43.0 "	3.5 + 96.5		68.0	2.4 + 65.6	
41.3 "	0.0 + 100.0		64.0	0.0 + 64.0	

Colour at Scale.	Green. Blue.		Luminosity of Spectrum.	Luminosity of Components.	
				Green.	Blue.
42.0 matches	99.9 + 0.1		62.5	61.9 + 0.06	
41.0 "	99.65 + 0.35		57.0	56.8 + 0.02	
40.0 "	99.35 + 0.65		50.0	49.67 + 0.33	
39.0 "	99.0 + 1.0		42.5	42.08 + 0.42	
38.0 "	98.6 + 1.4		36.0	35.48 + 0.52	
37.0 "	98.0 + 2.0		29.5	28.91 + 0.59	
36.0 "	97.4 + 2.6		24.0	23.8 + 0.62	
35.0 "	96.7 + 3.3		18.2	17.60 + 0.60	
34.0 "	95.9 + 4.1		14.2	13.62 + 0.58	
33.0 "	95.0 + 5.0		10.5	9.97 + 0.53	
32.0 "	94.0 + 6.0		8.5	7.89 + 0.51	
31.0 "	92.5 + 7.5		7.0	6.47 + 0.53	
30.0 "	90.3 + 9.7		5.6	5.06 + 0.54	
29.0 "	88.5 + 11.5		4.8	4.25 + 0.55	
28.0 "	86.5 + 14.5		4.0	3.42 + 0.58	
27.0 "	81.5 + 18.5		3.5	2.85 + 0.65	
26.0 "	77.0 + 23.0		3.0	2.31 + 0.69	
25.0 "	71.5 + 28.5		2.6	1.86 + 0.74	
24.0 "	64.5 + 35.5		2.3	1.48 + 0.82	
23.0 "	50.0 + 50.0		1.9	0.95 + 0.95	
22.0 "	25.0 + 75.0		1.65	0.42 + 1.23	
21.2 "	0.0 + 100.0		1.55	0.00 + 1.55	

The following are the remaining luminosities of the spectrum, which are represented by the red and blue of the two media :—

TABLE V.

Scale No.	Luminosities.	Scale No.	Luminosities.
	Red.		Blue.
64	0.00	14	0.80
63	1.00	13	0.70
62	2.00	12	0.60
61	4.00	11	0.55
60	7.00	10	0.52
59	12.50	9	0.50
58	21.00	8	0.48
57	33.00	7	0.45
56	50.00	6	0.40
	Blue.	5	0.37
21	1.50	4	0.35
20	1.40	3	0.27
19	1.35	2	0.5
18	1.20	1	0.23
17	1.10	0	0.21
16	1.00		
15	0.90		

These luminosities (which are, of course, the luminosities of the spectrum itself) are shown in Fig. 18, as the continuation of the red and blue curves respectively.

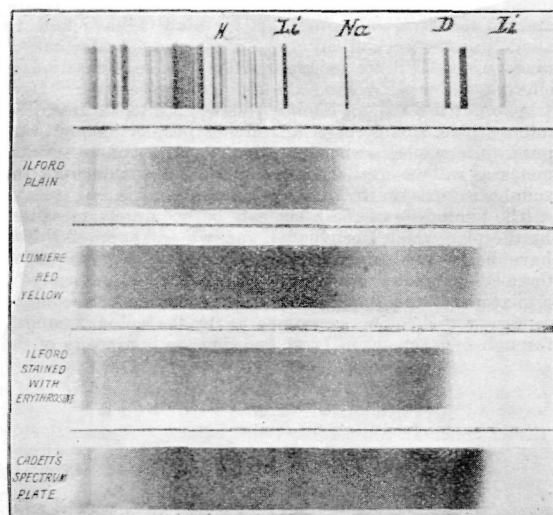
Table VI. gives the wave-length values of the scale numbers of the spectrum, and applies to diagrams 12, 13, 14, 17, and 18.

TABLE VI.

Scale No.	Wave-Length.	Scale No.	Wave-Length.
64	7217	32	4924
62	6957	30	4848
60	6728	28	4776
58	6520	26	4707
56	6330	24	4639
54	6152	22	4578
52	5996	20	4517
50	5850	18	4459
48	5720	16	4404
46	5596	14	4349
44	5481	12	4296
42	5373	10	4245
40	5270	8	4197
38	5172	6	4151
36	5085	4	4106
34	5002		

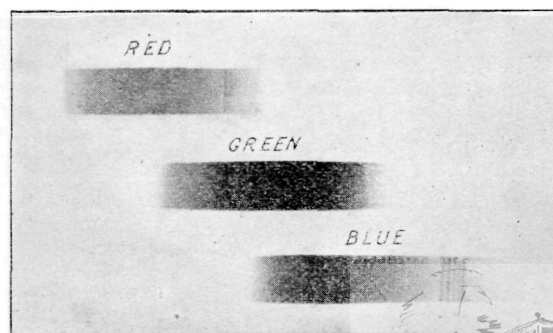
The practical question of obtaining the negative remains. Now I may at once state that except with the ordinary bromide plate, which with very prolonged exposure is sensitive into the red, no theoretically perfect negatives are possible so far. The photographic plates (Fig. 19) which can be impressed by the red through the use of dyes in the films have curves of sensitiveness so jagged that no absorbing screen has been found which can follow the jaggedness and cut off exactly the right proportions. See also curve II., Fig. 11, which is that of a bromide plate dyed with eosine.

Fig. 19.



With the ordinary bromide plates, in which the curve of sensitiveness shows no contraflexures (see curve I., Fig. 11),

Fig. 20.



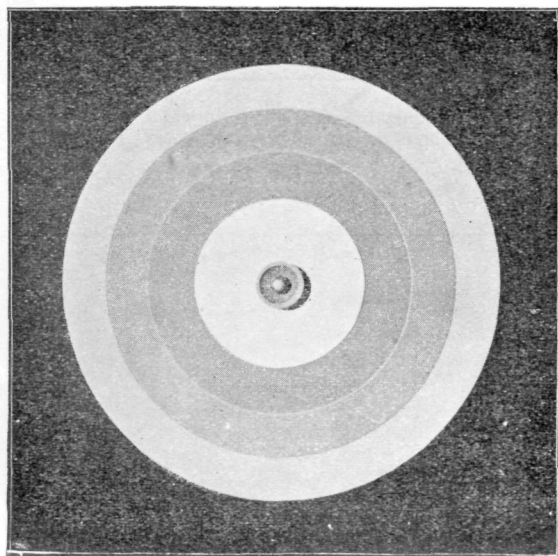
proper absorbing screens can be found, but the exposure for the red negative is so prolonged that their use becomes impracticable. I have myself photographed a spectrum with such a plate with proper screens, which reproduced very accurately the spectrum (Fig. 20). The absorbing screen to cut off the different parts of the spectrum is the crux, and it is evident that even with plates sensitive to the red and yellow (see Fig. 19) a compromise with theoretical perfection has to be made, owing to the irregularity in the curves of sensitiveness alluded to.

Objects in nature are seen by reflected light, and all objects so seen not only show their own local colour and light and shade, but they also reflect a fair proportion of white light. The very small percentage of white light, therefore, which is found in the mixture of the colours coming through the screens becomes inappreciable; and if that local colour be rendered correctly, any small percentage of white light which is in excess becomes negligible.

Again, colours of objects are *not* pure spectrum colours, but if examined by the spectroscope are found to occupy a considerable space in the spectrum, and this enables a system of compensation, which the irregularity of the sensitiveness of the plate requires, to be brought into play, for it must be recollected that the colour *as a whole* has to be photographed. For example, an orange colour will show on spectroscopic analysis that it is made up of a band of colours lying from the extreme red end of the spectrum up to the yellow. Now a plate may be very sensitive to the yellow and much less sensitive to the red. If, then, a screen be found which admits such a quantity of yellow that the deficiency of sensitiveness to the red is made up by the excess sensitiveness to the yellow, the integral result may be the same as if the plate were properly yellow and red sensitive. Bearing this in mind (I have here to allude to some work of my own), a colour apparatus can be made which will let us know when the compensation is effected without invoking the aid of the spectrum. If we take an intense red, an intense yellow, an intense green, an intense blue, and perhaps an intense purple pigment, and pure white paper, we can construct such a colour sensimeter as will be available for practically working out the suitable screens on the principle of compromise.

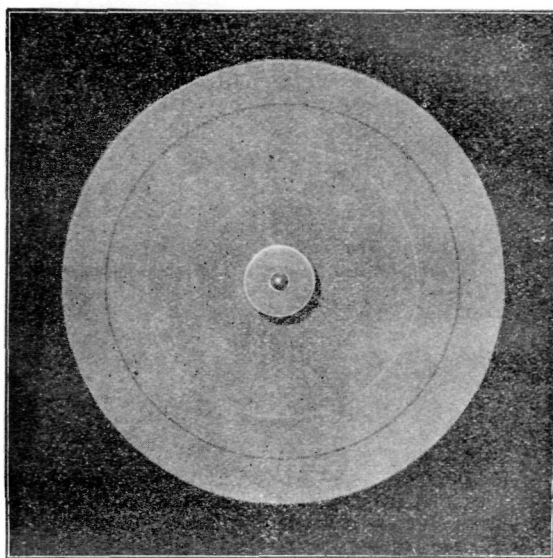
The luminosity of these six can be accurately measured by the plan which I originated, and we will suppose this to have been done and that they have to be reproduced by three-colour photography. If we match them severally by a mixture of the spectrum colours which most accurately represent the colour sensations or by the colours coming through such screens as Ives', knowing the luminosity of the

Fig. 21.



three colours which make the mixture, we can lay down that the luminosity, say of the yellow, is made up of so many parts of red and so many parts of green luminosity, and the purple of red and blue luminosity. Suppose we want to find the screen through which to take the "red" negative, we first must calculate how much the luminosity of the yellow must be reduced so that the red component in it is equal to that in the purple. We must in fact reduce the luminosity of the red, yellow, and white till all the red components are of equal value. Thus, if the luminosity of the yellow is 80, 40 of which are red and 40 green, and of the purple, 2 red and 2 blue, we must reduce the yellow to  $\frac{2}{40}$ ths of its luminosity, and then the red in each will be the same, and so on. To effect this the colours are placed as rings on a rotating disc, part of each ring or annulus is covered up by a dead black (the light reflected from it being taken into account). When the disc rotates we see that the luminosities of the rings are very different; but, notwithstanding, the red components in every ring are the same.

Fig. 22.

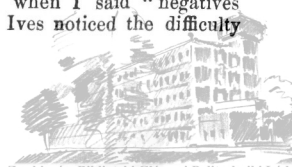


For the "red" negative to be correct it should show all the rings as equally dense. Without a screen we shall get very different results from this (Fig. 21), but by placing an orange screen in front of the lens we shall see it is better, and by gradual alteration of the colour or by using a parti-coloured diaphragm we shall at length obtain one which will effect what we require (Fig. 22). The same method is gone through for a green and a blue screen. The screens will vary according to the kind of plate we are using.

I have here another form of colour sensimeter, but based exactly on the same principles. Instead of pigments we use glass, the luminosity being reduced by means of rotating apertures placed in front or by neutral tint pigment. The glass is very convenient, as there is no white reflection to mix with the true colour as there is with pigments.

Having obtained these light filters we can photograph any object with a knowledge that we shall get negatives which are very nearly correct. From these negatives transparencies are made, and these have to be illuminated with the light passing through the three colour screens and the images superposed.

It might appear, after what has been said, that all the difficulties in taking the negatives (and from them the positives) are surmounted when we have got the light filters (screens) in the manner described, but that is not quite the case, for I entered a "caveat" when I said "negatives which are very nearly correct." Ives noticed the difficulty



of getting the red negatives of the same gradation as the other two. Mr. Chapman Jones and your lecturer each independently investigated the action of light of different colours in producing gradation in a plate, and both came to the conclusion that the gradation given by the red rays was steeper than that given by the green or the blue. Your lecturer used the rays of the spectrum in his investigations, and, thanks to the method of graphically showing gradation introduced by Hurter and Driffield, he was enabled to ascertain by a long series of experiments that the gradation increased for rays on each side of that ray of the spectrum which had the maximum effect on the simple salt of silver which was used in the plate.

Fig. 23.

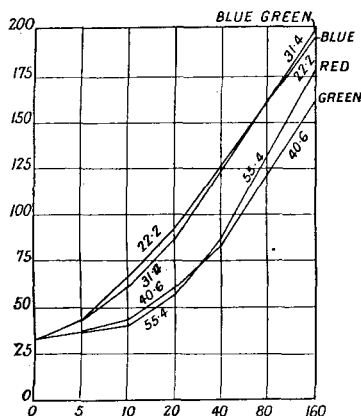
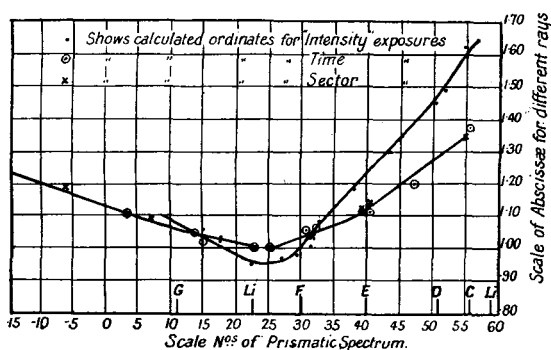


Fig. 23 will enable you to see the gradation given with four rays of the spectrum, and Fig. 24 will show the general variation in gradation which is given throughout the whole spectrum. It will be noticed that while the gradation given for the blue and the green are not far different to one another, that given by the yellow to the red is much

Fig. 24.



steeper. The reason of this is not quite apparent at the present time, but I think it can be explained by the theory which applies to the vibration of an atom when increased by shocks of ether waves, as described previously. If, eventually, it is found that such is indubitably the case, the phenomenon observed will be one which may give us a further insight into, and a measure of, the viscosity of the ether, and form a contribution to molecular physics of a very significant character.

In practice the shades, say, of orange and yellow must be a little bit untrue, for if a certain depth of yellow is correctly produced, it is evident that in some cases the lighter shades will be a trifle either too red or too green.

In Fig. 24 it will be noticed there are two curves, one more concave than the other. The former represents the alteration in gradation when the intensity of the light is altered whilst the time of exposure remains the same. This

is the curve which would apply to camera images, where the time of exposure of the image to the plate is, of course, the same. The other curve is that given by the measures where the time of exposure is altered and the intensity of the light remains the same.

[Some of Mr. Ives's slides were then projected by a triple lantern.]

This may be called the first important method of the photography of colour, and very beautiful it is.

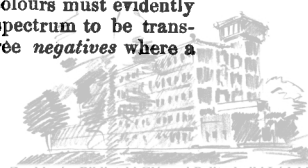
There is a modification of Ives' process which must be mentioned, and it is a very remarkable proof of the practical use that a man of science may make of his theoretical knowledge in its application to an art. Ives requires three negatives, it will be remembered, to produce a triple projection, but Prof. Joly has compressed the three negatives into one. He rules on a glass three sets of fine lines of appropriate colours touching one another. One set is orange, another greenish-blue, and the third blue. Each line has a breadth of some  $\frac{1}{100}$ th of an inch, and they follow and touch one another in the order given. The colours are the same as those found for Ives' screens for taking the negatives when the same plate is used. When such a ruled surface is placed next a plate the camera image has to pass through the lines to the sensitive surface, and we have one-third of the whole negative formed by light of the colour passing through each of the three-line series respectively. If we back the transparency obtained from this negative with a plate covered with lines similarly spaced, but which are red, green, and blue, and cause the red line to coincide with the lines of the transparency which, in the negative, were taken through the orange lines, the green with the green-blue, and the blue with the blue, and then send a beam of white light through the transparency to form an image on a screen, we have the three transparencies of Ives in one, only shown in lines. The eye makes the small portions next one another blend together, and the effect is to see the object or view in colours. The brightness of the picture must, however, be inferior to that given by triple projection.

There are several difficulties to cope with in this process. The first is to get exact equality in the width and spacing of the lines in both the "taking" and the "viewing" screens, and the second is to make all the lines of the exact depth of colour which is required. These difficulties Prof. Joly overcame, and some of his lantern slides are shown on the screen.

It should be noted that an American, Macdonough, claimed priority in the process; but I have no time or inclination to enter into the discussion of the rival claims.

Having shown how colour can be produced by triple projection, we can next proceed to see how projection can be made by the superposition of three coloured transparent films—a process which was explained by Mr. Ives and since worked out most successfully and on a commercial scale by Mr. Sanger Shepherd. The colour on a screen by mixing beams of white light passing through two or more coloured media is by no means the same as that obtained by sending one beam of white light through the same coloured media.

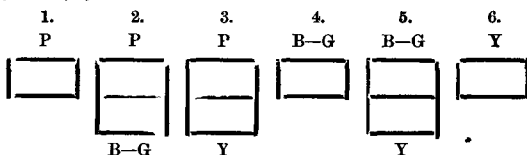
We have seen that the mixture of white light, after passing through a red, a green, and a blue medium respectively, can be made to give white. If the same coloured media, superposed one over the other, be interposed in the path of a beam of white light, they will cut off all light, or transmit only a dim light which has no dominant colour. In other words, for white we should get black. We have also seen that a mixture of green and red light gives yellow; if white light is transmitted through superposed media of these colours they would give a dark colour, tinged, perhaps, with one or other of the two colours red or green. Theoretical considerations show that the colours chosen must be totally different for superposition to those chosen for triple projection. What are these colours? is the question to be answered. (It may be stated once for all that the negatives which are suitable for giving triple projection transparencies are suitable for obtaining coloured transparencies by superposing three coloured prints from the negatives when suitable colours are chosen.) In the first place, the colours must evidently allow large overlapping bands of the spectrum to be transmitted. We shall find that in the three negatives where a



spot of white is to be reproduced, the developed image of the spot in each is opaque. Again, if we have a black spot in the object, each of the three negatives employed will show that spot as transparent. If we take three prints (one from each negative) on a transparent colourless film, such as gelatin, sensitised with bichromate of potash, the thickness of gelatin left after development with hot water will take the place of the silver opacity found in the positives used for projection. These images we can dye with different dyes, and the depth of colour of every part will be proportional to the thickness of gelatin left at that part. Suppose we have photographed a bright yellow object, the "red" negative would show it as opaque, as also would the green negative, whilst the "blue" negative would show it as transparent. In the gelatin prints from the red and green negatives, all gelatin would be washed away where the image of the yellow object had been impressed, and it would be of maximum thickness in the print from the blue negative. This shows that the colouring matter used for the print from the blue negative must be a yellow. Hence we have an indication that the general colour of the print from the blue negative should be complementary to the blue used for the triple projection. By similar reasoning it will be found that a pink must be used to stain the print from the green negative, and a blue-green the print from the red negative. Now it is found by some that there is a difficulty in choosing the colours which will absolutely fit the negatives, such as we have described for the triple projection process, and the question of stability of the colour is one which it is necessary to consider. Thus, it may happen that some slight modifications of the screens for taking the negatives may be necessary; and I propose to show you the principles on which they may be obtained. Let us take strips of gelatin or glass plates which are stained with the three colours respectively which it is proposed to employ, and let us superpose pieces of them on one another and also have some alone.

First of all, these colours must be of such a depth that when all are superposed one over the other, the resulting light passing through must be a dark grey. If we superpose the green-blue and the pink, the light passing through must be a blue. If the pink be superposed over the yellow, a red must pass, and if we have a yellow over the green-blue, a green colour must pass. The colours available are then red, yellow, green, green-blue, blue, and pink. Now let us make a sensitometer of these colours in square patches. The diagram shows these in section.

Colours passing through 1, Pink; 2, Blue; 3, Red; 4, Blue-Green; 5, Green; 6, Yellow.



Let us consider (1), (2), and (3). In order to reproduce the pink (1), blue (2), and red (3), we must try to obtain some screen which will allow light of such a colour to pass through that the densities of 1, 2, 3 are almost nil (for the same pink is a component of each of the three) on the photographic plate, whilst the others should show opacity. It will be found to be a green screen, and so on with the others. It will be evident that only a close approximation can be obtained, since in (1) the pink itself is the only obstructing medium.

For the three negatives—

- (I.) A green screen should make 1, 2, and 3, nearly transparent; 4, 5, and 6, nearly opaque.
- (II.) A red screen should make 2, 4, and 5, nearly transparent; 1, 3, and 6, nearly opaque.
- (III.) A blue screen should make 3, 5, and 6, nearly transparent; 1, 2, and 4, nearly opaque.
- (I') A gelatin print from (I.) will, if stained pink, give 1, 2, and 3, pink; 4, 5, and 6, bare.
- (II') A gelatin print from (II.) will, if stained blue-green, give 2, 4, and 5, blue-green; 1, 2, and 6, bare.

(III') A gelatin print from (III.) will, if stained yellow, give 3, 5, and 6, yellow; 1, 2, and 4, bare.

(I'), (II'), and (III'), superposed will give the original colours: pink, blue, red, blue-green, green, and yellow.

The sensitometer may be made still more delicate by choosing half tints of each colour and superposing them on whole tints of the other colours. The principle is shown, however, in the figure.

[Some beautiful recent slides by Mr. Sanger Shepherd were thrown on the screen. They illustrated the applicability of the process to various kinds of objects and views.]

The results which have been obtained by Mr. Sanger Shepherd in the beautiful colour positives which I have shown you, ought to be equally well obtained by careful three-colour printing in transparent colours on a white ground. Instead of superposing dyed gelatin prints, the same effect ought to be obtained by printing from collotype surfaces, prepared from suitable negatives, in transparent inks of the colours laid down above. (It may be remarked that the ink used for the first printing may be opaque, but the others must be quite transparent.) It is usual to make the first printing a yellow, and the printing from the "pink" and the "blue-green" surfaces in transparent inks. Specimens of such a printing we have before us in work done by Messrs. Waterlow. The different stages of the printings are shown, and also the finished picture. The portrait in colours which I exhibit is a very fine piece of chromatic printing, and is worthy of every study.

Now, it is not always convenient to use collotype surfaces from which to obtain prints, and the great desideratum is to obtain blocks which can be printed in the ordinary printing press. All of you are aware of the vast strides that have been made in reproducing half-tone photographs in black and white for illustrations in books. The means of doing this has not been by altering the depth of colour of the printing ink used, but by introducing a grain in the negative from which the printing block is produced. The amount of ink is the same wherever there is any ink, but the half-tones are produced by an admixture of more or less white, given by the uncovered surface of the paper on which the impression is made. In the same way printing blocks for each of the three colours can be produced, which, if printed alone, would give every detail of light and shade by the above method. The difficulty that must occur is the alteration of light, shade, and colour in different prints when the three impressions are made on the same surface, owing to difference in "register." Dr. Clay has called attention to this fact in an admirable paper he contributed to the Royal Society, and there is no doubt that something has to be done in improving the method before it will be competent to take rank as a real commercial success.

The question of printing in opaque inks is another form of picture-making which need be only briefly considered. It is evident that each printing must give an image in the form of dots more or less mixed by the eye with white. It must be remembered that of all the colours used for triple projection the green is the brightest, and that the brightness of the yellow light is due to "the green brightness plus the red brightness." When colours are placed side by side and mixed by the eye, the brightness is only the mean of the two brightnesses. Now, a green and a red pigment, however bright, are always inferior in brightness to a bright yellow pigment; hence, the yellow given by blending by the eye will always be too small relatively to the green and red unless the latter are toned down. As pictures must be bright, and not of a dull colouring, some means must be adopted for getting a yellow pigment into the scheme of printing, and this can best be done by producing a fourth negative, intermediate between the green and the red. This can readily be accomplished by splitting up the spectrum into four parts, working out the curves, and finding a fourth screen for use with the fourth negative. This process has not been worked to any extent except in the way of experiment.

Time has forced me to bring my lecture to a close. The subject I have chosen was perhaps too large for a single hour (which has, I am sorry to see, been expanded into nearly two). It is one, however, which is at present uppermost in the thoughts of many photographic workers, some



of whom are not aware of all that is necessary to ensure success. I have met some who are excellent manipulators, but who are thoroughly innocent of a knowledge of the scientific basis which underlies the process, but who have more or less succeeded by numerous trial and error experiments—experiments which might have been reduced in number had they been guided by theory. My object has been to help workers as far as I could by giving an outline of such theory, and I am grateful for the patience with which I have been heard.

Mr. GEORGE BEILBY said that in the regrettable absence of the President of the Society, it had been suggested that he should propose a vote of thanks to Sir William Abney for his interesting lecture. He regarded it as a great privilege to do so, and moreover he regarded it as a privilege to have been present. In bringing about such a gathering they were indebted to Sir William Abney and to his great name in connection with this subject. There was something peculiarly satisfactory to them in having with them one of the great authorities on this important work. Sir William Abney had kept his own personality very much in the background, but those who had followed what had occurred during the last 20 years in connection with this work must feel that they were in the presence of a great master. They were proud that Sir William Abney had come to associate himself with the work of the revered Dr. Hurter and to give them the result of the magnificent scientific work he had accomplished in connection with photography. He had to propose a very hearty vote of thanks to Sir William Abney for his address.

Mr. E. K. MUSPRATT seconded the vote of thanks, and

SIR WILLIAM ABNEY briefly acknowledged.

## London Section.

Meeting held on Monday, November 4th, 1901.

MR. OTTO HEHNER IN THE CHAIR.

### COLORIMETRIC METHOD FOR DETERMINING OXYGEN DISSOLVED IN WATER.

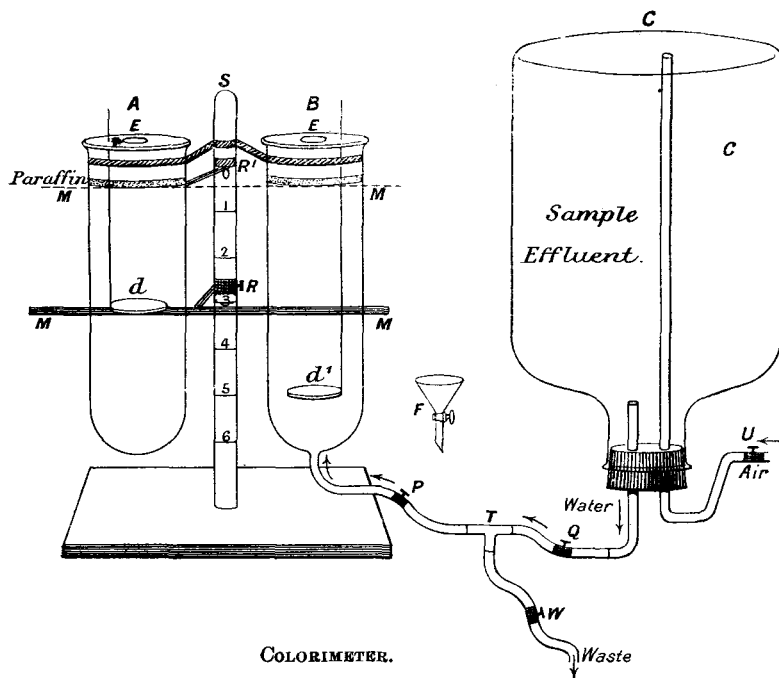
BY PROF. WILLIAM RAMSAY, F.R.S., AND MISS IDA HOMFRAY.

THE estimation of dissolved oxygen in effluents from sewage farms, and in rivers and drinking waters, forms a valuable adjunct to the numerous chemical and bacteriological tests for contamination which are at present applied, and in conjunction with a determination of nitrates and, perhaps, incubation, may even supplant them. It occurred to us that it would be advantageous if some quick and easy process could be devised for estimating the free oxygen in water—some process which could be safely entrusted to persons who had received no training in chemical manipulation.

Boiling out the gases, and their estimation by the usual process of gas analysis, are obviously unsuited for the purpose, and the method of which a description is now given

appears likely to be one which may be generally available in the hands of the managers of sewage farms and of inspectors at testing stations. We have also devised a portable apparatus which can be taken to the effluent or brook, and which enables a rapid estimation to be carried out in a few minutes on the spot.

It is well known that an ammoniacal solution of cuprous chloride is colourless, but turns blue owing to oxidation immediately on exposure to air, with formation of cupric chloride. If the water in which the cuprous chloride is dissolved contains oxygen in solution, the same blue colour is produced; when atmospheric oxygen is effectually excluded, the amount of cupric chloride formed is found to be an accurate measure of the dissolved oxygen present;



the estimation of cupric chloride lends itself especially to colorimetry, as, owing to the intensity and purity of the blue colour obtained in ammoniacal solution, very slight differences of tone are easily detected.

The apparatus used resembles Mills' colorimeter, modified so that sample waters can be protected from aeration in transference from the collecting bottle, and during the experiment; this is effected by means of a layer of paraffin oil, as shown in the diagram. The necessity for displacing air from the whole apparatus by an inert gas, as shown in Thresh's process, is thus obviated.

**The Apparatus.**—A and B in the diagram are two glass comparison-tubes, each 12 ins. long and 2 ins. in diameter; they are closed with movable caps (E); (d) (d') are opal glass discs supported by brass wires, which slide with friction through holes in the caps, and which hold the discs in any required position.

The tubes are supported on a stand with a vertical brass stem (S), which is graduated in inches and tenths. The levels of the discs and of the liquids in the tubes are read off against this scale with the help of the horizontal levelling rod (M M), which slides along (S) and can be fixed at any point by the clamp (R). The bottle (C) contains the sample water or effluent.

Just before the experiment, it is unstoppered and fitted with rubber corks and tubes as shown. Care must be taken that no air bubbles are included; the bottle is then inverted, supported on a tripod stand, and connected by thick rubber tubing through a T-piece with (B) as shown





**To Fill (B) from (C).**—The clamp (R) is set at 0.3 division on (S), reading by the bottom of (R). Open screw clips (Q), (W), and (U), and run off some water till all air is displaced from (C) to (W). Close (Q). Remove cap from (B) and pour in some paraffin. Replace cap and disc. Run off enough paraffin through (W) to displace air from tube (B) to T-piece, leaving a depth of 1–2 cm. in (B). Close (W) and open the other clips. Water is thus allowed to flow quietly into the colorimeter tube (B) until level with the rod (MM). The paraffin layer rises above the water, acting as a liquid stopper and preventing absorption of atmospheric oxygen for a considerable time.

**To fill (A) with Standard for Comparison.**—All corrections are eliminated by using as a standard, distilled water saturated with air, both being at the laboratory temperature, which must be noted. The water is shaken in an open flask till air bubbles are seen, and is allowed to stand until they have disappeared. It is poured quietly into a colorimeter tube (A) till its level reaches (MM). After making sure that no air bubbles are entrapped below the disc, a layer of paraffin is poured on to the water. The ratio of concentrations of oxygen in the waters in (A) and (B), can now be measured. The clamp (R) is moved to zero. (MM) is then the final level.

**Addition of Reagents.**—Cuprous chloride is found to keep from oxidation best in the form of a powder protected from light and damp in a stoppered black test-tube. It is made by warming a solution of cupric chloride with scraps of metallic copper, and then pouring into water. A white precipitate is filtered off, washed with boiling water, the water is displaced with alcohol, and the alcohol with ether; it may then be dried by help of a suction pump on the filter which retains it. It must not be used unless quite white, otherwise it may contain some cupric chloride. A little of the powder is poured into a small tap funnel (F) and covered with concentrated hydrochloric acid; a dark brown solution results. Some is then run into (A) and (B), avoiding air-bubbles. A white precipitate forms and aqueous ammonia is at once added from a pipette, till the liquid in both tubes reaches (MM); on stirring gently with the discs, solution takes place at once; the cuprous ammonium double salt is soluble and colourless, but some of it is oxidised to the cupric condition at the expense of all the oxygen dissolved in the water. A very small quantity of this powder suffices to ensure the removal of all the dissolved oxygen, but some excess does not interfere if sufficient ammonia is at once added. If any white precipitate remains, it turns yellow, for it is very sensitive to light, and interferes with the readings. The solutions (if coloured) should be of a pure greyish blue. A greenish colour indicates that an insufficient amount of ammonia has been added, unless the sample water itself was coloured or opalescent.

The colorimetric estimation consists in determining the ratio of the concentrations of the coloured cupric double salt in the two tubes.

**The Determination.**—As with Mills' colorimeter, the observer, looking through a hole in the cap of each tube, adjusts the levels of the opal glass discs ( $d$ ,  $d'$ ), so that, when seen from above through the liquids, the intensity of colour appears the same in both. The supports of the tubes are hinged to move in a horizontal plane, and their distance apart is regulated by the observer so that he can look down both tubes at once. (MM) is made to coincide with the level of each disc in succession, and readings are taken by (R) or (S), as shown in the figure. This gives the depth of ( $d$ ) below the level of the liquid at the zero.

Let the readings be  $p$  and  $q$  in tubes A and B respectively.

Intensity of colour, i.e., the total selective absorption of light in travelling through the lengths  $2p$ ,  $2q$ , respectively, of the solutions is the same by adjustment.

But this absorption depends only on the number of molecules of solute encountered, the reflecting disc being white and the solvent colourless.

Hence, in the two solutions

$$pc = qc',$$

where  $c$ ,  $c'$  are concentrations in A and B,

$$\therefore \frac{c}{c'} = \frac{p}{q}.$$

Thus,  $p/q \times 100$  expresses the percentage oxygen saturation compared to water saturated at known temperatures.

The final results are best expressed in c.c. per litre at N.T.P. The numbers are accurately known for distilled water at ordinary temperatures, and are given in Sutton's Volumetric Analysis from 0° C. to 30° C.

In very many cases the water to be tested contains much lime, which causes a turbidity on adding a solution of ammonia. In this case, 2 or 3 c.c. of a hot saturated solution of ammonium chloride is added before the other reagents to both tubes; dilution corrections are thus eliminated.

The effluents to be tested are often rather yellow in colour. The addition to the standard of a trace of an alkaline solution of paranitrophenol after the other reagents usually gives a colour similar to that of the effluent. The settings are thus somewhat facilitated, but the difficulty of dealing with coloured effluents is much less than appears at first.

Numerous trials of the method have been made to test its accuracy and reliability.

(1) **Comparison of the Colours obtained by Different Strengths of Cupric Chloride.**—The same volume of water was poured into A and B. 6 c.c. of  $\text{CuCl}_2$  solution was added to A, and successive quantities of the same solution was dropped from a burette into B. Readings by the colorimeter were taken after each addition of 0.3 c.c. to B. The readings were in all cases accurate to 1 or 2 per cent. This shows that the readings obtained are proportional to concentrations of cupric chloride.

(2) Negative results with ammoniacal cuprous chloride and un-aerated water were repeatedly obtained. The water used was boiled in a flask for some time under reduced pressure, paraffin sucked into the vacuum after cooling, and the usual testing process applied. No trace of colour was produced.

(3) With varying quantities of oxygen in the water, aliquot portions of the un-aerated water were run out and replaced by fully aerated water. Comparisons were made with the undiluted standard and a very good agreement was found. This method is not a very convenient one, as aeration gradually takes place through the paraffin which absorbs gases very readily.

In order to obtain more satisfactory and independent check upon our experiments Thresh's iodometric method was used side by side with the  $\text{Cu}_2\text{Cl}_2$  method. For particulars of Thresh's method which, though rather longer and more complicated, is very convenient and reliable, see Sutton's Volumetric Analysis.

It will be seen that the  $\text{Cu}_2\text{Cl}_2$  method and Thresh's method are so entirely different, as are also the sources of error, both chemical and personal, that they afford an excellent mutual check. The concordance was almost always very good, the errors being well within the limit required for practical purposes.

**Portable Modification.**—The cuprous chloride colour test described above has been arranged in a portable form, all necessary sample collecting apparatus, reagents and colour standards being contained in a small wooden box. Experiments can be carried out at the river side and the condition of the water tested in a few minutes at the required points of its course. The apparatus has been tried on the Brent near London with satisfactory results and also on the Thames. The oxygen found varies from 5.5 c.c. to 1 c.c. per litre according to rapidity of flow, the entrance of bad effluents and the presence of aerating boards along the course of the river.

The standards of colour in this apparatus consist of six sealed tubes about six inches long containing various strengths of ammoniacal cupric chloride. The experiment is carried out in a similar shaped tube with ground glass stopper, under paraffin oil. The standards are supported vertically in the box with a space between each into which the test tube can be inserted for comparison. Light is reflected up through the tubes by an opal glass plate set at



45°. It is thus easy to determine which of the standards is most nearly like the sample of the test tube in respect to intensity of blue colour.

To fill these standard tubes a stock solution (A) is made as follows: 3 grms. of pure copper wire are dissolved in nitric acid and the solution is evaporated to dryness several times with hydrochloric acid; it is then heated till all the free acid has been driven off. The residue of cupric chloride is dissolved in distilled water, and ammonia and hydrochloric acid are added till a clear deep blue solution is obtained smelling strongly of ammonia. The solution is then diluted until it occupies one litre. It can be kept for some time but gradually loses ammonia, becoming more or less green in tint. Hence it is usually necessary to add ammonia in making up the diluted solutions for the standards from it.

It is most essential to the permanence of the standard tubes that there should be a large excess of ammonia in the solutions with which they are filled.

This stock solution is then diluted in definite proportions so as to correspond colorimetrically to definite concentrations of oxygen in the water to be tested.

No. of Standard.	No. of c.c. of A per Litre.	No. of c.c. of O <sub>2</sub> per Litre.
1	3.6	1
2	7.2	2
3	10.8	3
4	14.4	4
5	18.0	5
6	21.6	6

The quantity of ammonia added in diluting is immaterial so long as enough is present to ensure that all the copper is present in the form of cuprammonium, and not of cupric ions. It should smell strongly of the gas.

It is easy to calculate the concentration of oxygen in water, which should correspond theoretically with a given strength of cupric chloride, say with Standard No. 6.

1 litre of A contains 3 grms. Cu = 6.4 grms. CuCl<sub>2</sub>.

1 litre of No. 6 contains  $\frac{6.4 \times 21.6}{1.030}$  0.1382 gm. CuCl<sub>2</sub>.

But 16 grms. O<sub>2</sub> are equivalent to 267.2 grms. CuCl<sub>2</sub> in the reaction.

Therefore 1 litre No. 6 is equivalent to  $\frac{0.1382 \times 16}{267.2}$  = 8.28 mgrms. O<sub>2</sub>.

= 5.8 c.c. of O<sub>2</sub> at N.T.P.

Therefore Standard No. 6 is theoretically equivalent to 5.8 c.c. of oxygen per litre. By the colorimeter it corresponds to 6 c.c. per litre—an agreement as near as can be expected. The result was not worked out till long after the standards had been empirically fixed, and is interesting as showing that the colour estimated is really a measure of the reaction between the cuprous chloride and the dissolved oxygen.

The numbers for standards were obtained from numerous experiments with varying concentrations of oxygen in water and by comparing the standards with one another.

A large number of actual effluent samples were procured from the Beddington and Aldershot sewage farms. The oxygen determination was carried out by the colorimeter, by Thresh's method, and in all the latter cases by the portable test tube method. The results obtained were compared with those obtained on duplicate samples at the testing station by the gas analysis method, on the same day. It was found, however, that the comparison with the duplicate sample often gave a not very satisfactory agreement when the three other methods agreed very closely among themselves. This was evidently due to aëration of the samples irregularly in the process of collecting, and also to partial incubation of impure samples when kept for some days.

A few compared results are given below. As the proportionate accuracy obtainable with smaller quantities of oxygen is less, and that required is also less, it is useless and misleading to work out discrepancies of readings in percentages. An accuracy of about 0.3 c.c. is as much as is required in estimations.

A good many concordant negative results were obtained, but are not quoted. The last effluents were very poorly

Cu <sub>2</sub> Cl <sub>2</sub> .	Thresh.	Test Tube.
1.90	2.26	2
5.12	5.16	..
0.70	0.53	..
6.92	7.07	..
2.87	2.92	..
5.40	6.65	..
2.44	2.70	2-3
1.00	1.27	1
1.86	1.80	2
0.82	0.86	1
3.06	3.08	3
0.60	0.45	0-1
7.70	7.50	7
4.35	4.17	4-5
0.00	0.15	0-1
0.00	0.00	0
Trace	0.35	0-1
5.20	5.80	5

aërated, and after saturating and incubating for three days no oxygen remained. These were examined microscopically and were found to abound in bacteria.

A number of experiments were made with a view to testing approximately the amount of aëration which takes place when boiled water is allowed to stand undisturbed in a large beaker for varying lengths of time. The object of these experiments was to determine how rapidly aëration might be expected to take place in an effluent or impure brook water when standing at rest. Of course, running water would aërate more rapidly.

In all these experiments distilled water was boiled under reduced pressure for some time, and in a corked wash bottle provided with india-rubber tubes and clips. After it was cold, the end of the tube was opened under paraffin, which was sucked into the vacuum, and the aëration of the water was at once tested. The results were as follows:—

Time when taken.	Time of Standing.	Depth.	Method of Treating	No. of c.c. per Litre.
	Hours.	Cm.		
12.45 p.m.	0	10	Boiled out	0.0
3.10 p.m.	2.25	5	After standing	0.5
4.45 p.m.	4	1		1.5
5.30 p.m.	0	Boiled out	Taken from flask	0.3
11.45 a.m. Saturday	18	5	From beaker	5.3
12.30 p.m. Monday	0	Boiled out	Taken from flask	1.4
12.30 p.m.	48	10	From beaker	5.9
2.30 p.m.	50	1	.. ..	5.7

The slight difference in the last two experiments is probably due to a chance bubble of air in collecting. These experiments seem to show that the absorption of oxygen by still water is very slow, and that it does not proceed to complete saturation. But after 12 to 24 hours, about 5 or 6 c.c. per litre should be present in any water, unless active decomposition is going on.

The tests for aëration in the transference of water from one vessel to another, as in collecting samples, were as follows (Thresh's method for estimation was used):—

Method of Collecting.	No. of C.c. of O <sub>2</sub> per Litre.	Gained.
	C.c.	C.c.
(1) Partly boiled out, air admitted, collected under coal-gas.	4.3	..
Run into beaker poured into funnel....	4.4	0.1
Run into beaker poured into flask and back.	4.4	0.1
(2) Boiled out and collected from flask....	2.0	..
Run into beaker and collected under gas	2.3	0.3
Run into beaker poured into funnel....	2.9	0.9
(3) Boiled out.....	0.9	..
Run into beaker, poured into funnel....	1.4	0.5
Run into beaker and collected in small flask by immersing, as in collecting river samples.	2.1	1.2
(4) Boiled out.....	0.14	..
Run into beaker and collected as in last	1.26	1.12



For poorly aerated samples precautions seem necessary.

To avoid aeration in collecting samples, the can fitted to the testing box is open at one end and has a valve opening inwards at the other. The stream can thus be allowed to flow right through, displacing all air. The valve closes when the can is lifted out full of water.

A number of samples were sent in duplicate for incubation tests. In each case one sample was tested at once and the other kept tightly stoppered in a thermostat bath at about 20° C. for a day or two. It was then tested and the aeration of the sample before incubating was compared with that of the duplicate sample after incubation. This is apparently the crucial test for bacterial contamination. Some very poorly aerated samples were saturated with air by shaking, previous to incubation.

The following series may be quoted. All estimations were done by the three methods and the results agreed very well. For clearness the mean is given :—

Kind of Sample.	Before Incubation.	After Incubation.
Brook water.....	7.7	4.3
" " below effluent outfall.	5.2	0.3
" " lower down..	5.3	0.0
Effluent.....	0.0	0.0
	(after saturation as explained).	

Experiments were made using alkaline pyrogallol instead of cuprous chloride and ammonia, the facility of obtaining and preserving pyrogallol being an obvious advantage. The colour obtained is good and easily estimated by most people. The results seemed satisfactory so long as distilled water was used. When, however, tap water was used a deep colour was obtained, even with boiled out water. This appears to be due to the lime contained, for the excessive coloration was obtained always with distilled water to which had been added a small quantity of calcium chloride.

This was taken to be a fatal objection, and the use of pyrogallol was therefore abandoned. Small quantities of nitrates do not affect the cuprous chloride experiment, as the solution is necessarily alkaline.

#### DISCUSSION.

Dr. S. RIDEAL said that he was greatly interested in the subject of the paper, and quite agreed with the authors as to the extreme importance which attached to the determination of dissolved oxygen and nitrates in sewage effluents. He had recently had an opportunity of reviewing the various methods proposed for this purpose and the aeration test for effluents, and his papers had been published in the *Analyst* for June and August last. (Rideal and Stewart, *Analyst*, 26, 141—148; Rideal, *loc. cit.* 196—202). When he heard of the paper on the subject before this Society, he hoped that Prof. Ramsay would have seen and would reply to his criticisms. Prof. Ramsay unfortunately had not seen the papers in the *Analyst*, and therefore he felt at some loss in making any remarks.

Prof. RAMSAY: You had better go on.

Dr. RIDEAL, continuing, said that Prof. Ramsay had told him a year ago about the apparatus devised by him, and had suggested it to him for trial. He at once procured one and tried it, and the results were recorded in the papers referred to. At first he was very pleased with its working: the colours were sharp, the apparatus worked easily and gave good results with river waters. The first objection he had to make was that the standards did not go quite far enough. At the present time of year (November) the dissolved oxygen per litre of water would be about 7.4 c.c. per litre, but the highest standard provided with the apparatus before them was 6 c.c. per litre, and he therefore suggested that additional standards up to 8 c.c. per litre should be added. A minor point was as to the accuracy. The difference between the colours of two tubes represented 1 c.c. of dissolved oxygen per litre. There was a marked difference between any two tubes, and it was easy to read to half a c.c. His assistant, Mr. Stewart, claimed that he

could read the tubes to one fifth of a c.c. For practical use by the manager of a sewage farm, however, half a c.c. was near enough; and, indeed, Prof. Ramsay only claimed that the method was to be regarded as an approximate one. The next point was with regard to the cuprous chloride. He had found it difficult to get a colourless solution with that salt. Prof. Ramsay had just demonstrated that the test required a certain amount of time; and if it took as much time to dissolve at the waterside as it had that night, during that period they had a hydrochloric acid solution exposed to the air and the cuprous chloride taking up oxygen; therefore all the precautions as to the layer of kerosene on the surface of the water were of no avail. He had found that a blank experiment with boiled water containing no oxygen gave a blue coloration equal to 1 or 2 c.c. per litre. He therefore concluded that most of the readings were too high by  $\frac{1}{2}$  to 1 c.c. by reason of the error thus described. He had also made some experiments with regard to the action of the kerosene in this test; but he would not go further into that matter at present, as kerosene was known to be an absorber of oxygen to some extent, and after all it was a minor point under the circumstances. Experiments on this point he had described in the paper referred to, which showed the rate of absorption of oxygen by the water when protected by the kerosene layer. Then with regard to nitrites, although dissolved oxygen and nitrates were the two most important bodies in this connection, it must be remembered that nitrites were **always** formed, and indeed in the bacterial method they were sometimes present in large amount—as much as 5 parts in 100,000. Their effect must therefore be taken into account, and also that of organic matter. In dealing with an effluent containing 2 or 3 parts of nitrites and coloured organic matter, he had found that the colour obtained was modified by these two constituents. He had tested the influence of nitrites on the colour, and, putting it roughly, the presence of 5 parts of nitrous nitrogen per 100,000 threw the colour up to an extent equal to 2 c.c. per litre. He quite agreed with Prof. Ramsay that a simple method giving approximate results was very desirable. One did not want refinement down to even 1 c.c. of oxygen; all that was necessary was to know if the effluent was more or less than half saturated. If more, it was probably all right; if less, it was probably all wrong. The most serious criticism that had to be made was that the standards supplied were not constant. He had not discovered this until quite recently. On seeing Prof. Ramsay's paper announced, he thought it desirable to repeat some of his experiments, and on doing so he found that his tubes were much fainter than they were a year ago. In view of this fact, he thought it quite possible that what he had attributed to nitrite interference might in part have been due to the gradual depreciation of the colour of the tubes. Still the nitrite in the acid solution obviously must act as an oxygen carrier, and therefore during the air contact one would get more colour than when nitrites were absent. On comparing his tubes, he found that at present the 3 c.c. tube was of a darker blue than the 5 c.c., and there were lesser variations in the others. With regard to an alternative method, he had lately been using a modification of the Winkler process. Winkler originally suggested the use for this purpose of a manganous salt. Manganous chloride had the great advantage of being thoroughly stable. The colour produced on the addition of caustic soda and an iodide by the liberation of the iodine after acidulation was as intense as the blue colour in the tubes under notice; and it was quite possible to determine the relative amount of dissolved oxygen in water by guessing the brown colours which showed, without the use of standards, whether the effluent was half or a quarter saturated. In order to compare the colour of a sample, one had merely to take some of the same effluent, shake it for a minute, until it became fully saturated, and add the reagents, when one had a standard for that particular temperature. This overcame the difficulty met with in coloured effluents and that due to nitrites if the reaction took place in an ordinary stoppered bottle, so that exposure of the acid solution to the air was prevented.

Mr. W. T. BURGESS asked for further information with regard to the amount of nitric acid found in what was sold





as pure sulphuric acid. He had very seldom himself had to reject a sample owing to the quantity of nitric acid interfering with the estimation of nitrates by Harrow's process, and he was therefore rather surprised at the statement made by the authors.

Mr. E. J. BEVAN suggested that pure sulphuric acid might be secured by simply passing through it a stream of sulphurous acid gas and boiling off the excess.

Prof. RAMSAY said that he had tried that method but without success.

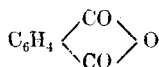
The CHAIRMAN said that Dr. Rideal had discovered so many points in which the process overestimated the dissolved oxygen, that it was difficult to see how he had ever managed to find so low a proportion as 6 c.c. at any time. In making a sewage analysis, the chemist ought to distinguish between methods which aimed at the highest accuracy obtainable and such as enabled him to form an opinion sufficient for practical purposes. And in the case before them the chemist could probably judge without figures whether a sewage was fairly aerated or not. In practice it made no difference whether a sample of sewage contained 2.5 or 2.7 of dissolved oxygen, especially as sewage fluctuated rapidly in composition; and therefore it seemed to him useless to attempt to get an exact estimate. While fully appreciating the value of Dr. Rideal's criticisms on the method, he took it that Prof. Ramsay had mainly in view a method and apparatus that would suit the foreman of the sewage farm. He felt no doubt that for that purpose Prof. Ramsay's method was more suitable than the most ingenious and accurate method worked out by Dr. Rideal. That method was admirably adapted for the hands of a skilled chemist; but at the riverside or sewage farm nothing could be simpler than Prof. Ramsay's process.

Prof. W. RAMSAY, in reply, said that he was quite unprejudiced as to the method of working. What he and his colleagues on the Commission wanted to find was the simplest and most reliable method of working, and he should certainly try Dr. Rideal's method. He thought that the cuprous chloride difficulty might be dismissed, because one need not run it all in. With regard to the kerosene, he quite agreed with Dr. Rideal that it took a long time for oxygen to pass through; but it always did enter in the course of a few hours. A much more serious matter was the deterioration of the standards, and in that case he feared they must say "peccavi." Much more ammonia should be present, and then they would not deteriorate. As to nitrites, he admitted that, if present, they ought to be counted as free oxygen, the same as nitrates. They were to be reckoned as "available oxygen," and that was all they needed to know about them.

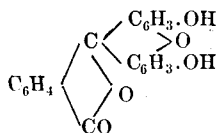
#### A NEW GLYCERIDE: GLYCEROL PHTHALATE.

BY WATSON SMITH.

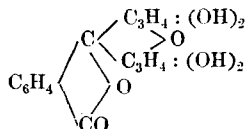
I HAD originally conceived the notion that as the diphenol, resorcinol,  $C_6H_4(OH)_2$ , unites on heating with phthalic anhydride—



to form resorcinol-phthalein or fluorescein—

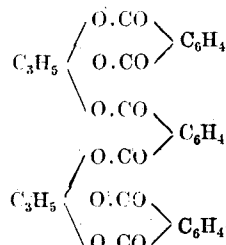


so some similar compound or analogue might be formed, if for resorcinol, the trihydric alcohol, glycerol, were substituted. The formula of the phthalein thus speculated, would be—



and a chromogen containing aliphatic constituents would have possessed much interest.

On heating glycerin with phthalic anhydride alone, however, no such reaction occurred. On heating with zinc chloride, the product appeared to be substantially similar to that obtained by heating phthalic anhydride with glycerin alone. The idea now was that probably a glyceride was formed, especially as on heating with alkali hydrates, entire decomposition or saponification took place. Acting upon this idea, proportions of glycerin and phthalic anhydride were taken corresponding with the Diglyceryl triphthalate.



The mixture was heated for a long time nearly to boiling, and on cooling, a solid, resin-like, perfectly transparent and strongly refractive substance was obtained. This substance is insoluble in water, but soluble in an excess of glycerin on heating. From the solution in glycerin, white oily drops separate on addition of water, soluble in alcohol and ether. Whether zinc chloride were used or not, similar compounds were obtained, consisting of thick resin-like masses, which deliquesced and became sticky on exposure to air. As the substance evidently still contained free glycerin and was not uniform in composition, it was distilled *in vacuo*. This method answered the purpose, and it was found that when a certain point was attained in the distillation, at which apparently all excess of glycerin had come over, the mass in the retort began to froth up, and then on removing the vacuum the substance at once solidified to a mass resembling glass slag.

It was found that whether excess of glycerin or excess of phthalic anhydride were taken in the mixture employed, after distillation *in vacuo*, one and the same product was obtained, viz., the hard glassy substance. This substance is easily decomposed or saponified with caustic soda solution, and hence the fact indicated the method which was adopted for determining the phthalic anhydride. Hot normal caustic soda was used and the excess was titrated back with decinormal acid. Thus figures were obtained pointing unmistakably to the fact that under the conditions stated, the diglyceryl triphthalate is formed. But it is extremely difficult to get rid of the last traces of glycerin, for at this final stage, the mass heated *in vacuo* shows an extraordinary tendency to froth up.

Thus, when glycerin was in some excess in the mixture taken, the percentage of phthalic anhydride found was 74.00.

Adopting a slight excess of phthalic anhydride, the percentage was 75.34.

Adopting a further excess, it was 75.91.

The theoretical number for diglyceryl triphthalate, is 77.35, that for a possible diglyceryl diphthalate being 66.66.

The difficulty is that the phthalic anhydride is much more volatile than the glycerin, and at the high temperatures needed for the formation of the body, some of the former is lost, and the preparation never quite reaches absolute completion, though as the figures show, it very nearly does so. If the distillation process *in vacuo* be stopped at various stages before the glycerin is all expelled so as to reach the frothing-up stage, products of varying degrees of fusibility are obtained, containing varying amounts of glycerin, which continued distillation would have expelled. Some of these are of the fusibility of sealing wax, and may be drawn out in long silky filaments of extraordinary length. I show here some specimens of monographs stamped upon some of this material, used just as sealing wax is.

Just at the moment when the mass froths up in the retort at the final stage of the distillation *in vacuo*, it is noticed



that a very distinct rise of temperature takes place. The maximum temperature reached was  $235^{\circ}\text{C}$ ., but on removing the retort from the hot sand bath, at this point, the further rise was to about  $242^{\circ}\text{C}$ .

This glass-like glyceride is characterised chiefly by its extraordinary insolubility in almost all solvents. It is practically insoluble in alcohol, ether, and benzene, also in petroleum and petroleum spirit. It does not dissolve appreciably in boiling water, but the water becomes slightly acid, probably through slight hydrolysis of the compound. It dissolves very slowly and slightly in cold or hot glycerin, and this also becomes slightly acid. It is quite tasteless, and softens on chewing. This glyceride is insoluble in hot olive oil, and also in other oils and fats. It simply softens in them and becomes plastic. Its best solvent appears to be cold acetone, but even in this it is but sparingly soluble. On pouring some of the solution on a watch-glass and letting it evaporate spontaneously, no sign of crystallisation was evident, but the clear transparent resin deposited in minute drops, solidifying to hard transparent masses of the tasteless resinoid body. I tried the experiment of taking a large excess of glycerin in preparing the triphthalic diglyceride, and obtained first the transparent refractive substance first described; this body I found was strongly acid. On distilling this product *in vacuo*, however, first water, then glycerin and acrolein vapours, then phthalic anhydride pass over, and finally the mass froths up as before, yielding the inert, glass-like glyceride. The fact is, reaction proceeds but slowly at these lower temperatures, or until, indeed, such high temperatures are attained, that under ordinary atmospheric pressure, decomposition of the whole would ensue. But under the reduced pressure, the necessary point of union is attained, excesses of glycerin and phthalic anhydride passing over. If instead of phthalic anhydride, phthalic acid be employed, a similar reaction occurs, the same glyceride being obtained.

Some of the glyceride was heated in a test-tube with thermometer, and connected with the vacuum pump. The compound proved very refractory to fusion, decomposition point and melting point lying very near together. Incipient fusion took place at  $190^{\circ}\text{C}$ ., and then the mass began to decompose and finally to char, at about  $220^{\circ}\text{C}$ ., and yet was not completely melted. Crystals of phthalic anhydride deposited on the sides of the tube. A bit of the glyceride ignited readily takes fire and burns with a bright flame, but does not melt, only softens. It will continue thus to burn till only a slight charred residue is left. The substance on charring emits an odour somewhat similar to that of burning fats. It can be moulded into forms like that of a taper, and will then ignite and burn with a bright flame without melting, but there is no advantage to offer over cheaper articles for the purpose. I have found that a very satisfactory cement for glass and earthenware can be made from this glyceride.

Since glycerin will not unite with phthalic anhydride to form a phthalein, but rather a phthalate, I thought it interesting to discover if resorcinol, which does so readily form the phthalein, fluorescein, would, on application of heat, displace the glycerin from combination with phthalic anhydride in this diglyceryl-triphthalate. I find that this reaction easily takes place, and thus an interesting test is obtained for the presence of the phthalic anhydride.

**Sulphonated Products.**—On treating the powdered substance with warm or hot concentrated sulphuric acid, sulphonation takes place, though by no means quickly. The substance shows no signs of charring in the process. With fuming sulphuric acid the action proceeds much more quickly, and no charring results. I have already observed two sulphonic acids, but have not as yet been able to examine them more closely. One is readily obtained when the sulphuric acid is used in excess, and on diluting the liquid and heating, and then allowing to cool, it readily separates in groups or aggregates of minute prisms, soluble in hot water, and quickly crystallising out. I am inclined to think this is the monosulphonic acid, but analysis must decide this later. Now, on treating a little fuming sulphuric acid with a large excess of the powdered glyceride, and heating gently, I was much surprised, after dilution with

water and filtering, to observe an oil separating. On evaporating and allowing to stand, quite a considerable quantity of this oily body separated, and also a smaller quantity of the crystalline sulphonic acid. The oil, which is heavier than water, and less soluble than the crystalline acid, was separated from the latter and washed with water. It then occurred to me that there might be some analogy here with those sulphonated oils which form the basis of the Turkey-red oils and oleines of commerce. The sulphonated oils, on treatment with ammonia, unite with it, giving perfectly clear oils, quite soluble in water. On neutralising with ammonia, my oil at once cleared, and became perfectly soluble in water, and the ammonia compound obtained, possessed many of the properties characteristic of the sulphonated oleine oils mentioned.

**Action of Nitric Acid.**—Strong nitric acid has by no means powerful action on this glyceride. To nitrate it freely, it was necessary first to treat with concentrated sulphuric acid, and then add nitric acid, and heat.

In conclusion, my best thanks are due to my friend, Dr. H. Pauli, of the firm of Meister, Lucius and Brüning, for a supply of pure phthalic anhydride, and to my assistant, Mr. Albert Shonk, for considerable help in carrying out the experiments just recorded.

#### DISCUSSION.

Mr. ARTHUR MARSHALL considered the new substance described by the author a very interesting one. It was evidently a true colloid, but unlike most other organic colloids it had a known and definite constitution, though it resembled them in being more soluble in a cold solvent than in a hot one. The same was the case with nitrocelluloses, most of which could be dissolved in ether or alcohol alone if only the temperatures were reduced low enough. Cellulose itself was also more soluble in Schweitzer's reagent at a low temperature than at a high one.

#### MANUFACTURE OF VARNISH BY THE PRESSURE PROCESS.

By ARTHUR J. SMITH.

On heating fossil gums in an open vessel the following changes take place:—The gum softens, and first water, then inflammable vapour, and later inflammable liquid of doubtful or unknown composition are driven off. The gum is converted finally into a black carbonaceous mass. The amount of volatile matter, which can be driven off from gums in this way, varies according to the gum. In the manufacture of varnish 40–50 per cent. of the gum is very often volatilised in the melting process.

The effects of this on the physical properties of the gum can easily be seen, by comparing some of the original gum with the residue left in the melting vessel, which, when allowed to cool, is generally brittle and can be easily powdered. Varnish made from a decomposition product of this description cannot have the wearing capacity, viscosity, or light colour of a varnish made from the whole undecomposed gum. It has been tried to make "whole gum" varnish, by dissolving the gum in spirit, mixing into oil, and then distilling off and recovering the spirit. This method does not seem to have been much used.

The majority of gums can be rendered miscible in oil without loss, by heating in a closed vessel under a small pressure, and this observation has been applied to varnish manufacture on a commercial scale. There is no risk of fire, and all noxious vapours are avoided. The 40–50 per cent. of the gum, usually volatilised and thrown away, is thus saved and becomes incorporated with the varnish, and appears to greatly increase its covering, wearing, and lasting capacities.

The result of saving this 40–50 per cent. is shown by the fact that the usual formula, 1 part of gum, 2 of oil, and 3 of turpentine, will not hold good, the product being quite unworkable. 5–6 parts of turpentine is the amount usually required to be added to make a workable varnish. Also being under pressure the gum mixes into the oil at a much lower temperature, and as a result the varnish is very little darker than the original oil, and, if properly refined oil has been used, a very pale varnish is the result.



It will be obvious to anyone familiar with varnish manufacture what a great saving this will effect in the cost of the varnish, by getting 8 or 9 parts instead of only 5, from 1 part of gum, and at about the same cost of manufacture.

A large number of quantitative experiments have been made in the laboratory, on nearly every variety of gum, to test this process on a small scale, the operation being carried on in a specially constructed "Jena" glass tube, fitted with pressure gauge and safety valve, the heat being furnished by a Bunsen or spirit lamp.

To take one experiment as an example, 100 grains of gum kauri, which had been exposed to the air in a powdered condition for some days to get rid of the majority of its water, was melted into 200 grains of refined linseed oil under a pressure of two atmospheres. After cooling and being open to the air for some time, 300 grains of turpentine were added, and the whole heated under pressure for some 20 minutes. On cooling, the resulting mixture was too thick for use with a brush, and it was found necessary to incorporate a further 230 grains of turpentine to bring it to the consistency of ordinary varnish, thus making  $5\frac{1}{3}$  parts of turpentine added.

It was then placed in a jar in the sunshine, and became clear in 10 days. It weighed 832 grains, and measured  $2\frac{1}{2}$  fl. oz. The sediment weighed 3.0 grains. A further increase of weight was found to have taken place after the varnish had been kept for a few weeks.

The phenomenon of gum melting into oil under pressure has been observed before, but the observation does not seem to have been applied with any degree of success to the manufacture of varnish on a commercial scale. I have successfully manufactured varnish in melters of up to 10-gall. capacity, and am now working on even a larger scale.

#### DISCUSSION.

Dr. J. LEWKOWITSCH thought the Society ought to be grateful to the author for bringing forward a subject of which one heard so little. The manufacture of varnish was at present only an art, its chemistry being practically unknown; and the men who understood the art held sway and kept everybody out of it. One disadvantage chemists had to contend with was that they did not know the constitution of even the commonest gums, such as the copals. In the course of reading the paper he interrupted the author to ask what gum was referred to in the case given; but even with the answer "kauri gum," very little information was conveyed, as dealers were pliable in their practices, and often named a gum to suit the wishes of their customers. The copals covered such a variety of exudations from different plants, that it was next to impossible to classify them, even if one knew the country or "coast" from which they came; for little was known as to the manner in which copals were mixed for the market in the washing and sorting establishments. Gums were not like fats and oils, which could be discerned by chemical methods; and, in fact, they knew very little about them. They knew that in the varnish manufacture it was necessary to melt the gums; but so long as they did not know what chemical processes occurred, and how the bodies decomposed and changed, and why they became soluble, very little progress would be made from a scientific point of view. He was afraid he could not take so sanguine a view of the new process as the author did. Everybody who was conversant with the trade knew how difficult it was to introduce a new varnish. And that was quite natural; for a coach-builder who turned out carriages worth several hundred pounds each was not likely to risk spoiling his reputation by using a new varnish of which he knew nothing.

He would like to know whether experiments had been made with this process on a large scale. They were told that it required more turpentine, and that seemed to agree with some experiments published only last month by Lippert. This chemist had tried this process, and confirmed the statement that some gums, when melted under pressure, dissolved in linseed oil. But he also stated that when run with turpentine, the gum separated out, and hence he condemned the process. He himself could not say that he had practical experience with this process to endorse that view; he

merely quoted what appeared in the experimenter's statement, and that was the experience of a practical varnish maker. The author appeared to have met with the same difficulty, and to have obviated it by adding more oil of turpentine; but he must remember that the turpentine would volatilise and so much less of a coat would be left for a given weight of varnish. Therefore, he would like to know whether the author had manufactured varnishes by his process on a large scale, and had tried his product by exposing varnished articles for a long time to the atmosphere, and what sort of coat the varnish gave under those circumstances; whether it was hard, brilliant, and possessed good lasting powers.

Mr. W. F. REID agreed with the views expressed by Dr. Lewkowitsch. He thought it was probably owing to the lack of publication that the authors did not know that a similar process was carried out years ago and was still used on the large scale, though not in exactly the same way, in the manufacture of linoleum. Kauri gum and other resins or gums were put into a closed vessel provided with a safety-valve and warmed under pressure, and a varnish thus produced.

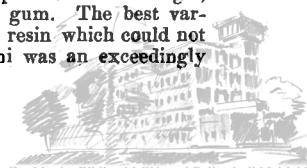
The CHAIRMAN: Under what pressure?

Mr. REID, continuing, said the pressure might be anything desired; but if one went beyond a certain temperature the whole mass was charred. He thought that the reason why the oil would dissolve the fused gum and remain clear, whereas, as Dr. Lewkowitsch had pointed out, gum not first fused would dissolve in oil and precipitate out on dilution, was that those portions of the gum which were volatile were retained when fusion took place under pressure. He had made quantities of varnish under pressure, but had always found that a varnish made in that way would "bloom" in course of time. He would like to hear from the author what was the result of exposing this varnish to the weather. His experience had been that it did not remain bright. Moreover, the gain in weight referred to was altogether fictitious. In fusing the gum in the ordinary way, one only drove off the volatile part of it, and the decomposition that took place made the gum much harder. Working under pressure in the present process prevented that, and so what one gained in quantity was lost in quality. The name of a gum was no guarantee of its composition. One might drive 10 per cent. of volatile matter from one sample and 20 per cent. from another. It was all a matter of experience, and no doubt the author would find that out when he came to work on the large scale.

Prof. W. RAMSAY said that there was one point which was interesting to him from the physico-chemical point of view. How was the pressure produced? Was it air pressure, or was it produced by the gases evolved from the gums themselves on heating?

The CHAIRMAN observed that, *à propos* of Prof. Ramsay's question, it was obvious that something was kept in the background by the author of the paper. The small apparatus shown would not bear much pressure, he it due to decomposition and evolution of gas, or to mere expansion. A pressure of two atmospheres, which entirely disappeared on cooling, could not have much effect on the reaction. From one expression—"oxidised gum"—used by the reader of the paper in the course of the evening, he gathered that possibly the gum went through some preliminary process of preparation before it was applied to the manufacture of varnish, and he asked the author whether this was so or not.

Mr. PETER MACÉWAN said to his mind the chief point of the paper was the statement that the old-fashioned method of treating insoluble resins by "running," so as to render them soluble, need not necessarily be carried out. He judged from the sample of varnish passed round that the simple method of solution proposed by Mr. Smith was effective, but it was not generally known. They now had the fact published that the use of the process of solution under pressure would do away with the "running" method by which 40 per cent. of the resins was lost. But Mr. Smith did not appear to have carried his experiments far enough; he seemed to have used only kauri gum. The best varnishes were made from gum animi, a resin which could not be compared with kauri. Gum animi was an exceedingly



hard and beautiful resin which no solvent would touch, and the new process should be tried on it as well as on copal and other varnish resins. He considered that a distinct service had been rendered to the varnish industry by this communication, for although much work had been done in it there was nothing quite conclusive. He recalled a paper which had been published in the Society's Journal some years ago. There the author showed that when the resins were so heated that the volatile products were passed over the resins, the latter were so altered as to become soluble. He could not help thinking that there was bound to be an immense difference between a varnish containing the whole of the constituents of the resin and one so altered in character by heating as to change from the insoluble to the soluble condition. Varnishes prepared from materials of widely different character were bound to give coatings equally different. He hoped the author would look into this thoroughly and also experiment with every kind of varnish resin of the "insoluble" class in the market.

Mr. A. J. SMITH said in reply, that whether the gum decomposed or merely fused was an open question at present. If the vessel which he used were left to cool, it was found that a partial vacuum was produced. The evolved gas all condensed back into the mass. It was to that fact that he attributed the extreme viscosity of the product. He did not think the gum was decomposed during the melting under pressure, but it certainly was in the open pan. Whether oxidation did not take place for lack of oxygen, he could not say; but no acids were found above the surface after cooling; hence he assumed that there was no decomposition in the process. That appeared to him to be an important point in connection with the wearing power of the fossil gum itself.

## Manchester Section.

*Meeting held on Friday, November 1st, 1901.*

### CHAIRMAN'S ADDRESS.

#### NOVELTY IN PATENTS ACCORDING TO GERMAN PATENT LAW.

BY DR. J. GROSSMANN.

A PATENT is a monopoly which is granted to an inventor under certain conditions. These conditions under the statute of monopolies are that the applicant should be the true and first inventor, and that the invention shall not be contrary to law nor mischievous to the state by raising prices of commodities at home, by hurt of trade, or generally inconvenient. The original statute of monopolies has been extended, though not entirely superseded, by the Patent Act of 1883 and amended Acts passed after that date. Like other Acts of Parliament, these Acts have been consolidated by judicial decisions; their scope has been gradually extended; manufactures have been held to include processes, and inventors have been held to include importers.

It is clear that the granting of a patent which confers a monopoly on the inventor is meant to be a reward for the work which the inventor has done. It is now recognised that a man is entitled to the benefits arising out of any application of his mind. On the other hand he should only be entitled to such benefits if there is a certain amount of originality in the outcome of his mind, something which has not been done before; but whilst it is very easy to decide in ordinary literary productions whether an author has simply copied from another author, and whilst in literary productions the same thoughts put into different language by different men would still be original as far as form is concerned in each case; it is different in the case of industrial inventions. The form in that case has nothing to do with the question whether an invention is novel. It is here simply a question of fact. Now although the law recognises

that an inventor should be recompensed by having a monopoly for his invention it does not in any way discriminate between the relative importance of different inventions; the monopoly is given whether the invention be of great importance or of very little importance; whether it shows a great amount of originality, or only very little; whether it is almost the natural outcome of previous improvements, or whether it is an entirely new departure. As a matter of fact nearly every invention is only the outcome of previous observations and publications, and the last inventor has only gone a little step further than his predecessor; and it may be safely assumed, in nearly every invention, that if that invention had not been made by the person who applied for the patent, it would not have been lost to humanity, but sooner or later another person would have found the same thing. The ordinary invention may be really compared to the worm which is caught by the early bird; but if that bird had not got up so early, the worm would have been doomed just the same, as the next early bird would have caught it. This is the reason why patents are limited to a comparatively short period, far shorter than the rights and privileges which are granted to literary productions. It must also not be forgotten that in the latter case little harm can be done by granting special privileges and exclusive rights, whereas in the case of industrial processes, rights and privileges granted in the wrong place may be of incalculable harm to the country which grants such privileges without careful investigation. It has been the custom in this country to grant patents indiscriminately to every one who applies, and although there has been a nominal alteration by which the Comptroller-General may interfere in the case of most glaring plagiarism, the fact is broadly as I have stated. The consequence of this is that there has been of late a feeling growing, amongst those conversant with patents, that an alteration in the English patent laws would be desirable, and as the outcome of that feeling a commission was appointed by the Board of Trade to inquire into the working of the Patent Acts on certain specified questions.

One of these questions was "whether, and if so, what additional powers should be given to the Patent Office to impose conditions on or otherwise limit the issue of later patents in respect to inventions which are obviously old, or which the information recorded in the Patent Office shows to have been previously protected by Letters Patent in this country."

One of the conclusions arrived at by the committee is as follows:—"That we are of opinion that the grant of invalid patents is a serious evil, inasmuch as it tends to the restraint of trade and the embarrassment of honest traders and inventors, and that this fact, coupled with the result of the foregoing inquiry, is, in our opinion, a cogent argument in favour of some inquiry as to anticipation by prior Letters Patent."

The "foregoing inquiry" referred to is one in which the committee endeavoured to ascertain approximately how many patents which were applied for had not been anticipated, and from which it appeared that only 57 out of every 100 could be classed as such, and that 43 per cent. of the patents applied for were simply repetitions of previous patents. The report continues: "We are therefore of opinion that, in addition to the existing inquiries, an examination ought to be made at the Patent Office into the question whether any invention claimed in a deposited specification has been claimed or described in any and what specifications of letters patent granted in the United Kingdom dated less than 50 years previous to the date of the application. That this inquiry should not be extended to provisional specifications which have been published, but not followed by a complete specification, and that, consequent upon the limitations of this inquiry an enactment should be passed to the effect that a publication of an invention in specifications of letters patent granted in the United Kingdom dated 50 years or more previous to the date of the application or in a provisional specification of any date of the kind before mentioned shall not of itself be deemed an anticipation of the invention." It will be noticed that these recommendations as to search for novelty only apply to such matter as has been published in previous patent specifications, and leave out any matter

which may be common knowledge through other publications, so that a patent granted under the committee's recommendations might still be granted for an obsolete invention or for no invention at all. This has been recognised by at least one very eminent member of the Committee, by Sir Edward Fry, who, in a note appended to the report, expresses himself as follows:—"I regret the omission of the inquiry whether the invention claimed is obviously old. I think that the inquiry, if made in conjunction with that into anticipation by previous specification, would add very little trouble to the examiner, and I fear that the omission of this inquiry may let through some of the patents which we desire to check."

Considering that there seems to be a general feeling in favour of alterations in the patent laws to the effect that patents should not indiscriminately be granted by the Patent Office, but that they should be examined for novelty, it may be useful to study the conditions as to what constitutes novelty in patents from the official examiner's point of view, and as the German Patent Office has been deciding these questions in many thousands of cases for the last 24 years, it may be of interest to put before you the latest opinions as to what constitutes novelty in patents in Germany.

Although the German Government does not undertake any guarantee that patents granted are necessarily valid, it examines the essential points of novelty in such a manner as to establish to a great extent the validity of a patent; whilst, therefore, the law in this country as to what constitutes novelty in an invention has had to be established on the basis of precedents taken from decided law cases, ranging over the last 60 or 70 years, and even further back, the German conception of novelty is supported by a much greater number of cases, which outside of law courts have been decided upon by the Patent Office before granting a patent. In order to have a clearer understanding as to the German Patent law, it is necessary to follow its history further back than 1877, the time when it was passed. Before the Franco-German war, Germany consisted of a number of independent states, each of which had a patent law of its own, which naturally was very ineffective, as would be the case for instance if each county in this country had a patent law of its own; in that case if a person happened to have a patent in Lancashire but not in Cheshire, it might be used openly by one almost within a stone throw of its owner. The Patent Law of 1877 was passed in order to consolidate all these different patent laws into one, and it became necessary to refer in some way to the different states and to their jurisdiction as regards patents in the former times. The general principles of the new patent laws, and their relative position to former patent laws, were therefore stated in special terms giving the motive of the act as follows:—

"The law cannot determine under what suppositions an object or a process for which a patent has been applied presents as a matter of fact something new. The office which examines the application has to judge on this according to the circumstances of each case."

The draft, however, in substantial accordance with the laws prevalent in Germany, denies the character of novelty to those inventions which, at the time of application, are either described in public print in such a manner, or are being used in the land already so openly that their use by other experts appear possible. Whether the publication of the printed matter is found in German or foreign publications shall be immaterial. The former use of the invention shall however exclude its novelty only if carried on openly, and within the realm.

The motives lay stress on this point, that the act is to be drafted in essential accordance with the law as it is expressed in Germany. With regard to that law, the first consideration at that time was the agreement with the different governments respecting the granting of patents and privileges, dated 21st September 1842. That agreement contains the principles which the different governments should observe with regard to the regulations of their Patent departments, especially also the patentability, and in that direction it says in article 1:—

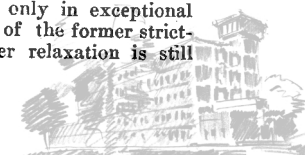
"Patents shall only be granted for such objects as are in reality new and characteristic; a patent must therefore not be granted for objects which before the date of the application have been worked within the combined states or which were current or in any way known at that time; patents should particularly not be granted for any matter which may already have appeared in publications, whether in German or any foreign language, and which is represented by description or drawing in such a manner that its working could be carried out by any expert. The decision as to novelty or individuality of the object to be patented remains within the judgment of the Government." The combined governments were accordingly bound to the foregoing conditions, but they were not prevented from taking even stricter views as to patentability or from restraining the granting of patents altogether.

A later Act of Parliament, that of 7th April 1891, enacts "that Patents are granted for new inventions which admit of industrial utilization, but the invention is not considered as new if, at the time of application, it has been described in public within the last 100 years, or has been used within the realm in such a manner, and so openly, that its use by other experts appears possible." This differs from the older enactment of the 25th May 1877 in this, that the words "within the last 100 years" have been added.

It will be noticed that the German law speaks of a "new invention," and this expression has caused a great amount of controversy; it is assumed by many that an invention must necessarily be new, others contend that something can be new without being an invention, and that there may be an invention which is not new. Caro puts the matter very clearly: 1st. "Patents are granted for inventions"; 2nd. "The invention must moreover be new"; 3rd. "A new invention must be of industrial applicability," and the patent office is therefore bound to conduct the examination in a threefold direction.

Without going into metaphysical speculations as to whether there are new inventions and old inventions, it appears that there is a great difference of opinion as to whether the German patent law exemplifies or defines the cases in which there is no novelty. Some authorities refer to the motives which say, that novelty is barred only when the invention has been carried on openly and within the realm. Others found opposite opinions on the passage in the motives, that the Government may judge novelty according to the circumstances of each particular case. The fact is that it is impossible to determine by law what is new, for nearly every invention may be divided into a number of well-known elements which are simply combined, and it is left to a great extent to individual opinion to determine whether this combination is of such a character as to constitute novelty or not. One thing stands out clearly in following the decisions of the German Patent Office through the last 25 years, viz., that the officials are adapting themselves to the requirements of the changes which have taken place in technology, and are justly taking broader views as to novelty.

A highly interesting article touching partially on this point has been published in the "Zeitschr. f. angew. Chem.," 1901, Nos. 36 and 37, by Dr. Julius Ephraim, and I have in the following taken the liberty of partially abstracting and partially translating this paper, as it gives us the latest views on points of interest in German law. The author commences by explaining that the natural development of the chemical industries and the natural evolution of patent law must necessarily lead to new points turning up, which in former days did not come into consideration. These questions will affect more or less every industry, and in discussing some of the questions which particularly apply to the chemical industry, he first considers previous publication as far as it affects chemical patents. It appears that for the last two years the examination of applications for patents in Germany has been carried on chiefly so as to ascertain whether the object of application has been completely published in literature; whereas the examination as to whether the object constitutes an invention is now in most cases neglected, and takes place only in exceptional cases. This is no doubt a relaxation of the former strictness, and yet it appears that further relaxation is still



desired by a certain class of technologists. As stated before, under the present law, an invention is not considered new if it has been published in print during the last 100 years in such a manner that it may be used by experts. Those who require further relaxation now claim that such publication shall only prohibit the granting of a patent if it describes the invention in a technically applicable form. But it would not always be an easy matter to determine this point, as the following instance will prove:—"When Buchner published his first investigations on zymase, there could be no doubt that the process, however minutely described, was a purely scientific one. No doubt an expert would be in a position to follow that description, and to get the same results. The question is, was that invention described in a technically applicable form? At the time C. Wehmer declared that he would not believe in Buchner's discoveries until the product could be obtained in commerce." Thus we see that an expert gave it as his opinion that the process did not represent a technically applicable process. If, therefore, the new relaxed forms of the German Patent Office were to be accepted, an application which contained nothing else but the description which Buchner himself had originally given would be patentable, as long as the applicant could show that this process, contrary to original views, was technically applicable. After Buchner's publication his discoveries were further followed out. Albert succeeded in finding a new and considerably simpler process for the preparation of zymase, and the inventor expressed the hope that soon Wehmer's requirements would be fulfilled. Without doubt Albert was of opinion that he had found a process which was technically practical, so that his publication could be locked upon as a prior publication with regard to future applications for patents. On the other hand, Albert was not quite certain, as he only expressed a hope on the point; so that it is clear that even if the Patent Office should relax its rules and extend the scope of prior publication, many doubtful cases would occur.

The further relaxation of the German patent law would particularly affect organic chemistry. Many products which at one time were considered far too expensive to ever enter into industrial application have become quite accessible through later fortunate discoveries. Take the case of two substances, one a substance of industrial importance, another the raw material for the preparation of that substance. Suppose the final product could not be commercially made because the known methods for the manufacture of the raw material were too expensive. Suppose a discovery is made by which the raw material can be cheaply prepared, should it then be possible to obtain a patent for the preparation of the final substances, which, since the new process for the raw material has been found, has become technically important? If scientific publications are not to be considered as prior publications, it might lead to very serious and unpleasant consequences.

The nature of chemical science is such that in many cases the scientific results can be at once transferred to technical work. This fact has been acknowledged in German patent law; in France, on the other hand, the laboratory experiment, even when it has been published, does not prevent the taking out of a patent for the same substance. The differences in the views which the law takes are particularly prominent in the case of Bullier's Calcium Carbide patent. The facts were the same in Germany as in France. The patent claimed by the German patent No. 77168 is as follows:—"Process for the preparation of carbon compounds of the metals of the alkaline earths consists in heating a mixture of the oxides, carbonates, &c. of the alkaline earths with carbon in an electric furnace." The question arose then whether this claim is anticipated by the previous publication of Moissan. "At the same temperature (3,000°) carbon quickly reduces calcium oxide. The metal is copiously produced, and combines easily with the carbon of the electrodes, forming a red hot fluid calcium carbide, which can be easily collected." In Germany this publication was considered as prior publication, in France however it was decided as follows:—"A communication made by a member of the Academy of Science, and which does not lead to an industrial result, but establishes simply a case

which has been observed in the course of laboratory experiments, that is a scientific fact, cannot constitute a prior publication with regard to a patent which is taken for a process and a new product." The consequences of adopting the French views are clearly seen in this example. As long as on repeating a scientific work it appears that industrial results can be obtained, a patent could be taken according to French law, which as a matter of fact would not disclose more to an expert than the original scientific publication. Thus the conditions necessary to convert the laboratory experiment into a manufacturing process must be worked out in each case by the expert, no matter whether the process is intended for technical or scientific purposes. As a matter of fact it appears that in Bullier's carbide patent those conditions which were found to be necessary in order to get over the difficulties of making carbide on a large scale, and without which it would have been impossible to introduce the process as a manufacturing process, were neither mentioned in the German nor the French patent application; so that what might have been unquestionably patentable remained unpatented, and was not disclosed to the public. The law thus protected what experts already knew, whilst the further knowledge which was absolutely necessary was not published. The conception of an invention as constituted by the present law is particularly suitable to prevent such cases, and should therefore not be altered. The restriction to 100 years in prior publications with regard to novelty is of some importance to chemical industry. In an action for novelty against the Lanoline patent, passages from Pliny and from Pharmacopœa of the middle ages were quoted against this patent, which as a matter of fact has become the foundation of an entirely new industry. Still on reading these old publications, any modern chemist would assume that statements mentioned in them, and not confirmed by later observers, were likely to be incorrect. The conditions under which experiments were carried out and observations made in those days were so different from those prevailing at the present time that these old statements require confirmation by later investigation. On the other hand, by the light of present knowledge a great deal may be found in old publications which has some remote bearing on modern problems. We need only draw attention to the fact that for instance Cavendish threw out hints with reference to the existence of Argon, but that it was always assumed that there was an error in his observation. But although these old publications, even when they describe a process completely, are of no value to the modern technologist in his practical work, still they may be brought forward as objections to new inventions. In Marggraf's *Chymische Schriften* it is stated that a rosin smelling like musk may be obtained from Amber Oil and Nitric Acid. But Amber Oil contains Butylxytol, from which, by nitrating, Baur obtained artificial musk. The use of liquefied air in mines might be considered to have been anticipated by A. von Humboldt's proposal of using Oxygen. A description of the safety explosives, Roburite and Helhofit, may be traced as far back as the 16th century, that is, to a time when no one would have thought of safety explosives. Such publications will not always interfere with the priority of a patent, but they may cause the patentee considerable trouble. It is, therefore, perfectly correct to leave out the question of old publications, particularly as they are often written in obscure language, and as we cannot examine them in the light of our present views. The question will, of course, arise, whether the limit put down by the German law is the most suitable limit. The fundamental idea of a restricted period is no doubt this, that only such literature should come into consideration which existed at the beginning of our modern industries. But as we are advancing in years, whilst the origin of the modern industry is practically a fixed point, it would be better to fix the date, and assume the year 1800 as the date from which publications should be admissible. This question is of considerable importance, as in the course of time the 100 years limit would bring us to a period when industrial processes were fully discussed and published in such journals as Dingler's journal.

Dr. Ephraim proceeds to consider how far analytical processes should be admissible for patents, but this is a





question which, after all, though of course important, is not of such general importance as the previous and the following subject, and as I am limited to time, I leave this part of his paper out, and proceed to the conclusion, which treats of an extremely important subject, that is the consideration of processes for the preparation of intermediate products. It is on this point particularly that the difficulty of determining the novelty or the patentability of a process is most marked. These intermediate products have become of the greatest importance in the coal tar industry. Take, for instance, the Azo colours, which owe their development mainly to the naphthol sulphonic acids, dioxynaphthalinsulphonic acids, naphthylaminsulphonic acids, amidonaphtholsulphonic acids, and so on. The importance of these intermediate products is also particularly evident in the process for the manufacture of artificial indigo. As long as it was only a question of analogous processes, that is, of the application of methods which had been used for practically the same purpose with compounds of analogous constitution, the views and decisions of the Patent Office with regard to new processes varied. For some time patents were granted for the preparation of new substances, even if only a certain application of the compound would yield a colouring matter of new technical effect, and a patent was granted for the preparation of an intermediate product. But the Patent Office sometimes changed its position, and would at another time refuse a patent if by an analogous compound a new chemical compound had been obtained by using which the applicant produced a new available colour. In such cases a patent only for the preparation of the new available colour would be granted.

The difference between the two views taken in judging these matters is as follows. When the process for the preparation of an intermediate product is generally protected the use of the product by another party for the preparation of a colour depends entirely upon the permission given by the owner of the patent for the intermediate product. If, however, the process for the manufacture of a special colour is patented only in such a way that the preparation of the intermediate product forms but a link in the preparation of the colour, the intermediate product may be made by anyone, so long as it is used for the preparation of another colour or product. The patent for the preparation of the colour makes intermediate products and its preparation generally known without protecting the inventor further than as far as its use for the preparation of a particular colour is concerned. As in most cases, an intermediate product may be used, not only for a single colour, but for a further series of colours, it leaves it open to others to use every intermediate product for experiments, which may lead to important results in other directions than those pursued by the original inventor. In that case, of course, the original inventor would receive no remuneration, whereas, otherwise, he would have a claim on any further inventions, dependent upon the use of his intermediate product.

It is indifferent as far as patentability is concerned whether the product is of technical importance, unless technical importance is just the point on which the inventor bases his claim, and which can only occur when the product is a new chemical compound. Thus, if the process for the production of phthalic acid, according to patent 91,202, had not been patentable for any other reasons, its patentability would not have been justifiable on the ground that phthalic acid was an article which was wanted, and of importance. But the circumstance that the presence of metallic salts, during the action of sulphuric acid on naphthalene, produce such an extraordinarily favourable yield in phthalic acid justifies the grant of the patent, assuming that it makes no difference in patent law, whether phthalic acid was at the time of technical importance or not.

Up to quite lately both the German Patent Office and the German Courts of Justice agreed in their views that there was no doubt as to the technical applicability of intermediate products. But in the latest decision of the High Court of Justice on the ionon patent, there is a passage which denies the industrial applicability of intermediate products.

The claim of the ionon patent, 73,089, is as follows:—"A process for the preparation of a new perfume, called ionon, from citral, and acetone, consisting in condensing the two compounds mentioned by alkaline agents, first into a new ketone, pseudo ionon, and in converting the latter or its condensation products with phenylhydrazin or other ammonia derivatives by acids into a ketone isomeric with ionon." The decision of the High Court was as follows:—"The invention, therefore, did not exist in this process, but only in the process, as far as it yielded a new substance, valuable in technology. The intermediate product was not an industrially applicable new substance."

The whole question, no doubt, is in close connection with the conditions of the German patent law, by which an invention is patentable, where it shows a new technical effect. What forms a technical effect is independent of scientific or theoretical considerations, but should be decided by those who are directly interested in the matter, that is, those who are engaged in chemical industries. If technologists can agree how a technical effect is to be interpreted in general cases, the law must follow the views which the technologists take, but in that case it would be necessary that all technologists should agree on the point, and not only those who have particular interests in a particular case. Here we come to a most important point, which does not only affect the German law, but the English law, that there are many clashing interests in connection with the patent question, and that the interest of the inventor, and the interest of those who may through that invention either gain or lose money are different points. As far as the German law is concerned, it is clear that if the views as regards patents of intermediate products undergo any alteration they will effect an alteration in the future as to the rescinding of patents. In section 11 of the Patent Law, No. 2, it is laid down that after the lapse of three years from the date of publication of the patent, the patent can be rescinded, if in the public interest the granting of licenses appearing necessary the patentee has refused to give licenses against adequate remuneration, and sufficient security; but so far, according to decided cases, "public interest" is not affected, if the later inventor without permission of the holder of the raw material patent may not use his own invention. If the question of patenting intermediate products is to be decided on a broader basis, this point will have to be altered and be decided in a broader sense. If the necessity for giving licenses is not increased, patents on intermediate products may prevent the carrying out of later inventions in a considerable degree.

I have gone particularly into the matter of intermediate products more fully as it is a development of patent law, which though particularly appertaining at present to organic chemistry, may be of far reaching effect in the course of time even in inorganic chemistry. It is a subject also which shows more clearly than any other the clashing interests which have to be considered in the formulation of patent laws, and in the present unsettled state of the English patent laws, I trust it may be useful to draw attention to the fact that even in the German patent law, which is generally and justly looked upon as a model of patent law, points have arisen on which those interested in the matter are evidently not satisfied. In any alteration of the patent laws in this country, it is to be hoped that not only will patent agents, lawyers, and manufacturers who work inventors' patents be consulted, but that the inventor also will have something to say in the matter. There is one particular lesson that we should learn from German experience, and that is that in searching for novelty it will be no use to go back only to published specifications of patents, but that it will be necessary to consult literature in general. But perhaps the most important point, and one that I have not been able to touch on this occasion is this, that in any alteration of the patent laws it should be clearly laid down that wherever anything has been mentioned in previous patent publications, which has evidently never been worked or even tried on a practical scale, and which has only been put into the specification for the purpose of extending its scope in an unlimited manner, and of preventing others from ever touching the subject, should not be considered as previous publication.



## THE REPORT OF SIR EDWARD FRY'S COMMITTEE ON PATENT LAW.

BY IVAN LEVINSTEIN.

SEEMING that we can no longer depend on the expansion of our staple trades for finding employment for our ever-increasing population, it is the duty of the Government to encourage the establishment of new manufactures and industries in this country. Patent laws were originally enacted for the very purpose of finding work for our people, by encouraging inventors and others to introduce new manufactures within this realm.

Our present laws, however, do the very opposite. The most important question, with regard to which amendment is wanted, is whether a foreign subject to whom we have granted a monopoly in this country should manufacture the patented article in our empire, or whether he need only produce it abroad and import it into this country. If we grant a monopoly to any person, foreign or otherwise, and allow him to produce the article abroad, our industrial interests are seriously injured, for the following reasons:—

1. We support foreign labour, and assist in the development of foreign industries.

2. We throw out of employment a certain number of our own workpeople, as a newly introduced article generally replaces one already in use.

3. We stifle invention, and increase the cost of production by closing the door to our own inventors and manufacturers, whilst our foreign competitors are at liberty to make and vend the patented article without any restriction or royalty, since very often the foreign patentee has not succeeded in obtaining a patent in his own or other countries.

4. We grant the foreign patentee in many instances a practically permanent or continuous monopoly, since, at the expiration of the patent, there is no skilled labour in this country trained in this special manufacture.

If, however, the monopoly is worked in this country, the advantages to our trades and industries are:—

1. The monopolist finds employment for our workpeople.

2. He contributes to our local and Imperial taxation.

3. He greatly benefits other collateral industries.

As an illustration of the benefit derived from working a foreign patent here, take the case of the Otto gas engine. Messrs. Crossley succeeded in getting a license from Otto, an Austrian subject, who had obtained a patent for an improvement in the production of a gas engine. During the existence of this patent, Messrs. Crossley paid in wages and salaries to British workmen nearly two millions sterling, and they made for themselves a great fortune, much of which has been most generously devoted by them to British hospitals and charitable institutions. On the expiry of the patent some half-dozen other makers started to produce these engines, and to-day we are among the largest producers of gas engines. If this patent had not been worked in this country, the manufacture of gas engines would probably be to-day, and for years to come, a foreign monopoly, like many other articles. Take the case of Messrs. Brunner, Mond, and Co. Dr. Mond succeeded in getting a license from Solvay, a Belgian, to make in this country ammonia soda, and thus was this firm established in this country. They have paid in wages to British workmen more than four millions in money, besides largely benefiting, like Messrs. Crossley, other collateral industries. When the patent expired, other makers started the production of this article, and, but for the establishment of its manufacture in this country, a large portion of our chemical industries would have been ruined. There are probably ten times more foreign inventors than British—assuming the ingenuity of Americans, French, Germans, &c., to be equal to our own—simply because the population of the Continent and America is ten times that of Great Britain.

The Government appointed a Committee under Sir Edward Fry to consider, *inter alia*, the question of working patents, and the result of that Committee's enquiry is a recommendation that a patentee may work his patent abroad and give no license here so long as he supplies the requirements of the British public.

No more suicidal policy has ever been recommended by any Departmental Committee. The Committee in this case consisted principally of lawyers, patent agents, and officials, with only two outside members, who knew very little of patent law.

During the proceedings Mr. Bousfield, K.C., who was examined by Lord Alverstone, a member of the Committee, stated that he was under the impression that it was most desirable to have as many manufactures as possible worked in this country. To which Lord Alverstone replied, "the economic question is not material to our present enquiry." This reply requires no comment. After the publication of Sir Edward Fry's Committee's report the Manchester Chamber called a conference of industrial chambers, which met in Manchester in June last, when a number of resolutions were carried, among which was one disapproving of Sir Edward Fry's Committee's recommendation on the question of working a patent. The author was delegated by the Manchester Chamber to bring these resolutions before the autumnal meeting of the Associated Chambers of the United Kingdom, which met in September last at Nottingham, and these resolutions were all confirmed with practical unanimity. An amendment was proposed by a member of Sir Edward Fry's Committee approving of the report, but it was defeated by an overwhelming majority.

There is only one practical solution of this question, and this is, that, if a patentee will neither work his patent himself in this country nor let others work it, it may be revoked, as is the law in France, Germany, Russia, Austria, &c.

It is contemplated to organise a powerful deputation to wait on Mr. Gerald Balfour, the President of the Board of Trade, to put before him the resolutions of the Associated Chambers of Great Britain, and to urge him to take steps for the speedy amendment of the law.

## New York Section.

Meeting held on November 24th, 1899.

MR. T. J. PARKER IN THE CHAIR.

### THE DEATH OF Mr. WALDRON SHAPLEIGH.

Before the business of the evening commenced, Prof. C. F. CHANDLER said:—

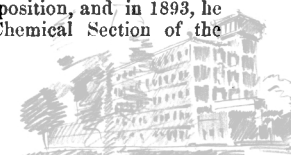
I rise to perform the painful duty of announcing the death of our beloved friend and fellow member, Waldron Shapleigh, which took place at his summer home in West Lebanon, Maine, on August the 30th, 1901.

Mr. Shapleigh was the son of the late Marshall Spring Shapleigh, of an old Maine family. He was born at Philadelphia in 1848. He studied chemistry in Philadelphia under the late Prof. Williams, and made such rapid progress that at the age of 19 he was appointed superintendent of the zinc works in Blair County, Pennsylvania. A year later he accepted a position as instructor of chemistry at Lehigh University at South Bethlehem, Pa. He remained there several years, and took charge of the chemical department *ad interim* on the death of Prof. Wetherill.

He then decided to devote his attention to the beet-sugar industry, and went to France, where he devoted two years to the study of this subject. An article by him on "The Cambrai Central Beet-Sugar Works, Nord France," appeared in the *American Chemist* for February, 1874. On his return from France he was appointed chemist to the King's County Sugar Refining Company of New York, then engaged in the manufacture of glucose—a position which he held for ten years, until the company was dissolved. He was for a time at Peoria, Illinois, in charge of a glucose establishment.

When the inventions of Welsbach were introduced into the United States, Mr. Shapleigh was appointed chief chemist to the Welsbach Company, and he maintained his connection with this company until his death.

In 1873, he was appointed by the Governor of Pennsylvania a Commissioner to the Vienna Exposition, and, in 1893, he was appointed a Judge of the Chemical Section of the





Columbian Fair at Chicago. He was a Fellow of the London Chemical Society, the American Chemical Society, the Society of Chemical Industry, and the Franklin Institute.

He is chiefly known by his work in connection with the rare earths associated with thoria in the monazites and the various other minerals in which it occurs. He showed distinguished ability in devising methods for separating these earths, and from the foundation of the American Welsbach Company he was able to prepare all the materials required by that company, so that the company was not obliged to purchase its mantle fluid from Vienna, as all the other companies were. It is very unfortunate that the confidential relations of Mr. Shapleigh to the Welsbach Company made it impossible for him to publish the results of his investigations upon these earths. This fact will deprive him of the credit to which he is justly entitled of unravelling to a remarkable degree the intricate chemistry of these earths. He has received medals for the beautiful collection which he prepared of the rare earths, especially for his salts of neodymium and praseodymium.

Mr. Shapleigh was a man of very unusual character. His mind was strong and clear and his temperament energetic, and he always gave the impression of possessing a great amount of reserve strength; at the same time he was very gentle in his disposition. He made a great many friends, and always very warm friends, by whom he was deeply loved, and his loss will be much lamented. He leaves a widow, a son, and a daughter.

### PROPORTIONS OF LIQUID FATTY ACIDS IN SOME FATS AND OILS, AND THEIR IODINE VALUES.

BY N. J. LANE, B.S.C.

In a paper published some years ago (J. Amer. Chem. Soc. 1893), I suggested, as a valuable improvement of Muter's method in determining the amount of liquid fatty acids and

their iodine values, boiling the lead salts with ether under a reflux condenser, and then allowing to cool slowly.

The method was also tried with petroleum ether, with very satisfactory results.

The following results were obtained with samples kindly furnished by Mr. Goulard, of Thomas Goulard and Co., the official inspectors for the New York Produce Exchange:—

	Steam Lard.	Oleo- Stearin.	Cotton Oil.
Liquid acids by ether (per cent.).	60.03	23.02	71.98
Liquid acids by petroleum ether (per cent.).	57.51	25.86	72.31
Iodine absorption by ether method.	100.8	75.70	140.1
Iodine absorption by petroleum ether method.	101.1	80.58	142.2
Titre test (° C.).....	..	50.2	34.3

A sample of artificial lard, prepared from the above fats in the following proportions—

	Per Cent.
Lard .....	70.00
Oleo-stearin .....	10.00
Cotton oil .....	20.00
	100.00

gave, by the petroleum ether method:—

	Found.	Calculated.
Liquid acids .....	Per Cent. 56.84	Per Cent. 57.31
" iodine absorption .....	113.9	111.6
Cotton oil .....	20.22	20.00

The following tables contain a number of results obtained by the improved Muter method:—

Sample of	Liquid Acids.	Iodine Value of Liquid Acids.	Titre Test of Fatty Acids.	Iodine Value of original Fat		Saponifica- tion Value.	Acidity as Oleic Acid.	
				Found.	Calculated from Iodine Value of Liquid Fatty Acids.			
Commercial lard.....	Per Cent. 69.60	95.94	° C. 33.1	..	..	..	0.45	Contained 4 per cent. of oleo-stearin. Run with ether.
Steam lard.....	65.76	97.18	36.9	..	..	..	0.48	
Cocoonut oil .....	30.21	18.58	23.0	..	..	..	4.08	
Oleo-stearin .....	29.41	77.82	48.7	..	..	..	0.50	
Cotton-seed oil .....	75.51	157.8	82.4	..	..	..	..	
" .....	76.85	157.3	33.2	..	..	..	..	Solid acids, grav. 13.61.
Bleached cotton-seed oil ..	70.50	149.8	32.7	103.2	106.1	..	0.72	
Olive oil .....	89.97	97.20	..	..	..	..	..	
Cod liver oil .....	69.90	167.6	..	..	..	199.0	..	
Sesame oil .....	78.05	139.9	21.4	..	..	189.2	..	
Peanut oil .....	78.63	116.2	..	..	..	191.0	..	Fresh sample. Old sample.
" .....	77.25	112.9	..	..	..	190.1	..	
" .....	81.64	119.5	..	..	..	190.5	..	
" .....	78.89	111.0	..	..	..	185.5	..	
Chinese peanut oil .....	80.26	131.0	..	..	..	..	..	
Japanese wood oil .....	89.62	179.7	..	..	..	194.8	..	
" " old sample.	87.80	178.4	..	..	..	191.4	..	

	Liquid Acids, Volumetric Determi- nation.	Liquid Acids, Gravimetric Determi- nation.	Iodine Value of Liquid Acids, calculated on Volumetric Determi- nation.	Iodine Value of original Fat found.	Iodine Value of original Fat calculated (I. val. L.A. × L.A. %).	Saponifi- cation Value.	
Castor oil.....	90.28	91.28	93.27	..	..	..	Liquid acid as ricinoleic. Petroleum ether and ethylic ether gave same result.
Walnut oil, fresh .....	91.09	..	167.0	150.2	152.3	191.6	
" rancid .....	85.57	87.32	146.7	129.5	125.5	..	With petroleum ether. Ethylic ether.
Cocoonut oil .....	44.22	..	17.86	7.71	7.90	257.4	
Tea-seed oil .....	93.22	..	99.61	90.49	92.85	190.1	With petroleum ether.
" .....	88.06	..	104.4	90.49	91.96	190.1	



## Nottingham Section.

Meeting held on Wednesday, October 30th, 1901.

MR. L. ARCHBUTT IN THE CHAIR.

### THE HEAT PRODUCING POWER OF FUEL.

BY ARTHUR ADAMS.

(*This Journal*, October 1901, 972.)

#### DISCUSSION.

Mr. WM. THOMSON wrote, stating that he had always used oxygen which had stood for 24 hours over water in a gasholder before use, and had made tests to find out whether carbon monoxide was produced during the combustion, but had failed to detect it. He had generally found the ash of the fuel left as grey-fused pellets, free from carbon. As the bubbles of gas were broken up four times by passing through the wire gauze surrounding the glass diving bell, he thought it might be taken that the gas left the apparatus at the same temperature as the water in it. In regard to the statement that an error of 1 mm. in reading the water level caused an error of 0.25 to 0.3 per cent., he never measured the water in the calorimeter itself, but either weighed it or measured from a litre flask delivering 1,000 grms. weight of water, and he took into consideration the radiation by taking half the fall of the thermometer during the period required to make the experiment, and adding it to the total rise of temperature. It had been suggested that the solution of the carbonic acid in the water would tend to raise the temperature of the water in the calorimeter, but he thought this not a serious error, because on burning pure carbon the results obtained were very close, if not identical, with those obtained by Favre and Silbermann.

Mr. R. M. CAVEN could well appreciate the importance of the two points dealt with in Mr. Adams's paper; the first, however, seemed to be met by Mr. Wm. Thomson's statement that he always employed oxygen saturated with aqueous vapour. With regard to the supposed incomplete combustion of the fuel, owing to the dryness of the oxygen, it seemed doubtful if the gas supplied direct from an ordinary cylinder would be "dry" in the sense of the term as used by Dixon and Baker. It was to be regretted that the author did not analyse the issuing gas, so as to be able to state the actual amount of carbon monoxide present.

Mr. J. T. WOOD said he understood that for all accurate estimation of calorific values, Mahler's Calorimetric Bomb was now used. This was not mentioned by the author of the paper.

The CHAIRMAN remarked that Mahler's calorimeter was largely used. It consisted of a strong steel shell, nickel plated on the outside and enamelled inside, in which the coal or other combustible substance was burnt in oxygen compressed to 25 atmospheres. The apparatus, though rather costly, was simple, and easily manipulated, and with a substance of known calorific value, such as pure naphthalene, it gave exact results. Mr. Reddrop, the late chemist of the L. & N.W. Railway, who had used this apparatus for several years, had obtained some very interesting comparative values by testing the same coals both in Mahler's and also in Lewis Thompson's calorimeters. The results given in the table, which he had Mr. Reddrop's permission to publish, were obtained with four samples of coal.

These results show that Lewis Thompson's apparatus, though admittedly imperfect, gave results which would be sufficiently accurate for many practical purposes, the error ranging in the above coal from +1.4 per cent. to -5.5 per cent. The error was greatest with coals yielding high percentages (80 per cent. and upwards) of coke, probably owing to the carbon of such coal not being completely burnt in the Lewis Thompson calorimeter. Wm. Thomson's calorimeter was a much more perfect apparatus than Lewis Thompson's. The author's remarks were a little misleading, for in the original description of the apparatus

Description of Coal.	Calorific Value determined by		Sulphur. Per Cent.	Coke. Per Cent.	Ash. Per Cent.
	Mahler's Calorimeter.	Thompson's Calorimeter.			
Lancashire ...	7,552	7,370	0.62	67.4	7.5
Staffordshire. {	7,968	..	1.33	..	..
	7,991	8,085	1.33	65.5	2.8
	7,977	..	1.25	..	..
Monmouthshire	8,327	7,965	0.74	82.5	6.1
Glamorgan-shire. {	8,411	..	0.74	..	..
	8,425	..	0.74	..	..
	8,450	7,920	0.69	89.6	3.2
	8,448	..	..	..	..

(*this Journal*, 1886, 581) a gasholder of special construction was described, in which the oxygen was stored over water, and was forced from the holder by admitting water at the bottom through a tube connected with the town main. As, however, the use of compressed oxygen direct from cylinders was now so common in laboratories, the author had done some service in calling attention to the necessity of saturating this oxygen with water before it was used. He thought, with Mr. Caven, that it was a pity the author had not made his paper more complete by giving some analyses of the products of combustion passing from the calorimeter when unsaturated and saturated oxygen were used.

Mr. F. J. R. CARULLA asked whether any calorimetric experiments had been made to show what difference there was in heating power between small and large coal from the same seam. Better results were obtained in practice from screened coal than from the screenings of the same coal, and yet the calorimeter might give the same value for both.

The CHAIRMAN said that the Lewis Thompson calorimeter would be well adapted for such comparative tests, as it was very expeditious in use. He should think that the large coal would give the higher results in the calorimeter.

Mr. WARD said that small coal generally contained more ash than the larger coal.

Mr. ARTHUR ADAMS said, in reply, that on reference to Mr. Wm. Thomson's original paper, it would be found that although a special gas holder was suggested, the importance of the presence of water vapour was in no way indicated, and in probably 99 cases out of 100, oxygen direct from the bottle, unsaturated, would be used. The Berthelot-Mahler calorimeter was more exact, as the products of combustion did not come into contact with water, thus preventing certain errors; it was far more expensive and less used than Mr. Wm. Thomson's form. The comparative results contributed by the chairman were of much interest, particularly in the case of the Staffordshire coals, where the result in Lewis Thompson's calorimeter was higher than in the Mahler calorimeter; this was probably due to the sulphur being burned with the formation of potassium sulphate, in the former case, while it would be mainly burned to sulphur dioxide in the Mahler calorimeter. The results seemed to prove that Lewis Thompson's form was not satisfactory, as the results were not uniform. A series of comparative experiments with the Thomson and Mahler calorimeters would be of considerable use and interest.

It was pointed out in the paper that compressed oxygen was not "dry" in the same sense as that of Dixon and Baker. It was saturated when compressed to 120 atmospheres, that is, the expanded gas would contain less than 4 milligrams per cubic foot, about 0.01 per cent., and it was sufficiently dry to cause incomplete combustion. There was a perceptible difference in the burning when moist oxygen was used; the combustion appeared to be slower. Want of time prevented the analysis of the gases, and in the last three months, since sending in the paper, it had not been possible to continue the experiments. Mr. Thomson's remarks showed that there was complete combustion with moist oxygen, while Fischer's experiment, quoted in the paper, and all the classical experiments of Favre and Silbermann, Berthelot, &c., showed that there was incomplete combustion generally. As to the measurement of water, weighing was



undoubtedly the best, but was little practised. It was not possible to make an error in measuring, of 1 mm. i.e. 0.25—0.3 per cent. without carelessness, so that measurement by noting line on beaker was near enough in most cases. The solubility of the gases in water, more particularly the sulphur dioxide, which varied with the sulphur present in the coal, was a possible source of error, which required examination. Screened coal gave higher values than screenings, and contained less ash.

### THE EFFECT OF TANNING EXTRACTS CONTAINING BISULPHITES ON LEATHER.

BY DR. J. GORDON PARKER AND AUGUST GANSSER.

Contribution from the London Leather Industries  
Laboratory, Herold's Institute, Bermondsey.

THE following research was commenced by us in order to clear up a question of great interest to tanners, and which it was impossible to solve until Prof. Procter devised a rapid and exact method for estimating mineral acids in leather.\*

The questions to be solved were whether tanners were correct in their view that leather, tanned with extracts which have been treated with bisulphites, and which smell of sulphurous acid, is brittle and liable to crack, and, if so, is the brittleness due to the presence of bisulphites, and to their production of mineral acids in the leather?

We have found no researches or published results on which tanners could have based their theory that the sulphurous acid oxidises into sulphuric acid, and so renders the leather liable to crack. It was therefore desirable to see whether leather, produced by the aid of this extract, does contain free mineral acids.

The extracts on the market, which have been treated with bisulphites of soda, are patented,† and are rendered perfectly soluble in cold water or cold liquor. An analysis of such an extract showed that sulphurous acid was present, partly as neutral sodium sulphite, partly as bisulphite, and partly in such a form that it was not liberated by the addition of strong mineral acids. This last was found to be nearly proportional to the temperature which had been used in making the extract, and appears to combine with and render soluble the phlobaphenes, which are difficultly soluble tannin matters. The proportion which is so bound has not been estimated, but no free sulphuric acid is formed from it, nor can sulphurous acid be liberated from the combined sodium sulphite.

The extracts referred to are known on the market as "Mimosa D," Tanning Extract A.T., and Quebracho Extract S.F. (manufactured by A. Huillard and Co.); but besides these, there are also other extracts, which, although not decolourised by means of sodium bisulphite, contain 1 to 2 per cent. of this material, which has been added to the extract subsequent to manufacture solely for the purpose of preserving it, and, to a slight extent, of brightening the colour.

The extracts produced by the Lepetit, Dollfus and Gansser patent, smell so strongly of sulphurous acid, that tanners expect that the leather tanned with them will contain free mineral acid, especially when the leather dries brittle. Unfortunately, all the conditions which go to cause leather to become brittle are not known.

On investigating several samples of leather tanned in different tanneries, and samples tanned under our own supervision, we found every sample absolutely free from all traces of sulphuric or sulphurous acids. The brittleness of the leather, therefore, could not be put down to the action of acid. The question thus resolved itself into an investigation whether brittleness was more prevalent in leather tanned with these extracts than in leather tanned otherwise. One is often liable to use a highly clarified,

rapid tanning extract in great strength in order to add weight to the leather, forgetting that these light-coloured extracts are in reality very strong solutions of tannin.

The brittleness can, in some cases, be put down to the use of extracts which have been improperly decolourised, and by a deposition of difficultly soluble tannins on and in the surfaces of the leather. Light-coloured extracts are used largely by sole leather tanners in the later stages to lighten the colour of the leather tanned in the earlier stages with cheaper and darker coloured materials, and where in such a case a brittle leather is produced, one is liable to put it down to the last extract used.

It is, therefore, of the highest importance to find out, if possible, the cause of leather drying brittle, and if this be not possible, then at least to ascertain which of the numerous theories put forward by tanners is correct.

**Experimental Part: A. Trials with "Mimosa D."**—Two pieces of calf grain, marked 1 and 2, were tanned with two different samples of commercial "Mimosa D," and the leather so tanned was afterwards tested for acid by Procter's method. At the same time, the alkalinity of the ash was estimated as follows:—

Three grms. of the leather were reduced to ash in a platinum dish. To this was added 10 c.c. of N/10 HCl. The soluble part was then titrated with N/10 Na<sub>2</sub>CO<sub>3</sub>, using methyl orange as indicator. In a similar manner a piece of goat skin, which had been tanned in a third sample of extract, was similarly treated. As the following results show, the three samples of leather, instead of containing any free mineral acids, were each in an alkaline condition.

The estimation of acid gave the following result:—

	Sample 1.	Sample 2.	Sample 3.
Amount of added N/10 HCl ....	c.c. 30.0	c.c. 30.0	c.c. 30.0
Amount of N/10 soda required ..	23.6	27.1	26.7
Difference .....	3.4	2.9	3.3
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub> ..	0.61	0.51	0.58
Calculated in percentage of CaCO <sub>3</sub> ..	0.53	0.48	0.54

The alkalinity of the leather ash gave the following results:—

	Sample 1.	Sample 2.
Weight of ash in grms. ....	0.0433	0.0395
Amount of N/10 HCl added .....	c.c. 10.0	c.c. 10.0
Amount of N/10 soda required ...	4.5	4.4
Difference .....	5.5	5.6
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub> ..	0.97	0.98
Calculated in percentage of CaCO <sub>3</sub> ..	0.91	0.93

None of the samples of leather above tested showed the slightest signs of being brittle. The leathers were air-dried, and without the addition of any oil.

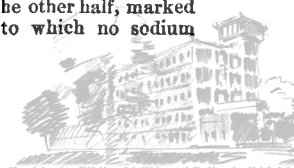
Each sample of extract which was used smelt strongly of sulphurous acid, yet the samples of leather were absolutely free from any trace of sulphurous or sulphuric acid, as the results above show.

To test the effect of light upon the samples of leather so produced, portions were exposed to strong sunlight for four weeks. The effect was very slight, the leather not being appreciably darkened.

**B. Trials with Chestnut Extract**—A calf skin was divided into halves; one half, marked 1, was tanned with a decolourised chestnut extract, to which had been added 2 per cent. of free sodium bisulphite; the other half, marked 2, was tanned with the pure extract, to which no sodium bisulphite had been added.

\* "Leather Trades' Review," January 9th, 1901 (this Journal, March 1901, 237).

† Lepetit, Dollfus and Gansser, D.R.P. No. 91,603, 21. IV., 1896; Zusatzpatent No. 103,725, 30. VI., 1897; E.P. 8582 of 1896, 23. IV., 1896, and 2603 of 1898, 21. II. (this Journal, 1897, 436) (*ibid.* 1899, 285), 1899.



The estimation of acid gave the following results :—

	Sample 1.	Sample 2.
Amount of N/10 HCl added.....	c.c. 33.0	c.c. 30.0
Amount of N/10 Na <sub>2</sub> CO <sub>3</sub> required	28.75	27.59
Difference .....	1.25	2.59
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	0.22	0.44
Calculated in percentage of CaCO <sub>3</sub>	0.20	0.41

The alkalinity of the leather ash was as follows :—

	Sample 1.	Sample 2.
Weight of ash in grms.....	0.0143	0.0197
Amount of N/10 HCl added.....	c.c. 10.0	c.c. 10.0
Amount of N/10 soda required ...	4.7	4.65
Difference .....	5.3	5.35
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	0.23	0.94
Calculated in percentage of CaCO <sub>3</sub>	0.83	0.89

That the small addition of bisulphite would have no action on the composition of the leather was to be expected, although it may be noted that these samples of leather were absolutely free from any traces of mineral acids.

The same samples on exposure to four weeks' sunlight had not altered in colour or in firmness.

C. *Trials with Oakwood Extract.*—In the same manner, as with B, a skin was divided into two parts, and both tanned with the same quantity of oakwood extract. To one half, marked 1, 2 per cent. of bisulphite was added; the other half, marked 2, was tanned in the original extracts, without any additions. The following are the results of the tests for acids :—

	Sample 1.	Sample 2.
Quantity of N/10 HCl used .....	c.c. 30.0	c.c. 30.0
Quantity of N/10 Na <sub>2</sub> CO <sub>3</sub> .....	27.5	24.6
Difference .....	2.5	5.4
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	0.44	0.95
Calculated in percentage of CaCO <sub>3</sub>	0.41	0.89

The alkalinity of the leather ash gave the following results :—

	Sample 1.	Sample 2.
Weight of the ash in grms.....	0.0260	0.0395
Amount of N/10 HCl added .....	c.c. 10.0	c.c. 10.0
Amount of N/10 Na <sub>2</sub> CO <sub>3</sub> used....	3.0	3.4
Difference .....	7.0	6.6
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	1.23	1.16
Calculated in percentage of CaCO <sub>3</sub>	1.16	1.09

The above results show that the leather was not affected in the slightest degree by the addition of the sulphite.

D. *Trials with samples of sole leather of known tannage, from different tanyards.*—Sample 1 : Sole leather, of light leather, good cut, firm but not brittle. The sample had been tanned with decolourised chestnut extract and "Mimosa D." Sample 2 : Similar appearance and characteristics to sample 1, slightly darker in colour. The leather had been tanned with undecolourised chestnut extract, quebracho extract, and "Mimosa D." Sample 3 : Heavily tanned sole leather, very brittle, dark colour, was of mixed tannage. A small proportion of "Mimosa D" had been used in the tannage.

Acid determination :—

	Sample 1.	Sample 2.	Sample 3.
Amount of N/10 HCl used .....	c.c. 30.0	c.c. 30.0	c.c. 30.0
Amount of N/10 Na <sub>2</sub> CO <sub>3</sub> .....	26.8	25.5	27.5
Difference .....	3.2	4.5	2.5
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	0.56	0.80	0.44
Calculated in percentage of CaCO <sub>3</sub>	0.53	0.74	0.41

Alkalinity of the leather ash :—

	Sample 1.	Sample 2.	Sample 3.
Weight of ash in grms.....	0.0738	0.1275	0.0272
Amount of N/10 HCl added .....	c.c. 10.0	c.c. 10.0	c.c. 10.0
Amount of N/10 Na <sub>2</sub> CO <sub>3</sub> required ..	4.5	2.75	6.0
	5.51	7.25	4.0
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	0.97	1.28	0.70
Calculated in percentage of CaCO <sub>3</sub>	0.91	1.20	0.66

As the results above show, samples 1 and 2, and also the brittle sample 3, are absolutely free from mineral acids, and the brittleness of sample 3 is, therefore, not due to the presence of bisulphites, but is evidently due to over-tannage.

E. *Trials with a soluble, decolourised quebracho extract containing bisulphites.*—Leather, marked 1, was a sample of calf leather heavily tanned by drumming. The leather was brittle and over-tanned, and parted with a lot of its tanning by washing in water. Leather, marked 2, was a sample of the same leather, which was treated for five hours in a 2 per cent. solution of bisulphite, and afterwards put for half an hour in a drum with the solution of bisulphite; it was then taken out of this, allowed to drip, and dried. The leather so produced had completely lost its brittleness, and dried out a good colour. Sample 1, previous to such treatment, was extremely brittle and harsh, and by treating it with bisulphite it lost its harshness. From this result one can scarcely continue under the impression that bisulphite produces brittleness, since, in this case, an opposite result is obtained.

The determination of acids gave the following results :—

	Sample 1.	Sample 2.
Quantity of N/10 HCl used.....	c.c. 30.0	c.c. 30.0
Quantity of N/10 Na <sub>2</sub> CO <sub>3</sub> required	25.8	25.9
	4.2	4.1
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	0.74	0.72
Calculated in percentage of CaCO <sub>3</sub>	0.69	0.68

Alkalinity of the leather ash :—

	Sample 1.	Sample 2.
Weight of ash in grms.....	0.0511	0.0687
Amount of N/10 HCl added .....	c.c. 10.0	c.c. 10.0
Amount of N/10 Na <sub>2</sub> CO <sub>3</sub> required	5.82	7.05
	4.18	2.95
Calculated in percentage of Na <sub>2</sub> CO <sub>3</sub>	1.05	1.24
Calculated in percentage of CaCO <sub>3</sub>	0.99	1.17

In the above examples the analyses demonstrate that the fear of using extracts containing bisulphite is without foundation.

Firstly, as the above results show, samples of leather, tanned with these extracts, were in each case free from any trace of mineral acids; secondly, there was no indication that even an excess of bisulphite had any injurious effect upon the leather. The weight of the ash from such leathers and the alkalinity, were practically the same as from other leathers. The bisulphite was not taken up by the leathers, but appeared to assist in the penetration of the extract into the leather, and, as the extract diffused in, the bisulphite dissolved out.



In order to make perfectly sure, with regard to the absorption of bisulphites by leather, the following experiments were undertaken:—

Two thick bellies, after the usual preliminary treatment of liming, &c., to remove the hair, were put in a paddle and delimed in a mixture of acetic and lactic acid. No. 1 piece was tanned in an extract containing bisulphites, "Mimosa D" being again selected. It was tanned slowly in a paddle, more extract being gradually added until the belly was thoroughly tanned. The other belly, No. 2, was treated in another paddle in exactly the same way, but each time extract was added a quantity of bisulphite of soda was also added, in the proportion of 1 part of bisulphite to every 10 parts by weight of extract; in other words, for every 100 grms. of extract added to strengthen the liquor, 10 grms. of bisulphite were added. The liquors were thus strengthened until they had a relative density of 1.045. The tannage lasted 10 days. At the end of this period, the leather was taken out, allowed to dry, and half of each piece was dried right out, and the other half was oiled on the surface and dried out slowly. Therefore, from the experiment, four separate samples were obtained, two oiled and the other two unoiled. Neither the former nor the latter, when dry, showed the slightest tendency to crack. Fifty grms. of each of the unoiled samples were reduced to ash in a platinum dish, with the following results:—No. 1 gave 0.3450 gm.; No. 2 gave 1.7160 gm.

These residues were now treated with a slight excess of sulphuric acid, and strongly heated with the blow-pipe. Ammonium carbonate was then added, and the ash again heated until the weighings were constant.

	Sample 1.	Sample 2.
Weight of residue.....	Grms. 0.4222	Grms. 1.7900
Calculated as sodium sulphate....	Per Cent. 0.84	Per Cent. 3.38
Calculated as sodium bisulphite..	0.61	2.62

From the remaining leather a further 50 grms. were taken from 1 and 2. The leather was cut into thin slices, and shaken gently in a shake machine for five or six hours with a litre of distilled water, filtered, and filtrate from each evaporated to dryness. The remaining leather was then dried and burnt to ash, as in the above.

	Sample 1.	Sample 2.
Weight of ash from washed leather	Grms. 0.1492	Grms. 0.2460
Weight as ash from filtrate .....	Lost	1.4813

In the same manner as above, these four residues were treated with sulphuric acid, strongly heated, and weighed.

	Sample 1.	Sample 2.
Washed leather (a).....	Grms. 0.2912	Grms. 0.2863
Liquor (b).....	0.1561	1.5540
Calculated as percentage of sodium sulphate (a).	Per Cent. 0.38	Per Cent. 0.55
Liquor (b).....	0.26	3.18
Calculated as percentage of sodium bisulphite (a).	0.42	0.40
Liquor (b).....	0.19	2.31

The other inorganic salts, which in the above were weighed as sodium sulphate, were found to be so small in quantity that they might be neglected.

It will be noticed, from the foregoing figures in experiment 1, that 0.42 per cent. of sodium bisulphite remains in the leather after a thorough soaking, and that 0.19 per cent. is found in the liquor, which gives a total of 0.61 per cent. of sodium bisulphite. This amount corresponds to the quantity of bisulphite which was found in the first experiment when the leather was originally treated.

Further, it is peculiar, with the figures got by experiment No. 2, that about the same amount of bisulphite is retained in the leather as in experiment No. 1, i.e., 0.40 per cent., which, together with the 2.31 per cent. which was found in the liquor, makes 2.71 per cent., thus corresponding within a tenth of a per cent. of the amount obtained by analysing the leather which was tanned with "Mimosa D" plus bisulphite.

We have endeavoured to show by the above simple experiments and analyses that sodium bisulphite does not appear to be absorbed by the leather; in fact, it is easily removed, and when present it remains chiefly in the tan liquors. The small percentage of sodium bisulphite which is retained by the leather is in each of the above experiments almost exactly the same, in spite of the use of varying quantities.

Further, no sulphuric acid is formed either in the leather or in the liquors, when such extracts are used; nor was there any indication of brittleness or cracking of the grain in any sample of leather which was tanned with tanning extracts treated with bisulphites.

Besides the above small experiments detailed, a much larger series of practical tests was carried out. Calf and sheep skins, and also hides, were tanned practically with varying quantities of tanning extract, to which, in one case, as much as 100 per cent. of bisulphite of soda was added (i.e., equal quantities of sodium bisulphite and tanning extract). A good colour was obtained in each case, and the leather, although it was dried fairly rapidly and without being oiled, showed no signs of cracking. Samples were taken from each, and, on analysis, showed no trace of free acid, either sulphurous or sulphuric.

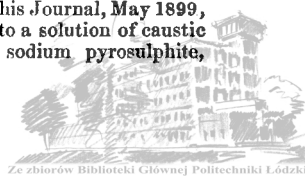
#### DISCUSSION.

Mr. S. T. PENTECOST thought that any acid remaining in the leather must be held by a lime soap, or a body of that nature. In bleaching cotton goods all traces of mineral oil might appear to have been removed, and yet after a time yellowish stains appeared. Might not the acid in the leather be imprisoned in a similar way? The intensity of the action of the bisulphite would depend upon the concentration of the tanning bath. Certainly, in the case of silk, treatment with bisulphite affected the fibre.

Mr. G. B. CLEMONS explained that the use of bisulphite was solely to render soluble in cold water certain tannins. He wished to know whether the tintometer could be taken as a reliable means of judging the colour of a tanning extract, and whether the tintometer results were always borne out in the tanpits.

Mr. J. T. WOOD regretted the absence of the authors of a paper of such importance to tanners. His firm had used extracts made according to the patents mentioned, and had experienced no difficulty. The patent stated that 300 kilos. of extract were heated with 100 kilos. of sodium bisulphite, in a closed vessel, to 115° C. for some hours. The product obtained from quebracho extract was perfectly soluble in cold water. (This Journal, 1897, p. 436.) As to the decay of bookbinding leathers, mentioned by the Chairman, a committee of the Society of Arts had recently published a report in which suggestions were made as to the prevention of this evil. The fumes of burnt gas were certainly fatal. The action of light was also of great importance. The amount of bisulphite in the strongest liquors would not be so strong as in the case Mr. Pentecost mentioned; it would not amount to more than 2 grms. per litre, and this could scarcely affect the leather. In reply to Mr. Clemons, while the tintometer was most useful, he did not find it any guide as to the value of an extract. He knew of a case in which an extract with a worse tintometer reading than sample actually produced a better-coloured leather.

Mr. R. M. CAYEN asked what was the precise nature of the substance known technically as "bisulphite of soda." The question was of theoretical, if not of practical, interest, because of the doubt which has been thrown on the existence of NaHSO<sub>3</sub> (see discussion by Divers, this Journal, May 1899, p. 451). When SO<sub>2</sub> gas was passed into a solution of caustic soda until the liquid was neutral; sodium pyrosulphite,



$\text{Na}_2\text{S}_2\text{O}_5$ , was contained in the solution. It would be of interest, therefore, to know whether the solution employed in leather dressing was acid in reaction, and, if so, what advantage it possessed over a solution of sulphurous acid.

Dr. J. GORDON PARKER, in reply, wrote that the tintometer was a perfectly reliable means of judging the colour of a tanning extract, provided one only compared tanning extracts of the same make one against the other. One could compare different deliveries of oakwood, one against the other, and different deliveries of chestnut or quebracho, but not an oakwood against a quebracho, as there was a different colouring matter present in each.

Mr. Wood's remark about the extracts at the end of his reply was correct, if two different extracts made of different materials were being compared, but would not be correct were the two extracts made from identical raw materials.

In reply to Mr. Caven, the bisulphite of soda which was used was supposed to be  $\text{NaHSO}_3$ . It possessed a great advantage over  $\text{SO}_2$  gas, as the sodium bisulphite appeared to form soluble compounds with the difficultly soluble tanning materials, whereas  $\text{SO}_2$  gas appeared merely to bleach them slightly, without dissolving them.

## Scottish Section.

*Meeting held in Glasgow, on Tuesday,  
October 29th, 1901.*

DR WM. FREW IN THE CHAIR.

### IMPROVEMENTS IN SUGAR-REFINING DURING THE LAST TWENTY-FIVE YEARS.

BY T. L. PATTERSON, F.I.C., F.C.S.

SUGAR-REFINING is a physical and mechanical, rather than a chemical process. Briefly stated, it consists in the dissolution of raw sugar in water; filtration of the liquor through cloth to remove insoluble impurities, and through animal charcoal to remove colour; and finally, concentration of the decolorised liquor to obtain crystals. But it depends largely on chemical analysis for the determination of the composition and value of the raw material and for checking the various processes in the refinery. Hence the staff of no well organised refinery is complete without one or more chemists, although very few chemicals are used in the process itself.

On the Continent, chemical processes are wholly in use for the removal of colouring and nitrogenous matters from beetroot juice. These consist for the most part in the treatment of the juice with alkaline earthy bases, zinc, iron, and tin salts; carbonic, sulphurous, and hydrosulphurous acids. Electricity, too, is on its trial for the same purpose and for the partial removal of mineral salts. Some of these chemical processes have been adopted for the treatment of cane-juice in the Colonies, but none have been introduced into this country, except perhaps in a very modified way, because they are not applicable to British methods of refining. Sucrose undergoes inversion and decomposition so easily, and is so closely related in composition to some of the impurities to be removed, that treatment with strong chemicals is out of the question. For this reason animal charcoal still maintains its position as a decolorant in the production of the highest class products, as well on the Continent as in this country.

No revolutionary change has taken place in the refining industry during the last 25 years, but many improvements on the old physical and mechanical processes have been made and introduced. The first of these to which I am about to direct your attention consists in improved methods of washing raw sugar in the centrifugal, just introduced by Weirich prior to this period, with steam, or with hot or cold water. The process was afterwards improved by Ducean and Newlands, Walker and Patterson, and others. Theoretically, raw sugar may be said to consist of pure sugar crystals and molasses. The object of the process is

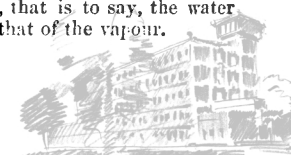
therefore to wash out the molasses, and raise the standard of the raw material—chiefly beetroot sugar of low quality—to that of the higher grades, and make them suitable for the production of the highest quality of refined sugar. For this purpose raw beetroot sugar of 75 per cent. to 80 per cent. net analysis is filled dry into the centrifugal, or mixed with syrup or water, and transferred to a centrifugal machine. After spinning out the syrup added or made, the sugar in the machine is washed with water or steam until most of the molasses is removed. The product which still contains all the insoluble impurity and some of the colouring matter has been raised in quality, though reduced in weight, to an analysis of 90 per cent. to 95 per cent. net. The process separates the raw sugar into two products, viz., (a) the partially purified or washed crystals, which are submitted to the ordinary process of refining for the production of white crystals, cubes, or granulated; and (b) the washings, containing the molasses which go to make yellows and syrups.

In the filtration of sugar liquors, filter-presses have been introduced with marked advantage, but in most refineries the old Taylor filter, with in some cases trifling modifications, still holds its own. There is room, however, for a greater use being made of the filter-press than has yet been done. Sometimes when filtration is difficult, a precipitate of phosphate of lime is brought about in the solution which coagulates gummy and gelatinous matters; or sand, kieselguhr, or other inert matter is added, to prevent them from choking the pores of the filter cloth, and thus facilitate filtration.

Animal charcoal is still the chief or only decolorant used in the refining industry. It is supplemented in many refineries by the addition of sulphurous acid in the gaseous state or in solution to the filtered liquid. The acid bleaches the yellowish liquors from the char to a considerable extent, and any excess is expelled in the after process of concentration. With the exception of phosphoric acid solutions, carbonate of lime, lime, and sulphuric acid for syrup inversion, sulphurous acid is the only chemical now in use.

The process of concentrating and graining the sugar solutions—technically called boiling—is always conducted in vacuum pans which boil at a low temperature. No improvement worthy of remark has taken place in the apparatus itself. But improved evaporators have been introduced by Yaryan (1886) and Lillie (1888) in America for the concentration of cane-juice, char, and other sweet waters, which have found their way into the refining industry of this country. They are built either vertical or horizontal, on the multiple effect system so largely used on the Continent and in the Colonies. They consist of one or more vessels divided into three compartments. The central or heating chamber is filled with pipes which connect the feeding chamber at one end with the collecting chamber at the other. This liquor is supplied in a regulated flow to the feeding chamber, where it is distributed in films over the inner surface of the pipes heated by low pressure steam, and thence into the collecting chamber more or less concentrated. The vapour evaporated from the liquid is conveyed to the heating and the partially concentrated liquor to the feeding chamber of the next vessel, and so on for as many vessels as are in series, usually three. A good vacuum is maintained in the last vessel and the manipulation is the same as that in use with the old triple effect. The advantage of these evaporators is attributed to the great economy of heat, the small quantity of liquid submitted to heat at one time, and to the rapidity of evaporation. The liquid is only in the apparatus a few minutes before it is discharged concentrated to the desired density. During this time cane juice at 14° Brix can be raised to 51° Brix and water, equal to 72 per cent. of the weight of the juice evaporated.

Whilst no great advancement has taken place in the construction of the vacuum pans in which sugars are grained, improvements have been made on the condensers, so that now two or more pans are worked in connection with one condenser. And in order to economise water, the condensers are made counter-current, that is to say, the water flows in the opposite direction to that of the vapour.



Some progress has been made in the method of boiling crystals on the lines of a "seeding" process introduced by Lebaudy of Paris some time prior to the commencement of the period under review (1865). This process has grown in favour and been largely adopted in the refining industry. To make the advantage of this improvement plain, it will be necessary in as few words as possible to describe the process of boiling. Large and well crystallised sugars, other things being equal, bring a better price on the market than small badly crystallised goods. Hence in boiling white sugars, except where granulated and cubes are required, the object of the refiner is to produce as large crystals as possible. With this end in view, it used to be necessary to start the pan with nearly pure sugar solutions, and when full of masse-cuite—that is a magma of sugar crystals and syrup or mother liquor—the pan was "cut" as it is technically called, that is half emptied, and the boiling process started again by feeding in more liquor. The crystals left in the pan grew larger with every increment of sugar solution, the motion of the boiling mass, and rapid concentration, until the pan was again full, when it was again cut. This process of cutting had to be repeated several times until the crystals attained the desired size, when the pan was completely emptied. Practically it is not possible to go on cutting indefinitely, because the boiling requires to be slowed down as the crystals increase in size, and the process would have to be greatly prolonged, making it very difficult to prevent the formation of new small crystals which must be sifted out, and injuring the colour of the syrup from which the after products are boiled. In this way several lots of sugar are obtained, the crystals of which increase in size from the first cut to the last. As all are of the same quality, though differing in size of grain, the refiner's aim is to turn out each lot with large well formed crystals, which command the highest price.

To obtain this result, reduce the cutting and shorten the time of boiling, refiners now proceed as follows, *viz.*, they concentrate some liquor in the pan to nearly the crystallising point and add to it a proportion of the dried sugar from the first or earlier cuts of a former boiling. The operation is technically called "seeding" the pan. The added crystals or "seed" grow as boiling proceeds, and attain full size much sooner than they can do under normal conditions. A refinement of this operation consists in using foreign sugar crystals as seed and growing the sugar in the liquor on them. When the different pans from this method of work are mixed, all the crystallised products can be turned out of large and uniform quality.

With the same object in view, when two pans stand together, it is a common practice to connect them with a pipe closed with valves. One pan is boiled full of masse-cuite, which may be started from liquor alone or from liquor and seed, the connection between them is opened and the second pan partially filled from the first. Boiling then proceeds until both are full and ready to cut, or be emptied, as the size of the grain warrants. No improvement has taken place in the boiling of mediums and yellows.

The masse-cuites are dropped from the pans into mixers, whence they are delivered to the centrifugal machines in which the syrup is separated from the sugar. The sugar is more or less washed, discharged into bins, air-dried, and filled into bags for market. Hitherto the syrup and washings have been collected together, and a second quality of sugar crystals boiled from the mixture. In the process of washing, rendered necessary to remove the mother syrup which adheres to the crystals, a considerable proportion of the sugar is dissolved and lost to the first product. This has always been a difficulty which refiners try to obviate by using as little water as possible, and carefully regulating that used to the class of sugar washed. But notwithstanding every care, 6 per cent. to 15 per cent. of the first products are washed into the second products, for which a lower price is obtained. A recent patent of my own (1897) has made it possible to separate in the centrifugal itself the washings from the syrup or mother liquor, so that washing may now be practised with impunity. The washings, having the same purity as the liquor from which the sugar was boiled, are returned to the pan separately from the syrup and boiled up with liquor to produce first product

sugar. In this way the loss from washing has been avoided. Several large refiners are presently erecting machines to work this process and good results are anticipated.

In describing the process of boiling crystals we excluded "granulated" and "cubes." Both are practically products of the last 25 years. No seed is used for either of these sugars as only small crystals are required. The masse-cuite for granulated sugar is treated in the centrifugal to separate the syrup, after washing the moist sugar is completely dried in a Hersey, Newhall, or Gibb revolving cylinder through which a current of hot air passes. It is then cooled and packed for sale.

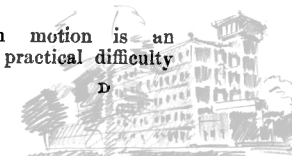
There are many patent processes for making cube sugar. Those which have been worked in this country are the Langen, Duncan and Newlands, Walker and Patterson, and the Adant. All are manipulated in much the same way. Masse-cuite boiled to a small grain is filled into moulds divided by partitions, so that the spaces between may form, when full, plates or prisms of sugar about five-eighths of an inch thick. The moulds are either a kind of centrifugal basket, or made to fit neatly into centrifugal baskets. The masse-cuite is allowed to set and cool in the mould, when the crystals become cemented together by a secondary crystallisation which takes place during the cooling process. The cooled moulds are then placed on a centrifugal spindle or in a centrifugal basket, and the syrup spun out. Whilst spinning the sugar plates are washed with clairce—a saturated solution of pure sugar in water—and when this too is spun out the moulds are removed and taken asunder. The plates are carefully separated and placed in a continuous mechanical or other stove, where they are dried in a current of hot air. The dry plates or prisms, now so hard that they have a metallic ring, are then transferred to a special cutting machine which chops them into cubes for market.

Besides the moulded masse-cuite processes just described, there are several others in which a kind of artificial masse-cuite is produced by mixing crystals, or crushed sugar, with a hot saturated solution of sugar. This mixture is then moulded into plates or prisms. Or again the sugar is moistened with water and pressed into moulds to form cubes or tablets, which are dried in the usual manner. None of these processes yield such a first-rate product as cubes made directly from masse-cuite, and need not be further referred to. The cube is such a convenient form of dry hard sugar, and its manufacture is now so perfect, that it has entirely supplanted the old form of loaf sugar in this country, the cutting of which was a most disagreeable household experience. Loaves are still made on the Continent, but their manufacture is rapidly dying out.

Passing to the residual products of the refinery we will consider an improved method of treating molasses, which in the beetroot sugar industry, because of the presence of a large percentage of lime and alkaline salts of organic acids, is the refuse of the process only suitable for cattle feeding and distilling. It is called "Crystallisation in Motion," many modifications of which are at work. The process was introduced on the Continent about 20 years ago, for granulating out the last available sugar that will crystallise. Patents were granted to Bock, Huck, and others, the latest improvement being perhaps that of Grossé. The apparatus consists of large cylinders or tanks, provided with a stirring arrangement kept in very slow motion, and in most cases surrounded by a jacket in which hot water circulates to control the cooling. It is often fitted with an air-tight door and connected to the vacuum pump and air pump by pipes, that it may work under reduced pressure and be emptied by increased pressure when finished.

The low syrups or molasses are put into this apparatus after they have been boiled to the granulating point, when they are in a supersaturated condition. Stirring then commences and is repeated at intervals during 50 or 60 hours, whilst the contents cool down very slowly to about 50° C. controlled by the hot water jacket. Under this treatment the small crystals of sugar at first formed grow in the viscous mother liquid or molasses, and become large enough to be easily separated from the exhausted molasses in the centrifugal machine.

The process of crystallisation in motion is an interesting one because it overcomes a practical difficulty





in a very simple way. It may be explained by considering what takes place when partially exhausted, concentrated molasses is slowly cooled at rest. Such molasses is a thick viscous supersaturated liquid, in which the free sugar molecules are held apart by the large proportion of organic salts and other organic bodies present in solution, and in which that freedom of molecular movement favourable to the formation of large crystals is entirely absent. Practically crystallisation proceeds continuously as the solution cools, but theoretically, it is intermittent, and we can assume that several free molecules arrange themselves around another molecule in innumerable centres in the mass, to form small crystals. After withdrawing the free molecules in their immediate neighbourhood, crystallisation stops at these centres, and the crystals formed drop to the bottom of the cooler, or remain suspended out of the sphere of action. As cooling proceeds the solution again becomes supersaturated at the lower temperature, and new crystals form in new centres, to drop out of action as before. In this way supersaturation and crystallisation proceed together in different centres, and succeed each other, until the mass is cool, with the result that most of the sugar is crystallised in a very fine state of division, the greater part of which passes through the perforated linings of the centrifugals along with the molasses, and cannot be recovered. The motion of the mass under the influence of very slow cooling overcomes the difficulty of the formation of small crystals and prevents the mother syrup from reaching a supersaturated condition. The crystals first formed are transferred to new spheres of action, and the free molecules arrange themselves on the crystals already formed in preference to forming new ones. Thus the crystals grow as the mass cools and the final product is a crop of crystals large enough to be easily separated from the exhausted molasses in the centrifugal machine. When the molasses contain very little sugar, as they often do, to yield a fair crop in this way, the masse is seeded with a proportion of crystals from a previous boiling to form crystallising centres, and all the sugar that will crystallise is recovered. On the other hand, when the masse is too thick and viscous, a little thinner molasses is added to give that freedom of motion required. Usually the molasses from this process go to the distillery, or for cattle feeding. But as nearly all the syrups in this country are refined and go into consumption as golden syrup, the process has only been adopted by one or two refiners.

The manufacture of golden syrup has greatly increased during the last quarter of a century. The quality has been very much improved by the use of high class syrups and sugars as the raw material from which it is made. On the other hand it has been much impaired by the use in large quantity of liquid Indian corn glucose for diluting poor syrups, improving the colour and preventing granulation. Good well inverted syrups do not require this addition, nor is it practised by the best makers.

The syrups and sugars used for making golden syrup require to be partially inverted to prevent the granulation of cane sugar in the finished product. For this purpose they are heated with a little sulphuric acid, which is afterwards neutralised with carbonate of lime and the sulphate of lime filtered out. The liquor is then passed through charcoal and boiled to the required density. Some refiners use yeast instead of sulphuric acid, according to Tompson's 1884 patent more or less modified. The action is slower than with acid, but it enables them to work without chemicals. Yeast added to strong solutions of sugar and heated to 50°–70° C. does not set up alcoholic fermentation. An objection to yeast is the introduction of organic impurities into the syrup which are not removed by charcoal.

White sugar is made on the Continent direct from beet-root juice by the chemical agents already referred to, without the use of charcoal. Many of the refineries, too, work without charcoal, but the products are never so pure and white as they are when this decolorant is employed. The best results and finest products can only be obtained with charcoal. Consequently in this country, where the public demand the best of everything, all refiners use char. As large quantities are required, at least one ton

for a ton of sugar, it may be taken for granted that the char department is the largest and most expensive in a refinery. Any useful improvement introduced into this department will therefore be of value. And progress has to be recorded here as elsewhere in a refinery. It is chiefly mechanical, and consists of improved methods of handling the char. Formerly the char, wet from the filters, was delivered by hand to kilns with fixed burning pipes, drawn by hand from the coolers below into barrows and wheeled to elevators, often after having been spread on a cooling floor by hand, because the kiln coolers were too short to do their work.

Now, the wet char is delivered mechanically on to kilns in which the reburning pipes do or do not revolve. In its passage down the pipes the char is raised to a low red heat, which destroys the organic matter absorbed from the sugar. Below the kilns it enters ample coolers which are a continuation of the burning pipes, whence it is automatically discharged into receivers ready for use. Then, as it is required, endless bands carry it from the receiver to the elevator. Thus the char is almost altogether mechanically handled, little or no labour is needed from the time it leaves the filter till it returns to it, except that required to control the machinery, and distribute the char on the kiln-heads and filters.

Char kilns have been greatly improved by placing driers on the top heated by waste gases from the kiln fires, an adaptation introduced from American practice. Wet char passing through these is delivered into the pipes dry. Thus, the driers do outside what was formerly done inside the pipe, and make it possible for each kiln to reburn the maximum amount of char.

Char is the great sugar refiner and much care is bestowed on its manipulation. With repeated revivification, even in the best kilns, it gradually loses its decolorising power, and after a year or two's use has to be turned out. The cost of renewing it is a serious item in the upkeep of a refinery. Two causes are chiefly responsible for the deterioration. One of these is the reduction of porosity brought about by the shrinkage of the calcium phosphate during successive reburnings. The other is due to the deposition of vegetable carbon, which has no decolorising power on its surface and in its pores, by the carbonisation of organic matter absorbed from the sugar. No remedy has yet been found for the former evil; but it has long been recognised that, were it possible to remove the vegetable carbon the latter would be taken away, the decolorising power would be greatly improved and the life of the char prolonged. This is the problem refiners are now trying to solve, and already two or three apparatus are on trial in which char is burned in a limited supply of air. Chief amongst these is a cylinder with revolving paddles patented by Weirich in 1896. It is in use in two or three refineries and good results are said to be got from the process. The patentee claims that impurities are oxidised at a temperature of 300° to 400° F. when this apparatus is used instead of ordinary kilns, but that a temperature of 600° F. is required when it is used as an auxiliary to these kilns. About the same time I was independently experimenting on the oxidation of vegetable carbon in char, and found that it was not removed in any appreciable quantity below a low red heat. At this temperature, however, the char is greatly improved by partial oxidation, and I am very hopeful that this process will prove of great value to the refiner.

Passing from improvements in the process itself, let me close this paper by a short reference to the commercial position of the industry during the last 25 years. Whilst there have been fluctuations, amounting to prosperity at times, though oftener to failure and loss, the industry, as a whole, may be said to have been on the down grade throughout this period. The decline was not caused by any want of enterprise on the part of refiners. What I have said to-night is evidence that they are not slow to adopt new processes, or improvements on old ones, which promise any chance of success. It has been almost wholly brought about by the action of foreign States who give bounties to rival refiners, which enable the latter to undersell home-made sugar in British markets. A few statistics will make this plain.





The consumption of sugar in this country, which was 860,000 tons in 1875, equal to 60 lb. per head of population, grew to 1,489,000 tons in 1900, equal to 81.2 lb. per head of population. During the same period, the sugar refined in this country fell from 760,000 to 610,000 tons. And the refined sugar imported—chiefly from Germany, Austria, and France—steadily increased from 100,000 tons in 1875 to 950,000 tons in 1900. In other words, the consumption increased 81.5 per cent., whilst home refined sugar decreased 20 per cent., and foreign refined sugar increased 950 per cent. In 1875, 88.5 per cent. of the sugar consumed in this country was refined in Britain and 11.5 per cent. abroad. Whilst in 1900 only 39 per cent. was refined in Britain and 61 per cent. abroad. These figures speak for themselves, and show the enormous injury done to this country by the imposition of foreign bounties. But for them the growth of sugar-refining would almost certainly have kept pace with the growth of consumption, and many of the refineries closed during the last quarter of a century would still be working profitably.

It is worth noting in this connection the diminution of refineries during the period under review. In 1875, 18 firms were refining sugar in London, and two new refineries were started a few years later, making in all 20 for London. There were 9 in Liverpool, 3 in Bristol, 2 in Manchester, 1 each in Earlestown, Plymouth, and Newcastle-under-Lyne, 13 in Greenock, 1 in Leith, and 1 in Dublin; total, 52. In 1900 only two were working in London, 3 in Liverpool, 1 in Earlestown, and 5 in Greenock; total, 11. Deducting the latter total from the first we get 41 closed. To this has to be added another refinery, making 42 in all. For, some years ago, a firm of Liverpool refiners courageously tried to stop the import of foreign refined sugar on the east coast of England by establishing a refinery at Rawcliff, near Goole, which they worked on a continental model. But after a run of two or three years at great loss, had to abandon the project.

Thus we have seen that 42 refineries have been extinguished in Great Britain and Ireland during this comparatively short period. Speculation and other causes were in a measure accountable for the closing of some of these. But the baneful influence of foreign bounties was noticeable here too. For it caused great fluctuations in the production of beet-root sugar on the Continent, with even greater fluctuations in the price of the raw material. And refiners here, who have to buy ahead to supply their wants, became the victims of these unnatural movements. Although most of them made heroic efforts to survive, we need not wonder that some refiners succumbed in the struggle for existence against such odds. The wonder rather is that there are any left. The remnant struggle on, buoyed up with the hope that in the near future our Government will yet be brought to reason, and take some action which will prohibit the importation of bounty-fed sugar into this country, or by some other means equally effective, put a stop to the unfair competition of foreign refiners, and save from total extinction an old, honourable and legitimate industry, which gives employment, even yet, to large capital and a considerable number of workmen.

When in spring last it became known that a duty was likely to be put on sugar, refiners were hopeful that the Chancellor of the Exchequer would make it differential, so as to recover the greatest duty from the sugars which received the largest bounties, but when the duty was declared it was found to bear with equal incidence on sugars of all origin, leaving the position, so far as the refining industry is concerned, practically where it was.

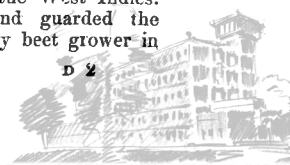
#### DISCUSSION.

Prof. HENDERSON congratulated the Section on having received so valuable an account of the present state of the sugar industry by an authority on the subject, like Mr. Patterson, to whom he was also personally indebted for information which had enabled him to compile a brief report on the same subject for another purpose. He suggested that Mr. Patterson might give the meeting some account of the results of the experiments on the cultivation of sugar beet in Scotland.

Dr. W. C. ANDERSON remarked on the low temperature (300–400° F.) at which Weinrich claimed to be able to oxidise the organic matter contained in the char, and asked whether the author's experiments tended to confirm this statement.

Mr. A. MACDONALD said that the paper seemed to cover every modification and improvement during the last 25 years. He was not sure if the lime process had been introduced within that period, possibly not; hence the omission of any reference to it. It was painful to mark the enormous curtailment which had taken place in the number of refineries of Great Britain, and though it was true that those remaining had increased in productive power, still the enormous increase in the imports of foreign-refined, coupled with the decline in the production of home-refined, was a clear indication of the disadvantages under which the home-refiners laboured, by reason of the bounties granted to foreign refiners. The margin between raw and refined beet sugar was now so small that a very trifling advantage one way or another was bound to determine the flow of business. For example, the price of raw beet sugar on the day of the meeting was 7s. 3d. per cwt., f.o.b., continental ports, on a basis of 88 per cent. The exact purity of 100 per cent. on this basis, apart from the arbitrary irritating increase adopted by the trade, was 8s. 2½d. per cwt. Now, as the price of foreign refined granulated sugar was then 8s. 3d. per cwt., f.o.b., it was clear that all the skill—all the scientific improvements in the world—could not effect economy enough to enable British refiners to compete against foreign refined sugar of the same character. It was only by variety, by greater skill, in enabling them to produce a better quality and an article of finer appearance, that the British refiners still existing had been able to hold their ground at all, in spite of the advantages of the differential rates between raw and continental refined sugar which enabled the refiner abroad to sell his refined product at practically the same price as raw.

Prof. Henderson had asked some questions about the cultivation of beetroot and the extraction of sugar therefrom in this country. The cultivation of beet and the extraction of sugar on a practical scale in this country had only taken place at Lavenham, under the initiation of Mr. James Duncan, and at a time when the higher price of sugar, as compared with the present price, should have made the enterprise a success. Mr. Duncan did not himself grow the roots, but relied upon the co-operation and hearty support of the farmer. He merely laid down a factory with all the necessary means of gathering in the roots, but while he had equipped his factory for working a large quantity of roots, he never could get more than about a quarter of the quantity which his factory was capable of overtaking. For this reason alone the venture proved unprofitable. Nevertheless he wrote in the most confident manner about the value of beet as a crop and as an industry, and said, "no one knows better than I do the value of beetroot to a country, for, not only does its cultivation mean fertile lands and rich after crops, but it means abundance of cattle, and a contented and happy peasantry, because of the profitable occupation it affords to many people." Besides this practical experiment—for after all it was only an experiment—many trials had been made in the cultivation of beet, and within 12 miles of Glasgow, viz., at Bishopton, beetroot had been grown indicating a yield of as much as three tons of sugar per acre. What this meant could only be realised by comparison, but when it was seen that the German crop averaged about 31 cwt. per acre, and that of France only about one ton, the possibilities of this country became plain. Indeed it might be laid down as a law that the moist climate of this country would enable a larger yield to be produced from our root crops than that of any other country. The question of ripening had yet to be answered by trial. This, our grievance against bounties, was as much because of the hindrance they had been to agricultural development and expansion, as it was because of the injury they had inflicted upon British refiners and growers of cane in the West Indies. For, had the Government fostered and guarded the cultivation of beet, at a time when every beet grower in



Germany was driving in his carriage to church while other farmers walked, the agricultural situation of this country would have been saved, the depopulation of the agrarian districts stemmed, and the enormous and alarming increase of the larger cities checked. Mr. Patterson's paper was a valuable resumé of the sugar trade, a kind of stocktaking for the last quarter of a century, and in a society connected with industry such as this was, it seemed desirable to take a look backwards in order to ascertain how far they had travelled and to take note of anything which had hindered their progress.

Mr. D. J. PLAYFAIR said that Mr. Patterson had made no reference to the strontia process for sugar refining, and asked if it were still in successful operation, or if it had been abandoned. With reference to the electrolytic process for refining sugar, Blount explained it to consist in using attackable anodes of either zinc or aluminium without a diaphragm, the resulting effect of which was to produce a hydrate of zinc or alumina, which on settling withdrew some of the impurities from the sugar solution. Some bleaching effect might also take place due to electrolysis of the soluble chlorides present, and perhaps Mr. Patterson could say if this happened, and if so, whether it had the effect of producing any inversion of the sugar.

Mr. T. L. PATTERSON said in reply to Prof. Henderson that the experiments on the growth of the sugar beet were conducted in Scotland for four or five years and were on the whole satisfactory. The roots grew well and yielded good crops, with a fair average percentage of sugar, especially in the small and medium sized roots. The yield of sugar per acre was high in some districts, higher even than Mr. McDonald had mentioned; whilst in others it was much below the average. The low yields were due more, perhaps, to inattention, or to ignorance of the culture of the beet on the part of the grower, than to any climatic or other cause; hence such yields might be disregarded. But field experiments such as these were often misleading and generally showed better results than could be obtained on the large scale. This was the case in the United States, and it would doubtless be the case here; therefore too much reliance should not be placed on the results. One fact brought out very clearly in Scotland was the large proportion which leaves bore to roots. This was evidently due to the cold humid sunless weather generally experienced in October and November, just when the roots should ripen and mature. Instead, they kept on growing so that even in December the roots were not ripe. The experiments on the whole went to show that it might be possible, notwithstanding somewhat unfavourable climatic conditions, to grow beet on the lowlands of Scotland for the manufacture of sugar. But until the bounties given to foreign producers by their governments, referred to by several speakers, were withdrawn, there was no probability of this being done at a profit. In reply to Dr. Anderson, his own experiments showed that it required a low red heat to oxidise the carbon deposited in the char by the carbonisation of the organic matter absorbed from the sugar, and he did not think a temperature of 400° F. would be sufficient to oxidise the organic matter alone. He had not referred to the lime process, as it was introduced prior to the period under review. The process has been long in use in one refinery. In reply to Mr. Playfair, the strontia process was not used in sugar refining. It was introduced by Scheibler in Germany for recovering sugar from molasses in the beet industry, but was now almost wholly superseded by Steffen's lime process. In this country there were no molasses to treat, and as the syrups went into consumption the sugar was not extracted from them. He had no experience of the electrolytic process of treating sugar solutions, nor was it used in this country. Electricity was on its trial on the Continent for treating beet-root juice, and good results were said to have been obtained. Experiments just published, made on the large scale in a French factory, went to show that the purity of the juice was greatly improved by treating it with lime to alkalinity, and with powdered manganate of lime in small quantity, during the passage of an electric current of 7 to 10 volts. It was claimed that the sucrose in solution was not injured by the treatment, and that only the organic impurities were oxidised. But further experi-

ments were required to confirm these statements, for it was well known that sucrose was as easily oxidised as many of the organic impurities which accompanied it in the beet.

## Worshire Section.

Meeting held on November 25th, 1901.

MR. G. WARD IN THE CHAIR.

### THE SOLUTION THEORY OF DYEING.

BY R. B. BROWN AND J. MCCRAE.

ACCORDING to the theorem of Nernst (Theoretical Chemistry, p. 412) when the value of the ratio of distribution of a dissolved substance between two solvents is constant and independent of the concentration, the molecular weight of the dissolved material must be the same in the two media. If then a dyestuff distributes itself in a constant ratio between liquid solvent and fibre, it follows that, provided we may assume that the fibre acts merely as a solvent for the dye, the molecular weight of this in the two media must be the same.

Witt (Färber-Zeit. 1890-91, 1) has attempted to explain the process of dyeing by the assumption of the formation of solid solution. Using this as a working basis, Walker and Appleyard (J. Chem. Soc. 1896, 69, 1334) investigated the distribution ratio of picric acid between water and silk, and found that the distribution coefficient is not independent of the concentration, but that  $s/\sqrt[2]{w} = \text{const.}$ , where  $s$  is the concentration of picric acid in the silk and  $w$  is the concentration in the water. This would indicate that in the aqueous solution the molecular weight of the picric acid is 2.7 times that in the silk. It has, however, been proved by cryoscopic and ebullioscopic determinations that, apart from ionic dissociation, picric acid in aqueous solution exists as simple molecules. Consequently, if Walker and Appleyard's result be interpreted from the solution point of view we come to the absurd conclusion that in silk, picric acid is present as  $1/2.7$  molecules.

Georgevics (Monatshft, 1894, 15, 707) does not find that the distribution ratio of Indigo Carmine between silk and water is independent of the concentration, but that  $\sqrt{w}/s = \text{const.}$ , where  $w$  is the amount of the dyestuff in 100 c.c. of the dye liquor after dyeing and  $s$  the amount of colour taken up by 100 grms. of silk. From the pure solution point of view this indicates that if indigo carmine exists as simple molecules in the silk, in aqueous solution it is present as double molecules. There is nothing improbable in this and it may therefore be regarded as supporting, to a certain extent, the theory of Witt.

Schmidt (Zeit. physikal. Chem. 1894, 15, 56) has shown that Eosin and Malachite Green do not distribute themselves in a constant ratio between water and silk at varying concentrations, nor does picric acid between water and cellulose. This fact, namely, that Henry's law does not apply to those substances, leads Schmidt to conclude that the taking up of dyestuff by fibre is "absorption" (purely a surface action). It may be noticed that Schmidt's experiments were carried out with solutions very much more concentrated than those actually employed in dyeing operations.

Sisley (Bull. Soc. Chim. 1900, [iii], 23, 865; J. Chem. Soc. 1901, 80, i, 99) has also contributed results which apparently support Witt's contention.

Zacharias (Färber-Zeit. 1901, 149, 166) proposes a new theory which may be said to embrace that of Witt. He suggests that dyeing always takes place in two stages:

- (1) The dyestuff is absorbed by the fibre;
- (2) The dyestuff is fixed on the fibre as a dye.

This theory assumes then that in the first place the fibre acts as an absorbent, and after the dyestuff has been absorbed a change takes place; the nature of this second change may be polymerisation or it may be of a chemical nature. From Fick's law of diffusion it is then deduced that  $c/c' = \text{const.}$  must apply to the absorption part of the



process. The author believes that the constancy of the values found by Walker and Appleyard and by Georgievics is not to be read as meaning that the substance exists in different molecular species in the liquid and in the fibre, but that the resistance to absorption increases with the concentration and is dependent on the nature of the dyestuff.

Van't Hoff (Vorlesungen, II., 65) has shown that solid solution is in no way connected with high molecular association. It is, therefore, *a priori*, not improbable that in the dyed fibre the colour exists as simple molecules, and if it can be shown that the distribution coefficient is constant and independent of the concentration, then we would be justified in concluding that the fibre acts simply as a solid solvent and Witt's theory would receive experimental confirmation.

Against Witt's theory many authors have raised the objection that the process is not reversible as it should be if it is purely a case of solution. This assumption is gratuitous since it has not yet been proved that in any case to which Witt's theory is applicable, liquid solvent cannot extract dyestuff from the dyed fibre and so establish the same equilibrium from the other side. In all probability in such cases the liquid solvent would partially extract the dyestuff if allowed sufficient time. The time required to establish the equilibrium may be much longer than that necessary for the dyeing operation proper, as might be expected from the opposite nature of the process. Sisley (*loc. cit.*) has shown that silk dyed with Rosaniline on boiling with (amyl?) alcohol partially gives up the dye to the liquid solvent. In this case, evidently, the equilibrium is quickly reached from either side.

Witt assumes that all processes of substantive dyeing may be accounted for by his theory, and many have contested his views because instances have been found which certainly appear to contradict it. The supporters of other theories seem to have endeavoured to construct a single theory to suit all cases. From the very varying chemical nature of the dyestuffs employed it is highly probable that the laws regulating dyeing will vary in so far as the chemical aspect is concerned. Zacharias's hypothesis has much to recommend it, and, as we see, it is an extension of Witt's theory; he assumes that the absorption is always accompanied by a secondary chemical change. Cases so far investigated would appear to support this assumption, but below are recorded some results in which the ratio of the concentrations in the liquid and in the fibre is constant and independent of the concentration within the limits examined. This may then be attributed purely to absorption unaccompanied by polymerisation or any chemical change, that is, the results clearly confirm for those cases the proposition suggested by Witt.

We incline, therefore, to the view that in certain cases the operation of dyeing consists simply in the formation of a solid solution, and that to this solution Henry's law is strictly applicable, but at the same time we recognise that this does not hold good in general, for the non-reversibility of the process in most cases speaks volumes in favour of the view that a chemical or physical change takes place during the fixing.

#### EXPERIMENTAL.

An endeavour was being made to dye a series of standard patterns on wool with known amounts of certain colouring matters in connection with an investigation into the relative dyeing properties of wool and silk (R. B. Brown, this Journal, 1901, 20, 226). For this purpose, pieces of wool (weight 0.6 gm.) were dyed, each in 120 c.c. of water with different percentages of colouring matter. The amount of dyestuff remaining in solution was estimated at the end of the dyeing operation by means of the Duboscq Colorimeter and the actual amount on the wool calculated from this. It was noticed that within wide limits of concentration a constant percentage of dyestuff is taken up by the wool when the relative amounts of wool and water, the temperature, and the duration of the experiment (sufficiently long for the establishment of the equilibrium) are constant. No great accuracy has been aimed at in the experiments recorded below, but the values obtained for the distribution ratio are sufficiently constant.

It would be extremely useful from the point of view of the practical application of dyestuffs to be able to predict exactly how much colouring matter would be removed from the dyebath under given conditions of temperature, time, and concentration, and also to know the effect in this respect of additions of various substances to the dyebath.

Experiments having for their object the determination of these details, and the further elucidation of the solution theory are now in progress.

I.—Colouring matter: Acid Magenta (with addition of sulphuric acid). 0.6 gm. of wool in 120 c.c. of water. Dyed for 1 hour at 100°.

Original Concentration of Aqueous Solution.	Final Concentration of Aqueous Solution.	Final Concentration of Colour in Wool.	Distribution Coefficient.
$x$	$c_1$	$c_2$	$c_1/c_2$
Grm. per Litre.	Grm. per Litre.	Grm. per 5 gm.	
0.005	0.00068	0.00432	0.158
0.010	0.00137	0.00863	0.158
0.015	0.00229	0.01271	0.180
0.020	0.00260	0.01740	0.149
0.025	0.00327	0.02170	0.151
0.040	0.00516	0.03485	0.148
0.050	0.00720	0.04230	0.168
0.0625	0.00869	0.04850	0.158
0.075	0.00997	0.06500	0.150
0.0875	0.01059	0.07700	0.136

II.—Colouring matter: Chrysoidine FF. No addition to dye bath. 0.6 gm. of wool in 120 c.c. of water. Dyed for 1 hour at 60°.

$x$	$c_1$	$c_2$	$c_1/c_2$
0.015	0.00605	0.00605	1.158
0.02	0.01052	0.00948	1.110
0.025	0.01297	0.01203	1.078
0.03	0.01587	0.01413	1.123
0.04	0.02128	0.01872	1.137
0.05	0.02595	0.02405	1.078
0.0625	0.03281	0.02969	1.105
0.075	0.03907	0.03593	1.037
0.0875	0.04541	0.04209	1.079
0.1	0.05130	0.04870	1.053

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



## I.—PLANT, APPARATUS, AND MACHINERY.

## PATENTS.

*Condensed Steam and other Waters for Boiler Feed and other Purposes; Purification of* —. A. Harris, London. Eng. Pat. 18,266, Oct. 13, 1900.

THE purification is effected by the addition of two reagents, such as the soluble salts of alumina and an alkali, which, as described in Eng. Pat. 19,621, 1899, react upon each other to form a precipitate, in which the oil or other impurities will adhere, so that they can be removed by decantation or filtration. According to the present invention, the reagents are added successively, so that the first may be evenly distributed throughout the bulk of the water before the second is added, thereby securing an even precipitation. The reagents may be admitted successively at different levels of the vessel in which the water is treated, or either or both may be added to the crude water in separate vessels, &c., before both are brought together in the treating vessel. Several arrangements of apparatus for carrying out the invention are described in the specification.—R. A.

*Saturating Liquids with Gases; Apparatus for* —. F. Fischer and L. Kiefer, both of Karlsruhe, Baden, Germany. Eng. Pat. 21,494, Nov. 27, 1900.

THE apparatus comprises a collecting reservoir and a mixing cylinder, the feed liquid being supplied by pumps or from a high-pressure feed-pipe. When pumps are used, the supply of liquid to the mixing cylinder is regulated by a float and a valve chest containing three passages. One of the passages is in connection with a feed-water tank, and can be closed by a return-stroke valve, whilst a second passage communicates with the mixing cylinder. The third passage communicates with the collecting reservoir, and can be closed by a valve actuated by a float. When the float is in its depressed position, communication is set up between the pump and the feed-water tank, but when the float is elevated communication is set up between the pump and the collecting reservoir. When a direct high-pressure feed is used, the supply is regulated by a float, which, when at its highest position, closes a valve in the feed-pipe. The mixing cylinder is provided with a blow-off valve, which can be raised by a lever operated by a disc fitted with tappets, the disc being rotated by the liquid supplied to the mixing chamber.—R. A.

*Cooling or Aerating Liquids, and for Purifying Gases; Apparatus for* —. M. Kasper, Düsseldorf, Germany. Eng. Pat. 20,353, Nov. 12, 1900.

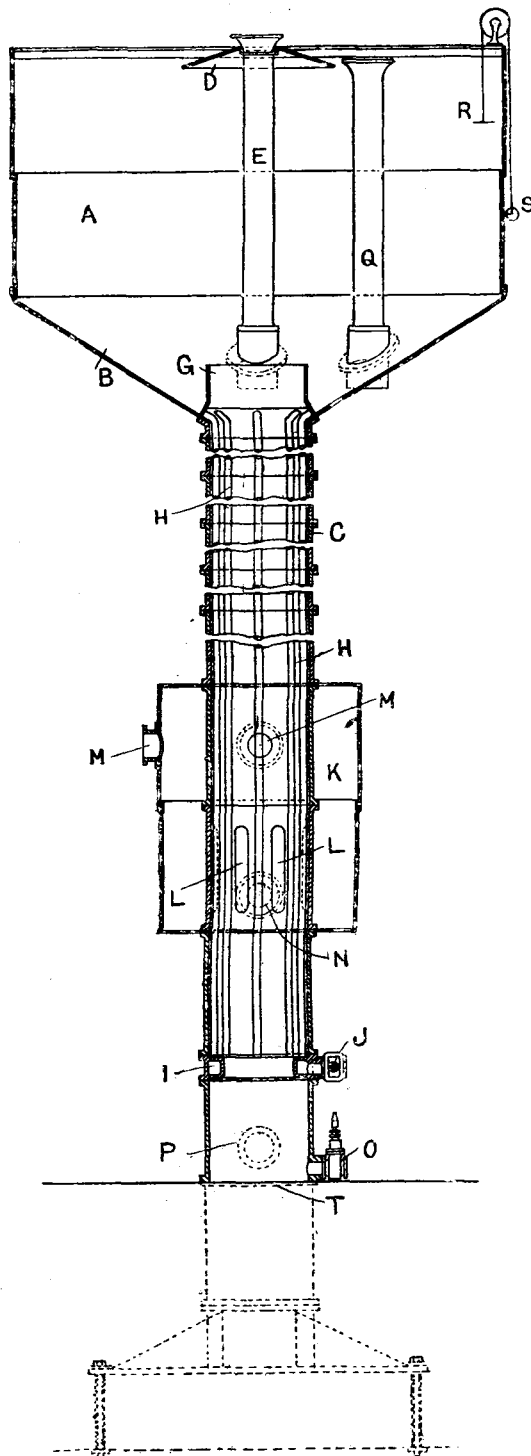
THIS apparatus consists of a number of open frames or perforated supports, mounted one above the other, and provided with natural or artificial stones, over the upper layer of which the liquid to be treated or to be used for purifying gases is distributed. The liquid is finely divided by the stones in its downward passage through the apparatus, thus ensuring a good surface contact between it and the air, &c. One or more vertical or inclined flues are provided through the frame or supports, for the purpose of inducing a good draught.—R. A.

*Solid Matters from Liquids; Apparatus for Separating* —. K. E. Markel and J. J. Crosfield, Warrington, Lancashire. Eng. Pat. 23,051, Dec. 17, 1900.

THE figure shows a sectional elevation of the part of the apparatus above ground, with the foundations in dotted lines.

A is a comparatively shallow tank with conical bottom, resting on a column of any required height, C, which forms part of the tank. The water which it is desired to clarify enters the tank through the pipe E, being distributed by the cone D, so as to disturb the water in A as little as possible. In this tank the dirt gradually settles, collecting mostly in the conical hopper around the lip G, and falling thence down the pipes H to the ring I, whence it is let off from time to time by opening the mud-cock J. Some of the mud passes with the water down the central column, but being of greater specific gravity than the water, and coagulating to a

certain extent, it falls with a slightly greater velocity than that due to the flow of water, and use is made of this difference in velocity to separate it from the water by drawing the latter out through lips L, unitedly of greater area than the pipe or columns C, into the chamber K, from which the clarified water passes away through openings M. The mud



in C descends to the base of the column and is let off, when necessary, by opening the valve O. The foundation is bedded in a mass of concrete.—L. A.



*Liquefaction of Air and other Aeriform Fluids.* C. Joly and E. J. Richardson, London. Eng. Pat. 15,511, July 31, 1901.

THE patentees describe and claim a system and several arrangements of apparatus for "the continuous liquefaction of air and other aeriform fluids the critical temperature of which is below the ordinary temperature of the atmosphere at comparatively low pressures, and any pressure which does not exceed that which is required for the condensation at the critical temperature." The liquefaction is effected by the cooling or expansion of the compressed gas in stages, the expansion of a portion of the compressed gas at the end of each stage being employed to effect the further cooling of another portion of the gas to the next stage, until liquefaction is attained. The heat of compression in the first stage of the operation is absorbed by running water, and thereafter liquid air, an alcoholic solution of calcium chloride, or other unfreezable liquid, or copper or iron filings, &c. is or are employed to aid in the transfer of heat from the compressed portion to the expanding portion of the gas.—R. A.

*Drying Fibrous and other Material [Pulp, Yarn, &c.], and Apparatus therefor.* H. H. Lake, London. From F. Hiorth, Christiania, Norway. Eng. Pat. 14,090, July 10, 1901.

THE materials are conveyed by a carrier through a drying channel, through which a volume of air is circulated in the same direction as the materials, the air being held in continuous circulation through the drying channel and an air-heating apparatus, by means of a fan, &c. A small volume of the moist air is allowed to escape, and a corresponding volume of fresh air is taken into the apparatus. Means may be employed to utilise the heat in the escaping moist air for heating the fresh air taken in.—R. A.

*Solids from Liquids; Centrifugal Apparatus for Separating* — H. H. Lake, London. From J. J. Berrigan, New York, U.S.A. Eng. Pat. 12,181, June 14, 1901.

A RECEPTACLE for the combined constituents to be separated, is constructed to rotate on an eccentric axis passing through it, so that, by the centrifugal force due to the rotation, the constituents are projected and deposited against a portion of the inner periphery of the receptacle. The feed conduit is arranged to direct the materials entering the receptacle against a definite portion of the inner periphery, and scrapers are provided for conveying the solid material from its place of deposit to an escape outlet. The receptacle is balanced during its rotation by liquid contained in an external vessel, in liquid communication with the receptacle, means being provided for preventing the entrance of solid material into the communicating opening.—R. A.

*Cement and other Materials; Apparatus for the Separation of the Flour or Finer Particles of* — W. F. Goreham. Eng. Pat. 15,344, 1900.

See under IX., page 1115.

## II.—FUEL, GAS, AND LIGHT.

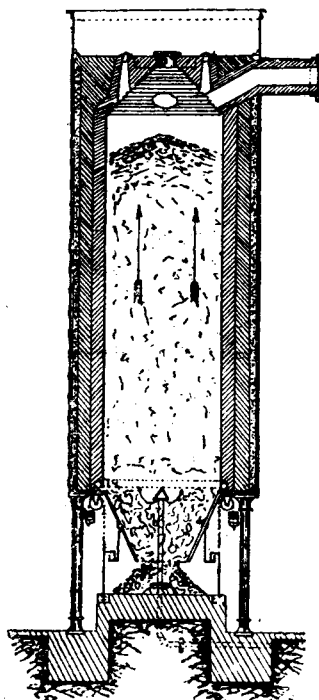
*Peat Briquettes.* Engineer, Aug. 30, 1901, 228.

A NEW kind of peat briquette called "press-coal," due to Schöning, has been introduced into Sweden; in 4 mins., at a temperature of 300°–500° F., and by immense pressure, peat-coal briquettes are produced, containing 66 per cent. of carbon and only 3.7 per cent. of moisture, and 2.9 per cent. of ash. The briquettes are lustrous, solid and compact, and have a specific gravity of 1.2, and a calorific value of 6,500 calories. Trials of the fuel are to be made on the Swedish State railways and at the naval station of Carlscrona.—J. W. H.

*Producer; Gobbe's "Quenching"* — F. Bruyère. J. Gas Lighting, 1901, 78, [2002], 778–779.

THE coke is drawn from gas retorts or coke ovens into tip waggons, which are emptied into the producer, shown

in the annexed illustration, through the door in its top. Steam or a fine spray of water is introduced at the bottom of the producer, and is heated by and cools the coke in the latter as it passes through it. The coke is gradually quenched by the ascending steam, and is removed



from the bottom of the apparatus, while the steam is gradually heated until, in the upper part of the producer, it attains the temperature (600° C.) at which the reaction  $H_2O + C = H_2 + CO$  occurs. The gas thus produced is stated actually to be purer than ordinary water-gas, and to contain only traces of nitrogen and carbon dioxide. It is said to be peculiarly well adapted for heating gas retorts.

The following calculation of the amount of gas obtained in the quenching of coke in this apparatus is given:—70 kilos. of red-hot coke (the residue from the carbonisation of 100 kilos. of coal) will bring into the apparatus  $70 \times 350 = 24,500$  calories. The decomposition of 1 kilo. of steam will require 3,222 calories for the formation of

hydrogen, while the carbonic oxide produced will yield 1,648 calories, making the net heat required =  $3,222 - 1,648 = 1,574$  calories. The gases liberated by the decomposition will carry off at 600° =  $\left(\frac{3.499}{9} + \frac{14 \times 0.245}{9}\right) \times 600 = 456$  calories. Hence the 70 kilos. of coke will decompose  $\frac{24,500}{1574 + 456} = \frac{24,500}{2,030} = 12.06$  kilos. of steam.

The decomposition of 12.06 kilos. of steam will require 8.04 kilos. of coke (? carbon), and as the weight of the gas made will be 0.67 kilo. per cubic metre, the volume of gas will be  $\frac{12.06 + 8.04}{0.67} = \frac{20.10}{0.67} = 30$  cb.m. This is equivalent to 3.730 cb.m. per kilo. of coke (? carbon) consumed. The gas thus produced is stated to be cheaper than either Siemens' producer-gas or ordinary water-gas, because no coke is consumed in raising the heat of the bed of fuel.

—J. A. B.

*Water-Gas in the Destructive Distillation of Coal; Utilisation of* — V. B. Lewes. J. Gas Lighting, 1901, 78, [2000], 623–627.

THE author refers to an earlier paper (this Journal, 1900, 647), in which it was stated that less benzol was required, to enrich to a given extent, a mixture of coal-gas and water-gas in fixed proportions, when the water-gas was mixed with the coal-gas in the foul main than when it was mixed with clean coal-gas. It was also suggested that if a stream of water-gas were passed through the crown of a retort during the carbonisation of coal, the consequent dilution and quickening of the rate of flow of the gas evolved would prevent the secondary reactions which commonly occur in the retort and result in many hydrocarbons valuable as illuminants being broken down into methane, hydrogen, and carbon. A considerable gain in candle-feet per ton of coal seemed likely to result, and the author now reports the results of two sets of trials made at the Crystal Palace District Gas Company's works with a view to ascertain the extent of the gain, if any. T



compare the value of the varying volumes of gas of different candle-power, these values are reduced to "candle-feet," obtained by multiplying the make by the candle-power and dividing by 5. Thus, in the case given below,—

$$\frac{10,468 \times 15.88}{5} = 33,246 \text{ candle-feet.}$$

The first set of trials was made in six (later, 12) beds of seven through horizontal retorts, 22 ins. by 16 ins. in cross section, with Derbyshire coal, which under ordinary conditions of carbonisation yielded 10,468 cb. ft. of 15.88 candle-power gas (= 33,246 candle-feet) per ton. Arrangements were made for admitting water-gas at the top of the ascension-pipes on one side of the bench, and by blocking up the dip-pipes on that side, compelling the water-gas to pass down to and through the retorts, and to escape with the coal gas through the ascension pipes on the other side of the bench. A trial showed that the closing of the dip-pipes on one side, *ceteris paribus*, resulted in the coal yielding 8,615 cb. ft. of 18.58 candle-power gas (= 32,013 candle feet) per ton, instead of the normal result quoted above. Hence it was clear that the normal working conditions had been altered, and that the results obtained in this set of trials, when water-gas was introduced into the retorts, should be taken with some reservation. After trials in which the proportion of water-gas introduced to coal-gas made, was varied, it was found that the highest return in candle-feet per ton of coal carbonised was 40,833, which was obtained with a mixture containing 29.1 per cent. of water-gas. Actually there were obtained 13,730 cb. ft. of gas of 14.87 candle-power. This represented a gain in candle-feet of 22.6 per cent. on the normal results of carbonising such coal.

The second set of trials was made with a bench of 70 inclined retorts, into each of which cold water-gas was admitted through a pipe in the upper mouthpiece, while the gas escaped through the ordinary ascension pipe at the lower mouthpiece. The following are details of trials of the coal (Derbyshire) used, both without the use of water-gas, and with the proportions of water-gas which gave the most favourable results:—

	Without Water-Gas.	Water-Gas added for 3 hours from time of charging the Retorts.	Water-Gas added for 3 hours from ½-hour after time of charging the Retorts.
Cubic feet of gas obtained per ton of coal .....	9,907	14,715	14,925
Illuminating power of gas (candles) .....	16.55	14.85	14.40
Candle-feet per ton of coal .....	32,792	43,703	42,984
Percentage increase in candle-feet over normal carbonising results....	..	33.2	31.0
Percentage of water-gas in mixture .....	Nil.	28.6	28.5
Percentage of carbonic oxide in mixture .....	7.19	..	14.0
Gallons of tar in hydraulic main per ton of coal .....	8.3	7.4	8.07
Gross calorific value of gas (calories per cubic foot) .....	152.6	125.8	127.2
Net calorific value of gas (calories per cubic foot) .....	138.8	118.7	116.8

The economy of admitting water-gas to coal-gas retorts in this manner is dependent on the relative costs of water-gas and coal-gas, and of purification of coal-gas and of the mixed gas. Figures furnished by C. Dellwik of the cost of crude water-gas made by his process with generator fuel at 12s. 6d. per ton, make it 3.12d. per 1,000 cb. ft. for a production of 500,000 cb. ft. per diem and 2.98d. for a production of 1,000,000 cb. ft. per diem. The gas should contain only 4 per cent. of carbon dioxide, and if it were passed while hot into the retorts, the author believes the carbon dioxide would be partially reduced by contact

with the carbon in the retort, though such reduction was not observed when the cold water-gas was used. The cost of purifying the mixed gas therefore might not greatly exceed the cost of purifying coal-gas. Taking the cost of coal-gas of 16.5 candle-power in the gasholder at 1s. per 1,000 cb. ft., and that of water-gas at 3½d., 1,000 cb. ft. of the mixed gas of 14.4 candle-power could be produced for 9.57d. Though the calorific value of the mixed gas would be considerably lower than that of the coal-gas, the temperature of its flame would be as high, and hence it would be equally efficient with coal-gas for lighting by means of incandescent mantles.

The illuminating power tests were made throughout according to the instructions of the Gas Referees, the gas being burnt in the Argand burner at such a rate that it gave a 16 candle-power flame. Had the gas been burnt at the fixed rate of 5 cub. feet per hour, the coal-gas recorded as of 16.55 candle-power would have been returned as of 16.22 candle-power, and the mixed gas containing 29.1 per cent. of water-gas would have been returned as of 12.79 instead of 14.87 candle-power. On the other hand, burning the gas at such a rate that it gave constantly a flame 3 ins. high, would have had the effect of returning the 16.55 candle-power coal-gas as of 17.90 candle-power, and the 14.87 candle-power mixed gas as of 16.78 candle-power.—J. A. B.

#### *Illuminating Gas from Coke-Ovens, Production of —.* F. Schniewind. J. Gas Lighting, 1901, 78, [2001], 691—703.

THE author refers to the large number of by-product coke ovens recently erected in the United States, some of which, as for instance an installation of 400 ovens at Everett, Mass., are designed to supply coke for locomotives and domestic use, and gas for illuminating as well as heating purposes. The following is an outline of the scheme by which the author thinks that the plant may be maintained in full operation throughout the year, while meeting the fluctuations in the demand for illuminating gas. The gas evolved during the first 15 hours of carbonisation is collected for illuminating purposes, and the gas subsequently evolved is used for heating the ovens, after benzol has been extracted from it. The plant is of such capacity that the first or illuminating fraction, amounting, with Dominion coal from Cape Breton, N.S., to 44.5 per cent. of the total, and having an illuminating power after purification, of 18.5 candles, suffices to meet the minimum consumption per diem. The increased consumption during the winter months is met by adding to the first fraction a sufficient volume of the second or heating fraction, and then bringing up the illuminating power of the mixed gas to the required standard by carburetting it with benzol. The benzol recovered from the heating gas would suffice to keep up the illuminating power of the gas distributed, to 20 candles throughout the year, and leave a considerable balance of benzol for sale.

An auxiliary producer or water-gas plant serves to supply heating gas to the ovens when sufficient of the second fraction of the gas evolved from the coal is not available for the purpose. The calorific power of the gas distributed, would vary only to a very small extent, say between 670 and 690 B.T.U. per cubic foot.

The author considers that this scheme would lead to coke-oven plant becoming a central station for the supply of light, heat, and power. Thus for lighting, the gas is of high candle-power, and of high calorific power, and would therefore be serviceable either for open-flame or incandescent burners. For heating, the high calorific power renders the gas the best artificial fuel gas available for domestic and small manufacturing purposes, while the coke would be a good smokeless solid fuel, and the tar might also be used as a liquid fuel. For the supply of power, the gas could be used in gas engines, or producer gas could be made from the coke, and similarly used. Even where boilers were retained, the coke would form a good fuel.

The operating results of a plant of 100 coke ovens and gas plant are given as follows:—





*Annual Expenditure.*

	Net Tons per Annum.
(1) <i>Coal</i> .—800 net tons average coking coal carbonised per diem.....	292,000
(2) <i>Wages, Materials, and Maintenance</i> .—Estimated at 30 to 50 cents per net ton of coal carbonised, inclusive of unloading coal, loading coke, delivering tar to storage tank, making concentrated gas liquor or sulphate of ammonia, and delivering illuminating gas into holder.	
(3) <i>Gas Auxiliary Expenses</i> .—Producer plant for use in winter months, consuming coke.....	6,333
Operating expenses, wages, and materials for producers will be 25 to 40 cents per ton of coke gasified.	
(4) <i>General Expenses</i> .	
(5) <i>Interest and Depreciation</i> .—Cost of plant depends greatly on the location.	

*Annual Receipts.*

(6) <i>Coke</i> (= 75 per cent. of coal).—600 net tons per diem.....	219,000
The value of the coke will depend on the demand, but usually metallurgical and foundry coke obtain better prices than domestic fuel.	
(7) <i>Tar</i> (= 5 per cent.).....	14,600
At present worth 14s. 6d. to 17s. 6d. in London.	
(8) <i>Ammonia</i> (= 1 per cent.) equivalent to sulphate.....	2,920
Sulphate at present quoted 10s. 12s. 6d. in Liverpool, but cost of sulphuric acid must be deducted. The weight of 60° B. acid required is about equal to the weight of sulphate obtained.	
(9) <i>Additional By-Products</i> , such as cyanides, light hydrocarbons, and sulphur, may be recovered. Their market prices are very variable.	
(10) <i>Gas</i> .—The gas sold, after allowing for leakage, &c., will amount to.....	Cb. Ft. 1,250,000,000
The value in the holder will depend on local conditions.	

—J. A. B.

*Gas-Pipes; Destruction of —, by means of Electricity.* W. Leybold. *J. Gas Lighting*, 1901, **78**, [2000], 630—631.

CAST and wrought iron gas-pipes ordinarily are serviceable for 25 to 50 years, cast being more durable than wrought iron ones. In recent years electrolysis has caused rapid destruction of gas-pipes in places where electric tramways exist. A current of about 500 volts passes into the overhead wire from one or more generating stations, and after running into the motor beneath each tramcar, returns by means of the rails to the stations. Each rail is jointed by thick copper wires soldered to the ends of the lengths of rail, but nevertheless the rails offer a certain resistance to the current, some of which passes on a favourable opportunity into the earth and underlying gas and water-pipes, which offer less resistance to it. Current flows from the rails to the pipes at spots remote from the electricity works, and, in the vicinity of the works, flows back from the pipes to the rails. The difference of tension between pipe and rail varied; in Hamburg at a time when the current was supplied from only one works it ranged from 0.2 to 1 volt in most places, but was as much as 4.65 volts near the works. The soil in Hamburg contains 0.006 to 0.004 per cent. of sodium chloride, and the tramway authorities employ salt or refuse sodium nitrate to melt ice and snow on the rails. Sodium chloride is split up into sodium and chlorine, the sodium forming with water sodium hydroxide and hydrogen. The hydrogen is liberated at the negative pole, while the chlorine forms ferrous chloride at the positive pole, and the sodium hydroxide precipitates therefrom ferrous oxide. The process continues until the positive pole is completely dissolved.

Efforts should be made to reduce the currents passing into the gas-pipes by laying rails of sufficient transverse section, well connected by soldered copper wire, or with the joints welded by means of thermite (this *Journal*, 1900, 908, and 1901, 253). The tension in the rails should also be reduced by fixing in many places insulated return transmission cables to carry the current back from the rails to the works.

Destruction of the drums of gas meters and the bells of station governors is observed sometimes, when saline water or glycerin is used in the meter or governor. Currents circulate in such a medium between the different metals used for the component parts of the apparatus.—J. A. B.

*Incandescent Gas Lighting; Discovery of —.* Auer von Welsbach. *J. für Gasbeleucht.* **44**, [36], 661—664.

WHILE engaged in the spectroscopic examination of the light emitted by erbia and other rare earths, when placed in a gas flame, Auer found that small fragments of the earths, held on platinum wire, did not give sufficiently bright spectra. To increase the available illuminating surface, he adopted the plan of impregnating pieces of cotton fabric with the salts of the earths, and on subsequently burning out the cotton the residual oxides were found to be sufficiently coherent for his purpose. Lanthanum oxide treated in this way glowed so brilliantly as to suggest the possibility of applying it to practical illuminating purposes, and thus the idea of the "incandescent mantle" originated. A mantle of lanthanum oxide, however, soon disintegrated and fell to dust when left in the air, owing to the absorption of moisture and carbon dioxide; and this fact led to the use of other oxides, notably zirconia and thoria, in admixture with the lanthana for the purpose of imparting stability to the mantle. Auer's view was that such oxides must be so intimately mixed as to form a "molecular mixture," such as is obtained when the salts are first brought into solution together and then converted into the oxides. Hatinger, who was assisting Auer, now discovered the value of small proportions of ceria in improving the illuminating power of the mantles.

Up to this stage in the development of the invention it had been commercially unsuccessful, and the fortunes of the companies concerned reached a very low ebb. Auer again turned his attention to the use of thoria, then a very rare and costly substance. As mentioned above, it had been used in the earlier mixtures and was found to give a brilliant light for a time; but after 50 or 60 hours the light fell till it was no better than that of mantles made without thoria. This led to an investigation into the nature of thoria, and crystallisation methods were devised whereby it could be prepared in a pure state quickly and in considerable quantities. The surprising discovery was then made that the purer these thoria preparations were, the lower was the illuminating power of mantles made from them, and finally a point was reached at which the mantles had very little illuminating power at all. An investigation of the purified thoria and of the mother-liquors showed that the light-exciting substance accumulated in the latter, and eventually it was identified as ceria, which clings persistently to thoria and can be separated completely from it only with difficulty. Cerium solution was now added gradually to a purified thorium solution, and at last the well-known thoria-ceria mixture, giving the brilliant results of the present day, was arrived at. This discovery at once brought success to the incandescent gas-light industry.

Nothing very definite is known as to the cause of the luminosity of the thoria-ceria combination. Auer's views are as follows:—Bodies capable of glowing brightly in a flame are produced when certain refractory oxides are brought into "molecular mixture" with each other. The main component, or refractory carrier, must remain unaltered in the flame, whilst the lesser component must be both readily reducible and readily oxidisable in it. The proportion in which the two components should be present appears to depend upon the pressure: for atmospheric pressure, in the case of thoria and ceria, 1 per cent. of the latter is the correct amount; but for 100 atmospheres it would probably be greater. In that part of the flame-sheath where the mantle must be arranged, oxidising and reducing actions alternate with extreme rapidity. The oxides, which are extremely finely divided, are thus bathed in gases which are now mainly oxidising and now mainly reducing. The principal component of the mantle is neither oxidised nor reduced under these conditions, but the lesser component assumes alternately the higher and lower degrees of oxidation. If, therefore, the two components are capable of combining when the lesser is in either one of its degrees of oxidation, the combination will

be destroyed when the other degree of oxidation is assumed. This combination and separation of the components will alternate as rapidly as the changes in the character of the flame gases, possibly many million times per second, and the molecular disturbances thus set up will lead to the excitation of light waves.

All incandescence bodies which are of this general character give a more or less useful light, and when examined in the form of salts are found to present certain peculiarities. The salts corresponding to the two components are in each case physically, though not chemically, similar; they crystallise together and form characteristic double salts, as in the case of thorium-cerium salts. In the alumina-chromium oxide combination, which gives a brilliant light, the alumina is the oxidisable and reducible member: chromium and aluminium salts form isomorphous double salts—the alums. Other combinations are those of uranium oxide with thoria, lime, or strontia, in which a small percentage of uranium oxide produces a brilliant light. Here it is the uranium oxide which assumes two degrees of oxidation. Their salts present the peculiarities referred to above; for example, uranium and calcium occur regularly together in certain compounds, as in uranocalcite. Their oxides, like the salts, can combine, and they separate whenever the uranium passes into the state of higher oxidation.—H. B.

*Incandescent Mantles; Photometric Examination of —.*  
Report of the Photometry Committee to the "Deutscher Verein von Gas- und Wasserfachmännern," Vienna, 1901. *J. für Gasbeleucht.* 44, [38], 697—699.

In Germany mantles are obtainable in two forms, those which have been burned-off and collodionised, and those which have not been burned-off. In preparing the former for testing, they should be hung from a hook and set fire to at the top, to burn away the stiffening material. In preparing the latter (which should be kept as dry as possible), they are carefully stretched upon a vertical wooden model, so that the meshes and the upper edge are as nearly horizontal as possible; any folds at the top are smoothed out; and the asbestos loop is adjusted evenly in the notch at the top of the model. The mantle is taken off the model by means of a hooked wire, and is burned off by playing a Bunsen flame round about its top and allowing the material to burn gradually downwards. It is recommended to suspend the mantle over a glass pyramid, to prevent the lower portions from coming together on burning. When the glow of the carbonaceous matter at the top has entirely ceased, the mantle should be shaped and hardened over a press-gas flame, the mantle being repeatedly raised and lowered, while the pressure is gradually increased. The pressure of gas to be used depends upon the kind of mantle. The burner consists of a special form of Bunsen burner, covered with wire gauze at its upper end and provided with a flame-spreader, which directs the flame out sideways. To give proper photometrical results the mantle should be so shaped as to fit the head of the testing burner closely.

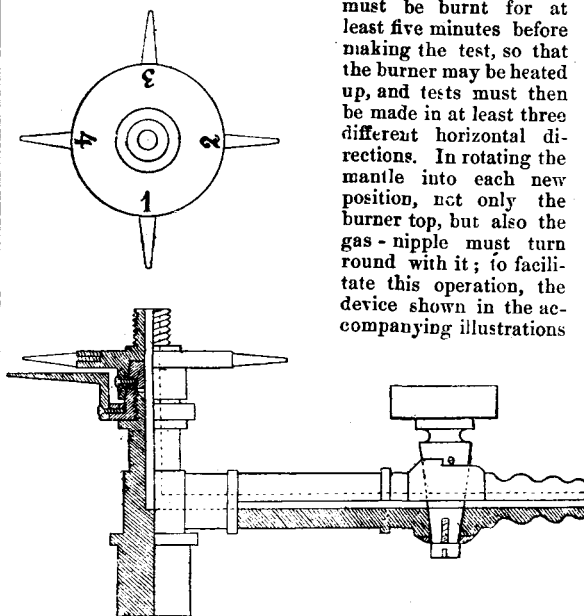
*The Testing Burner.*—As burners of different forms may give different results with the same mantle, the testing burners must always be of the same construction. They are first tested without mantles, to see that they burn uniformly and that the greenish-blue zones are of the standard form, and before each test they are blown through to clean them. The mantle supports must be so high that the distance between the burner rim and the upper edge of the mantle is at least 70 mm. The glass chimney should be a smooth unperforated cylinder, 25 cm. long.

*Regulating the Gas-nipple.*—The gas-nipple of the burner should be so regulated, that with a gas pressure of 35—40 mm. (1·4—1·6 ins.) the consumption will be 115—130 litres (4·0—4·6 cb. ft.) of gas per hour. Before regulating the nipple, the mantle is maintained at incandescence for at least half an hour on a burner with a consumption of about 130 litres per hour. The nipple is then adjusted by gradually increasing the apertures until, at a uniform pressure between 35 and 40 mm., the highest illuminating power is reached. To check this point the gas pressure should be reduced by about 5 mm., by means of the

micrometer screw of the gas-meter, when a diminution in the luminosity should be perceived; the proper pressure is then restored.

*Testing the Mantles.*—To determine the illuminating power of any make of mantle, at least four mantles must be tested, the gas pressure and consumption being adjusted to those for which the nipple of the burner has been

regulated. The mantle must be burnt for at least five minutes before making the test, so that the burner may be heated up, and tests must then be made in at least three different horizontal directions. In rotating the mantle into each new position, not only the burner top, but also the gas-nipple must turn round with it; to facilitate this operation, the device shown in the accompanying illustrations



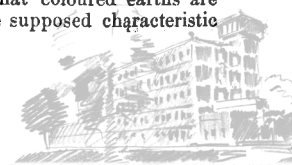
is recommended. It consists of a fixed cone, provided with a fixed arm or pointer, and a rotatable upper part, which is numbered and carries four pointers; its method of application is obvious.

*The Standard of Light.*—Experienced observers can obtain satisfactory results with the standard Hefner flame, but beginners find great difficulty owing to the marked difference in the colour of the two lights. Moreover, with mantles of 70—100 candle-power, the photometer screen comes so close to the Hefner standard, that very slight movements of the screen produce large differences in the reading. The use of an intermediate light standard is therefore recommended, consisting of either a small electric incandescent lamp, actuated by accumulators, or a "Liliput" incandescent gas lamp, provided with a pressure regulator. The "Liliput" lamp must have burned for at least 50 hours before it is used for photometric purposes. The value of the intermediate light standard is determined at the beginning and the end of the test by means of a Hefner lamp, the results being calculated to Hefner units.

*Durability Tests.*—It is sufficient to determine the illuminating power of the mantles at the outset, and after 24, 100, and 300 hours' burning respectively, a clean chimney being used during each photometric test. The gas must burn throughout at the rate and pressure prescribed above, the pressure of 35—40 mm. being particularly attended to, and with this object the burner stand should be provided with an efficient governor.—H. B.

*Luminescence Spectra of the Rare Earths.* E. Baur and R. Marc. *Ber.* 34, [12], 2460—2466.

MUTHMANN and Baur, on examining the luminescence spectra emitted by Yt, Gd, &c., when subjected to the action of cathode rays, obtained results (this Journal, 1900, 785) agreeing with those of former observers. The authors find, however, that Yt, Gd, and La, when in pure condition, have no discontinuous spectra, the spectra hitherto observed being really due to the presence of minute amounts of Er, Nd, and Pr. They have proved that coloured earths are necessary for the production of the supposed characteristic



spectra of Yt, Gd, and La, by adding  $\text{Er}_2\text{O}_3$ ,  $\text{Nd}_2\text{O}_3$ , and  $\text{Pr}_2\text{O}_3$ , to the purest  $\text{Yt}_2\text{O}_3$ ,  $\text{CaO}$ , and other suitable earths, and thus producing artificially the spectra in question, from materials which did not by themselves show them. These mixtures, when in proportions suitable for the production of spectra, may be regarded as "solid solutions" of the coloured body in the colourless one. Whilst spectra are emitted by very minute proportions of the coloured earths, they are developed most brightly when from 0.1 to 1.0 per cent. is present in the mixture; with 5 per cent. they are much weaker, and with 10 per cent. mostly disappear. The spectrum of the coloured earth varies, to some extent, according to the nature of the "solvent" (*i.e.*, the colourless body) employed; moreover, the coloured earths, when present together in the same "solvent," influence each other remarkably, even preventing entirely the emission of discontinuous spectra.

It was observed that when a specimen of yttria, giving the supposed typical Yt spectrum, was further fractionated to remove the last traces of Er, the whole spectrum diminished in brightness, whilst the so-called gadolinium spectrum made its appearance. It was then found that fractions which contained Nd always gave the "gadolinium" spectrum. The two spectra, therefore, appeared to be connected with the presence of Er and Nd. Using calcium sulphate as a neutral "solvent," and adding 1 per cent. of erbium sulphate, the entire spectrum hitherto ascribed to yttrium was obtained. Similarly, with 1 per cent. of neodymium sulphate the "gadolinium" spectrum was produced exactly. To make sure that these results could not have been due to the presence of Yt and Gd in the coloured earths, a specimen of the purest yttria (giving only a weak spectrum), and the  $\text{Er}_2\text{O}_3$  and  $\text{Nd}_2\text{O}_3$  used, were progressively diluted with  $\text{CaO}$ .  $\text{Yt}_2\text{O}_3$ , even at a dilution of only 1:100, barely showed the yellow line, whilst at 1:1000 no spectrum was visible. On the other hand,  $\text{Nd}_2\text{O}_3$  at 1:10,000,000, and  $\text{Er}_2\text{O}_3$  at 1:1,000,000, gave distinctly visible spectra. The spectrum exhibited by specimens of yttria is therefore due to the presence of Er and Nd, in traces not otherwise recognisable.

Praseodymium sulphate was diluted similarly with  $\text{CaSO}_4$  (1:100), when the spectrum ascribed to lanthanum (Muthmann and Baur, *loc. cit.*) was reproduced. The luminescence of lanthanum sulphate is therefore due to traces of Pr. The spectra of  $\text{La}_2\text{O}_3$  and of  $\text{CaO} + \text{Pr}_2\text{O}_3$  (1 per cent.) show considerable differences at first sight; but the lines coincide in position, and the differences are due to the difference of the "solvent." If the same solvent be used [comparing, for instance,  $\text{Pr}_2\text{O}_3$  in  $\text{Yt}_2\text{O}_3$  (1:1000) with  $\text{La}_2\text{O}_3$  in  $\text{Yt}_2\text{O}_3$  (1:100)], the spectra become identical. To prove that the spectrum of the  $\text{Pr}_2\text{O}_3$  was not due to its containing  $\text{La}_2\text{O}_3$ , dilutions with  $\text{CaO}$  were made as above. At a dilution of 1:1000,  $\text{La}_2\text{O}_3$  gave no longer a line spectrum; whereas the  $\text{Pr}_2\text{O}_3$  showed its characteristic lines even at 1:1,000,000.

In view of Crookes' experiments and conclusions as to the decomposability of Yt and Gd, it is interesting to note the effect of Nd and Er upon each other. If dissolved in sulphate of calcium or yttrium in the proportion  $1\text{Nd}_2\text{O}_3:4\text{Er}_2\text{O}_3$ , both spectra appear, complete and of equal intensity. When the  $\text{Nd}_2\text{O}_3$  is increased in proportion, the two red, the orange, and finally the typical yellow lines of erbium gradually disappear; whereas, if the  $\text{Er}_2\text{O}_3$  is increased, spectra are ultimately obtained in which all four lines of neodymia are absent, the first to go being the orange, then the yellowish-green, and finally the red, although in mixtures containing Nd, the orange bands are usually the most intense, and the yellowish-green band the weakest.

The presence of even 0.1 per cent. of iron reduces the luminescence very much, larger proportions preventing it entirely.—H. B.

#### PATENTS.

*Peat and other Moist Substances; Mode of Drying* —. T. A. Dillon, Kingstown, co. Dublin. Eng. Pat. 18,561. Oct. 18, 1900.

THE moist peat or other substance is placed in a chamber through which a steam coil or pipe passes. Air enters the

chamber through several holes at the end at which the steam enters the coil, and leaves it through the annular space between the widened open mouth of the coil, and the sides of an aperture at the other end of the chamber. The steam rushing from the widened mouth of the coil is said to create a vacuum in the chamber, and so maintain a steady continuous flow of hot dry air through it. Waste steam is employed for choice.—J. A. B.

*Peat or other Materials; Pulping, Mashing, and Moulding* —. G. T. Zohrab, Glasgow. Eng. Pat. 18,680. Oct. 19, 1900.

THE apparatus consists of a vertical cylindrical vessel, mounted upon wheels so as to be movable, and provided with an upper bell-feeding mouth for the peat or other like material. It has a central axis on which is a screw, which forces the peat downwards towards a horizontal perforated plate, above and against which rotate suitable knives. The material is forced through the plate to a lower compartment, where it meets other rotating knives serving to thoroughly knead and mix the material. At the bottom of the cylinder are a number of parallel horizontal archimedean screws, which force the pulp out through moulds of any desired shape.—R. S.

*Peat; Method and Apparatus for Treating* —. P. Jensen, London. From G. Heine, Høgsfjord, Norway. Eng. Pat. 3215, Feb. 14, 1901.

PEAT is obtained from the bog by first mixing it with water to make a pulp, and then withdrawing the pulp formed, by suction and conveying it to the factory. The pulp is next carried over cylinder machines, such as are used in paper-making, until it is sufficiently dry to be taken to a press for making briquettes.—R. S.

*Artificial Fuel*. A. Kahn, Helmstedt, and M. Heberlein, Eisenach, both in Germany. Eng. Pat. 2912, Feb. 11, 1901.

ABOUT 75 per cent. of gypsum is mixed with about 15 per cent. of carbonaceous substances, such as peat or peat refuse, leaves, sawdust, coal slime, &c., and the mixture is saturated with 10 per cent. of resin or oils. The mass is pressed and made into blocks.—R. S.

*Smoke-consuming Furnaces*. J. A. Crawford, Allegheny, U.S.A. Eng. Pat. 17,041, Sept. 25, 1900.

THE side walls and the bridge wall are provided with intercommunicating horizontal channels arranged above the grate. Vertical channels pass both upwards and downwards from this channel. The lower channels lead from air inlet openings below the grate, while the upper ones lead to downwardly-inclined openings in the walls of the upper part of the combustion chamber. Other short channels lead directly from the horizontal channels to the furnace. The air inlets below the grate bars are formed of open ended metal boxes, having regulating air inlet slides. Two tubes may lead to the stack, namely, a cold air pipe opening from the ash pit, and a hot air pipe, the exit of which is above the end of the cold air pipe, opening from the fire chamber.—R. S.

*Consuming Smoke and Gaseous Products of Combustion in Furnaces*. G. S. Gallagher, Manhattan, U.S.A. Eng. Pat. 15,811, Aug. 6, 1901.

THIS invention consists in forming the furnaces of steam boilers with special flues in the side walls, the bridge wall, the bridge wall extension, the front wall, and, when a central wall is employed, in that also. These flues are constructed of hollow tiles, fitting closely one against the other. In the side, front, and central wall they have two horizontal rows of perforations, allowing the air heated therein to enter the combustion chamber above the fuel. Those employed for the bridge wall have each two openings, one above the other, the lower an inlet for smoke and gases from the surface of the fuel, the upper an outlet for these gases mixed with air, into the combustion chamber. The inlet and outlet openings lead to a flue bringing heated air. The tiles for the bridge wall extensions are of tapering

form, and are provided with openings communicating from the interior of the hollow tiles to the outside or combustion chamber of the furnace.—R. S.

*Hydrocarbon Oils, and Rendering them Non-Explosive; Improved Process for Purifying* — J. W. Mason, Manchester. Eng. Pat. 16,134, Aug. 10, 1901.

EACH 100 galls. of the oil is treated with 4 oz. of salt and 4 oz. of slaked lime in a cylindrical boiler provided with a blow-pipe, by means of which air can be forced through the oil. After blowing for about 10 minutes, 8 oz. of borax and the same quantity of "borax dry soap" are added to the oil, when the air is again blown through for about one hour. After settling 15 minutes, the oil is run through a charcoal filter into a storage tank. The sludge remaining in the boiler may be used for cleansing purposes, 1 lb. mixed with 5 galls. of hot water being said to form a good soft soap.

—T. A. L.

*Coke Oven.* E. Stauber, Berlin. Eng. Pat. 19,143, Oct. 25, 1900.

THE coking oven consists of a vertical chamber having a feeding hopper, and communicating by a number of short passages, at different levels, with a concentric heating chamber, in which is situated a perforated coiled pipe, through which liquid fuel passes from an upper reservoir. The coal is first dried by means of a solid fuel box which is slid beneath the oven. The vapours then given off pass to the chimney. When vapours cease to be given off, the liquid fuel is turned on, and burns in the heating chamber, escaping from the coiled pipe through a number of openings, the necessary air being admitted through tubes passing through the walls of the oven. The gases given off during this period—the coking period—are drawn through a condenser by an exhaustor. On the completion of the process the fire-box is withdrawn, allowing the coke to fall into a lower chamber, where it is allowed to cool out of contact with air. It is subsequently discharged by means of a door into trucks.—R. S.

*Gas from Gas-Producers, and Heating and Moistening the Air supplied to the Producers; Apparatus for Purifying* — W. J. Crossley, Manchester, and J. Atkinson, Marple. Eng. Pat. 17,734, Oct. 6, 1900.

THE inventors claim, in combination with other gas-producing plant, the use of a tower constructed of superimposed sections, each alternate section being a gas section through which the gas from the producer passes, and in which it is cooled and more or less cleansed, whilst the intermediate sections are air sections, in which air is heated and moistened by a downward current of water which traverses both the gas and air sections, and is conveyed from the one set to the other by means of siphons. The gas takes a downward course, whilst the air moves in the opposite direction.—C. S.

*Heating-Gas from Gas Producers, Blast Furnaces, or Coke Ovens; Apparatus for Purifying* — W. J. Crossley, Manchester, and J. Atkinson, Marple. Eng. Pat. 17,993, Oct. 10, 1900.

THE inventors claim the use and construction of a rapidly revolving body, consisting of more or less bi-conical surfaces, provided with ribs, and enclosed in a corresponding bi-conical outer case, so that the tips of the ribs closely approach the inner surface of the casing. A central inlet is provided for the admission of gas at the one side, and a central gas outlet on the other, the gas passing round the edges of the larger diameter of the revolving body at a very high circumferential velocity, so as to throw impurities outwards into a slot and passage formed for this purpose, and thus separate them from the gas under treatment. The construction of the revolving body and ribs is such that, whilst considerable power is required to impart the high velocity to the gas in passing from the central inlet to the circumference, a large proportion of this power is returned as the gas is slowing down during its approach to the central outlet.—C. S.

*Oil-vaporising Apparatus.* A. Kitson, London. Eng. Pat. 19,848, Nov. 5, 1900.

THE apparatus comprises the combination of an externally-heated vaporising tube, nearly or quite horizontal, which is preferably charged with coke or similar material, a narrow perforated tube placed in the vaporising tube, and of chambers containing respectively water and oil, which substances are forced, under equal pressures, into the vaporiser tube and the perforated tube respectively. Two modifications are given.—C. S.

*Catalytic Substance for Use as a Gas Condenser, and for other Purposes; Manufacture of a Mineral* — A. J. Boulton, London. From A. Tissier, Paris. Eng. Pat. 18,035, Oct. 10, 1900.

A NATURAL quartz mineral, quarried at Antogny le Tiller par les Ormes, France, and composed approximately of 78·8 per cent. of silica, 1·44 per cent. of iron oxide, 6·96 per cent. of alumina, and 4·32 per cent. of magnesia, is powdered, made into a paste, moulded into suitable blocks, &c., and baked at about 2,000° C. The blocks, &c. are made into linings or rings in the smoke passages or flues of boilers, &c. Thus arranged, they are said to absorb "incompletely burnt and waste gases and the gases of combustion, and to combine them so as to ensure complete combustion." The blocks, if caused to absorb petroleum or other liquid hydrocarbon, and then placed on a furnace grate, are said to effect the complete combustion of the hydrocarbon without the emission of smoke.—J. A. B.

*Mineral Oil Gas for Heating Purposes; Apparatus for Generating and Burning* — W. H. Bennett, London. Eng. Pat. 17,004, Sept. 25, 1900.

THE oil is supplied, under pressure, to a central tube closed at the top by a cap, from which the oil passes to a tubular ring, which is heated by the flame of the burning gas, or, at starting, by oil lighted in a cup for the purpose. The oil is thus vaporised in the heated ring, and the vapour passes down to a larger tubular ring placed below, and having outlets at top, through which the vapour issues and burns. A cylindrical shell encompasses the burner, and an inner truncated inverted cone constitutes with the shell, an annular outlet for the flame.—J. A. B.

*Carburetted Apparatus; Construction of* — C. Karfunkelstein, Berlin. Eng. Pat. 14,513, July 16, 1901.

THE principal feature in this apparatus is that the compressed air, in flowing through the porous material impregnated with the hydrocarbon to be vaporised, is claimed to uniformly distribute the fluid in the upper layers of the said material, and to automatically replace the evaporated liquid from a reservoir in the lower part of the apparatus, so as to bring the ascending air well into contact throughout its whole course with finely-divided hydrocarbon, thus increasing the carburetted effect. The vessel used for this purpose is divided, by perforated partitions, into three superimposed compartments, the upper one for collecting the carburetted air, and furnished with outlet taps; the central compartment containing the porous material; whilst the air-supply pipe opens into the lower compartment (serving as reservoir).—C. S.

*Spirit and the like for Lighting, Heating, and Motive Power Purposes; Process and Apparatus for Vaporising* — K. Gossweiler, Ulm, Germany. Eng. Pat. 22,129, Dec. 5, 1900.

THE invention relates to a system for the preliminary heating of gas generators, the heat being produced "indirectly without direct ignition of a combustible connected with the gasification tube," namely, by blowing air through a portion of the spirit to be vaporised and igniting this vapour for the purpose of heating the gasification tube. In one modification of the apparatus a separate reservoir is provided for this portion of the spirit; in another, means are provided for raising the spirit to be vaporised into the hottest zone of the heating flame, in order to accelerate gasification; and in another, the wick for the combustible is separated from the remainder in the reservoir by means



of a casing, "for the purpose of limiting the cooling of the gas produced by the still ungasified combustible to the amount given out through the casing." Acetylene, or coal gas, can also be used for the preliminary heating.—C. S.

*Gas for Lighting, Heating, and other Purposes, and Apparatus for Use in connection therewith; Manufacture of —.* H. Lane, Manchester. Eng. Pat. 17,762, Oct. 6, 1900.

THE process claimed consists of the following stages:—(1) Combustion of fuel in a generator; (2) forcing the gaseous products of combustion downwards by means of an air-blast introduced beneath the raw fuel; (3) completing the combustion by a supplementary supply of air introduced preferably between the generator and the regenerator; (4) the upward flow of the gaseous products through the regenerator, which has internal chequer-work composed of firebrick below and preferably of metal above; (5) the spraying of water on the highly heated upper chequer-work of the regenerator, and the downward flow of the resulting steam; (6) the scouring out the remnant of the gaseous products of combustion by the steam aforesaid; (7) the flow of superheated steam from the regenerator into and up the coked and raw fuel in the generator; (8) decomposition of the superheated steam into its constituent gases; (9) destructive distillation (into vapour) of the raw fuel by the upward flowing gases; and (10) formation of new gaseous products by the admixture of the resulting vapour with the said gases.

The apparatus includes a generator, furnished with a fire-grate and water seal near the bottom, and a feed aperture and gas outlet, as well as a series of air-inlet pipes (automatically controlled) and passages at intermediate points; a passage communicating with a regenerator; an air-inlet pipe and passage for a supplementary air supply, introduced preferably into the passage between the generator and regenerator; also an arrangement of chequer-work within the regenerator, &c.—C. S.

*Gas; Apparatus for Purifying —.* J. E. Prégardien, Kalk, near Cologne. Eng. Pat. 13,788, July 6, 1901.

PURIFYING vessels, with vertical inlet and outlet pipes connected in series, have a bye-pass between the inlet and outlet pipe of each vessel. The bye-pass is closed or opened by alteration of the position of plugs in the inlet and outlet connections. These plugs provide a hydraulic seal for the ends of the pipes which are shut out for the time being.—J. A. B.

*Naphthalene contained in Illuminating and Heating Gases; Apparatus for Extracting the —.* L. Breittmayer, Bordeaux, France. Eng. Pat. 10,024, May 14, 1901.

THE gas to be freed from naphthalene passes through a closed vessel containing a suitable naphthalene solvent, the pipe or pipes by which the gas enters being very close to the surface of the liquid, so that the inrush of the entering gas agitates the solvent, continually exposing a fresh surface. The vessel is also provided with a gas exit and openings for charging and emptying.—T. A. L.

*Gas Igniters; Automatic —.* L. Mette, Berlin. Eng. Pat. 17,383, Oct. 1, 1900.

A FABRIC, preferably conical or bell-shaped, is impregnated with a solution of salts of aluminium, cerium, and platinum, in such proportions as to leave a residue, after burning, consisting of about 98·7 per cent. of alumina, 1 per cent. of ceria, and 0·3 per cent. of platinum. In the upper part of the conical fabric is fixed an igniting-ball of platinum black, and outside the conical body is arranged another conical body "of the toughest possible material, such as alumina, clay, asbestos, or similar substance." The device is fixed inside the upper end of the lamp chimney. The object of the strong outer conical body is to protect the inner fabric from injury by the explosion which accompanies ignition. The inner fabric may be replaced by a brush-shaped bundle of threads impregnated with rare earths and with platinum.—H. B.

*[Incandescent] Gas or other Burners; Apparatus [Caloric Engine] for Supplying Air, or other Combustion Supporter, to —.* A. J. Boulton, London. From The New Process Lighting Co. of Cleveland, Ohio. Eng. Pat. 21,622, Nov. 29, 1900.

THIS relates to an incandescent gas lamp supplied with air under pressure, the air-compressor consisting of a blower-fan driven by a caloric engine, which is situated in the upper part of the lamp and actuated by the waste heat from the burner. The caloric engine is of the type in which air is expanded and contracted, for furnishing the motive power, by being alternately shifted from one end to the other of a cylinder the ends of which are maintained at widely different temperatures.—H. B.

*Incandescent Lighting; Apparatus for Burning Mixtures of Air and Inflammable Vapour for Heating and —.* J. B. Leroux and P. J. Carmien, Nantes. Eng. Pat. 11,504, June 5, 1901.

ALCOHOL or mineral oil, contained in a bottle or other convenient receptacle, is conveyed by means of a capillary siphon tube to a distributor, which is provided with a chamber containing a filter, for removing impurities from the liquid, and with a valve for regulating the supply of liquid. A Bunsen mixing tube, which may be provided with a mantle at the top, is superimposed upon the distributor. To start the burner, the base of the mixing tube, into which the volatile liquid issues, is heated by the application of a spirit torch.—H. B.

*Bunsen Gas Burners.* M. S. Walker, Chicago. Eng. Pat. 15,139, July 25, 1901.

AT the lower end of the usual Bunsen mixing-tube is arranged a "primary mixer," consisting of a chamber provided with a gas-inlet nozzle of small diameter, and having air inlets at the sides as usual. Above the gas nozzle, and in line with it, is a second discharge orifice, through which the mixture of gas and air issues into the ordinary Bunsen tube, where a further admission of air may be made if desired. The second discharge orifice may have several forms; as, for example, simply a central perforation in a diaphragm which extends across the tube, or a vertical tubular piece fixed centrally in such a diaphragm. The burner may be used in incandescent gas lighting.—H. B.

*Incandescent Gas Burners.* H. Winkler, Berlin. Eng. Pat. 19,535, Oct. 31, 1900.

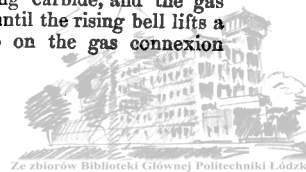
IN an incandescence burner having a rate of gas consumption at least twice that of the ordinary Welsbach burner, there is arranged, between the burner head and the mixing tube, a shallow intermediate chamber, into which the top of the mixing tube projects. This chamber is of greater diameter than the mixing tube, but narrows quickly, in the upward and downward directions, to the diameters of the burner head and the mixing tube respectively. The burner is supplied entirely or mainly with air drawn through the mixing tube, the burner head and mantle being surrounded by a globe, surmounted by a chimney for creating a strong draught.—H. B.

*Incandescent Bodies for Lighting Purposes.* G. Meyer, Prague. Eng. Pat. 19,859, Nov. 5, 1900.

A PROCESS of making incandescent bodies more resistant, consisting in knitting, sewing, or netting on the impregnated, and not yet incinerated, incandescent body or mantle, threads forming a rib or framework, which threads are impregnated with suitable chemicals with the object of vitrifying or stiffening and thoroughly attaching them to the mantle . . . For example, the stiffening threads may consist of cotton threads impregnated with silicic acid preparations.—H. B.

*Acetylene Generators, and Apparatus connected therewith.* L. Thorne, London. Eng. Pat. 16,960, Sept. 24, 1900.

WATER passes from the tank of a bell gasholder to one or more adjacent generators containing carbide, and the gas evolved, passes into the gasholder until the rising bell lifts a weight, and thereby closes a tap on the gas connexion



between the generator and holder. Gas subsequently evolved, forces back the water from the generator to the gasholder tank, and thus leaves the carbide dry until, by the descent of the bell, the gas connexion is re-opened, and the pressure in the generator thereby relieved.—J. A. B.

*Acetylene and other Gases; Purifying* — G. G. Smith, Florence, Italy. Eng. Pat. 20,973, Nov. 8, 1900.

THE gas is passed through pipes which dip beneath the surface of liquid vaseline, which is stated to remove impurities, in addition to water, as already claimed under Eng. Pat. 12,240 of 1900 (this Journal, 1900, 1001).

—J. A. B.

*Acetylene Generator with Automatic Supply of Carbide.* K. Gossweiler, Ulm, Germany. Eng. Pat. 2766, Feb. 8, 1901.

CARBIDE is contained in a receptacle, the position of which is varied through the movements of a bell or float, so that, when the bell contains little gas, carbide is discharged into water, and gas is thereby generated, and, when the bell is nearly filled with gas, the discharge of carbide is completely arrested.—J. A. B.

*Acetylene Gas Apparatus.* J. Sharpe and R. G. Code, both of Ottawa, Canada. Eng. Pat. 7286, April 9, 1901.

THE movements of a gas bell, into which the acetylene passes, operate, by levers and ratchet and pawl mechanism, a rotary water distributor, by which water passes through a movable intake to generating chambers placed below.

—J. A. B.

*Acetylene-Gas; Apparatus for Generating and Storing* — F. Schmitt, Mannheim, Germany. Eng. Pat. 9664, May 9, 1901.

WATER passes from a receptacle into one or more generating chambers, containing carbide, placed within the receptacle. The gas evolved passes to a gasometer and thence to an intermediate storage chamber, from which it expels water. The water so expelled is independent of the water which is used for supplying the generating chambers, and the gas is therefore said to be stored in the storage chamber under uniform pressure.—J. A. B.

*Acetylene Generators for Lanterns, Lamps, and other Purposes.* L. Blériot, Paris. Eng. Pat. 13,790, July 6, 1901.

A RESERVOIR of water, formed as a truncated pyramid or cone, in order to fit without play in a socket of corresponding form, contains a tubular box, with double bottom, in which is a basket containing carbide or acetylith. A screw, with winged head accessible from outside, opens or closes an orifice at the base of the box, by raising or lowering a conically shaped elastic plug. When the orifice is closed, the elasticity of the plug still admits of the escape of gas in the event of the pressure within the box becoming high. A purifier and cap with elastic ring joint are provided at the top of the box.—J. A. B.

### III.—DESTRUCTIVE DISTILLATION. TAR PRODUCTS, PETROLEUM.

*Petroleum; Filtering* —, through Fuller's Earth. C. Engler and E. Albrecht. Zeits. angew. Chem. 1901, [36], 889.

EXPERIMENTS were carried out to ascertain the nature of the processes taking place during the filtration of petroleum through Florida earth (see this Journal, 1900, 890, 1005). The results obtained are shown in the table.

Dimensions of filtering layer: 86 cm. high, 4 cm. diameter. Sp. gr. 0.7929 at 15° C.; and appearance of the crude oil: translucent, brown, with greenish fluorescence.

In order to preclude the possibility of chemical action during the filtration, a mixture of the oils which had already been once filtered through Florida earth, was filtered; this would consequently be free from any substances likely to be affected chemically by the filtering material. Similar results were obtained, thus showing that the separation is

effected mechanically. In a further experiment the filtered oil was drawn off at different levels from the tube containing the Florida earth. The results show that the lighter oil ascends through the filtering layer more quickly, whilst the heavier portions remain behind and are found in the lower part of the filtering tube, whence they are forced slowly upward by the fresh oil entering from below. The separation of petroleum into fractions of different specific gravity by means of Florida earth, appears to be due solely to capillary attraction. Experiments with other filtering media, as infusorial earth, either alone or mixed with sand, &c., did not give satisfactory results.

Fraction.	Sp. Gr. at 15° C.	Volume.	Appearance.
1	0.7812	About 70 c.c.	Water-white.
2	0.7870	" 65	Water-white, with faint fluorescence.
3	0.7897	" 120	
4	0.7905	" 70	Faint yellow; greenish fluorescence.
5	0.7913	" 50	
6	0.7919	" 110	Rather more yellowish.
7	0.7920	" 100	
8	0.7965	" 400	Yellow, with greenish fluorescence.
9	0.8020	" 50	
10	0.8032	" 100	Orange, with greenish fluorescence.
11	0.7976	" 300	
12	0.7962	" 500	

There is only agreement to a limited extent with the conclusion drawn by Day (this Journal, 1900, 890), that the differences in natural petroleum arise from their having undergone a similar process of capillary filtration through porous strata. But there is agreement that in individual instances a separation and transference of the lighter fractions from the place of formation to a centre of deposition at a distance, may have occurred, and also that if the communication was subsequently cut off or considerably obstructed, a permanent local separation of the light oils from the heavy ones may have been effected; but it is pointed out that if the original communication were maintained, the separation effected by capillarity would be gradually counteracted by diffusion. Further, the chemical nature of the oils from various adjoining districts is often so divergent as to prevent a common origin being ascribed to them.—A. S.

*Petroleum Inclusions in the Muschelkalk Formation of Roth-Malsch, Baden.* C. Engler and E. Albrecht. Zeits. angew. Chem. 1901, [37], 913—916.

THE samples of oil examined were collected from the cavities of certain fossil Gryphaeæ and Ammonites, occurring in the Muschelkalk (Lias a) at Roth-Malsch (Baden). Of the following specimens, No. 1 had lost its lighter fractions through exposure to the air, No. 2 was a thin brown oil taken from freshly broken rock, and No. 3 a thin yellow transparent oil from a mussel cavity containing crystals of calc-spar. The chemical composition was found to be—

—	I.			II.	III.
Carbon .....	87.22	87.66	87.20	86.73	86.59
Hydrogen ...	12.65	12.33	12.45	12.95	12.92
Total ....	99.87	99.99	99.65	99.68	99.51

with traces of sulphur and nitrogen. The oil is therefore mainly a hydrocarbon oil similar to that found in Baku crude by Markownikow and Oglobin.

To examine the properties of the oil and isolate any asphaltum, pitch, and paraffin, the sample was treated with petroleum spirit (ligroin) boiling below 50° C., and left to stand for several hours, the deposited asphaltum being collected on a filter, washed with the same spirit, dissolved in benzene, filtered, and evaporated to drive off the solvent. The first filtrate was distilled, the residue dissolved in ether, cooled in a refrigerating mixture, and treated with cooled





alcohol, the deposited sticky flakes being washed with ether-alcohol (1:1). The alcohol-ether solution on being re-cooled also deposited a pitchy substance, which was united with the other, the paraffin being extracted with alcohol under a reflux condenser, and the pitch left behind. The oil recovered from the filtrates was thick and dark brown, sp. gr. 0.9453 at 17° C.

The original assumption that this oil was formed from the fatty matter of the fossil fauna, was disproved by the quantity being too great for such an origin, and also by the presence of similar oil in accidental cavities in the rock. The assumption is, therefore, that the oil was forced into the fossil and other cavities, by pressure, from the surrounding bituminous rock; and the same process is considered by the authors to have probably occurred in the case of petroleum deposits found in sandstones and other porous rocks.—C. S.

*Benzol; Detection of Carbon Bisulphide in —.*

E. Votoček and R. Potměšil.

See under XXIII., page 1147.

#### PATENT.

*Preservative Oils for Coating and Impregnating; Process of Treating Tar Oils for the Production of —.*

H. Herborn, Wiesbaden, Germany. Eng. Pat. 3922, Feb. 23, 1901.

The heavy tar oils, after having been freed from their readily volatile constituents, are thoroughly agitated in the cold with 3 per cent. of concentrated hydrochloric acid and 2 per cent. of a saturated solution of zinc chloride or sulphate or aluminium chloride. The oil is then allowed to stand for several hours, when the clear portion is decanted. It contains about 2 per cent. of chlorine, and is of a characteristic reddish-brown colour; its specific gravity and viscosity have increased, and it has lost its unpleasant odour. On distillation, hydrochloric acid is given off at 200° C., and the distillate shows a characteristic red coloration. As an example of the method, a tar oil having a sp. gr. 1.097 and viscosity 2.93, took up 2.25 per cent. of chlorine, and then had a sp. gr. 1.122 and viscosity 6.00.—T. A. L.

#### IV.—COLOURING MATTERS AND DYE STUFFS.

*Yellow Dyestuff obtained from Sulphocyanide Salts; Sulphocyanogen, so-called Pseudosulphocyanogen, and the —.* A. Goldberg. J. prakt. Chem. 1901, 64, [15, 16, 17, 18], 166—181. (See also this Journal, 1901, 113, 238, 798.)

A DESCRIPTION is given of the production of pseudosulphocyanogen by the action of hydrogen peroxide and potassium or ammonium persulphate on potassium or ammonium sulphocyanide. The yield is much lower than by the processes given in the former papers. The analyses of the products show them to be mixtures of at least two substances, from one of which the small quantities of Canarin are derived. The product obtained by the action of one equivalent of bromine can be freed from oxygen by dissolving in pure sulphuric acid, filtering, and pouring into water; persulphocyanic acid,  $\text{H}_2\text{C}_2\text{N}_2\text{S}_3$ , is also formed. The alkaline solution of the anhydrous substance has the same tinctorial properties as the alkaline solution of the original pseudosulphocyanogen.

The dyestuff, Canarin, is always formed in much smaller quantity than the yellow compound; its empirical formula is  $\text{H}_6\text{C}_8\text{N}_8\text{S}_6\text{O}$ . Experiments showed that a favourable yield of Canarin could be obtained in the dry way by the action of halogen on sulphocyanides, if care were taken that the halogen acted uniformly and at a high temperature. Hence the process of Ger. Pat. 101,804. 8 kilos. of sodium sulphocyanide are heated in a ball-mill to 150°—180° C., whilst 3.5 kilos. (one equivalent) of dry chlorine are led in, the mill being in motion during the whole operation. The resultant brownish-red powder is washed with water, the residue heated to 60° C. with a solution

of 5 kilos. of caustic soda in 40—50 litres of water, allowed to cool, filtered, and the residue treated with 1—2 per cent. caustic soda solution. A pale yellow substance then dissolves. The residue is chiefly composed of the sodium compound of the dyestuff, which is separated by acids as a brownish-yellow voluminous precipitate. The sodium salt is soluble with difficulty, especially in solutions of salts of caustic soda. The potassium salt is much more soluble. Both salts are substantive cotton dyestuffs of similar properties. The finest and greenest shade is obtained with an addition of about 10 per cent. of soda to 2 per cent. of the sodium salt. Somewhat redder shades are given by sodium chloride, sulphate, and phosphate. The dyeing power approaches that of Chloramine Yellow or the chlorinated Primuline Yellow, to which latter the shade is most similar. The fastness to acids, soap, and fulling is very considerable; the shades also resist cold or hot strong alkalis. Canarin and its sodium salt appear to be non-poisonous.—A. C. W.

*Indigo; The Electrolytic Reduction of —.* F. Haber.

J. prakt. Chem. 1901, 64, [15, 16, 17, 18], 289—293.

(See also this Journal, 1899, 757, 1132.)

BINZ, in his account of the electrolytic reduction of indigo, makes no mention of the efficiency, which, however, appears to be low. The theoretical conclusions thus cannot be regarded as proved. The author does not consider that the reduction of indigo in such an unfavourable electrolyte as a paste of zinc oxide and caustic soda by means of a prodigal consumption of current can be regarded as a "solution of the problem" of the electrolytic reduction of indigo.—A. C. W.

*Anthragallol; Nitro-Compounds of —.* II. M. Bamberger and F. Böck. Monatsh. für Chem. 1901, 22, [7], 717—731.

As a further result of work on these compounds, the authors find that  $\alpha$ -nitro-anthragallol, obtained pure by recrystallising the product of the reaction of water on pseudonitro-anthragallol, from alcoholic benzene, gives on acetylation a derivative of the composition  $\text{C}_{14}\text{H}_4\text{O}_5\text{NO}_2(\text{O} \cdot \text{C}_2\text{H}_5\text{O})_3$ . Further, pseudonitro-anthragallol gives the following reactions:—By the action of methyl alcohol, it yields a derivative of a pseudo nature; aqueous hydrochloric acid converts it into  $\beta$ -nitro-anthragallol,  $\text{C}_{14}\text{H}_7\text{O}_5\text{NO}_2$ , whilst the alcoholic acid gives monochloro-anthragallol,  $\text{C}_{14}\text{H}_7\text{O}_5\text{Cl}$ ; formic acid has the same action as water, yielding  $\alpha$ -nitro-anthragallol; with pyridine, pseudonitro-anthragallol forms a salt,  $\text{C}_{14}\text{H}_8\text{O}_5\text{HNO}_3$ ,  $\text{C}_5\text{H}_5\text{N}$ ; whilst acetyl chloride gives a compound of the formula  $\text{C}_{16}\text{H}_{10}\text{O}_8\text{NCl}$ .—T. H. P.

*Anthragallol; Nitro-Compounds of —.* III. M. Bamberger and F. Böck. Monatsh. für Chem. 1901, 22, [7], 732—736.

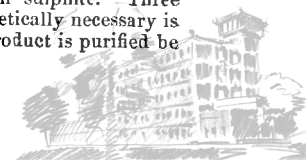
The anthragallolamide obtained by Georgievics by the action of ammonia on anthragallol, does not give the expected nitro-anthragallolamide on nitration. Dimethyl-anthragallol,  $\text{C}_{14}\text{H}_5\text{O}_5\text{OH}(\text{OCH}_3)_2$ , is obtained by the action of dimethyl sulphate.—T. H. P.

*Diazo-Sulphonates and Phenols or Amines; Action of Light upon Compounds of —.* Seyewetz and Blanc. Rev. Gén. des Mat. Col. 1901, 5, [56], 203—205.

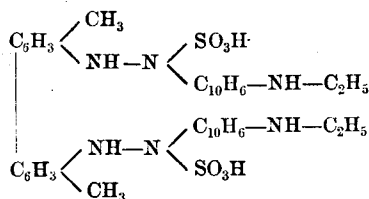
It is known that sodium sulphite gives, with di- and tetrazo compounds, bodies which, when mixed with amines and phenols, do not furnish dyestuffs. The latter are produced, however, as Feer has shown (this Journal, 1890, 1902), when such mixtures are exposed to light.

The authors find that the sodium sulphite compounds in question react with amines and phenols, forming compounds which are decomposable into sulphur dioxide and dyestuffs. The precise conditions of this action and the subsequent formation of dyestuff, have been investigated in the case of the compound formed from sodium tetrazodityl disulphonate and  $\beta$ -naphthylamine ether.

Sodium tetrazodityl disulphonate is obtained by acting upon tetrazotised tolidine with sodium sulphite. Three times the proportion of the latter theoretically necessary is required to give the best yield. The product is purified by



recrystallisation from a 10 per cent. solution of sodium sulphite. It was combined with  $\beta$ -naphthylamine ether by mixing it with a dilute solution of the latter at a temperature of  $12^{\circ}\text{C}$ ., an oily precipitate being obtained, which crystallises after standing for a few hours. This was recrystallised from 30 per cent. alcohol, being dissolved in the latter at a temperature of about  $80^{\circ}\text{C}$ . Above this temperature it undergoes partial decomposition into sulphur dioxide and a red dyestuff. The same change is brought about by the action of light. The crystals, which have an orange-yellow colour, decompose at about  $160^{\circ}\text{C}$ . without melting. They are insoluble in cold, and sparingly soluble in hot alcohol and insoluble in ether, benzene, and chloroform. Alkalis in alcoholic solution give a precipitate which is slightly soluble in alcohol and water. Dilute acids on boiling, and concentrated acids in the cold, decompose the compound, liberating sulphur dioxide. Its formula is—



The dyestuff, as obtained by boiling this compound with alcohol and cooling, is a red powder, decomposing at  $150^{\circ}\text{C}$ ., insoluble in cold and very slightly soluble in hot water, moderately soluble in alcohol, acetone, and acetic acid, and sparingly soluble in ether. It is insoluble in dilute but dissolves in concentrated acids with a blue or violet colour. Its identity was proved with the dyestuff produced by direct combination of  $\beta$ -naphthylamine ether and tetrazorised tollidine.—E. B.

*Fluorescein; Sensitiveness to Light of —, its Substitution Derivatives and Leuco Bases.* O. Gros. Zeits. phys. Chem. 37, 156. Zeits. angew. Chem. 1901, [37], 924.

THE sensitiveness to light was tested by soaking earthen plates with ethereal solutions of the substances under examination, and then exposing them to light, one-half of each plate being covered with black paper to serve as a check test. It was found that the influence of light on the colours was independent of any elevation of temperature due to the light itself, inasmuch as the concealed halves of the plates became warmer than the exposed portions.

The effect of different parts of the spectrum was also examined, by the interposition of coloured glasses. The coloration of the leuco bases was most extensive under rose-red; furthermore, in most of the leuco bases, a second maximum of coloration was recorded under the corresponding complementary colour of the colouring matter. The addition of salts was found, with few exceptions, to modify the velocity of the reaction; copper salts, barium chloride, and mercury chloride retarding, whilst salts of cadmium, manganese, lead, molybdenum, tungsten, uranium, &c., produced acceleration. The nitro compounds appear to possess a special affinity for oxidation, being the only members of the leuco bases that oxidise in the dark.

The sensitiveness of the colouring matters themselves to light is based on a process of oxidation, and not of reduction, as is sometimes assumed. This was proved by the direct measurement of the oxygen consumed in the course of the exposure to light, by means of a special apparatus.

The influence of the resulting colouring matter on the oxidation of the leuco bases is at first accelerative; and the sensitiveness to light is heightened by the presence of extraneous pigments. This catalytic action is probably stimulated by absorption of light; hence the sensitiveness of the leuco base extends not only to the rays absorbed by itself, but also to those absorbed by the resulting colouring matter.—C. S.

*Diphenylamine; Two New Nitro-amino Derivatives of—.* F. Kährmann and G. Steiner. Ber. 34, [12], 3089—3092.

*2-Nitro-3'-Aminodiphenylamine.*—A mixture of *o*-nitrochlorobenzene (25 grms.) and anhydrous sodium acetate

(25 grms.) is brought into a wide-necked flask and heated in an oil bath to  $170^{\circ}$ — $180^{\circ}$ . Metaphenylenediamine is gradually added and the heating is continued for 6—7 hours, while a slow stream of carbon dioxide is sent through the flask. The substance crystallises from alcohol in long red needles melting at  $112^{\circ}\text{C}$ ., which are nearly insoluble in boiling water, but dissolve readily in benzene, alcohol, ether, and glacial acetic acid. The average yield, calculated on the nitrochlorobenzene used, was 40 per cent. of the theoretical.

The sulphate (thick brown prisms or fine yellow needles) is easily soluble in boiling water and alcohol.

On reduction the hydrochloride of 2,3'-Diaminodiphenylamine is obtained in long snow-white clustering needles. This salt is stable, when dry, in the absence of light, but becomes pink when exposed to light or to the atmosphere.

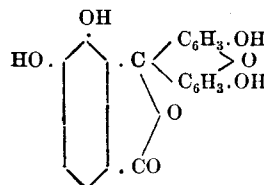
2-Nitro-2'-Aminodiphenylamine was obtained by heating together *o*-phenylenediamine, sodium acetate, and *o*-nitrochlorobenzene in an atmosphere of carbon dioxide. The yield was extremely poor. The substance crystallises from a large amount of boiling water in yellow-red needles melting at  $103^{\circ}$ .—H. L.

*Dyestuffs of the Æsculetin Series.* C. Liebermann and F. Wiedermann. Ber. 1901, 34, [12], 2608—2617.

By treating the bisulphite compound of æsculetin with ammonia, Rochleder, in 1867, obtained a remarkably fluorescent dyestuff, which he termed *Æscorkein*. The bisulphite compound has the formula  $\text{C}_{18}\text{H}_{13}\text{O}_8\text{NaHSO}_3$ , but is not an ordinary double compound. When treated with dilute mineral acids, no sulphur dioxide is given off; the residue consists of an organic compound containing sulphur, and is, in fact, dihydro-æsculetin sulphonic acid. On treatment of the sodium salt with ammonia or ethylamine vapour, it is converted into æscorkein sodium sulphonate, which dissolves in alkalis with a blue colour and a brilliant blood-red fluorescence. The product gives a dibromide which dyes blue shades on silk, wool, and chromed cotton. The reduction product from æsculetin with sodium amalgam, æscorkein, is probably identical with Rochleder's hydro-æsculetin. Both products have the formula  $\text{C}_{18}\text{H}_{14}\text{O}_8$ , melt at about  $300^{\circ}\text{C}$ ., and give the same acetyl compound melting at  $274^{\circ}\text{C}$ . On treatment with ammonia vapour, hydro-æsculetin is converted into a product having the formula  $\text{C}_{18}\text{H}_{13}\text{N}_2\text{O}_7$ , which is soluble in water with a blue colour, but no fluorescence, and does not dye wool or mordants. On investigating other æsculetin compounds like methyl-æsculetin, æsculetin- $\beta$ -carboxylic acid, and its esters, the authors find that like æsculetin itself, no dyestuff is formed by the action of ammonia. This reaction only takes place with the hydro-derivatives of these compounds obtained by the action of bisulphite or of nascent hydrogen as in the case of the dihydro-æsculetin sulphonic acid or of dihydro-æsculetin.—T. A. L.

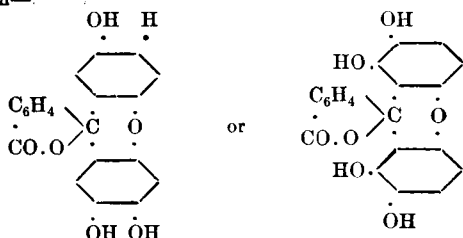
*Dihydroxyfluorescein.* C. Liebermann. Ber. 1901, 34, [11], 2299—2303.

SOME years ago the author, with the object of obtaining fluoresceins containing *o*-hydroxyl groups, and with a view of examining their behaviour towards mordants, prepared fluoresceins from hemipic and norhemipic acids. That from the latter acid has the formula—

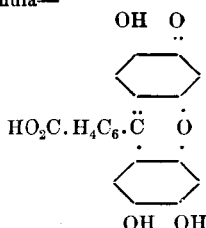


and gives golden-yellow shades on alumina and dark brown on iron mordants, the corresponding eosine dyeing alumina mordants pink. The shades, however, were of no particular value, and as norhemipic acid was not obtainable in any appreciable quantity, the research was abandoned. More recently Thiele (Ber. 31, 1247) has prepared oxyhydro-

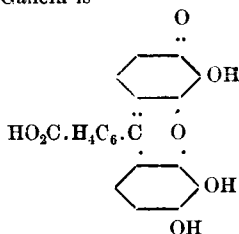
quinone, and this product on condensation with phthalic anhydride, gives a dihydroxyfluorescein having the constitution—



or the corresponding quinonoid formula. The condensation with phthalic anhydride takes place readily, as in the case of ordinary fluorescein, a good yield being obtained. The excess of phthalic anhydride is removed by boiling with water, and the product is recrystallised from dilute alcohol, forming microscopic greenish-golden shining plates. It dissolves in alcohol with a strong yellowish-green fluorescence and in alkalis with a cherry-red non-fluorescent colour. The solution in concentrated sulphuric acid is pure canary-yellow. This reaction and the fluorescent alcoholic solution show that the product obtained is not purpurin. On treatment with acetic anhydride and sodium acetate, the substance readily yields the tetra-acetate having the formula C<sub>20</sub>H<sub>8</sub>(OC<sub>2</sub>H<sub>3</sub>O)<sub>4</sub>, which crystallises in small faintly-yellowish prisms, melting with decomposition at 264° C. Dihydroxyfluorescein can also be produced from oxyhydroquinone triacetate (Thiele, *loc. cit.*) by heating 10 parts by weight of the triacetate, 7 parts of phthalic anhydride, and 4 parts of concentrated sulphuric acid for a few minutes to 145° C., but is somewhat difficult to purify when obtained by this process. The product dyes unmordanted wool and silk orange shades. On an alumina mordant it gives a bright orange, on iron a bright violet, and on chrome a rose-red shade. All these shades are fast to soap and do not fluoresce. The affinity of dihydroxyfluorescein for mordants is very marked, the fabric being dyed even in the cold, but preferably at about 50°–60° C. The same is the case with Gallein, and it therefore appears probable that dihydroxyfluorescein is represented by the quinonoid formula—



whilst that of Gallein is—

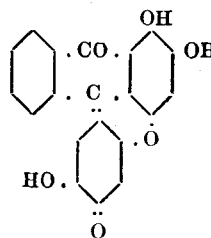


The latter gives reddish-violet shades on alumina and dark bluish-violet on iron mordants, due probably to the different arrangement of the hydroxyl groups, which may also account for the lack of fluorescence in this compound.

The action of bromine on dihydroxyfluorescein yields a dihydroxyeosin, which, however, according to analysis appears to be a dibromodihydroxyfluorescein. It crystallises from acetic acid in small greenish-golden prisms, whilst the colour of its solution in alkalis is not very different from that of dihydroxyfluorescein. The shades on mordants are redder, but the difference is not so great as might have been expected.—T. A. L.

*Dihydroxyfluorescein.* J. Thiele and C. Jaeger.  
Ber. 1901, 34, [12], 2617–2620.

THE authors refer to Liebermann's publication (see preceding abstract) on this subject. They obtain dihydroxyfluorescein in the same way, but purify it by extracting the crude melt with 30 per cent. sulphuric acid and washing with water. When suspended in four times its weight of glacial acetic acid and treated with four atomic proportions of bromine, it is converted into a dibromide, which dissolves in sodium acetate to a faintly-green fluorescing eosine solution, becoming rose-coloured, and non-fluorescent, on treatment with alkalis. On heating dihydroxyfluorescein with 7–8 times its weight of concentrated sulphuric acid for two hours to 120° C. and precipitation with water, a product is obtained to which the name Violein has been given. The substance dissolves with a violet colour to a certain extent in nitrobenzene, glacial acetic acid, and aniline, and gives indigo-blue salts with alkalis. It probably has the following constitution:—

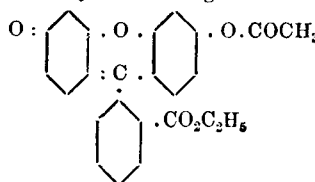


and is thus an anthraquinone derivative similar to Cœrulein. The tetra-acetate forms a dark brownish-violet powder, easily soluble, with a reddish-violet colour, in glacial acetic acid, acetone, acetic ester, and chloroform, but very slightly soluble in water.—T. A. L.

*Oxyhydroquinonephthalein; Fluorescein Esters.* W. Feuerstein, M. Dutoit and J. Wallach. Ber. 1901, 34, [12], 2637–2642.

SINCE oxyhydroquinone by Thiele's researches has become a tolerably readily obtainable substance, and in view of Kostanecki's mordant theory, according to which the introduction of two o-hydroxyl groups into any chromophore, confers mordant dyeing properties upon the substance, it appeared of interest to examine the action of phthalic anhydride on oxyhydroquinone. The authors confirm Liebermann's results (see preceding abstracts), and his conclusions as to the formula of the substance which they support by some new facts. They find that the dyestuff contains a carboxyl group, since by heating with alcohol and sulphuric acid it is converted into the ester which crystallises from alcohol in green plates melting at 326° C. The product differs from the original one in being quite insoluble, even in boiling water. By treatment with acetic anhydride and fused sodium acetate, the ester yields the tetracetyl compound which forms orange-yellow crystals melting at 239° C. The tetracetyloxyhydroquinone phthalein they find similar to Liebermann, crystallises from acetone, and melts at 264° C., whilst the tetrabenzoyl derivative separates from a mixture of benzene and light petroleum spirit in large crystals.

*Fluorescein Ester.*—Contrary to what has generally been assumed, it is found that fluorescein, when heated with twice its weight of absolute alcohol and one and a half times its weight of concentrated sulphuric acid, is converted into its ethyl ester. This product melts at 242° C., is insoluble in sodium bicarbonate, and when boiled with dilute soda lye yields ethyl alcohol and fluorescein. On boiling with acetic anhydride and sodium acetate, it yields acetylfluorescein ethyl ester having the formula—



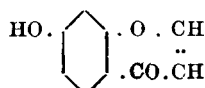
and crystallising from acetone in orange-coloured needles melting at 191° C. Fluorescein methyl ester melts at 253° C., and forms shining green crystals with a reddish reflex, very sparingly soluble in the usual solvents.—T. A. L.

**Malachite Green; Hydroxy - Derivatives of —.**  
E. Votoček and J. Jelinek. Chem.-Zeit. Rep. 1901, 25, [76], 274.

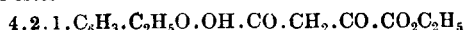
The authors find that in the condensation of carbinols, the phenol hydroxyl does not always enter the para position as would be expected, but also the *m*- and *o*-positions. They have condensed tetramethyldiaminodiphenylcarbinol with  $\beta$ -naphthol, phenetol, anisol, guaiacol, and resorcinol methyl ether and dimethylaniline with anisaldehyde *p*-ethoxybenzaldehyde, protocatechualdehyde, piperonal,  $\beta$ -resorcylaldehyde, and  $\beta$ -naphthaldehyde. The introduction of more than one hydroxyl can change the colour of the parent substance Malachite Green to violet. This effect depends upon two factors, the mutual position of the hydroxyls and their position with regard to the methane carbon, and does not therefore follow any simple law. The substitution of the hydroxyl hydrogen by acetyl produces a regular change in the shade of the dyestuff from violet towards the green portion of the spectrum, whilst the replacement of the hydroxyl hydrogens by alkyl groups has scarcely any effect on the shade of the dyestuff. The thiophenols react with tetramethyldiaminodiphenylcarbinol in a different manner from the phenols, and in presence of phosphorus oxychloride, no triphenylmethane dyestuff is formed, but a substance containing sulphur which belongs probably to the aromatic pinacones.—T. A. L.

**3-Hydroxychromone; Synthesis of —.** St. v. Kostanecki, L. Paul and J. Tambor. Ber. 1901, 34, [12], 2475—2479.

It having been shown that the oxidation product of brasilin is 3-hydroxychromonol, the authors propose to continue their investigations in the chromone group with a view of subsequently synthesising brasilin and hæmatoxylin. The chromone derivatives hitherto synthesised have all contained alkyl groups in the  $\beta$ -position of the  $\gamma$ -pyrone ring, whereas the brasilin oxidation product is a derivative of chromone itself. The authors have succeeded in obtaining 3-hydroxychromone—



synthetically as follows :—The Resacetophenone ethyl ether condenses in presence of metallic sodium with oxalic diethyl ester to form 4-ethoxy-2-hydroxybenzoylpyruvic ethyl ester—

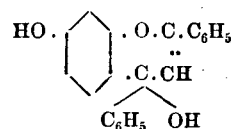


which crystallises from alcohol in well-formed white prisms, melting at 100° C. On cohobating the alcoholic solution with hydrochloric acid (sp. gr. 1.19) a voluminous precipitate is formed after some time, consisting of 3-ethoxychromone- $\beta$ -carboxylic acid. This product separates from alcohol in groups of prismatic needles, melting at 234° C., giving off carbon dioxide and yielding 3-ethoxychromone, which crystallises from dilute alcohol in long needles, melting at 121° C. This differs from the foregoing compound in its behaviour to concentrated sulphuric acid, in which it dissolves to a colorless solution with a violet-blue fluorescence, whereas the carboxylic acid gives a yellow non-fluorescent solution. On boiling the product with hydriodic acid (sp. gr. 1.9) for some time, the alkyl group is removed, and 3-hydroxychromone is formed. This substance crystallises from water in tufts of white needles, melting at 218° C. It dissolves in soda lye to a colorless non-fluorescent solution, and behaves to sulphuric acid like its ethyl ether.—T. A. L.

**1.4-Benzopyranol; Derivatives of —.** III. C. Bülow and W. von Sicherer. Ber. 1901, 34, [11], 2368—2385.

In continuation of previous work (see Bülow and Wagner, this Journal, 1901, 704 and 799), and with a view of investigating the stability of the hydroxyl group of the

pyranol ring, the authors have condensed dibenzoylmethane, the aromatic "homologue" of acetylacetone, with resorcinol in glacial acetic acid by means of gaseous hydrochloric acid, and have obtained 2.4-diphenyl-7-hydroxy-[1.4-benzopyranol] having the formula—

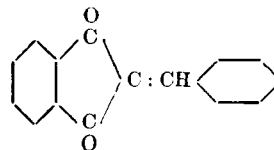


The hydroxyl, attached to the carbon atom 4 has carbinolic rather than phenolic properties, and on heating the product with hydrochloric acid under pressure, this hydroxyl is replaced by chlorine, which can be split off by suitable treatment with water. Moreover, a diacetyl or dibenzoyl compound can be readily obtained, or an acetylbenzoyl derivative. A dimethyl ether has also been prepared. Like the previously prepared pyranol derivatives, the new product gives a hydrochloric acid addition product, and the sulphate and picrate have also been obtained, together with the platinum and aurichloride salts. By the action of 15—25 per cent. caustic potash at a high temperature, 2.4-diphenyl-7-hydroxy-[1.4-benzopyranol] is decomposed quantitatively into acetophenone and benzo-resorcinol. Bromine in acetic acid solution gives a crystalline scarlet precipitate, dissolving to a non-fluorescent solution in concentrated sulphuric acid. Reducing agents convert 2.4-diphenyl-7-hydroxy-[1.4-benzopyranol] into 2.4-diphenyl-7-hydroxy [1.4-benzodihydropyran], which also gives a non-fluorescent solution in concentrated sulphuric acid. The pyranol derivative only contains the phenolic hydroxyl, which is readily identified by its behaviour to alkalis and capability of yielding an acetyl compound. The pyranol derivative gives, with sodium nitrite, a crystalline nitroso compound, in which the nitroso group enters the position 8. This product shows Liebermann's nitroso reaction.

—T. A. L.

**Indandion [Diketohydrindene]; Derivatives of —.**  
E. Noeltig and H. Blum. Ber. 34, [12], 2467—2471.

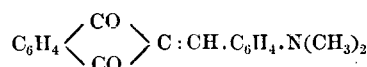
INDANDION [diketohydrindene] reacts readily with benzaldehyde (Kötze and Wislicenus, Annalen, 252, 72) to form benzal-indandion—



This substance has the properties of a chromogen according to Kostanecki (Ber. 1897, 30, 1183), who showed that a dihydroxy derivative (with the hydroxy groups in ortho position to each other) was a mordant dyestuff.

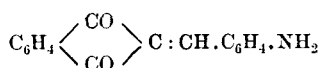
The authors have prepared amino derivatives of benzal-indandion, which prove to be weakly basic dyestuffs in accordance with Kostanecki's view of the parent substance.

**Dimethyl-*p*-aminobenzal-indandion—**



is prepared by boiling molecular quantities of indandion and dimethyl-para-amino-benzaldehyde in alcoholic solution, or by heating an intimate mixture of these substances to 150° C. in the oil-bath. The substance (m. pt. 99°) is sparingly soluble in alcohol, from which it crystallises in splendid steel-blue needles. It is more soluble in benzene (deep red crystals), but the best solvent is glacial acetic acid. The hydrochloride is stable only in the absence of air, and is immediately dissociated by water. Bright red shades are produced on tanned cotton, and also on wool and silk, from a weakly acid bath.

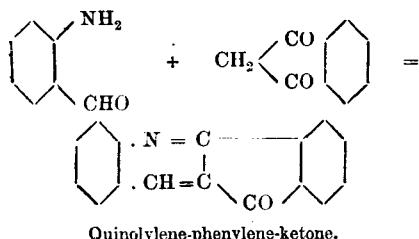


*p*-Aminobenzal-indandion—

*p*-Aminobenzaldehyde polymerises on standing and does not react in this condition. It can be depolymerised by dissolving in hydrochloric acid and reprecipitating by ammonia. The substance is now soluble in alcohol, and the solution reacts readily with indandion. *p*-Aminobenzal-indandion (lustrous blue leaflets from alcohol) decomposes at 247°. The solubility in all solvents is less than that of the dimethylated product. Tanned cotton, and also wool and silk, are dyed bright yellow shades.

3'-Nitro-4'-dimethylamino-benzal-indandion. — The reaction between indandion and nitrodimethyl-*p*-amino benzaldehyde is carried out as above. The yield, as in the preceding condensations, is nearly quantitative. Owing to the influence of the nitro group, the substance is not capable of forming salts, and therefore cannot be fixed on the fibre.

CONDENSATION OF *o*-AMINO BENZALDEHYDE WITH INDANDION. — Indandion reacts readily, not only with benzaldehyde, but also with aniline; and therefore with *o*-amino benzaldehyde, a double condensation takes place, two molecules of water being lost.

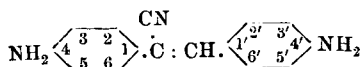


Quinolyene-phenylene-ketone may be described as a fluorene ketone in which a quinolyene nucleus replaces one benzene nucleus. The reaction is carried out by heating an intimate mixture of the two substances (in molecular proportions) on the water-bath. The yield is quantitative. The substance melts at 175° (from alcohol). Insoluble in water or alkalis, it dissolves in not too dilute acids. An oxime and phenyl-hydrazone are described.

Quinolyene-phenylene-methane is obtained by distilling the ketone with an excess of zinc dust and zinc oxide. M. pt. 166°—167° (white crystals from alcohol). When the body is heated with lead oxide a red substance is produced, which is doubtless analogous to *Bis*-diphenylenethane, the red hydrocarbon obtained by Graebe from fluorene.—H. L.

## 7-Cyano-stilbene; Isomeric Diamino Bases of —. M. Freund. Ber. 34, [12], 3104—3108.

THE author shows that 4.4'-diamino-7-cyano stilbene—



and the isomeric bases 3.3'-diamino-7-cyano stilbene and 4.3'-diamino-7-cyano-stilbene (just as *p*-diamino-stilbene or Benzidine) have the property of forming powerfully substantive tetrazo dyestuffs.

4.4'-dinitro-7-cyano-stilbene (silky yellow needles, m. pt. 215°) is prepared by the nitration of 7-cyano-stilbene (V. Meyer. Annalen, 250, 124). The yield is 60—70 per cent. of the weight of substance taken. The corresponding diamino base is obtained on reduction in the form of red-brown needles (from alcohol) which melt at 188°. When this is tetrazotised and combined with H-acid, a deep blue dyestuff is formed; with R-salt a violet-blue is produced.

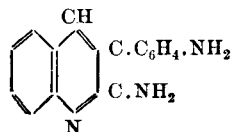
3.3'-dinitro-7-cyano-stilbene is the condensation product of *m*-nitrobenzyleyanide with *m*-nitrobenzaldehyde. It is a crystalline brown substance melting at 204°. The diamino body crystallises from alcohol in flat red needles, m. pt. 145°

—146°. The H-acid dyestuff dyes cotton a blue-red, the R-salt combination dyes a salmon-red shade.

4.3'-dinitro-7-cyano-stilbene has been described by Remse (Ber. 23, 3135). The diamino base obtained on reduction melts at 108°—110°. The dyestuffs obtained are intermediate in shade between those obtained from the two isomeric bases described above.

Pachorr has shown that the cyano-stilbene derivatives prepared from *o*-nitrobenzaldehyde are converted by a molecular transposition to derivatives of amidoquinoline (Ber. 29, 496).

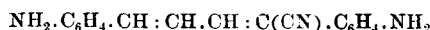
The author has obtained *py*-1-amino-*py*-2-aminophenyl-quinoline —



by the action of tin and hydrochloric acid on 4.2'-dinitro-7-cyano-stilbene. The latter substance melts at 184°, and is obtained by condensing *p*-nitrobenzyl-cyanide with *o*-nitrobenzaldehyde by means of sodium ethylate (Remse, loc. cit.). An intermediate body (not described by Remse) was obtained, viz., 2'.4-dinitro-7-hydroxy-7-cyano-diphenylethane—NO<sub>2</sub>.C<sub>6</sub>H<sub>4</sub>.CH(OH).CH(CN).C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>—melting at 138°. Concentrated sulphuric acid or sodium ethylate converts this substance into 2'.4-dinitro-7-cyano-stilbene. —H. L.

*Di-p*-aminophenyl-cyano-butadiën; Substantive Nature of Azo Dyes prepared from —. M. Freund. Ber. 34, [12], 3109.

*Di-p*-AMINOPHENYL-CYANO-BUTADIËN —

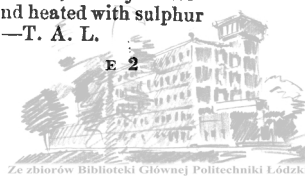


a base analogous to cyano-diamino-stilbene (see preceding abstract), but in which the two benzene rings are separated by a chain of four carbons, is likewise capable of forming substantive azo dyestuffs. *p*-Nitrocinnamic aldehyde condensed with *p*-nitrobenzyl cyanide yields *di-p*-nitrophenyl-cyanobutadiën. This substance (m. pt. 276° C.) crystallises in yellow needles, which are very sparingly soluble in the usual solvents. On reduction with sodium sulphide, *di-p*-aminophenyl-cyano-butadiën is obtained. The product is crystalline, of a yellow-brown colour, and melts at 196° C. When combined with H acid, it yields a fine blue dyestuff, which dyes unmordanted cotton well. The dyed materials are fast to light, to washing, and to acids.—H. L.

## PATENTS.

Colouring Matters [Black]; Production of New —. I. Levinstein, C. Mensching, and Levinstein, Ltd., Manchester. Eng. Pat. 18,756, Oct. 20, 1900.

THE azo dyestuffs obtained from *o*-nitrophenol and its homologues and diazo-nitranilines give black dyestuffs for cotton when treated with sulphur and sodium sulphide. For example, the azo product obtained in the usual manner from 13.8 kilos. of *p*-nitraniline and an alkaline solution of 13.9 kilos. of *o*-nitrophenol is mixed with 140 kilos. of sodium sulphide and 60 kilos. of sulphur, the whole being heated to 170°—180° C. for three hours, water being added occasionally to prevent the melt becoming dry. It is then heated for one hour to dryness at 180° C., and after grinding, may be at once used for dyeing purposes. The azo dyestuff may also be boiled with 126 kilos. of sodium sulphide and 46 kilos. of sulphur in 150 litres of water, either in an open vessel or under a reflux condenser, for about 24 hours. The reaction product is then made acid, or air is blown through the liquid, until the dyestuff is precipitated. After filtering, the product may be used for dyeing, together with sodium sulphide. The azo products may also be mixed with dinitrophenol, picric or picramic acid, or oxydinitro-diphenylamines or their homologues, and heated with sulphur and an alkaline sulphide as described.—T. A. L.



**Colouring Matters [Violet] belonging to the Diphenyl- $\alpha$ -naphthylmethane Series; Manufacture of Sulphonated** —. C. D. Abel, London. From the "Actien-gesellschaft für Anilinfabrikation," Berlin. Eng. Pat. 22,456, Dec. 10, 1900.

THE patentees sulphonate a diphenyl- $\alpha$ -naphthyl-methane derivative with fuming sulphuric acid at the ordinary temperature. For instance, 20 kilos. of Victoria Blue 4 R. (from tetramethyldiaminobenzophenone and methylphenyl- $\alpha$ -naphthylamine) are stirred into 80 kilos. of fuming sulphuric acid (30 per cent.  $\text{SO}_3$ ) at a temperature not exceeding 20° C. The melt is then heated for about one hour to 35° C. until a sample dissolves completely in dilute ammonia, when the whole is poured on to ice, redissolved in sodium carbonate solution and the sodium salt of the new sulphonic acid which separates is filtered off. It dyes wool a pure deep violet from an acid bath.—T. A. L.

**Nitro-, Azoxy-, Azo-, and Hydrazo-Compounds; Improved Process for Reducing** —. R. J. Urquhart, Manchester. From Weiler-ter-Meer, Urdingen a/Rhein, Prussia. Eng. Pat. 15,706, Aug. 3, 1901.

THE patentees employ iron and caustic soda as reducing agents, the quantities in the case of nitrobenzene, which can be successively reduced to azoxy-, azo-, or hydrazo-benzene or aniline, being as follows:—1,000 kilos. of nitrobenzene and 750 kilos. of iron are heated together in a boiler to about 90° C. 800 kilos. of caustic soda lye (55° B.) are then gradually added, the temperature being maintained at 100°–120° C., by steam or by cooling. When the smell of nitrobenzene has disappeared and the product solidifies at 31° C., the azoxybenzene may be drawn off. In order to produce azobenzene, further 250 kilos. of iron and 30 kilos. of caustic soda lye are added. The solidifying point of the product falls to 25° C. and subsequently rises to about 63° C. denoting the formation of azobenzene. A further quantity of 250 kilos. of iron and 300 kilos. of caustic soda solution yields hydrazobenzene the solidifying point of the product first falling to about 55° C. and afterwards rising to 122°–125° C. In this case the temperature is allowed to rise to 130° C. in order to prevent the product solidifying in the apparatus. In order to carry the reduction on to aniline, another 250 kilos. of iron and 300 kilos. of caustic soda lye are added and the mixture is further heated. The aniline formed is subsequently separated by distillation. All or any of these proportions may be altered as may also the temperatures, and examples are also given in which the reduction takes place in an indifferent medium such as a high-boiling hydrocarbon.—T. A. L.

**Marking Inks, for Marking Linen, Cotton, and Silk Fabrics, and the Like, and an Improved Vessel for Holding the Same; Impt. in Coloured** —. A. Raynes, London. Eng. Pat. 21,063, Nov. 21, 1900.

THE process described consists substantially in forming Aniline Black on the fibre.—E. B.

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

**Silk; The Fibroin of.** E. Fischer and A. Skita. Chem.-Zeit. Rep. 1901, 25, [76], 274.

THE only decomposition products of silk fibroin which have hitherto been identified are tyrosine, an aminopropionic acid and glycocoll. The authors now find that the tyrosine is identical with the *l*-tyrosine of casein and other proteids, whilst the aminopropionic acid is identical with *d*-alanine. In order to separate the other amino acids present in fibroin, recourse has been had to the esterification method. It is found that the composition of fibroin is more complicated than had been previously assumed, and when decomposed with hydrochloric acid, it yields, in addition to tyrosine, aminopropionic acid and glycocoll, *l*-leucin, *l*-phenylalanine, and some other amino acids. The separation of glycocoll from the amino acids richer in carbon, is

best effected by conversion into the esters and crystallisation of the glycocoll hydrochloride from alcohol. In order to determine the relation between the optically active form of alanine and of lactic acid, the authors have decomposed *d*-alanine with nitrous acid, obtaining *d*-lactic acid, so-called sarcosolactic acid. Hence the *d*-alanine corresponds in constitution to *d*-lactic acid. They obtained from 100 parts of fibroin, 10 parts of *l*-tyrosine, 21 parts of *d*-alanine, 36 parts of glycocoll, and about 1–1.5 parts each of *l*-leucine and *l*-phenylalanine, and are at present investigating the composition of silk gum, which, in contradistinction to fibroin, is rich in diamino acids.—T. A. L.

**Gelatin for Albumin in Calico Printing, Substitution of** —. F. Binder and C. Sunder. Bull. Soc. Ind. Mulhouse, 1901, 330–331. (Also Report on the above Paper. L. Baumann, *ibid.*, 329.)

THE following compounds render gelatin insoluble when printed in admixture with it and steamed for an hour: Sodium carbonate (4 per cent. of the weight of the gelatin), sodium acetate, aluminium hydrate, chromic hydrate, ferrous hydrate, ferrous carbonate, copper oxide, copper carbonate manganese oxide, magnesium carbonate, zinc tannate stannous hydrate, stannic hydrate, and lead acetate. An addition of vegetable or mineral oil, vaselin, &c., increase, the brightness and fastness of the prints. The copper, iron, and manganese compounds and zinc tannate give dull effects. Magnesium carbonate gradually thickens the printing mixtures, and finally coagulates them. The best results are obtained with zinc acetate. The colours made with this keep well and coagulate after 4 minutes' steaming. Reserve effects may be produced with advantage under Aniline Black by the use of the mixture of gelatin and zinc acetate, along with suitable pigments, in place of the usual mixtures containing albumin. A reserve thickening suitable for this purpose consists of 25 parts of glue-gum tragacanth paste (prepared by boiling for 6 hours equal parts of glue and gum tragacanth paste), 10 parts of zinc acetate, 15 parts of sodium acetate, and 20 parts of olein.

Anent the foregoing process, Baumann states that the colours obtained are very bright, and they cost less than the similar colours fixed by means of albumin. On the other hand, however, they are less fast than the latter.

—E. B.

**Chrome Mordants ("Half Discharges"), Action of Alkali Phosphates upon** —. F. Binder and C. Zundel. Bull. Soc. Ind. Mulhouse, 1901, 332–333. (Also Report upon the preceding by C. Schoen. *Ibid.* 331–332.)

WHEN a tissue padded with chromium sulphate-acetate is printed with thickened solutions of the ortho-, meta-, and pyrophosphates of soda respectively, and, after being steamed for a minute, is passed into a hot solution of sodium carbonate, an apple-green colour is obtained where the ortho-phosphate has been printed, the pyrophosphate giving a grey-purple, and the metaphosphate a dull green colour. On dyeing with Galloeyanine, the compounds thus produced attract the dyestuff considerably less than does the chromic hydrate in the ground of the tissue, "half discharge" effects resulting. The compound formed from the ortho salt dyes more than that produced from the meta salt, while that from the pyro salt is very slightly coloured. To apply the reaction in practice, the tissue is first padded in a solution of chromium sulphate-acetate and dried. It is then printed with citric acid to produce a white reserve, steamed 1–2 minutes at 100° C., printed with sodium pyrophosphate, again steamed, for a length of time depending upon the degree of "half discharge" desired, fixed in sodium carbonate solution, dyed with any suitable colouring matter, cleared with bleaching powder, soaped and finished.

Schoen who has examined the process, confirms the authors' statements.—E. B.

**Methylenesorcinol as a Mordant for Basic Dyestuffs.** A. M. Newjadomsky. Färber-Zeit. 1901, 12, [19], 293–294.

C. FAVRE has observed (this Journal, 1901, 710) that "methylenesorcinol," a compound produced by the action of formaldehyde on resorcinol, is capable of acting as a mordant for basic dyestuffs, being, it is stated, more





efficient in this respect than antimony tannate. The author finds that the mixture suggested by Favre for producing upon the cotton fibre the compound in question, deposits a precipitate after standing for 1—1½ hours and is, consequently, not suitable for use on the large scale. A mixture of 500 c.c. of a 0·2 per cent. solution of resorcinol, 10 c.c. of a 40 per cent. solution of formaldehyde, and 10 c.c. of a solution of sodium bisulphite at 21° B., remains clear for 2—3 days and gives satisfactory results when padded upon cotton tissues. To develop the mordanting compound, the tissues may be hung in a cold room for 60 hours, as Favre directs. It is more convenient in practice, however, to accomplish the development by a short steaming or to pass the tissues into boiling water. The dyeing of the mordant takes place in 20—30 minutes. The colours obtained are fuller than those produced upon an antimony tannate mordant. They are faster to soap but less fast to light than the latter. It is difficult to obtain discharge effects upon them. In printing, a mixture of 1 grm. of Methylene Blue, 20 grms. of acetic acid at 6° B., 1 grm. of resorcinol, and 10 c.c. of formaldehyde (40 per cent. solution) (the thickening agent is not mentioned), printed and steamed for 15—10 minutes, gives a bright blue, which possesses little fastness to soap and light. Prolonged steaming dulls the colour, a change which is also noticed with other dye-stuffs, for instance, Capri Blue and Torquoise Blue. The dyestuffs of the triphenylmethane series, along with Rhodamine and Thioflavine T, withstand an hour's steaming. The fastness of the lakes produced from these dyestuffs is also greater than that of other lakes of the kind. All the lakes are brighter when formed upon oil-prepared tissues. Additions of metallic salts, namely, tartar emetic and zinc chloride, increase the fastness of the lakes; thus, a mixture of 1 grm. of Methylene Blue, 15 grms. of acetic acid at 6° B., 200 grms. of gum solution, 1·5 grms. of resorcinol, 10 c.c. of formaldehyde (40 per cent.), and 10 grms. of zinc chloride, gives a lake which is slightly faster than that obtained as above described. Tartar emetic acts similarly and is, in general, a more suitable addition than the zinc salt, as it is without the dulling action upon the colour lakes of certain dyestuffs which the latter salt exhibits.—E. B.

*Silk; Method of Detecting certain Weighting Matters in* — A. Romann. Bull. Soc. Ind. Mulhouse, 1901, 328.

See under XXIII., page 1147.

#### PATENTS.

*Artificial Horsehair; Improved Process for Making* — F. Lehner, Zürich, Switzerland. Eng. Pat. 17,759, Oct. 6, 1900.

THE improvements claimed consist in passing a thread of "cotton, ramie, cellulose, viscose, nitrocellulose, artificial silk, or the like," or several such threads combined to the thickness of a single horsehair, through a solvent such as ammoniacal copper, zinc chloride, ether-alcohol, sulphuric acid. The separate fibres and threads are thus caused to soften and form a homogeneous thread. They are then drawn through suitable hardening liquids or exposed to the air, to prevent excessive action of the solvents and to preserve the form of the thread. The following is an example of the process for a thread composed of cellulose:—The dry thread is drawn slowly through a solution of "16 parts by weight" of freshly precipitated oxide of copper in 1 litre of ammonia (sp. gr. 0·91). After being freed from excess of the solvent by means of a pair of rollers through which it is drawn, it is passed slowly through a 5 per cent. solution of sulphuric acid, and it is then washed, wound on a reel, dried, and finally bleached in a dilute chlorine bath, and washed and dried. The threads obtained, as described, may be further treated with solutions of india-rubber or gelatin, or with collodion, to fill any defective places in them.—E. B.

*Yarn in the Form of Cops, Bobbins, or the like; Treating* — W. Coventry, Salford. Eng. Pat. 20,332, Nov. 12, 1900.

THIS apparatus is an improvement capable of being applied to the cop-dyeing machine previously described (this

Journal, 1899, 912). The modified dye vessel is fitted with a central vertical air-pipe surrounded by movable cop-bearing carrier, making joint with a sleeve fixed on the bottom of the vat. The space between the central air-pipe and the interior of the cop-carrier and sleeve forms an annular chamber through which the dyeing liquid circulates. The upper part of the central air-pipe is enlarged and fits the interior of the cop-carrier like a piston. As the carrier slides upwards over the enlarged upper end of the air-pipe, an air-valve fitted in the latter opens and places that portion of the carrier which is above the piston end in communication with the air-pump. The enlarged end of the central pillar also serves to cut off that portion of the carrier which is still in contact with the liquid from the action of the air, and this avoids any risk of drawing some of the solution into the air-pipe.—G. T. M.

*Dyeing Machines; Apparatus connected with Revolving Swift Hank* — A. B. Burrows, Liversedge, Yorkshire. Eng. Pat. 20,595, Nov. 15, 1900.

THE swift is lifted from the dye vat by means of a mechanism which stops automatically when the frame is raised to the required height. This result is attained by attaching, by means of brackets or hangers, the uppermost horizontal side of the swift frame to a horizontal rail, which can be moved in a vertical plane by means of chains fastened to each end, connected together by a hook, and attached to a winding-on boss or drum driven by worm gearing. The end of the movable rail nearer the driving gear carries a bracket through which passes a vertical rod having at its upper end an adjustable stop-nut. The lower end of the vertical rod is connected with one arm of a bell-crank lever, the latter operating a strap fork-rod, which shifts the driving belt from the fast to the loose pulley. The height to which the swift rises is determined by the position of the adjustable stop-nut on the vertical rod. As the bracket on the horizontal rail attains the required position, it lifts the stop-nut, thus causing the automatic stop-motion to come into operation.—G. T. M.

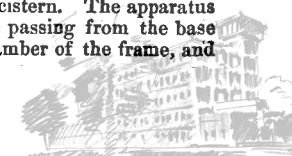
*Dyeing and otherwise Treating Yarn in "Cheese" and other Compact Form; Apparatus for* — C. Hartley, Seedley, Lancashire. Eng. Pat. 20,150, Nov. 9, 1900.

It is often extremely difficult, after a dyeing operation, to remove the ordinary perforated metal tubes from the paper tubes on which the yarn is wound in "cheese" or similar forms. To overcome this difficulty perforated metal tubes, having longitudinal slits, are employed, and a tapering ring or conical nozzle is inserted into the split end of the tube, causing it to expand and press against the paper tube. After the dyeing operation the ring or nozzle is removed, the tube then collapses and is readily withdrawn from the "cheese."

The perforated metal tubes carrying the "cheeses" are mounted on a horizontal plate, fitted with a series of hollow vertical pillars, and from the closed upper ends of each of these four radiating tubes proceed to the upper ends of the same number of "cheese" bearing tubes. The hollow pillars and the lower ends of the perforated tubes being placed in communication with the air-pipe, the suction action of the pump is simultaneously applied to both ends of the "cheese."—G. T. M.

*Dyeing and otherwise Treating Yarn in Cop and other Similar Compact Form; Apparatus for* — C. Hartley, Seedley, Lancashire. Eng. Pat. 20,158, Nov. 9, 1900.

THIS apparatus, consisting of a cistern fitted with fluid inlet and outlet, and having round its upper edge a channel communicating with the interior by means of a slot, contains a movable box or frame sliding vertically on the sides of the cistern and supporting the perforated cop-carrying spindles on hollow tables attached to the frame plates. These hollow tables have outlets leading into the inner chamber of the frame. When the frame is in position, its outlet is in contact with that of the cistern. The apparatus is also fitted with a vertical air-pipe passing from the base of the cistern, through the inner chamber of the frame, and



having at its upper end an air-chamber furnished with apertures corresponding with those of the hollow tables.

When the circulating pump is set in motion, the dyeing liquid passes successively through cops, spindles, hollow tables, frame-chamber and cistern outlet, and is then forced back into the channel round the upper edge of the cistern and thence into the cistern again.

After the dyeing operation the frame is slowly raised, and as the apertures of the hollow tables pass over those of the air-chamber, the cops are successively subjected to the suction action of the air-pump and drained of superfluous liquid.—G. T. M.

*Dyeing, Sizing or Slashing, Washing, and Similarly Treating Warps; Apparatus for* —. W. E. Heys, Manchester. From Adolphe Masseron, J. Pivert, F. Chaplet, E. J. Mueller and J. Caquelin, Paris. Eng. Pat. 14,619, July 18, 1901.

THE apparatus described consists of a system of rollers arranged to carry the whole or a portion of a warp through a series of dyeing becks, subsequently bringing the whole of the dyed and undyed warp into the sizing or slashing vats. Before passing to the slashing vat, the warp is subjected to the action of a hollow compensating roller, the function of which is take up any slackness of the warp and maintain uniformity of tension. Since the whole or only a part of the warp may pass under the compensating roller, it becomes necessary to control the amount of pressure exerted by the roller. Instead of employing several rollers, the required variation in weight is attained by the introduction or withdrawal of liquid through an opening in the hollow roller.—G. T. M.

*Power-Dyeing Machines used in Dyeing of Yarns and the Like* —. E. Preston, Kidderminster. Eng. Pat. 21,542. Nov. 28, 1901.

THE dye vat, made of sufficient length to accommodate one or more swifts placed in parallel, is deep enough to admit of the total immersion of the rotating frames in the dye liquor. An additional improvement is effected by closing the vat with aluminium or copper covers during the dyeing operation. The swift employed in this machine consists of a rectangular frame rotating about a horizontal axis by means of suitable gearing. Adjustable arms are arranged on the inner sides of the frame, the opposite arms being connected by rods or tubes carrying the hanks of yarn. The swifts are raised and lowered in the ordinary way.—G. T. M.

*Blue Shades Fast to Light; An Improved Process for Producing on Wool* —. H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 21,133, Nov. 22, 1900.

THE dyestuffs prepared by combining diazotised *p*-nitro-*o*-aminosalicylic acid ( $\text{OH}:\text{COOH}:\text{NO}_2:\text{NH}_2 = 1:2:4:6$ ) in an alkaline solution, with aminohydroxynaphthalene sulphonic acids containing the  $\text{NH}_2$  and  $\text{OH}$  groups in the peri-position, such as the 3-3' and 2-4 disulphonic and 4-monosulphonic acids of 1-1'-aminonaphthol, dye wool from an acid bath reddish shades, which do not resist light. By the action of copper salts these are turned from red to blue and are rendered fast to light.—E. B.

*Dyeing Patterns on Fabrics.* J. Bolland, Ashton-on-Mersey. Eng. Pat. 17,508, Oct. 3, 1900.

PORTIONS of textile fabrics or other materials are preserved from the action of dye liquors by enclosing them in soft metal forms before dyeing. After dyeing, the forms are removed and the materials are washed, &c., and finished as usual.—E. B.

*Wool; Rendering Incapable of Absorbing Dyestuffs.* O. Imray, London. From The Farbwerke vormals Meister, Lucius und Brüning, Hoechst a/Main, Germany. Eng. Pat. 19,668, Nov. 2, 1900.

WOOL is rendered incapable of absorbing dyestuffs of all classes except the basic class, by treating it with tannin substances in a hot bath and then with certain metallic salts; of these antimony and chromium salts being found to

be the most suitable. Aluminium and zinc salts have not sufficient fixing power to be of much use. Stannous salts may be used with advantage, especially when antimony and chromium compounds have been employed to fix the tannic acid, to strengthen the discharging action upon azo dyestuffs of the wool thus treated. Iron and copper salts are less applicable, owing to the dullness of the compounds which they yield. Titanium salts give a yellow compound which is not always advantageous. The following example illustrates the process:—100 kilos. of woollen yarn are boiled for an hour in about 3,000 litres of water with 25 kilos. of tannic acid and are then treated for half an hour in a separate bath with 15 kilos. of tartar emetic. The yarn is then rinsed and dried. The result is improved by adding to the rinsing bath 3 kilos. of sodium stannate along with the same amount of concentrated hydrochloric acid, and working for some time in this. The yarn thus treated is woven in appropriate designs with white, unprepared woollen yarn. Tissues are thus obtained which when they are dyed give two coloured effects, since the prepared wool absorbs acid and mordant dyestuffs less and basic dyestuffs more than the unprepared wool.

The effect may be varied by dyeing with two dyestuffs, for instance, an acid and a basic dyestuff, of different colours, or the wool, *e.g.*, in the loose state, before being prepared, may be dyed, for example, with Coerulein after being mordanted as usual with chrome, and after it has been prepared and spun with unprepared wool, the latter may be dyed with an acid dyestuff, for instance, Victoria Scarlet 3 R, which will not dye the prepared wool. A scarlet and olive coloured material is produced in the latter case.

For tissues, the process is especially valuable in printing. For example, a suitably thickened mixture, containing per litre 200 grms. of tannic acid and 100 grms. of acetic acid at 8° B., is printed upon a woollen tissue, and this is steamed for one hour and then treated for half an hour in a hot bath containing 5 per cent. (of the weight of the wool) of tartar emetic. After being well rinsed the tissue is dyed, various two-shade or two-colour effects being obtained according to the dyestuffs employed.—E. B.

*Printing Cotton with Sulphur Dyestuffs; New Process for* —. H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 19,670, Nov. 2, 1900.

THE commercial preparations of the sulphur dyestuffs (Vidal Black, Immedial Black, &c.), although they have acquired a high technical value in dyeing, have been employed only to a limited extent in calico printing. This is due to the fact that they contain inorganic sulphur compounds, such as sodium sulphide, which blacken the copper rollers used in printing. The present invention has for its object the preparation of mixtures suitable for printing, containing the organic sulphur compounds in a reduced state and free from injurious sulphur compounds. This is attained:—First, by dissolving the dyestuffs in water, precipitating them with common salt or acids, and treating with alkaline reducing agents the dyestuffs which have been thus purified. Secondly, by simultaneously precipitating the inorganic sulphuretted compounds and reducing the dyestuffs by means of suitable metals, such as zinc, along with caustic alkalis. Thirdly, by effecting the reduction of the dyestuffs by means of alkaline reducing mixtures, such as caustic soda and glucose, without metals, and then precipitating the hydrogen sulphide by the addition of suitable metallic salts, for instance, copper sulphate and "pyrolignite of iron." As an example of the second of these methods, a mixture of 15 grms. of Katigen Black S W, 20 c.c. of water, 10 grms. of zinc dust, and 18 grms. of caustic soda-lye at 48° B., is boiled for 2–3 minutes, until the reduction is completed, 35 grms. of gum tragacanth paste (65:1,000) and 2 grms. of glucose (to increase the stability of the printing mixture) are added, and the mixture, after cooling, is strained to remove the zinc sulphide which has been formed. The tissue printed with this mixture is dried, steamed for 10 minutes without pressure, rinsed, and soaped. A fast black colour is thus obtained.—E. B.



*Printing Textile Materials with Indigo.* B. Willeox, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen-on-Rhine, Germany. Eng. Pat. 22,402, Dec. 8, 1900.

Wool and silk, as well as cotton, are printed with Indigo without the aid of a strong alkali, by the use in the printing mixtures of a solid hydrosulphite, in particular, the very stable double salts, zinc sodium hydrosulphite and zinc calcium hydrosulphite. The reduction of the Indigo takes place readily on the printed materials in the presence of weakly alkaline substances, such as borax, magnesia, sodium silicate, carbonate, bicarbonate and phosphate, and soap.

The process is carried out either by mixing the Indigo with the hydrosulphite chosen and printing upon the material previously prepared with an alkali salt, or by directly printing a mixture of Indigo, hydrosulphite, and an alkaline substance, such as magnesia or sodium bicarbonate, which reacts only at a higher temperature. In either case the material, after printing, is steamed, when the reduction of the Indigo takes place and the Indigo White which is formed, is fixed upon the fibre. The materials are finally treated as usual to reproduce the Indigo Blue. For example, wool and silk, first padded with a 5 per cent. solution of borax, and cotton similarly treated with a solution of sodium silicate at 6° B., are printed with a mixture of 5—50 parts of "Indigo pure" in powder, 25—150 parts of zinc sodium hydrosulphite, 170—200 parts of water, and 800—6000 parts of gum thickening (1:1). The materials are next steamed 3—4 minutes in a Mather and Platt's steam-ageing machine, and are then washed in the usual way.

Wool and silk can be printed with Indigo by this process in shades ranging from a pale pearl-blue to a deep blue-black. The fastness of the colours is greater than has hitherto been achieved in printing, and the fibres are not weakened. As applied to cotton, the process offers the advantage that the Indigo can be printed without any exceptional care being taken in the steaming and other operations.—E. B.

*Carpet-cleaning Soap; Apparatus for Making* —. H. S. Jones. From S. Feder and J. van de Bücken. Eng. Pat. 14,085, July 10, 1901.

See under XII., page 1122.

*Fibrous and other Material [Yarn, &c.]; Drying of —, and Apparatus therefor.* F. Hiorth. Eng. Pat. 14,090, July 10, 1901.

See under I., page 1095.

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

*Electrolytic Printing.* Engineering, Aug. 16, 1901, 225.

By the method devised by Friese-Greene (this Journal, 1898, 841), the same presses and type can be used as heretofore, but, instead of inking the type, the latter is connected to the negative pole of a source of electricity. The paper is impregnated with suitable compounds, and the pressure rolls are connected to the positive pole. As the paper passes through the press, it is traversed by the current, and the impregnating material is decomposed, causing a sharp impression of the type to appear on the surface of the paper. The amount of impregnating material needed to give a good impression is stated to be small. It was originally proposed to use a mixture of manganese sulphate and nitrate of soda; this gives a dark brown impression, which tends to become blacker with age. It is stated, however, that means have been discovered for obtaining a perfectly black impression, sharper than can be obtained with ink. It is possible to produce about 36,000 impressions per hour, and to print on both sides of the paper. By using different impregnating materials, prints can be obtained in a greater variety of colours.—A. S.

*Leather Dyeing; Application of Titanium Salts for* —. M. C. Lamb. J. Soc. Dyers and Colorists, 1901, 17, [9], 214—216.

TITANIUM salts unite with the tannin matter of leather to form on the fibre a yellowish-brown titanium tannate, which is exceedingly fast to light and to the action of soap. Basic titanium oxalate, potassium titanium oxalate, and "tanno-potassium titanium oxalate" have been experimented with; they all produce a yellow-brown tint on the leather, and also fix any excess of tannin matter in the hide. Potassium titanium oxalate, on account of its cheapness, is the most useful. This salt is exceedingly soluble in hot water, and may be applied to tanned leather either with the brush or in the dye-bath. The colour obtained depends on the nature of the tannage, and also on the amount of salt used, the range being from a bright yellow-brown, resembling the shade produced on leather by "Phosphine," to a reddish yellow-brown, like that given by Indian Yellow R.

The basic colours especially give much fuller and richer shades with titanium than without.

*Application to Vegetable-tanned Leather.*—Dry crust skins are wetted down by steeping in water at about 45° C. and then washed in the drum for about half-an-hour at a similar temperature, to remove any large excess of tanning matter, and to distribute the remainder evenly through the skins. The goods are then worked in the drum or tray (preferably the former) with a solution of titanium salt for a period of 15 to 30 minutes at about 45° C., until the leather has become a good commercial shade of yellow-brown. They are then well washed in warm water and dried or topped with acid or basic dyestuffs as desired.

Calf skins should be well scoured with soda before treatment, whilst Persians and other foreign tannages should be scoured, soured, and retanned in sumach.

In order to obtain blacks, the leather must be first dyed with a neutral solution of logwood, and then passed through a bath of potassium titanium oxalate (2 lb. of oxalate to 50 galls. of water) at as high a temperature as the leather will safely stand (Procter).

When the leather has to be merely stained, the goods must be well scoured, set out, and allowed to "sam," then set out quite flat on the table, grain side up, and brushed from the middle with a 1 per cent. solution of titanium salt.

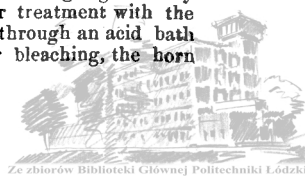
*Application to Chrome-tanned Leather.*—The leather, after tanning, either by the single or double bath method, is washed in borax solution to remove free acid, and then in water, and finally mordanted with a vegetable tannin substance. Three lb. of gambier and 1 lb. of sumach extract are dissolved in water and diluted with sufficient water at 50° C. to tan 100 lb. of leather (wet) in the drum or paddle. When a light yellow colour is produced on the goods, they are removed, washed, and placed in the titanium salt (2 to 3 lb. for each 50 galls. of water) in the trough. The tannin and titanium salt unite on the leather producing a yellow-brown tint. The goods are then washed, struck out, and fat-ligured.

*Application to Chamois Leather.*—A variety of shades may be obtained on this class of leathers by treating them with dilute alkali (mild) to remove oxidised oil, and then drumming with a solution of titanium salt (1 per cent.), and dyeing, also in the drum, at about 45° C. for half-an-hour without previous washing.

A list is given showing the colours obtainable with alizarin colours and natural dyestuffs.—R. I. J.

*Horn and Ivory; Dyeing and Bleaching* —. Textile Colorist, 1901, 23, [273], 285.

In order to produce light colours on horn it is necessary to bleach the horn previous to dyeing. The bleaching is performed by treating the horn in a bath made of red lead, quicklime, and potash lye, the quantities being regulated by the original colour of the horn. After treatment with the red lead solution, the horn is passed through an acid bath of about 20 per cent. strength. After bleaching, the horn



is dried in bran and polished with a fine linen cloth when it is ready to be dyed.

The dyeing is performed by steeping the horn in an alcoholic solution of a suitable aniline dyestuff. A yellow may be produced by steeping the horn in a warm solution of potassium chromate, until the desired shade is obtained.

Ivory can be bleached by exposing it immersed in water for three or four days, to the rays of the sun; a quicker process, however, is to immerse it alternately in a 4 per cent. solution of potassium permanganate and a 1 per cent. solution of oxalic acid; the ivory being allowed to remain half an hour in each solution, after which it is transferred to a hot mixture of bran, lime, and water, and is finally rubbed dry with sawdust.

Hydrogen peroxide may be used for bleaching; chlorine imparts a yellow tint to the ivory. Ivory is dyed in an exactly similar manner to horn.—M. C. L.

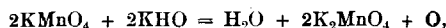
## VII.—ACIDS, ALKALIS, AND SALTS.

*Hydrochloric Acid Free from Sulphuric Acid; Preparation of* —. C. Scheuer, Linden, Hanover. Ger. Pat. 123,861, Dec. 5, 1900. Zeits. angew. Chem. 1901, **14**, [38], 954.

THE impure hydrochloric acid is run in a continuous stream into a boiling solution of magnesium chloride, boiling at 118° to 120° C. The hydrochloric acid distils over without loss, free from sulphuric acid and iron. When the magnesium chloride solution has taken up so much sulphate that the above-mentioned boiling point is no longer maintained, the solution can be used in the manufacture of magnesium sulphate.—C. A. M.

*Manganic Acid and Barium Manganates.* G. Kassner and H. Keller. Archiv, **239**, [7], 473—490.

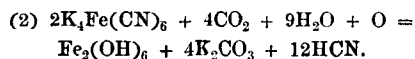
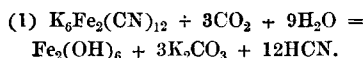
REVIEWING the work of Jolles, the authors find that the barium manganate obtained by precipitating potassium manganate with barium chloride in alkaline solution, is never pure, being contaminated with barium carbonate, oxides of manganese, and barium permanganate. Their results do not accord with the formula  $\text{BaMnO}_4 \cdot \text{H}_2\text{O}$ , since their salt only lost 2.97 per cent. in drying, as compared with the theoretical quantity, 6.57. They found that the reduction of barium manganate to manganite is best attained by means of hydrogen peroxide. When potassium ferrocyanide is employed for the purpose, the product is contaminated with iron; to remove this, prolonged washing, which results in dissociation, is necessary. The formula attributed to the salt  $\text{BaMnO}_4 \cdot \text{H}_2\text{O}$ , thus obtained, would appear to be correct, since after 12 weeks' drying it loses 6.8 per cent. in weight. They find that the theoretical quantities necessary for the preparation of potassium manganate do not agree quantitatively with the equation—



more potassium hydrate, equivalent to one and a half or twice the quantity thus indicated, being necessary to obtain the theoretical yield of manganate.—J. O. B.

*Potassium Ferro- and Ferri-Cyanides; Action of Carbon Dioxide on Aqueous Solutions of* —. J. Matuschek. Chem.-Zeit. 1901, **25**, [77], 815.

AT the ordinary temperature and away from the action of direct sunlight, carbon dioxide acts slowly on an aqueous solution of potassium ferrocyanide whilst a ferri-cyanide solution of equal iron content is unaffected. At 100° C. both solutions are attacked by carbon dioxide with formation of ferric hydroxide, potassium carbonate, and hydrogen cyanide, the reactions in the two cases being expressed by the equations:—



In both cases some of the hydrogen cyanide undergoes decomposition in the presence of potassium carbonate, with formation of ammonia and formic acid.—T. H. P.

*Alkalis and Acids; Micro-Chemical Detection of —. Ozone and Water; Detection of Small Quantities of* —. F. Emich.

See under XXIII., page 1142.

## PATENTS.

*Sulphuric Acid; Apparatus for Concentrating* —. C. A. Flanagan, Stretford, Lancaster. Eng. Pat. 3530, Feb. 19, 1901.

A SERIES of shallow open lead pans is set on brickwork, heated by the waste furnace gases, the pans communicating by pipes, and the acid being delivered from the last of the pans into the first of two or more stills by a siphon. The stills are of cast-iron, enamelled inside, each set in a separate furnace, at successively lower elevations. A conical lead hood is provided to each still, having on the outside a number of turned-up shelves, answering the purpose of troughs, the uppermost of which receives cold water, which successively overflows each trough, and is led away from the flange into which the hood is set. A siphoned lead draught pipe with a luted joint set in each hood carries the vapours into a main pipe leading to a condenser. The acid, as it is concentrated, passes by an overflow pipe from still to still until it reaches the lowest of the series, whence it is conducted to a cooling cistern, for siphoning into carboys. The condenser, receiving the fumes from the stills, is an open wood-lined cistern, containing a series of closed leaden boxes, connected, the first to the draught pipe, and then successively by siphons to the rest of the series, from the last of which the undensified gases pass to a chimney or water tower. The water of condensation is taken by a pipe from each closed vessel to a long trough connected to the lower end of the worm in the acid cooler, or to the drain.—E. S.

*Sulphuretted Hydrogen, and other By-Products from Alkali and other Works; Treatment or Utilization of* —. G. Wilton, Beckton, Essex. Eng. Pat. 15,468, July 30, 1901.

THE hydrogen sulphide is burned in a special furnace, and the sulphur dioxide is scrubbed in towers packed with limestone. Down these towers, waste liquor from the ammonia stills in the ammonium sulphate manufacture is caused to trickle; or this waste liquor may be used to absorb the acid independently of the limestone, to obtain an inoffensive effluent. Another process consists in passing the sulphur dioxide, with excess oxygen, through towers containing iron scrap, sprayed with water, or mother liquor derived from the crystallisation out of the ferrous sulphate produced in the process, tanks being provided to collect the effluent, which is concentrated so as to yield crystals. Apparatus is shown in which these processes are combined. The hydrogen sulphide is led into a brick-work furnace, immediately over burning coke on the firebars, and then passes into a second chamber, over refractory material below which a gas burner is set, the flames from which ensure combustion; baffle-plates are set above. Valved passages are provided for admission of air, which in this case is maintained in excess. The  $\text{SO}_2$  is taken from the furnace to near the top of a tower made of a lead casing, lined with acid-resisting brick, and supported by wooden framing. The first and second towers are charged with iron scrap, and the following tower or towers with limestone. The gas passes from the top to the bottom, and from the bottom to the top of each successive tower.—E. S.

*Bicarbonate of Soda; The Precipitation of* —. P. Naef. New York, U.S.A. Eng. Pat. 16,973, Sept. 24, 1900.

THE invention relates to the precipitation of sodium bicarbonate from ammoniacal solution of sodium chloride by carbon-dioxide, which may be derived partly from purified furnace gases, and partly from limekiln gases, the heat of such gases being first utilised in distilling ammonia from



the ammonium chloride solution of the ammonia-soda process. Instead of using furnace gases, producer gas may be first made, purified, burned for whatever purpose desired, and the gases of combustion be applied as described. The apparatus consists of two revolving cylinders, arranged horizontally, one above the other, the uppermost receiving the liquor to be treated, whence it flows through an external valved passage into the lower cylinder, and thence into a settling vessel, fitted with an agitator, and means for withdrawing the sodium bicarbonate deposited. Kiln gases pass into the lower cylinder in a contrary direction to the flow of liquor, and are led from the opposite end into the upper cylinder, into which also pass furnace gases, so led as to mix with the kiln gases: thus the liquor is constantly sprayed in an atmosphere of carbon dioxide. In another form of apparatus, a single horizontal revolving cylinder receives the liquor, which passes thence into a vertical column or tower after passage through a settling vat, and finally flows into another settling vat, both constructed similarly to that first described. The rich gases enter near the bottom of the tower, from the top of which they are led, after mixture with furnace gases, into the cylinder, this arrangement including the two systems of mixing the gas with the liquor, with, and without pressure. It is stated that thus the advantage is gained "that only a small part of the gas has to be pumped, and that against low pressure." Reference is made to Eng. Pats. 13,771 and 23,404, both of 1899 (this Journal, 1900, 1014 and 1901, 28, respectively). There are 37 claims.—E. S.

**Carbonate of Magnesia, Manufacture of** — W. Marsh, Manchester. Eng. Pat. 17,888, Oct. 9, 1900.

IN the manufacture of magnesium carbonate by the Paterson process (the treatment of burnt dolomite powder diffused in water with  $\text{CO}_2$  under pressure), the inventor adds 5 per cent. or more of vegetable or animal charcoal, or of petroleum oil, to the burnt dolomite diffused in water and passes in carbon dioxide under a pressure of about 160 lb. to the sq. in., with agitation. When magnesite is used, a small proportion of burnt dolomite or lime may be added to the liquid. The described process is stated to increase the yield of light magnesium carbonate from 2 per cent. to 5 per cent.—E. S.

**Alkali Cyanides; the Manufacture or Preparation of** — G. W. Johnson, London. From The Deutsche Gold und Silber Scheide-Anstalt vormals Rössler of Frankfurt-on-Main, Germany. Eng. Pat. 21,820, Dec. 1, 1900.

THIS is an improvement on the synthetic process for the production of alkali cyanides from an alkali metal, ammonia, and carbon, described in Eng. Pat. 21,732, 1894 (this Journal, 1895, 968). Sodium cyanide, for instance, is melted in a vessel built in a furnace, in which the heat can be efficiently regulated, and the calculated quantity of alkali metal and carbon to form an amide being added, at a temperature just sufficient to maintain fusion of the mass, ammonia gas is passed through the mixture. The alkali amide first formed combines with the cyanide to form a dialkali-cyanamide, with which the carbon combines to produce a cyanide. The temperature is somewhat raised towards the end of the operation. By "alkali metal" is understood to be included lead-sodium alloy, for instance. —E. S.

#### PATENTS.

**Electrolytic Apparatus. [Bleaching Liquor.]** M. Haas. Eng. Pat. 14,946, 1901.

See under XI. A., page 1120.

**Chlorine and Alkali; Electrolytic Apparatus for Producing** — J. Mactear. Eng. Pat. 16,874, 1901.

See under XI. A., page 1121.

**Alkaline Salt Solutions; Process and Apparatus for the Electrolytic Decomposition of** — A. J. de Brits e Cunha. Eng. Pat. 16,801, 1900.

See under XI. A., page 1121.

## VIII.—GLASS, POTTERY, ENAMELS.

**Lead Oxide; Volatility of** — M. Stoermer. Chem.-Zeit. 1901, 25, [77], 818.

THE author has made a number of experiments on the behaviour of lead glasses when heated, and from the results draws the following conclusions:—In the fritting of lead glasses a part of the lead oxide is lost, the loss being greater as the proportion of lead oxide in the glass increases. More lead is lost if the fritting takes place in a reducing fire. The ready-formed lead glasses do not lose in weight when again heated in an oxidising fire, but in a reducing (smoky) fire, the lead silicate is destroyed, lead being set free and vapourised. Lead silicate is hence not volatilised as such, but as metallic lead and during fritting as lead oxide. In the preparation of lead glazes, a smoky fire must, therefore, be carefully avoided, since it results in the volatilisation of part of the flux in the form of lead, the glaze being thereby destroyed, and rendered more difficultly fusible. The glaze will then not become bright at the same temperature, as if it were burnt in an oxidising fire.

—T. H. P.

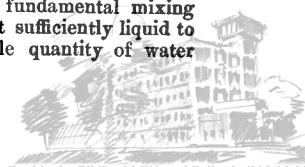
**Enamelling.** Engineer, 1901, 92, [2382], 194; [2384], 238; [2385], 264; [2387], 323; [2388], 347.

A DETAILED description of the manufacture of enamelled ware is given, with diagrams of the apparatus employed. In particular, attention is drawn to the decadence of enamelling in England, and it is shown how, on the other hand, the industry has undergone an enormous development in Austria and Germany. The enamelling industry includes:—(1) Hollow-ware enamelling for domestic use; (2) hollow-ware enamelling for chemical use; (3) enamelling locomotive and other tubes; (4) enamelling drain- and water-pipes; (5) signboard enamelling.

All enamelled ware first receives a fundamental or "grey" coating of a composition chiefly composed of glass. This is followed by a coating of "white" or glaze, any additional colour required being laid on above the white.

In signboard enamelling, the iron plates, after levelling or setting, are dipped into a hydrochloric acid (1:1) bath, then heated to redness to loosen the scale, cleaned in dilute sulphuric acid, rinsed in cold water, thoroughly scoured with fine sand, rinsed in boiling water, and allowed to dry. With hollow-ware, the hydrochloric acid bath may be omitted. The enamelling process should immediately follow the drying. The enamel mixings are applied to the metal either in the form of a powder or of a liquid paste, the latter method being the one more generally in use at the present time. When the enamel is applied as a powder, the metal is first rubbed with a cloth which has been dipped in a solution of gum, and the powder then carefully dusted through a sieve over the surface. When using the liquid paste, either the surface to be coated is dipped into the liquid, or the latter is carefully poured over it, any surplus being drained off, and any parts which are not to be coated, wiped clean with a cloth. The first or "grey" coating is dried at about 160° C., and then fused on in a suitable furnace (usually a muffle) at a cherry-red to white heat (about 1,200° C.). The object is then allowed to cool slowly and uniformly. The "white" coating which is next applied, should be made as liquid as possible, and should be fused on at a somewhat lower temperature (about 1,050° C.). The coloured coating is applied in a similar manner to the white.

In preparing the enamel mixings, the constituents are thoroughly ground and mixed, then fused together and re-ground. The fusing of the mixture is carried on in crucibles in suitable furnaces, and the melt is run into water, in order to render the material brittle and easy to grind. In grinding, it is essential that the construction of the mill be such as to prevent any iron-fitting from coming in contact with the material, as contamination with iron would cause discoloration of the enamel. The mixing of the powdered enamel constituents can be carried on in rotating barrels or churns. The "grey" or fundamental mixing should be kept in a condition only just sufficiently liquid to allow of being poured out, a suitable quantity of water



being added when the mixing is required for use. The "white" composition is mixed with water and kept in tubs until used; it should be carefully protected from dust.

Some of the more important points to be observed are:—The plates or other articles must first be thoroughly cleaned.

The "grey" coating must be "fused not only on but also into the metal" at a bright red—almost white—heat, and must fuse at a higher temperature than the "white" composition, so that it is not affected when the latter is applied. The compositions should have approximately the same capacities of expansion and contraction as the metals to which they are to be applied. After the application of the enamel coatings, slow and uniform cooling of the articles is essential. It is important both for the sake of economy and for obtaining good results, that experimental tests be made with each separate batch of enamel mixings.

The following are examples of the compositions employed.

For "grey" or fundamental coatings:—

(1)	Per Cent.	(2)	Per Cent.
Almost any kind of glass.	49	Glass (any kind).....	61
Oxide of lead.....	47	Red lead .....	22
Fused borax .....	4	Borax .....	16
		Nitre .....	1

(3)	Per Cent.	
Quartz.....	67.5	This is specially adapted for iron pipes.
Borax .....	29.5	
"Enamelling soda".....	3.0	

(4)	Per Cent.	
Frit of:—		Fused and then ground with— $\frac{1}{10}$ weight of silica frit. Clay, $\frac{1}{10}$ weight of silica frit. Magnesia, $\frac{1}{4}$ weight of white lead.
Silica powder .....	60	
Borax .....	33	
White lead .....	7	

(5)	Per Cent.	
Silica.....	65	This is a fair average of several mixings, and can be varied slightly to suit different conditions of work.
Borax .....	14	
Oxide of lead .....	4	
Clay.....	15	
Magnesia .....	2	

For "white" or glaze:—

(1)	Per Cent.	(2)	Per Cent.
Felspar .....	33	Cullet .....	20
Borax .....	22.5	Powdered flint.....	15
Quartz .....	16.5	Lead .....	52
Oxide of tin.....	15	Soda.....	4.5
Soda.....	8	Arsenic .....	4.5
Fluorspar.....	3.75	Nitre .....	4
Saltpetre .....	2.25		

(3)	Per Cent.	
Frit of:—		This should be ground up with the following:—
Silica powder.....	30	
Oxide of tin.....	18	
Borax.....	17	
Soda.....	8.5	
Nitre.....	7.5	
White lead.....	5.5	
Ammonium carbonate .....	5.5	
Magnesia.....	4	
Silica powder .....	4	

(4)	Per Cent.	
Frit of:—		This should be ground up with the following:—
Silica powder.....	26	
Oxide of tin.....	21	
Borax .....	20	
Soda.....	10.25	
Nitre .....	7	
Ammonium carbonate .....	5	
Magnesia .....	3.25	

(5)	Per Cent.	(6)	Per Cent.
Felspar .....	41	Borax.....	30
Borax .....	35	Felspar.....	22
Oxide of tin.....	17	Silica powder .....	17.5
Nitre.....	7	Oxide of tin.....	15
		Soda.....	13.5
		Nitre .....	2

(7)

Per Cent.	To this must be added, whilst being ground, the following percentages of the weight of the frit:—	Per Cent.
Borax..... 28	}	Silica powder ..... 18
Oxide of tin ..... 19.5		Borax ..... 9
Cullet ..... 18		Magnesia ..... 5.25
Silica powder ..... 17.5		Boric acid ..... 1.5
Nitre ..... 9.5		
Magnesia ..... 5		
Clay ..... 2.5		

(8)		
	Per Cent.	
Borax.....	26.75	To this should be added, whilst being ground, the following percentages of the weight of the frit:—
Cullet .....	19	
Silica powder .....	18.5	
Oxide of tin.....	19	
Nitre .....	9.25	
Magnesia .....	4.5	Per Cent.
Soda.....	3	Silica powder .....
		Magnesia .....
		Soda.....

Silica powder .....	1.75
Magnesia .....	1.75
Soda.....	1

No. 1 is for pipes, Nos. 2 and 3 for sign tablets, and Nos. 4—8 for culinary utensils. For colouring enamel frits the following are chiefly used:—Blue, silicate of cobalt or smalt; violet, peroxide of manganese; green, oxide of copper, with, if necessary, an addition of oxide of iron; yellow, Naples yellow; orange, red oxide of iron and an antimony preparation mixed so as to give the desired tint; red, sulphate of iron and alumina; brown, mixture of black and purple, oxide of iron mixed with a certain proportion of clay; black, oxides of iron, cobalt, and manganese.

—A. S.

*Silicate Analysis [Clays].* M. Stoermer.

See under XXIII., page 1143.

## PATENT.

*Molten Glass; Separating —, from Impurities.* J. M. Garrison, Belle Vernon, Pennsylvania. Eng. Pat. 12,933, June 25, 1901.

THE separator is a vessel closed at the bottom, and open at the top, and has a short elbow-shaped spout about half-way down. The inlet of this is contracted, and the lower end, opening into the vessel, is expanded. The separator is immersed till the spout inlet is below the surface of the molten glass, and it is stated that pure glass flows into the vessel, and impurities are left floating on the surface of the molten glass outside.—W. C. H.

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

*Puzzuolana; New Uses of —.* E. Leduc. Monit. Scient. 1901, 15, [718], 633—635.

THE author mentions that recently notices have appeared of the use of puzzuolana in conjunction with Portland cement, and gives an outline of the reasons that have led to the revival of its use. It is pointed out that the walls of the empty spaces left in a block of hardened cement, are coated with crystals of hydrate of lime, since the main cementing constituent, the silicate  $\text{SiO}_2 \cdot 3\text{CaO}$ , breaks down into hydrate of lime, and a less basic silicate. Determinations of this free lime vary with the process used, e.g., a strong solution of methylene iodide, or by means of sugar; probably 25 per cent. is about the true amount. Since good results are given by "fat" lime mortars and puzzuolana, it is probable that when puzzuolana is present, the lime set free during the hardening of cement, forms a new "puzzuolana cement." In the case of sea-water, the magnesium sulphate reacts with the lime, forming sulphate of lime,





which forms sulpho-aluminates, with the somewhat unstable aluminates present in cement. Four methods are suggested of avoiding the disintegration of the cement owing to the bulky form of these sulpho-aluminate crystals:—

1. To make blocks so compact that water cannot penetrate them.
2. To carbonate the surfaces, and so obtain the same result as in No. 1.
3. To use cements containing no alumina.
4. To prevent the formation of calcium sulphate by fixing the lime in a stable combination. It is stated that mixtures of cement and puzzuolana yield this result, for a sugar solution takes up a larger amount of lime from plain cement than from a mixture of cement and puzzuolana.

Two materials are mentioned that might be used with cement, like puzzuolana—the slag from blast furnaces, and a soft greyish-yellow rock, rich in silica, found abundantly in the Ardennes (described by L. Cayeux in the memoirs of the Société Géologique du Nord).

A mixture of cement and puzzuolana is stated to be less strong at first than pure cement, owing to the slowness with which the lime and silica unite, when the mortar begins to set.

A table, compiled by M. Séret, is added, giving crushing strength of cement alone, and with admixtures of different puzzuolanas, trass, santorin earth, blast-furnace slag, &c.

—W. C. H.

*Quartz Schist in place of Fire-clay in Lime- and Cement-Furnace Practice.* Chem.-Zeit. Rep. 1901, 25, [70], 259. From Stahl u Eisen, 1901, 21, 898.

IN course of a discussion on a paper by E. Cramer, in which, as the result of laboratory experiments, the resistance of quartz schist to attack by lime in lime-burning was stated to be greater than that of fire-clay, it was contended that this statement was not in accordance with actual practical experience, and cases were instanced in which quartz stones had been attacked. Although, however, an artificial stone, consisting of a mixture of quartz and clay, may have an inferior resistance, especially mechanically, to fire-clay, the result may be different when the quarried natural stone, absolutely homogeneous and dense, is used in place of that in which the various constituents must expand irregularly. It is stated that in cement works it has been practically shown that whilst the best Kulmitz fire-clay was melted in from two to three months, Krummendorf quartz schist lasted from four to six months, and the quartzose sandstone blocks now used held for eight to 12 months.—W. G. M.

*Cement Manufacture; The Rotatory Process of* —. W. H. Stanger and B. Blount. Proc. Inst. Civil Eng. 1901, 145, [3], 3—31.

THE authors describe the processes of Ransome (this Journal, 1885, 327; 1888, 850), Stokes (this Journal, 1888, 568; 1889, 622), and Hurry and Seaman (this Journal, 1896, 199; 1899, 147) for the manufacture of cement by means of continuous kilns, and discuss fully the advantages of this type of process over ordinary English practice. They arrive at the following conclusions:—

1. The rotatory process is so notably superior to older processes, both in economy and in the quality of the product, that it is likely to supersede them.

2. The chief advantages of the system are the mechanical and continuous handling of the materials, from the crushers of raw material to the stock boxes containing finished cement, and the production of an artificially-matured cement, thus dispensing with the cumbersome process of aeration by turning over and storage. The difficulties met with in preparing a refractory lining for the kilns, have been satisfactorily solved and powdered coal has been successfully applied as fuel.—A. S.

*Cement; Detection of Powdered Slag in* —. W. Fresenius.

See under XXIII., page 1143.

## PATENTS.

*Limekilns.* W. Siepen, Horrem, Rhine Province, Germany. Eng. Pat. 10,841. May 25, 1901.

THE lower part of the kiln is funnel-shaped, half the outlet being blocked by a horizontal iron plate. One semicircular loop or arch, extending further into the outlet, is bolted to the upper, and another to the under, surface of the iron plate, and bars are pushed in to rest on the lower arch at the back, and on a cross-piece in front, forming a grid to prevent lime in the centre of the kiln falling through, while lime which has fallen at the back is cleared out.—W. C. H.

*Refractory Materials; Manufacture of* —. R. J. Friswell, and The British Uralite Company, Ltd. Eng. Pat. 21,496, Nov. 27, 1900.

IN the process for the deposition of silica in the interstices of asbestos and the like by the decomposition of an alkali silicate by carbon dioxide, described in Eng. Pat. 4628, 1898 (this Journal, 1899, 585), it is now stated that the reaction ceases before the CO<sub>2</sub> theoretically available in the sodium bicarbonate present is exhausted, thus necessitating frequent renewal of the solution. The materials to be rendered refractory, containing asbestos, silica, chalk, &c., saturated with sodium silicate, are immersed in a tank containing sodium bicarbonate solution, into which a continuous stream of carbon dioxide is passed. Agitation is maintained. As modified, and claimed, the process consists in exposing the articles saturated with sodium silicate and still moist, in an air-tight chamber into which cooled and washed furnace gases, or CO<sub>2</sub> from other sources, are passed, until absorption ceases.—E. S.

*Cement and other Materials; Apparatus for the Separation of the Flour or Finer Particles of* —. W. F. Goreham, Belvedere, Kent. Eng. Pat. 15,344, Aug. 29, 1900.

THE claims are for a fixed oblong trough narrowed towards the bottom, having one end higher than the other, for holding the material to be treated, with a slit or jets for delivering air under pressure into the lower part of the trough along its whole length, and a casing to conduct the air which rises from the trough into a chamber, where the light powdered material carried over by the air can settle, and a means for supplying powdered material to be treated at the higher end of the trough, and for discharging the coarser parts of the powdered material at the lower end.

—W. C. H.

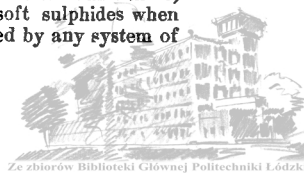
*Cement, Lime, and the like; Iron Shaft-Furnace for Burning* —. E. H. Stein, Hanover. Eng. Pat. 23,574, Dec. 24, 1900.

THE shaft-furnace is composed of an upper, or preparatory-heating, a middle or burning, and a lower, or cooling shaft. The whole is surrounded by a casing, through which air, for cooling the burning-shaft, passes; part of this air is said to support the combustion, and part to dry the material before being burnt. The burning and preparatory-heating shafts are separately carried by the outer casing, which transmits their weight to the base-plate direct. The several shaft portions are built up of rings, composed of segments, which are not fastened to each other, but to the outer casing by means of stay-bolts, the heads of which pass into slots formed by lugs on the outer surface of the segments. This arrangement allows of expansion of the segments, without the setting up of tension in the whole ring, and facilitates the removal and repair of damaged portions.—W. C. H.

## X.—METALLURGY.

*Concentrating [Ore] Systems.* Eng. and Mining J. 1901, 72, [11], 322.

FOR the separation of minerals according to their differences in specific gravity, the superiority of power jigs over other mechanical appliances, such as vanners or concentrating tables, is claimed. It is not only expensive to reduce the whole product of a mine to the state of fine sand and slime, but it is wasteful of value, because the soft sulphides when once ground to slime cannot be recovered by any system of



concentration. When, on the other hand, ores are crushed to, say, half-inch ( $12\frac{1}{2}$  mm.) size, the greater part of the sulphides are separated, whilst about 80 per cent. of the rock or quartz fragments are practically barren, and can be eliminated at this stage by running the material directly upon the coarse or rougher jigs; the pure coarse sulphides may be saved from further loss, and only the small proportion of mixed material requires to be re-crushed and re-jigged. Even when the mineral or sulphide is disseminated in such fine particles, that recourse must be had finally to table or buddle concentration, a large percentage of barren rock material can almost invariably be eliminated by jigging, before the ore-bearing material is reduced to sand and slime. As regards the choice of jigs, this does not lie so much between different styles, as between methods of use.

In the zinc-lead mining district surrounding Joplin, Mo., it is stated that the two-line or rougher and cleaner type of jig, supplemented sometimes with a line of sand-jigs, has held its own against all the other systems of jigging and concentrating tried. The crude ore from the mines rarely averages more than 2 or 3 per cent. of lead and 5—8 per cent. of zinc, usually in the hardest flint gangue, with no other elements of value than the lead and zinc. The products of the mills are, (1) nearly clean galena, averaging 76—83 per cent. of lead and rarely containing more than 2 per cent. of iron or zinc; (2) waste mundic or pyrite, containing 15—25 per cent. of zinc, practically all of which is recovered by magnetic separation; (3) clean blende, containing not more than 0.2 per cent. of lead and from 0.75 up to 10 or 15 per cent. of iron in the case of pyritic ores; (4) sand and flint tailings containing less than 0.2 per cent. of lead and generally not more than 1—2 per cent. of zinc; and (5) a small amount of slime or sludge, quite rich in zinc, which is treated in sludge-mills for the production of fine concentrates.—A. S.

#### *Magnetic Concentration; The Waring System of —.*

Eng. and Mining J. 1901, 72, [11], 328.

MAGNETIC separators may be classified under five distinct types:—

(1) the revolving cylinder or drum type (a) with magnetic fields created in peculiarly formed grooves extending annularly around the drum; (b) with the magnetic fields lying over or between pole pieces extending across the face of the drum. Machines of the type 1a produce an almost clean diamagnetic product at a single pass, but are not adapted for the classification of magnetic minerals.

(2) The double pole crossed or tangent belt type, including also a variety of belt machines.

(3) Machines in which the magnetic particles are deflected out of the course of a falling stream of material.

(4) Vertical single or multiple magnets revolving partly over a belt or pan carrying the material to be separated.

(5) Magnets oscillating from side to side directly across a flowing stream of material. Machines of this type are not so capable of producing absolutely clean diamagnetic products as those of type 1a, but they give excellent results as regards the paramagnetic products.

The Waring system of concentration and classification is a combination of types 1a and 5, and is stated to have given good results in practice. According to this system, the zinciferous or cupriferous concentrates are dried and passed through a screen of  $\frac{1}{4}$ -inch mesh into a mechanical calciner, where the pyritic minerals are partially desulphurised, and the ferric oxide, ferrous carbonate, copper minerals, &c., reduced to the magnetic condition. This operation is so carried out that the blende and other minerals which are subsequently to be separated by magnetic repulsion, are not affected; the heat generated by the burning sulphur of the pyritic constituents is usually sufficient to produce the condition required. The material is cooled and delivered to a magnetic separator of the drum type, falling into the intense magnetic fields lying within the peculiarly arranged grooves of the machine. The diamagnetic blende is expelled by magnetic repulsion aided by centrifugal force, whilst the paramagnetic particles, no matter how low their permeability may be, remain attached to the polar rings, whence they are released upon arriving at the opposite side of the axis.

The diamagnetic product requires no further treatment, as it is always quite free from iron-bearing or other magnetic minerals. The paramagnetic product, containing iron, copper ores, &c., as well as many garnet minerals, if such were present in the ore, may also contain particles of zinc blende not separated from the pyrites, &c. in crushing, and still adhering thereto. It is passed down an inclined plane in rapid flow under the magnetic classifier; the minerals are here separated according to their degree of magnetic permeability, the strongly magnetic pyrites first and the least magnetic minerals last into separate chutes on each side of the inclined plane. The last product, which is usually very small in amount, is either re-crushed and again run over the separator, or it is added at once to the appropriate class of finished material.

Two examples are given of the results obtained by this system of treatment:—

1. Ordinary pyriticiferous zinc ores from Galena, Kansas, averaging 44 per cent. of zinc and 15 per cent. of iron, and selling at 10 dols. per ton. These ores yield 64 per cent. of clean blende, containing 64.2 per cent. of zinc, absolutely free from pyrites, and selling at 28 dols. per ton; also 28 per cent. of burnt pyrites containing 49.5 per cent. of iron, and 2.8 per cent. of zinc.

2. Colorado mixed sulphide ore, containing 10 per cent. of lead, 14.6 per cent. of zinc, and 12.2 per cent. of iron, and worth 140 dols. per 100 tons, yields by wet concentration alone, products worth 697.57 dols., and by wet concentration combined with magnetic separation, products worth 1136.12 dols.—A. S.

*Iron; Passivity of —.* H. L. Heathcote. Zeits. Phys. Chem. 1901, 37, 368—373. Science Abstracts, 1901, 4, [9], 769.

THE author takes as evidence of passivity the fact that no action visible to the naked eye occurs on dipping the metal into nitric acid of sp. gr. 1.2 at 15°—17° C. Iron is in all cases rendered passive by nitric acid of sp. gr. 1.4 or 1.25, but only after some of the metal is dissolved. Electrolytically deposited iron (on copper) is rendered passive by 1.4 sp. gr. acid, but becomes active again after one or two minutes in acid of sp. gr. 1.2. Yellow, purple, or blue annealed iron is rendered passive by acid of sp. gr. 1.3, although no visible action takes place. In concentrated nitric acid, iron retains its passive state for a long time, but at the same time gradual solution of the metal takes place. Iron rendered passive by acid of sp. gr. 1.4 becomes active after about 20 hours' immersion in acid of sp. gr. 1.2, but the change takes place much more rapidly if the acid be kept in motion; for instance, if a stream of 1.2 sp. gr. acid be allowed to impinge on a passive iron rod immersed in acid of the same strength, the metal becomes active after one minute. Exactly the opposite is the case, however, when stronger acid (sp. gr. 1.275) is used. On immersion in other liquids the passive state persists for very different periods: in concentrated potassium nitrate solution, a few seconds; in twice normal caustic potash, 2—3 minutes; and in saturated solutions of potassium bichromate or ferric alum, several days. Iron can also retain its passive condition for some time, if kept *in vacuo*, after completely removing the acid. (See also this Journal, 1900, 1154.)

—A. S.

*Steel; Process for Hardening —.* O. Gentsch. Chem. Zeit. Rep. 1901, 25, [70], 260. Stahl u. Eisen, 1901, 21, 851.

THE first requirement for the satisfactory hardening of steel is the uniform heating of the tool. The author has constructed a furnace, with a coke fire beneath a chamotte plate, and provided with a good chimney. The firing of the furnace costs M. 1.5 for coke, or M. 10 for charcoal for a 10 hours' run. The furnace may be used for case hardening or for reheating. The tool to be hardened is painted over by means of a brush with a hardening composition, from which, on heating, the steel takes up a quantity of carbon corresponding to its structure, a fine-grained steel taking up less than a cheaper coarse-grained one, and the latter being markedly improved by the process. As soon as the tool is red hot, it is dipped into a water-bath contain-



ing whitening, then in rape oil, and lastly in mineral oil. After the first bath the tool is very hard; tempering is effected not, as hitherto, by heating from without, but—in the second bath—by the heat remaining within the tool. Hardening cracks are avoided. Experiments made by direction of the Minister of Railways with tools made from Böhler steel, and with others made from Bismarck steel hardened by the above process, showed, after three weeks, a difference of 25–30 per cent. in favour of the latter.

—W. G. M.

*Steel; Compression of* —. A. W. Zdanowicz. Chem.-Zeit. Rep. 1901 25, [70], 260. From Stahl u. Eisen, 1901, 21, 857.

In the Whitworth process for compressing fluid ingots, the pressure is so applied that only the solidified shell of the ingot is compressed, and the fluid metal within remains unaffected. In a process introduced by Beutter in the steel works of St. Etienne, pressure is applied to the ingot from below, and there is no cover to the conical chill, so that the effect of the pressure is to force the ingot further upwards, with the result that the tapered sides of the mould bring lateral pressure to bear upon the fluid metal within, cavities are prevented, and the metal is forced into the head. By this process (which is illustrated by drawings in the original) superficial fissures are prevented, the scrap is reduced to 4 per cent., porosity and internal strains are prevented, liquation is reduced, and the molecular condition and mechanical properties are improved. The reduction in waste causes a saving of 25 per cent. of the cost of the raw steel, but from this must be deducted the cost of compression, which amounts to about M. 8 per ton. A pressure of 500 kilos. per sq. cm. [3·17 tons per sq. in.] is required if applied until cold, or of 250 kilos. per sq. cm. if the block be removed at a red heat. Plant has been provided at the St. Etienne Works to apply pressure to small crucible steel ingots and to larger ingots requiring 30,000 or 60,000 kilos. gross pressure (according to whether they are to be removed hot or cold).—W. G. M.

*Gold; Curious Occurrence of* —. B. H. Bennetts. Inst. Min. and Metall., Oct. 17, 1901.

A DEPOSIT of gold on a piece of wrought iron, originally used in a mine track, and which had lain in the gutter carrying off the mine water, has been found at the Mount Lyell Mine, Tasmania. On removing the precipitated copper, the gold was found deposited in places, especially near three bolt holes, in the form of bright yellow plates. It was firmly attached to the iron, and had probably been deposited by the electric action of the iron-copper couple from traces of chloride in the acid mine water. No gold, however, could be detected in the mine water.—A. W.

*Copper Ores; Dry and Wet Treatment of* —. C. C. Longridge. Trans. N. Eng. Inst. Min. and Mech. Engineers, 50, 1901, 88–122.

THIS paper is a general summary of the operations involved in the extraction of copper from its ores, and treats in a cursory manner of the subject under the following headings:—Reduction and concentration; calcination; methods of calcining copper-ores and mattes; chemistry of copper-ore calcination; calcination losses; chemistry of copper-smelting; methods of copper-smelting: (a) German or blast-furnace process, (b) English or reverberatory-furnace process, (c) Welsh process, (d) Anglo-German process, (e) Pyritic process, (f) converter or Bessemer process, (g) Special processes; refining of coarse copper by dry methods; slags for copper smelting; blast furnaces for copper smelting; remarks on blast furnaces; reverberatory furnaces for copper-smelting; remarks on reverberatory furnaces; wet extraction of copper.—A. W.

*Copper-Tin, Copper-Zinc, and Tin-Zinc Alloys; New Determinations of the Specific Gravities of* —. E. Maey. Zeits. physik. Chem. 38, 289–291. Chem. Centr. 1901, 2, [15], 841.

THE composition was calculated from the quantities of the pure metals used in the preparation of the alloys. The following results were obtained.

#### Copper-Tin Alloys.

Tin.	Sp. Gr.	Tin.	Sp. Gr.
Per Cent.		Per Cent.	
28	8·903	70·3	7·972
39·2	8·980	78·4	7·726
42	8·791	80	7·735
56	8·357	100	7·284
59	8·210		

#### Copper-Zinc Alloys.

Zinc.	Sp. Gr.	Zinc.	Sp. Gr.
Per Cent.		Per Cent.	
0	8·862	67·8	7·878
19·8	8·459	68·4	7·833
52·4	8·140	76·4	7·714
53·5	8·143	85·0	7·595
61·4	7·976	87·9	7·481
63·7	7·951	94·1	7·311
65·4	7·904	100	7·087
7·1	7·873		

#### Tin-Zinc Alloys.

Zinc.	Sp. Gr.	Zinc.	Sp. Gr.
Per Cent.		Per Cent.	
0	7·284	75	7·110
5	7·233	100	7·087
0	7·190		

—A. S.

*Aluminium; Crystallised Metallic Compounds of* —.

O. Brunck. Ber. 34, [12], 2733–2735.

By melting together aluminium and various metals, and either pouring out the liquid alloy after a solid crust had formed on the surface, or allowing to solidify completely and then dissolving out all that 2 per cent. hydrochloric acid would dissolve, the author has obtained well-crystallised alloys of aluminium with copper ( $\text{Cu}_4\text{Al}_9$ ), iron ( $\text{FeAl}_3$ ), nickel ( $\text{NiAl}_3$ ), cobalt ( $\text{Co}_3\text{Al}_{13}$ ), manganese ( $\text{Mn}_2\text{Al}_7$ ), and platinum ( $\text{Pt}_3\text{Al}_{10}$ ). Stronger hydrochloric acid readily dissolves them all, save the last, from which it dissolves out all the aluminium.—J. T. D.

*Sulphur in Iron and Steel; Determination of* —.

W. A. Noyes and L. L. Helmer.

See under XXIII., page 1143.

*Molybdenum; Electrolytic Determination of* —.

L. G. Kollock and E. F. Smith.

See under XXIII., page 1145.

*Cyanide Solutions containing Zinc; Testing* —.

L. M. Green.

See under XXIII., page 1144.

#### PATENTS.

*Pyritic Ores; Hot Blast Furnaces for Smelting* —, &c. F. L. Rondebush, London. From E. T. Bradford, Denver, U.S.A. Eng. Pat. 18,028, Oct. 10, 1900.

THE essential feature is that the blast is made to pass through a pipe which is coiled within the throat of the furnace. It thereby becomes heated before it reaches the tuyères.—J. H. C.

*Metals such as Lead, Zinc, Copper, Iron, Silver, and Gold; Treatment of Mixed Ore for the Separation of* —. E. Ferraris, Sardaigne, Italy. Eng. Pat. 12,349, June 17, 1901.

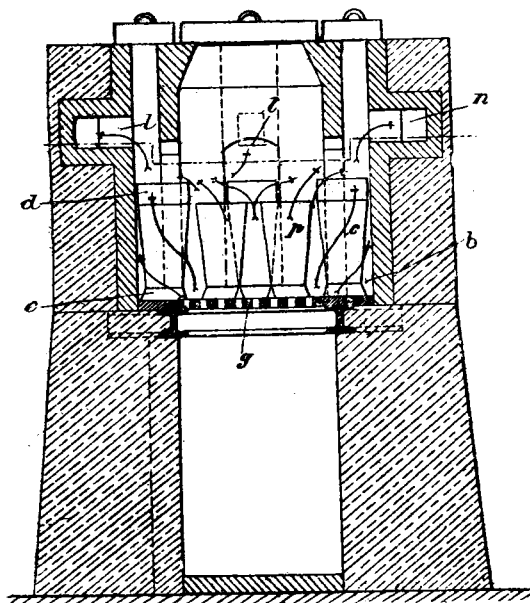
THE mixed sulphide ore is heated with concentrated sulphuric acid in a furnace, by which is produced metallic sulphates, sulphur dioxide, water, and sulphur, the last named being then burnt and the whole of the sulphur



dioxide converted into sulphuric acid. The sulphated ore is leached with water to dissolve the sulphates of copper, zinc, and part of the iron, the residue being then subjected to the usual process of lead smelting to recover the lead, silver, and gold. The copper and ferric iron are precipitated from the solution by zinc oxide and the copper further recovered as sulphate or metal. The ferrous iron is then precipitated by means of zinc oxide and compressed air, injected into the hot solution, the sulphate of zinc solution being concentrated by heat and air, and the salt crystallised. The solid zinc sulphate can then be converted into anhydrous oxide by heating with carbon, the sulphur dioxide thereby evolved being converted into sulphuric acid for re-use, whilst the oxide can be used or smelted as a high grade zinc ore, all these operations being claimed in parts.—A. W.

**Crucible Furnace; New or Improved** —. O. Michael, Freiburg, in Baden, Germany. Eng. Pat. 10,303, May 17, 1901.

THE furnace is built for the purpose of a preliminary heating of the pots or crucibles by the waste heat of the furnace, and is characterised by niches, *b*, in the walls of the shaft or furnace, the floors of which, *e*, are of the same level as the grating, *g*, carrying the melting pots or crucibles, *p*, so that metal difficult to fuse can be sufficiently heated within the crucibles, *c*, standing in the niches, by the waste gases passing into the branch flues, *l*, leading to the common flue, *n*, in preparation for the next smelting.



In this vertical section, the furnace is a horizontally rectangular one, and holds four central pots, *p*, surrounded with coke on the grating, *g*. In the centre of each of the four walls is cut a niche to hold one pot each, *c*, so that, while the four central pots are being finally heated, the four side pots with their contents are receiving a preliminary heating previous to taking the place of the central pots on the grating in the following heat.—A. W.

**Metals [Al, &c.]; Reduction of** —, and the Production of Alloys of the Same. H. S. Blackmore, Mount Vernon, U.S.A. Eng. Pat. 11,933, June 11, 1901.

THE process is for the reduction of metals, and consists in liquefying refractory metallic oxides at a temperature below their normal melting points by the action of an inert heated flux, then adding thereto a metallic carbide, the carbon of which is capable of reducing the oxide and liberating the combined metal, the bath being replenished with oxide and carbide from time to time as the metal is reduced. The details of procedure may be varied in accordance with the

21 claims, and the method is specially applicable to the production of aluminium and alloys of the same. The bath may consist of cryolite with added lithium fluoride, or of any inert salt or body which may be a haloid salt and free from hydrogen, and the metallic oxide may be added first and the metallic carbide after, or *vice versa*, the action between the two latter materials taking place at a temperature below the melting point of the oxide, and resulting in the combination of the two electro-negative constituents. Alloys may be prepared by using a carbide of a different metal to that of the oxide.

Aluminium is thus obtained by the action on its oxide of its carbide in a bath of cryolite, containing a fluoride to facilitate fusion and reduce specific gravity. The bath may be a metallic oxyhaloid, such as an oxyfluoride, and this itself may be heated with a metallic carbide, which will result in the abstraction of the oxygen and haloid element and the liberating and alloying of the metals. In this respect aluminium oxyfluoride and calcium carbide are heated either together or in a molten inert bath, the carbon combining with the oxygen, the calcium with the fluorine, and the aluminium being obtained as metal. Aluminium carbide may be produced in a molten bath of inert chemical salt, and the oxide added thereto.—A. W.

**Iron; Process of Melting** —, and Materials Employed therein. J. W. Maddison, and Wm. Rhodes, Wakefield. Eng. Pat. 16,484, Sept. 15, 1900.

A CUPOLA furnace is first charged with coke, then with a layer of wood, then a layer of mixed iron or steel, "scale" and "salt," and on the top of all, the charge of iron; this system of charging being repeated as often as desired. Commonly 8 lb. of "scale" and  $\frac{1}{2}$  lb. salt are used for each hundredweight of iron, with variations in the proportion of salt according to the size of the cupola.—J. H. C.

**Iron; Manufacture of** —. P. M. Justice, London. From B. Talbot, Pencoyd, Pennsylvania, U.S.A. Eng. Pat. 22,408, Dec. 8, 1900.

MOLTEN iron is mixed with lime or other oxidising material, or with puddling furnace slag, in a forehearth or refining furnace, provided with a basic lining; after which it is charged into the puddling furnace for working in the ordinary way.—J. H. C.

**Steel; Production of Highly Carburised** —. R. Dietrich, Sieg, Germany. Eng. Pat. 11,832, June 10, 1901.

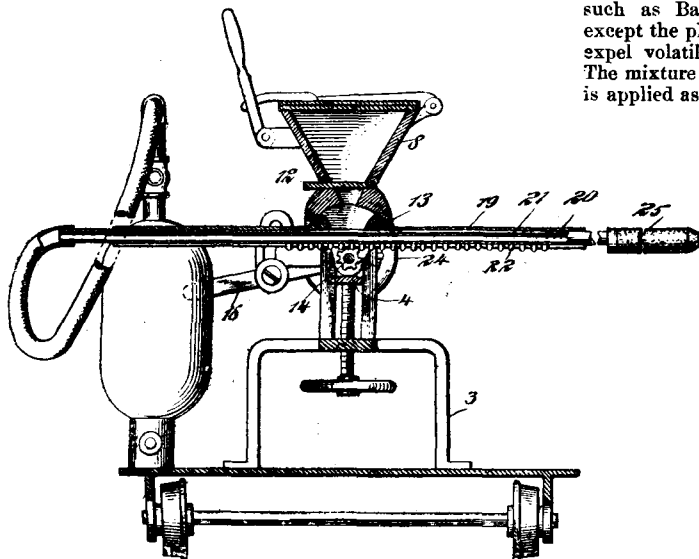
MELTED iron or low-carbon steel is poured in a thin stream into tar or other liquid or liquefiable carburising substance.—J. H. C.

**Iron or Steel; Machines for Use in Recarburising** —. J. W. Davis, Converse, U.S.A. Eng. Pat. 13,598, July 1, 1901.

THE machine for injecting carbon, carborundum or other powdered material into iron or steel is claimed as a whole or in parts according to the principles shown in the accompanying drawing. It consists of a platform, mounted on truck wheels, on which are arranged standards, 3, with a vertically moving frame, 4, which carries the feeding hopper, 8, and other working parts. The hopper has a top cover, closed by a locking lever, and in the lower portion a slide valve, 12. The lower end is curved in a segment of a circle to move against the curved block, 13, having an opening wider than the lower opening of the hopper. The shaft, 14, with bearings in the frame, 4, carries an extension of this block, whilst another extension is attached to a sleeve surrounding the shaft, to one of its ends and provided with a hand lever, 16, by which the sleeve may be rotated relatively to the shaft and the block consequently adjusted to move the discharging tube attached. Through the block, 13, is a transverse bore which is continued on either side by a guide tube, 19, registering with the bore. In this tube is the discharge tube, 20, provided at its upper side with a longitudinal slot, 21, through which the material can pass from the hopper. On its lower side is a rack, 22, projecting through a longitudinal slot in the bottom of the outer tube, 19. The rack is worked through a pinion by a hand wheel,



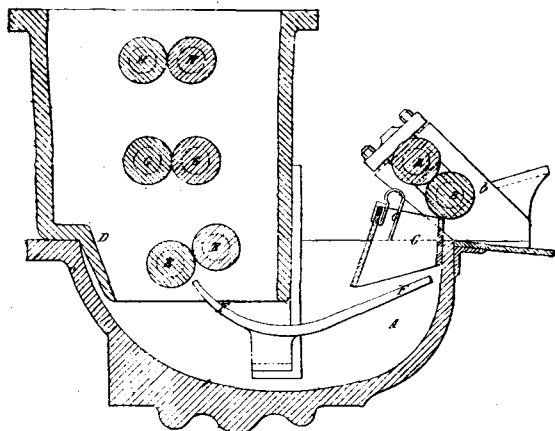
24, on the shaft. On one end of the tube, 20, is a fire-clay nozzle, 25, and on the other a flexible tube connected with a compressed air reservoir carried also on the base platform. The apparatus can thus be moved to the furnace,



the nozzle pushed forward through the opening, and the carbon or other material forced into the bath, by the air blowing through the central tube.—A. W.

**Steel and other Metal Plates or Sheets; Apparatus for Coating —, with Tin or other Metal.** W. P. Thomas, Abercarn. Eng. Pat. 18,844, Oct. 22, 1900.

THE apparatus comprises in one structure feed rollers or other devices, a flux box, a metal-coating bath or pot, and oil-bath or grease-pot, and one or more pairs of finishing rolls for the purpose of avoiding the use of more than one metal-coating bath, the pickled or prepared sheets being passed singly through the complete apparatus.



Two examples of the principle are given, of which the accompanying drawing represents the apparatus suitable for large sheets. It consists of feed rollers, B B, carried by brackets, b, at one end of the pot, A, a flux box, C, with guides, c, so placed that the sheets descend vertically through the centre of the box; guides, F, within the metal bath or pot, and one or more pairs of finishing rolls, E E, G G, and H H, in the grease-pot, D, preferably containing palm oil.

—A. W.

**Blackening for Foundry Purposes; Manufacture of —.** J. Dunn, Glasgow. Eng. Pat. 19,009, Oct. 24, 1900.

CLAIM is made for a preparation consisting of a mixture of carboniferous products, such as plumbago, gas-retort carbon, paraffin still coke, and the like, with an infusorial earth, such as Barbadoes tripolite. Each of the constituents, except the plumbago, is first calcined separately in order to expel volatile matters, and only the residues employed. The mixture is thoroughly ground into a fine powder which is applied as blackening in the usual manner.—C. A. M.

**Blast-Furnace Slag; Process of Treating Molten —.** A. D. Elbers, Hoboken, New Jersey, U.S.A. Eng. Pat. 8333, April 23, 1901.

THE slag is disintegrated by protracted cooling under diminished air-pressure by running it into kilns of special construction and there leaving it. The slag is said to be thus reduced, to such pulverulent and sufficiently soluble condition, as to form a cheap substitute for lime for manuring the soil.—J. H. C.

**Nickel; Preparation of —.** T. Michalowsky, Cracow, Austria. Eng. Pat. 17,569, Oct. 3, 1900.

POROUS nickel, suited for forming electrodes for electric accumulator batteries, is obtained by electrolytic precipitation from solutions containing iron, zinc, or other like metals.

—J. H. C.

**Aluminium; Preparing —, for Soldering.** C. P. Sørensen, Copenhagen. Eng. Pat. 16,864, Aug. 22, 1901.

THE parts to be soldered are heated to about 300° C., and then treated in a strong lye of soda or other alkali, which removes the oxide, after which they are washed in cold water, the soldering then being proceeded with in the usual way, without the necessity of a flux. The brazing alloys may consist of zinc and brass filings or zinc and tin filings.

—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (A).—ELECTRO-CHEMISTRY.

**Electro-motive Behaviour of Substances capable of different Stages of Oxidation.** R. Luther. Zeits. für Elektrochem. 1901, 7, [65], 897—899. From Zeits. phys. Chem. 1900, 34, 488—494; and 1901, 36, 384—404.

It is shown experimentally that with a substance which can have three stages of oxidation, the electro-motive force corresponding to the direct oxidation from the lowest to the highest stage is not the sum of the two electro-motive forces corresponding to the oxidation from the first to the second and from the second to the third stage respectively, but that the value lies between the two.

Incidentally it is shown that the difference in potential between a metal and its salt solution is independent of the anion. The second paper deals with the peculiar behaviour of chromium.—J. S.

**Electro-thermal Reactions and Syntheses.** W. Löb. Zeits. für Elektrochem. 7, [66], 903—921.

AN investigation on the behaviour of chloroform and carbon tetrachloride, when exposed to high temperatures produced by the electric current. The paper is divided into five parts, the first three of which deal with the historical, practical, and theoretical aspects of the subject, while the last two are devoted to the results obtained by the author with the two compounds named above. The apparatus used consisted of a decomposing flask, containing in its upper part the incandescent electric conductor, and of a series of absorption bottles, in which the gaseous products of the decomposition were absorbed.



by various reagents and retained for analysis. Nickel, iron, platinum, and platinum-iridium were used as materials for the incandescent spirals in the decomposing flask.

With regard to the absorption of the gases produced by the decomposition of chloroform, the author draws attention to the presence of chloroform vapour in the gaseous mixture, and to the error that would be caused by the liberation of carbon monoxide if caustic potash were used as the first absorbent. As cooling, even down to  $-18^{\circ}\text{C}.$ , failed to remove this constituent of the residual gases, the absorbing solutions were used in the following order:—Nordhausen sulphuric acid, phosphorus, acid cuprous chloride solution, caustic potash solution.

The results of the investigation may be summarised as follows:—

(1) The use of metallic conductors heated to incandescence by the electric current, affords a simple and accurate method for determining *qualitatively* the influence of temperature and the material of the conductor, upon the decomposition products obtained. Further experiments are, however, required to show how far the method can be applied to the *quantitative* determination of the energy absorption and reaction speed, of these decompositions.

(2) With regard to the thermal decomposition of chloroform, the results obtained indicate that an intermediate product—dichloromethane—is formed, but that the existence of this compound in the gaseous mixture is only of momentary duration.—J. B. C. K.

**Carbon; Formation of** —, in the *Electrolysis of Solutions of Ammonium Oxalate*. H. Verwer. Chem.-Zeit. 1901, 25, 792—793.

The many advantages of adding ammonium oxalate in the electrolytic analysis of a large number of metals has been demonstrated by Classen. Ammonium oxalate is particularly useful in the electrolysis of iron from the salts of organic acids, such as citrates and tartrates, in that the metallic precipitate can be obtained free from carbon. Heidenreich (Ber. 1896, 29, 1585) and others have shown that salts of citric and tartaric acid always deposit electrolytic iron associated with considerable quantities of carbon. Avery and Dales (this Journal, 1899, 300) have, on the other hand, stated that electrolytic iron separates from ammonium oxalate solutions contaminated with considerable quantities of carbon. The author (this Journal, 1899, 610) controverts this statement. He now, however, finds that under some conditions carbon is deposited with iron from solutions of ammonium oxalate, and after several experiments he asserts that the precipitated iron never contains the slightest trace of carbon if the current is stopped and the solution removed before all the iron is precipitated. If, after complete separation of the iron, the liquid be further electrolysed, whereby the precipitated iron acts as the cathode, it was always found to contain carbon.

The author next precipitated iron from a solution consisting of 10 parts of ammonium oxalate and 1 part of Mohr's salt, and removed the solution whilst it was still coloured yellow with iron. The precipitate contained no carbon. Using the precipitated iron as a cathode, a solution of ammonium carbonate and bicarbonate was electrolysed for several hours. Another portion of precipitated iron was used as a cathode to a solution of pure ammonium oxalate which had been electrolysed for 12 hours. In both experiments the iron contained carbon. Solutions of potassium and sodium carbonate behaved similarly, the precipitated iron containing carbon, though in a less degree. These results indicate that a reduction of carbonic oxide to carbon takes place in aqueous solutions. Tartrates behave quite differently; the precipitated iron always contains carbon. The author is able to confirm Avery and Dale's statement (*loc. cit.*) that with Moore's method (Chem. News, 1896, 53, 209), where a ferrous salt is electrolysed in the presence of ammonium metaphosphate, the precipitated iron is associated with a small quantity of carbon and a larger one of phosphorus.—J. L. B.

## PATENTS.

**Secondary Battery Plates; Manufacture of** —. B. Kuettnner, London. Eng. Pat. 17,490, Oct. 2, 1900.

LEAD oxide, with or without the addition of finely divided lead, is mixed with a solution of a soluble silicate, and formed into a paste which is made into a plate, and then dipped into a dilute alkaline solution, from which it is removed and placed in a second alkaline solution of greater strength until the hardening process is complete, after which it can be reduced or oxidised in the usual manner. The special form of grid consists of a thin lead plate or sheet, having an undercut rim on both sides equal in depth to the thickness of the plate. The grid is formed with tubular apertures which may project on both sides, or alternately on either side to the thickness of the plate, having their edges slightly bent over to form lips. The diameter of the apertures is about equal to their depth, and to the distance separating them, and the grid may be perforated with smaller holes between the tubes. In a modification of the grid, the tubular apertures are of half the length, and extend alternately on each side.

—G. H. R.

**Secondary Electric Batteries; Manufacture of Electrode Plates of** —. A. Nodon, Paris. Eng. Pat. 13,300, June 29, 1901. (U.I.C., Dec. 1, 1900.)

ACTIVE material for electrodes, especially anodes, is made by mixing minium or litharge with a solution of silicate of soda or potash, at  $30^{\circ}\text{B}.$  A thick paste is formed which is packed into grids or frames, of lead or lead alloy. These are exposed to air, and when dry and hardened are again immersed in the silicate solution. They are afterwards acted upon by a weak solution of sulphuric acid, with the formation of soluble sulphate, leaving insoluble lead silicate of brown colour which adheres strongly to the frames. The plates are water-washed, and are then ready for forming.

—J. C. R.

**Electrolytic Apparatus. [Bleaching Liquor.]** M. Haas, Aue, Saxony. Eng. Pat. 14,946, July 23, 1901.

THE electrolytic apparatus is an improvement on that described in Eng. Pats. 10,215 of 1899 and 9331 of 1900 (this Journal, 1899, 771, and 1900, 752), and consists of a vessel divided into several compartments by the electrodes and mounted in a storage receptacle, the only liquid connection between which and the several compartments consists in long narrow channels or pipes. Various modifications are described, in one of which long, narrow pipes are employed to carry off the liquid to a collecting vessel.

—G. H. R.

**Air, Gases, and Gaseous Mixtures; Apparatus [Ozonisers, &c.] for Electrically Treating** —. R. J. Yarnold, London. Eng. Pat. 19,393, Oct. 30, 1900.

To avoid loss of efficiency incidental to rise of temperature in ozonisers of the type described in Eng. Pat. 2629 of 1899 (this Journal, 1899, 923), it is proposed to employ hollow electrodes, through which a current or stream of water or other cooling medium may be continuously caused to flow. As a dielectric of high capacity for placing between the electrodes, compressed sheets of homogeneous compounds of mica and shellac are employed, preferably using the compound known as micanite or megomit.—J. C. R.

**Electric Furnaces for Dental and other Purposes.** R. Winter and V. Pappenheim, Berlin. Eng. Pat. 13,367, July 1, 1901.

A FURNACE for dental work is described. Use is made of a series of rods or lengths of refractory clay, mounted on the inside walls of the furnace casing, and provided with longitudinal holes for receiving heating wires of small cross section, whereby the wire, as well as the heating bodies surrounding them, on becoming incandescent, can expand independently of one another.—J. C. R.





**Electrolytic Cells.** J. H. Noble, Liverpool, and A. Merry, Liscard, Chester. Eng. Pat. 20,200, Nov. 9, 1900. (See Eng. Pat. 2372 of 1900; this Journal, 1901, 728.)

It is now proposed and claimed to (1) provide a head or pressure on the electrolyte in the porous (anode) cell considerably greater than that on the liquid surrounding the cathode cell, and proportional to the ampère used; (2) to avoid secondary action at the porous cell by louvers or a pervious inner cell open below, adapted to afford an easy and quick passage of the liberated gas to the top of the cell, but an obstructed one in the direction of the outer porous cell and cathode, preferably in the form of a truncated inverted cone; (3) to glaze the top of the porous cell so that chlorine, when chlorides are being decomposed, shall not pass through this portion near the closure; and (4) methods of feeding the electrolyte into the cell and drawing off the electrolytic products. Where brine is decomposed, it is proposed to utilise the caustic alkali produced by these, or mercury-using cells directly for soap-making in different ways.—J. C. R.

**Alkaline Salt Solutions; Process and Apparatus for the Electrolytic Decomposition of —.** [Intermediate Electrolyte.] A. J. De Brito e Cunha, Lisbon. Eng. Pat. 16,801, Sept. 20, 1900.

The porous diaphragms, which may be cylindrical or flat, are made of barium, or aluminium silicate, mixed with sodium silicate, sodium hydrate, brine, or any other suitable solution more or less concentrated according to the required porosity of the diaphragms. The mass is subjected for about 6 hours to 90 lb. pressure, and is afterwards moulded while hot, and hardens while cooling. Two diaphragms are employed, the intervening space containing an intermediate electrolyte designed to separate the alkaline solution from the original electrolyte, so as to avoid any recombination of the final products of electrolysis. Syphons are used for extracting the alumina, barium hydrate, and caustic alkali formed, and means are provided for the utilisation of the intermediate caustic alkali passed through by osmose action, and for producing efficiency of current and facility of putting out of circuit, changing diaphragms, and erecting the electrolytic cell.—G. H. R.

**Chlorine and Alkali; Electrolytic Apparatus for Production of —.** J. Mactear, London. Eng. Pat. 16,874, Aug. 22, 1901.

The cathode cell has in its bottom a central well in which is mounted a rotary propeller, and a space at the base of its wall communicates with the central well by ducts formed under the bottom. The anode cell is placed centrally in the cathode cell, the edges of its sides extending into a recess formed in the bottom of the latter, and it is supplied with pipes for supply, and for overflow of chloride solution, and for outflow of chlorine. The spindle of the propeller passes through the cover of the anode cell, and is supplied with a liquid seal, and a quantity of mercury fills the well, the space and the ducts forming a layer over the bottom of the cell.—G. H. R.

## (B.)—ELECTRO-METALLURGY.

### PATENTS.

**Aluminium and other Metals; Process for Obtaining [part Electrolytic].** G. Taddei, Turin, Italy. Eng. Pat. 13,379, July 1, 1901.

ALUMINIUM and other metals whose chlorides are less exothermic than that of sodium, are obtained by electrolysing sodium chloride at a high temperature (1,000° C. by a continuous current, at about 4.5 V.) in one vessel, and passing the chlorine into a second vessel heated to 1,300° to 1,500° C., where it forms the chloride of the metal to be obtained. The sodium is passed into a third vessel where it meets and decomposes the metallic chloride from the second vessel, and from which the sodium chloride formed is returned to the first vessel. The anhydrous chloride is obtained in the second vessel in the usual manner, by bringing the chlorine—in this case prepared electrolytically—into contact with an intimate mixture of

alumina and carbon (tar) at a high temperature. The chloride and sodium are brought together as vapour in a chamber, not separately heated.—J. C. R.

**Metals; Apparatus for Refining —, by Electrolysis.** H. H. Lake, London. From A. G. Betts, New York, U.S.A. Eng. Pat. 14,967, July 23, 1901.

THE electrodes are contained in an electrolytic vat, adjacent to which extends a track formed by rails on which a carriage travels to and fro, bearing a pair of pressure rollers in the path of the electrodes, and a frame capable of vertical, reciprocating movements on the carriage, and adapted to engage, raise, and lower the several electrodes between the successive movements of the carriage. The electrodes are severally provided on their upper ends with oppositely projecting lugs which fit on projections on the frame, which has screws and nuts, and is operated by an intermittent motor, while another, working alternately with it, moves the carriage along the track. A movable brake prevents movements of the carriage, and is released by the final descending movement of the frame, which also carries an automatic switch for reversing the motor at either end of the path of movement of the carriage.—G. H. R.

ERRATA.—This Journal, 1901, 727, col. 1, under XI. B., read references as follows:—"Elect. Rev. (N.Y.)," &c., and "Elect. World and Engineer," &c.

## XII.—FATS, OILS, AND SOAP.

**Mixed Glycerides in Cacao-Butter.** J. Klimont. Ber. 1901, 34, [12], 2636.

By means of fractional crystallisation from acetone, the author has separated cacao-butter into three parts:—(1) A fraction which after repeated crystallisation melted at 64° C., and had an iodine value of 0. Finally crystals melting at 70° C. were isolated from it. This fraction was judged to consist of a mixture of tripalmitin and tristearin.

The second fraction melted at 31°–32° C., and had a saponification value of 196.4 and an iodine value of 28.9, whilst its composition was  $C_{55}H_{104}O_6$ . It was concluded that it was a triglyceride containing palmitic, oleic, and stearic acid radicles.

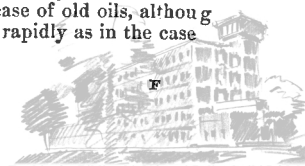
The third fraction consisted of a mixed glyceride which melted at 26°–27° C., and had an iodine value of 31.7, and a saponification value of 210.5. Its composition was  $C_{51}H_{95}O_6$ .

Triolein could not be detected in the sample of cacao-butter.—C. A. M.

**Sesamé Oil; Occurrence and Detection in Commercial Arachis Oil —.** P. Soltzien. Chem. Rev. Fett- u. Harz-Ind. 1901, 8, [10], 202–203.

THE author finds that Tambon's reagent (this Journal, 1901, 285) is not capable of detecting less than 2 per cent. of sesamé oil, and must therefore be regarded as much less sensitive than Baudouin's test. Referring to Tambon's statement that the fatty acids of arachis oil gave a red coloration with Baudouin's reagent, the author asserts that this must really have been due to the presence of small quantities of sesamé oil. All the commercial samples examined by him gave colorations corresponding to the presence of from 0.25 to 15 per cent. of that oil, whilst a specimen of arachis oil extracted by himself from the nuts gave absolutely no coloration. He was also informed by certain manufacturers that it was customary to add sesamé oil to the finest grades of arachis oil, with the object of influencing their behaviour towards cold and of improving their miscibility for salads.

The tin chloride test for the fatty acids of sesamé oil is, in the author's opinion, untrustworthy, since the reagent will give a brown coloration with the fatty acids of arachis oil, or even rose colorations in the case of old oils, although the reaction does not take place so rapidly as in the case of sesamé oil.—C. A. M.



**Walnut Oil; [Bulgarian] —.** N. Petkow. Zeits. Untersuch. Nahr. u. Genussm. 1901, 4, [18], 828.

Five samples of cold-pressed walnut oil gave the following results on analysis:—

—	1.	2.	3.	4.	5.	Average.
Sp. gr. at 15° C.	0.9260	0.9255	0.9260	0.9260	0.9256	0.9258
Refractometer number at 40° C.	67.0	67.5	63.0	68.0	68.0	67.7
Iodine value.	147.92	148.10	148.43	148.40	148.20	148.21
Acidity (Burstyn degrees)	3.17	4.26	5.97	5.25	3.25	4.38

—W. P. S.

**Cây-doc Oil of Tonkin.** Chamber of Comm. J., Nov. 1901, 258.

THE Annamites in the Province of Thái-Nguyên, in Tonkin, give the name of Cây-doc to a tree of the *Clusiaceæ* species, the oleaginous grains of which produce an oil for burning purposes, which causes an important local trade. This tree, says the *Moniteur Officiel du Commerce*, is a pine-like conifer, 10 to 15 metres in height. The Director of Agriculture in Indo-China sent home a sample of the Cây-doc oil prepared by the natives by an unknown process. Examination showed that the composition of this oil of the "*Garcinia Tonkinensis*" is as follows:—

Glycerides .....	90.04
Resin acid (soluble in alkali) .....	4.21
Resin (insoluble in alkali) .....	1.17
Essential oil .....	4.58
	100.00

This oil contains 43.2 per cent. of olein, and yields 11.96 per cent. of glycerin. The aggregate of the solid fatty acids, when separated from the oily acid, melts at 55°. The raw oil is easily saponified; the soap produced can easily be dissolved in water, excepting 1.17 per cent. of its weight. This residuum consists of resin, soluble in benzene, but not soluble in alkalis. The oil supplies a soap of a brown colour, having the appearance and, up to a certain point, the pleasant odour of palm oil; it can absorb much water. The oil of the "*Garcinia Tonkinensis*" seems likely to play a part in the soap industry of Europe. The tree is cultivated in Tonkin, but is slow of growth, yielding a fair crop of seeds only after the twentieth year. The value of the crop of seeds from a tree at the place of production is said to be a Tonkin piastre, equal to about 2 frs. 50 cents. It appears that Tonkin is not able to supply much either of seeds or of oil prepared by the natives, to European factories.

**Oils; Determination of Sulphur in —.** F. Jean.

See under XXIII., page 1147.

**Vegetable Fats in Animal Fats; Detection of —, by the Phytosterol Acetate Method.** Bömer.

See under XXIII., page 1147.

#### PATENTS.

**Oils and Fats; Bleaching of —.** J. C. W. Stanley and The Cotton-Seed Oil Sydicate, Ltd. London. Eng. Pat. 17,443, Oct. 2, 1900.

THE claims of this patent are, for the most part, the same as those of Eng. Pat. 11,419 (this Journal, 1901, 910). The oil is circulated in a thin film from spreading nozzles and subjected to the action of artificial light, substantially as described before. In this patent, however, no claim is made for the simultaneous introduction of air or other oxidising gas.—C. A. M.

**Detergents and their Manufacture.** G. Bamberg, London. Eng. Pat. 17,166, Sept. 27, 1900.

CLAIM is made for a detergent powder prepared by mixing together soap and chemically treated oatmeal or other

cereal. The finally-powdered meal (say 23 lb.) is treated with a solution of caustic soda (say 7 lb.) in sufficient water to give a specific gravity of 1.27, and a suitable quantity (say 22 lb.) of soap shavings. The temperature of the mixture rises and ammonia is given off in abundance. Finally the product is granulated and exposed to the air until the whole of the excess of caustic alkali has been converted into carbonate. The resulting dry soap powder is stated to be readily soluble in cold water, and to possess considerable cleansing properties.—C. A. M.

**Soap; Apparatus for Making Carpet Cleaning —.** H. Sefton-Jones, London, from S. Feder and J. van de Bücken. Aachen, Germany. Eng. Pat. 14,085. July 10, 1901.

The object of this invention is to secure the thorough incorporation at the right moment of the special substances (glycolic acid, taurocholic acid, &c.) added to remove stains and preserve the pattern. The apparatus claimed, consists of a vessel surrounded by a tightly-fitting chamber to prevent loss of heat, and provided with an air-tight bipartite lid. The vessel rests upon an iron plate, which is exposed to the heat of a fire. Through the centre passes an axle, bearing agitator-blades which revolve in opposite directions. A special apparatus is fixed to the side of the casing near the bottom, through which the additional substances are introduced into the unfinished soap by means of a piston rod and a delivery pipe, whilst a discharge pipe on the other side leads to the mould divisions. In each of the moulds, movable pegs are loosely inserted at a certain height, and these being pressed out by the liquid soap, indicate when the mould is filled. The vessel is provided with a thermometer and a safety-valve.—C. A. M.

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

#### (A.)—PIGMENTS, PAINTS.

**Red Lead; Determination of Foreign Impurities in —.** A. Jousser.

See under XXIII., page 1144.

#### (B.)—RESINS, VARNISHES.

**Rassamala Resin.** A. Tschirch and L. van Italie. Archiv, 239, [7], 541—547.

THE resin of *Alumnia excelsa*, the so-called Malayan storax, as at present prepared, has few characteristics in common with true storax. Two kinds of the resin were experimented with, *Rassamala bodas*, white (or yellow) rassamala, and *Rassamala beurem*, red rassamala, consisting of hard, dusty, readily pulverisable pieces, with a glassy fracture and an aromatic odour, recalling that of cinnamon, pepper, and turpentine. The melting point ranged from 65° to 80° C. It was partially soluble in alcohol, methyl alcohol, carbon bisulphide, and glacial acetic acid, more soluble in ether, acetic ether, chloroform, and benzene, but slightly soluble in soda solution, and in acetone. The alcoholic solution contained tannin. From the ethereal solution, a small amount of cinnamic acid crystallised out. Treatment with sodium bisulphite showed the probable presence of a little benzoic and cinnamic aldehyde. Oxidation with chromic and sulphuric acids afforded evidence of the presence of benzoic acid. The resin gave no indication of the presence of esters. It is evident, therefore, that this resin differs in every respect from the storax, a difference which may be due to the method of preparation.—J. O. B.

**Copals; Fusion of —, under Pressure.** W. Lippert. Chem. Rev. Fett- u. Harz-Ind. 1901, 8, [9], 177—178.

In the ordinary method of rendering copals soluble in oil, by a partial destructive distillation ("*Brenzreaktion*"), the loss in the form of gas and copal oil varies from 25 to 50 per cent., according to the nature of the copal; and even if the copal oil be recovered it is of little value. Various attempts have therefore been made to avoid this loss during



the fusion, of which mention may be made of those of Violette and of Precht, who have proposed fusing the copal in closed vessels.

The author points out that Violette's statements are at variance, since in one place he asserts that a copal melted at 350° C., and in another that it melted at 100° C. The copals thus treated were said to dissolve readily in linseed oil and turpentine. In repeating Violette's experiments, the author heated 10 grms. of hard Manilla copal for a long time in a glass bomb at 100° C., but no fusion occurred until after raising the temperature to 180° C. The copal then appeared as a light-yellow mass, full of bubbles. On opening the bomb the pressure was found to be very slight. On heating the contents with linseed oil, bubbles of water escaped, but the copal still remained insoluble.

Cowrie copal heated in the glass bomb for some hours at 300° C. left a light-yellow fused mass, which was completely soluble in hot linseed oil. It was found that turpentine and siccative could be added to the solution to some extent, but that too much, precipitated the copal. On cooling, the copal separated from the varnish, but was redissolved on heating.

Manilla copal treated in the same way yielded a very dark mass, which, in the author's opinion, was superfused, seeing that in the first experiment the copal melted at 180° C. The mass was soluble in hot linseed oil, the solution in this case, too, becoming turbid on cooling, and the copal being precipitated by turpentine and siccative.

Zanzibar copal (Animé), when subjected to the same process, remained practically unchanged, only an aromatic odour and a few drops of water being perceptible.

From these experiments the author concludes that copals do not fuse at 100° C. when heated in a closed vessel; and that those melted at a high temperature under pressure, although soluble in hot linseed oil and ethereal oils, separate out on cooling, so that the process cannot be regarded as technically practicable.—C. A. M.

#### PATENTS.

*Rosin; Process of Treating* — to obtain Rosin of Low Melting Point. F. Müller and G. Rossbach, Schwandorf, Bavaria, Germany. Eng. Pat. 17,427, Oct. 2, 1900.

ACCORDING to this patent, the melting point of the rosin is lowered, without any addition of foreign substance, by first melting it, and then heating it in a vessel by any suitable means. The longer the heating is continued, the greater the reduction in the melting point.—C. A. M.

*Varnishes and Products resembling Resins and Balsams; Manufacture of* — A. Kronstein, Karlsruhe, Baden, Germany. Eng. Pat. 17,378, Oct. 1, 1900.

THE process claimed, consists of heating a fatty oil or other unsaturated organic compound to a temperature short of decomposition, with the exclusion of oxygen or any oxidising agent, so that polymerised substances of high molecular weight are produced. The process may be applied to the manufacture of varnishes from drying oils, fish oils, rapeseed oil, castor oil, &c. Of other unsaturated organic compounds suitable for the purpose, the following are among those chiefly employed:—Allyl cinnamate, styracin, allyl- and diallyl malonates, or "allyl salts of substituted malonic acid, of indene, and of substances of the type of eugenol, saffrol, iso-eugenol, and styrol and its substitution products."

An admixture of 50 per cent. of Japanese wood oil with oils ordinarily used in the manufacture of varnishes yields a product of the consistency of "stand oil," when thus heated to a temperature below the decomposition point.

The substances formed vary with the nature of the compounds used. Thus, amber-like products are obtained from bodies of the type of allyl cinnamate, styracin, and allyl or diallyl malonate. A compound resembling glass with the properties of a resin is yielded by styrol and its derivatives, whilst esters of diallyl malonic acid produce a substance resembling natural balsam, which is soluble in the ordinary solvents. Allyl cinnamate or diallyl malonate are converted into resin-like bodies which are soluble in benzene and ethylene bromide, but insoluble in alcohol and ether.

By heating a solution of styracin in allyl cinnamate, in the absence of oxygen, a compound resembling Peru balsam is formed, whilst saffrol, eugenol, iso-saffrol and iso-eugenol give soluble solid substances resembling natural soft resins.

A special claim is made for the manufacture of oil of high viscosity, by heating wood oil, or mixtures containing more than 50 per cent. of that oil, with other oils or unsaturated organic compounds, in the absence of air, to about 200° C. until solidification occurs, and then raising the temperature to 300° C. until the mass liquefies, forming an oil which only becomes hard when exposed to the air.

—C. A. M.

#### (C.)—INDIA-RUBBER, &c.

*Caoutchouc of French Indo-China.* Ch. of Comm. J., Nov. 1901, 260.

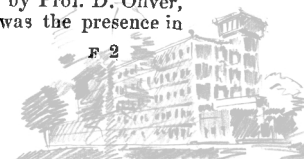
THE *Monit. Officiel du Commerce* publishes a detailed report on four kinds of caoutchouc from Upper and Lower Laos. No. I. appears in the form of balls (having an average diameter of 5 to 10 cm.), of a pretty regular shape, the weight of which varies from 40 to 120 grms. The general colour of this caoutchouc is more or less dark. In the cells in the circumference, as well as in the centre, vegetable refuse is met with, also fragments of moss, bark, wood, and very small particles of earth. These are only small impurities not due to fraud. The odour of this caoutchouc is weak, of the usual kind; there is no odour of acid or putrid fermentation, no igneous smell. It is a remarkably dry, sinewy caoutchouc, no portion of which is resinous nor fatty. This kind has been quoted by brokers at 8 frs. per kilo. Its preservation is remarkable, even under very defective circumstances. Sample No. II. differs from No. I. in the following particulars. It is in lumps, 15 to 20 cm. long on an average, with a diameter of 5 to 7 cm., weighing on an average from 250 to 270 grms. The cell-like structure resembles that of No. I. Two striking features may be noted: the almost black colouring and the stickiness of this sample. This sort is valued at 6 frs. to 7 frs. 75 cents per kilo. Sample No. III. shows crooked and irregular masses, weighing 417 grms. and valued at only 5 frs. 50 to 7 frs. 75. No. IV. is very similar to No. I., but the balls have a smaller diameter and weigh only from 30 to 80 grms. The loss from washing of these four samples is very trifling, and ensures therefore one of the most profitable industrial results. These samples (as well as the objects made from them, also photographs and tracings of the mechanical tests concerning them) may be seen at the Office National du Commerce Extérieur, Paris.

*India-Rubber; Behaviour of* —, towards Nitrous Acid. C. Harries. Ber. 1901, 34, [12], 2991—2992.

THE yellow compound produced on rubber tubes and stoppers by the action of nitrous acid is a nitrogenous derivative of acid character, probably a polynitrosate rather than a polynitrosite. It may be prepared by dissolving rubber (West African) in heavy petroleum and passing a rapid stream of nitrous acid (from arsenic and nitric acid) through the solution, or, more simply, by merely soaking the rubber in petroleum for 24 hours and churning the swelled mass with the current of gas until a test shows that the product is soluble in acetic ether and in alkalis. In either case a colloidal mass is obtained which breaks into golden scales easily dissolved by acetic ether, and precipitated therefrom when ether is added. The substance has the empirical formula  $C_{40}H_{62}N_{10}O_{24}$ , sinters at 95°—100° C., decomposes at 135° C. with evolution of gas, dissolves readily in alkalis and is precipitated, unchanged, when acid is added. The molecular weight determinations gave unexpectedly low results.—R. L. J.

*Gutta-percha from Eucommia Ulmoides.* Imp. Inst. J., Nov. 1901.

SPECIMENS of a peculiar Chinese plant, known as "Tuchung," were forwarded to Kew more than 10 years ago for examination, with the statement that the tree is cultivated for its bark, which is a most valued medicine. The plant was named *Eucommia ulmoides* by Prof. D. Oliver, and the chief interest attaching to it was the presence in



the tissues of an elastic gum, resembling caoutchouc or gutta-percha. If the bark be broken across, this appears in the form of innumerable silvery threads, and in a later examination Prof. F. E. Weiss found the yield from the dry bark to be about 3 per cent. Since then further material has been obtained, as well as living specimens of the plant, which are at present growing in several botanic gardens in Paris, and at Kew, and with these a much more satisfactory examination has been possible (*Kew Bulletin*, Nos. 172—174, p. 89).

The order of the plant is difficult to decide, owing to its peculiarities, and while Oliver and Ballion agree in placing it among the *Trochodendraceae*, it is referred to *Hamamelidaceae* by Solereder. Wherever it is placed according to its morphological characters, however, the allied plants do not yield rubber or gutta-percha. The elastic gum produced by the plant is found to be of the nature of gutta-percha rather than caoutchouc, and the vessels containing it are much more similar to the cells which yield true gutta-percha from *Dichopsis* than to the lactiferous vessels of the common rubber-yielding trees. It occurs in every part of the plant except the wood and the outer parenchymatous layer of the young roots. Some preliminary experiments, to ascertain the yield and quality of the gutta, have been made by MM. Dybowski and Frow on material obtained from living plants in Paris. The dried leaves yielded 2.25 per cent. of substance soluble in toluene, which, considering that the fresh leaves contained 70 per cent. of moisture, is not a high return. The fruits, however, gave a much better result, as when undried, containing 7.4 per cent. of moisture, they yielded over 27 per cent. of substance soluble in toluene. The product obtained was of brown colour, and behaved exactly like gutta-percha. When immersed in hot water it softened, and could then be stretched out into thin sheets, like gold-beaters' skin, or moulded, whilst on cooling, it again became quite hard. As gutta-percha it was stated by experts to be of good quality.

The plant has proved to be very hardy, for at Kew a specimen obtained in 1897 from Paris has grown successfully in the open without protection, and in Paris two unprotected plants have survived exposure through two winters to as low a temperature as 18° or 19° F.

*Eucommia ulmoides* may therefore prove of value as a source of gutta-percha, and the Jardin Colonial is already making experiments with the plant in Annam, Tonkin, and North Africa.

#### XIV.—TANNING; LEATHER, GLUE, SIZE.

*Quebracho Extracts Soluble in the Cold, and their Chemical Analysis.* J. Paessler and W. Appellius. *Deutsche Gerber-Zeit.* 44, [33—42, and 45]. *Zeits. angew. Chem.* 1901, [37], 930.

In examining different Quebracho extracts soluble in the cold, the authors found that Vienna hide powder contains about 1 per cent. of free hydrochloric acid, and is therefore unsuited for testing Quebracho extracts containing sulphite, this acid acting on the latter in the hide filter. They therefore give the preference to neutral Freiberg hide powder, and consider that such powders as are capable of producing chemical changes like the one mentioned, should not be used for tannin analysis.—C. S.

*Horn and Ivory; Dyeing and Bleaching* —. Textile Colorist.

See under VI., page 1111.

#### PATENTS.

*Hides and Skins; Process for Removing Hair, Wool, Fur, and the like from* —. H. R. Riches, and The Wool, Hide, and Skin Syndicate, Limited, London. Eng. Pat. 14,727, Aug. 16, 1900.

THE raw skins, after being softened in water and broken over the beam with the fleshing knife, are hung in specially constructed chambers and exposed to the combined action of ammonia, and the heat communicated by water vapours at a temperature of about 80° F., for a period of time which

varies according to the class and thickness of the skins. The wool or hair which is unimpaired by the treatment can then be removed by hand.

The method, "ammonia and heat," and the structural details of the steam-chambers, are claimed.—R. L. J.

*Chrome Tanning or Dressing of Skins, Hides, and the like.* F. Valentiner, Leipzig-Plagwitz, Germany. Eng. Pat. 19,831, Nov. 5, 1900.

FLUORINE compounds of chromium, such as the one referred to below, form leather much more rapidly than the compounds usually employed in chrome tannage, and with greater security to the hide substance.

*Preparation of "Fluorinated Chromium Hydroxide."*—Sodium bichromate (100 kilos.) is dissolved in water (400 kilos.) and mixed with formaline (5 kilos.). Hydrofluoric acid (200 kilos. of 10 per cent. strength) is added, and the whole heated at 60°—70° C. until a test portion treated with excess of caustic potash gives the characteristic emerald-green coloration.

*Application.*—A hide or butt (20 kilos.) is placed in a vat containing 40 kilos. of a solution of about  $\frac{1}{2}$  per cent. strength of fluorinated chromium hydroxide, stirred frequently and left until test portions, cut from the thickest part of the hide, show that tanning is complete. The hide is rinsed, drawn through a weak alkaline bath, and finished in the usual manner.

The process may be combined with a vegetable tannage, which should follow rather than precede the chrome treatment.—R. L. J.

*Tanning Hides and Skins.* P. Bez and E. Bez, Lérans, Ariège, France. Eng. Pat. 12,372, June 18, 1901.

THE skins are placed in weak tan liquors of gradually increasing strength, the density of each liquor being artificially raised by addition of a neutral salt, such as barium chloride, so as to produce a rapid penetration by osmotic pressure. Thus, pits may be filled with the following liquors:—I. A tan-liquor of density 1° Baumé, increased to 5° B. by addition of barium chloride; II. A liquor of density 2° B., raised to 10° B. by adding barium chloride; III. A liquor of density 3° B., raised to 15° B.; and, IV. A liquor, of density 4° B., raised to 20° B. in the way indicated.

On the same principle, uncombined tannin may be rapidly washed out by placing the tanned hides in a still denser solution of barium chloride in water only.

Extreme rapidity (hours instead of days) and uniformity of tannage are claimed for this process.—R. L. J.

#### XV.—MANURES, Etc.

*Perchlorate in Chili Saltpetre; Determination of* —. Grimm.

See under XXIII., page 1144.

*Blast Furnace Slag; Process of Treating Molten* —. A. D. Elbers. Eng. Pat. 8333, 1901.

See under X., page 1119.

#### XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar; Alkalinity of* —: *Revision of the Methods of Determination.* A. Herzfeld. *Zeits. Vereins Deutsch. Zucker-Ind.* 1901, Sept. [548], Part I, 381—386.

THE author deals with certain suggestions made by a Silesian Commission for alterations in the standard method of testing the alkalinity of sugars. The method consists in dissolving the sugar in faintly alkaline water containing phenolphthalein, and prepared in such a way that the pink color is discharged by the addition of not more than 0.5 c.c. of centinormal acid.

The alterations proposed by the Silesian Commission related to the cases where neutral sugars, when dissolved in neutral phenolphthalein water, give no red coloration, and it was suggested: (1) that from 1 to 3 c.c. of centinormal alkali be added to the 10 per cent. sugar solution, and if a red coloration ensue, as compared with the original, that the



sugar be returned as neutral, or not acid; (2) that a sample of the sugar be placed on litmus paper, and if a blue coloration be obtained, the sugar be described as not acid; (3) that the spotting method with turmeric be employed.

According to the standard method the maximum error that may occur is, that a sugar containing 0.0005 per cent. of acidity would be returned as alkaline. The wording of the Silesian suggestions appears to indicate that the phenolphthalein water has not been properly prepared, and that it was employed slightly on the acid side instead of on the alkaline. This is the only explanation of clause (1), and if the suggestion were carried out on a properly prepared phenolphthalein water, the sole result would be to magnify the maximum error indicated above. As regards the two other clauses the author points out that, since they relate to other indicators than phenolphthalein, they are quite out of the question. The "spotting" method, it is true, is the most delicate way of testing with turmeric, but litmus and turmeric alkalinities are very different from those found with phenolphthalein, and sugars which are distinctly acid to phenolphthalein, and which suffer inversion on heating in solution, may frequently show an alkaline reaction towards turmeric. The author concludes that there is no reason for modifying in any way the present method of testing.—J. F. B.

*Glucose; Derivatives of.—Remarks on Koenigs and Knorr's Paper.* A. Colley. Ber. 1901, **34**, [12], 3205—3207. (Compare this Journal, 1901, 626; also preceding abstract.)

THE author remarks that he had already prepared acetobromoglucose long before the publication of the work of Koenigs and Knorr, but had not published his investigations.

In preparing acetobromoglucose from acetyl bromide and glucose, some difficulty was experienced in moderating the reaction so as to avoid the production of free bromine, and consequent decomposition of the product; it was necessary to work at a temperature below  $-10^{\circ}\text{C}$ . If protected from atmospheric moisture acetobromoglucose may be kept for a long time without change. The author confirms the observation of Koenigs and Knorr, that acetochloroglucose readily reacts with silver and barium acetates, with the formation of the pentacetylglucose melting at  $131^{\circ}\text{C}$ . He further attempted to replace the chlorine by a divalent radicle, by the interaction of acetochloroglucose with silver sulphate; but, instead of the sulphuric acid derivative of glucose expected, there was formed a considerable amount of the isomeric pentacetylglucose melting at  $112^{\circ}\text{C}$ , together with by-products which have not been investigated.—J. F. B.

*Desiccation of Beetroots; Influence of—, in the Bull. on Storage in Silos.* G. Levesque-Hérault. Field. de l'Assoc. des Chim. de Sucre et de Dist. 1901, **19**, [1 and 2], 159—161.

WITH the crop of beets obtained last year, the summer of which was very warm and dry, it was found that, when the beets were stored in silos for a few days before being used in the factory, dry rot very quickly set in. No rain fell for the first three weeks of working, and although the roots received, grew steadily richer during this period, yet, owing to the storage in silos, the value of the roots when used was found to gradually diminish. This result was due to the continued action of the drought on the roots, which had the effect of predisposing them to rapid change.—T. H. P.

*Beetroot Juice; Krause's Method for the Determination of the Quotient of Purity of—.* Drenckmann. Zeits. Vereins Deutsch. Zucker-Ind. 1901, **51**, [546], 655—663.

THE use of finely ground pulp offering much inconvenience, finely grated, or minced chips were employed in the experiments made to compare the ordinary process of alcoholic digestion (clarifying with faintly basic lead acetate according to Weisberg) and Krause's method, varying the time of digestion and the temperature.

The results show that, by extending the time of digestion and de-aeration from 10 to 45 minutes, no increase in the sugar content occurs, but an increase of total solids, and a lowering of the quotient by 1 to 2 per cent.

On reducing the temperature of digestion from  $90^{\circ}$  to  $70^{\circ}\text{C}$ , the duration of time being 10 minutes, the extraction of sugar was unaltered generally, the dry substance not at all, so that a temperature of  $70^{\circ}\text{C}$  is sufficient for digestion. Throughout the various conditions the sugar obtained was 0.2 to 0.3 per cent. higher than by alcoholic digestion.

Errors were to be expected from the greater tendency of aqueous solutions to retain air, as compared with alcoholic solutions, but no increase of volume due to this cause was found.

Strohmer's observations agree with the above. He finds that the determination of sugar, and therefore the sugar content of the beetroot, is very slightly, if at all, influenced by the time of digestion and de-aeration and the temperature, or by the fineness of the pulp; but that all these conditions affect the hydrometer, and consequently the quotient, and suggests that, as has been agreed upon in Prague, a uniform method of procedure be adopted to make the results obtained in different factories comparable.

Sachs is of opinion that it is better to work cold, the results being more comparable with those of diffusion; and the higher purity found by this method is no proof that it is wrong.—L. J. de W.

*Vacuum Pan Construction [Sugar Manufacture].* Greiner. Zeits. Vereins Deutsch. Zucker-Ind. 1901, **51** [546], 701—708.

THE greater magnitude of operations arising from adoption of the diffusion process, and heating by double and triple effect, led to the increased size of vacuum pans which are now introduced in many factories, where a single pan may take the whole of the first product. The question then arises, whether the safety and quality of the work do not suffer from dealing with such large portions of massecuite. The great fluctuations in the demand for steam, whether direct or indirect, are felt in the boilers, and economy of fuel is more likely to be assured where two or more pans are used, the boiling periods of which overlap, so that greater regularity results.

At the moment when the first grain is formed in the pan, the volume is the smallest during the whole boiling, representing 28 to 30 per cent. of the whole liquor used; where larger crystals are to be produced, less still; and the heating coils must not rise above this, since they should never be uncovered by liquor. Why not, then, as Rillieux proposed, carry out the concentration, formation, and growth of grain in separate vessels? The reason is, that the steam available at the end of the evaporating station, indirect steam being now used for heating, is no longer sufficient for boiling a mass with a boiling point of  $120^{\circ}$  to  $130^{\circ}\text{C}$  under atmospheric pressure. The operation of concentrating should be interrupted when ready for graining, and the rest of the process continued at a spot where direct steam is available.

There are many faults in boiling. The obtaining of soft, floury grain is often ascribed to low steam when the real cause is want of exchange of heat. To this end, movement is necessary, as Hagemann and Jelinek have shown, since a heating surface may transmit three times more heat where there is movement of the mass than where there is none. To the same end, and with movement of the mass, the heating coils need be only one-third the size as would be required without such motion. A boiling mass can never be without motion, but the slight displacements observed take place rather in the body of the syrup than at the heating surface. The heating coils often fill the whole horizontal section of the pan, leaving only about 1-25th part free in the centre, and a small ring at the walls, thus preventing circulation. It is therefore no paradox to state that by lessening the heating surface the heating effect may be increased, if a portion is removed which impeded circulation. The author has found that by shutting off the outer ring, or, in large pans, the outer two, a fine and regular product was obtained. When the massecuite, however, has become so stiff that this motion no longer proceeds, the outer coils are then heated, the circulation of the mass being no longer necessary, as the grain will not deposit, owing to the great frictional resistance.



Fine or false grain is caused either by stagnation of a portion of the massecuite in the angular parts of badly-formed pans, or by drawing in fresh liquor which, instead of becoming distributed among the mass, rises unmixed to the surface, where it undergoes active evaporation. The danger of false grain increases towards the end of the boiling as the difference in density between liquor and massecuite increases. This is to be avoided by introducing the liquor in thin streams.—L. J. de W.

*Syrups; Process and Apparatus for Regulating Supersaturation in the Boiling of*—H. Claassen. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1901, 18, [12], 1035—1042. Ger. Pats. 117,531 and 117,532.

HITHERTO the concentration of syrups for crystallisation has been determined by experience—the string proof. This proof has no precision, owing to the variable composition of the syrup as regards purity and viscosity. For good work it is necessary that an appropriate temperature and water content should be established to maintain the necessary supersaturation, and that this optimum should continue long enough for the syrup to have time to deposit its sugar on the crystals already formed. These conditions are fulfilled by the new process.

To maintain the temperature long enough at the desired degree, corresponding to water content of the syrup and the degree of vacuum, without causing evaporation, the best means found is the introduction of steam into the massecuite.

As boiling proceeds, the purity of the syrup diminishes continuously by the precipitation of sugar, and the water content must also diminish with a corresponding rise of temperature. These two determinations are made by the instrument described below, and its use enables the conditions suitable for crystallisation to be kept constant. With decrease of purity, viscosity increases, and to remedy this obstacle to crystallisation the temperature must be raised, and the concentration diminished; that is, as boiling proceeds the quotient of supersaturation of the hot syrup is diminished. In this way the syrup may be reduced to a purity of 65.

The further desugarising is carried out in mixers. Here account must also be taken of the viscosity caused by cooling, and this viscosity can only be lessened by lowering the supersaturation. Thus, preposterous as it may seem, an addition of water may be the means of accelerating the deposition of sugar by lessening the viscosity more than the supersaturation. This should be so done that, after centrifugalising, the supersaturation is lowered approximately to 0. The control apparatus consists of a manometer, a thermometer, and two double scales. There is also a series of tables giving the water content corresponding to various temperatures and degrees of purity, as determined by the author's experiments. The purity of the syrup to be boiled being known, a reference to the tables will give the water content corresponding to the vacuum and temperature as read. On setting the index of the scale in accordance with these data, a reading is obtained of the temperature to which the syrup is to be brought by concentration. This point being reached, the valve is opened for direct admission of steam to keep the quantity of water fixed while grain forms. During the first hour after graining an advance is made from the next figure on the tables, and so on.

The claims are for (1) a process for regulating the supersaturation in the boiling of syrups, consisting in evaporating the syrup at first in the ordinary manner up to a concentration determined empirically. The heating is stopped and the temperature regulated according to the degree of the vacuum by the introduction of dry steam direct into the syrup until crystals are formed. The boiling is continued by introducing syrup, but maintaining a determinate concentration by means of the ordinary steam, or by steam introduced directly, to obtain the necessary temperature. During the whole boiling the mass is kept in movement by an agitator.

(2) The determination of the temperature necessary according to (1) by the establishment of an apparatus to control the degree of vacuum and water content; this latter is determined by the quotients of supersaturation, which are themselves dependent on the purity.

(3) The control apparatus described.

(4) The further treatment of the massecuite thus obtained in crystallisers with agitators, and with the addition of water or dilute sugar juices.

The supplementary patent proposes the addition of ready-formed sugar as seed, instead of graining the syrup itself. During the introduction of the seed the supersaturation of the syrup is to be kept lower than when graining, and as the seed is colder it should be introduced gradually, to avoid forming false grain.—L. J. de W.

*Grosse's Process; Boiling and Crystallising Low Products by*—E. Carlson. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1901, 18, [12], 1043—1044. From Deutsch. Zucker-Ind. 1901, 553 and 923.

THE crystallisation of the last traces of sugar with a supersaturation of 0 would be too slow; the concentration therefore has to be carried to a degree a little higher than that indicated by the tables. To arrive at the exact point, the concentration is pushed a little further than actually required; then, from a sample taken, the degree Brix is determined, and the quantity of water calculated which is required to be added to restore it to the desired degree Brix. The water is not added directly, nor as sweet water from the filters, for the mixing would be too slow, and there would be danger of sugar being dissolved; it is preferable to use a quantity of molasses at 75° Brix heated to 50°—60° C.

It appears from the tables that, in order to "spin off" a syrup of given purity, the concentration must be greater in proportion to the purity of the massecuite. When this purity is too great the concentration required is so great that crystallisation is impossible, and the purity should, in this case, be lowered by additions of undiluted molasses at 70° to 75°, but it is better to modify the previous stages of work, so as to have the product of relatively low purity.

Beyond the questions of temperature and concentration one has to consider the purity of the drained syrup; it corresponds to the limit of crystallisation of the product worked. If, with successive boilings of approximately equal quality, but with a gradual increase of concentration and time of crystallisation, there is not found a parallel diminution in the purity of the spun syrup, but rather an increase in viscosity and difficulty of centrifugalising, it is fairly certain that the limit of crystallisation has been closely approached.

For massecuites of sensibly similar properties, and conditions of working more or less constant, there exists a fairly constant difference between the purity of the massecuites of the second and last syrups. This difference remains so long as the limit of crystallisation is not approached, whether the initial purity rises or falls. As soon as this limit is neared, the difference in question loses its constant character, diminishing when the purity of the massecuite falls and *vice versa*; on the contrary, the purity of the "syrup off" remains fairly constant. If it be found that by additions of molasses to the boiling mass the diminution of the quotient of the massecuite does not run parallel with that of the "syrup off," but that the difference only varies correspondingly, it is fairly certain that the limit of crystallisation is nearly reached, and it is evident that fresh additions of molasses are then of no utility.

After boiling, the massecuite is mixed either in the pan itself or in another mixer. If under each pan there is a mixer capable of holding the entire boiling, mixing is continued 36 hours in the pan, and a further 20 to 30 hours in the mixer, making a total of 75 to 80 hours for the whole operation. On mixing in a vacuum, it is observed that the mass becomes frothy, swells up, and seems to become fluid. This is not due, as generally believed, to the destruction of crystals by the wipers, but to the entrance of air through badly-made joints. Mixing 30 to 40 hours in a vacuum removes 0.5 to 0.7 more water.

Cooling should be conducted so slowly as not to supersaturate the mother-liquor unnecessarily. At the end of the cooling there is always found an increase of supersaturation, due to the fact that, at this limit, crystallisation does not proceed as quickly as the cooling. If the mother liquor is too viscous and too difficult to spin, the supersaturation may be suppressed by an addition of diluted molasses (75° Brix,





about), heated not above 50°—60° C., or, more simply, by reheating in the mixer if arrangements for this exist, or in the centrifugal casing. The two last methods should be used very carefully, not to lower the concentration of the mother liquor below the point of saturation.—L. J. de W.

**Crystallisation Products, particularly Sugar Candy; Process and Arrangement for facilitating Removal of —, from Wire or Tubular Carriers.** S. Bornett, Vienna. Zeits. des Vereins Deutsch. Zucker-Ind. 1901, 51, [547], 761—762. Ger. Pat. 121,870.

This method at present in use for preparing sticks of sugar candy is by allowing it to crystallise on woollen threads, the use of wire or metal tubes offering difficulties, as the sugar could not be removed from the carriers. This is to be facilitated by passing an electric current so as to heat the carriers to about the melting point of the product.

—L. J. de W.

**Caramelan; Investigations on —. IV. The Products of Hydrolysis of Caramelan.** F. Stolle. Zeits. des Vereins Deutsch. Zucker-Ind. 1901, Sept., [548], Part II. 836—838.

CONTINUING his researches on caramelan (see this Journal, 1900, 859), the author has investigated the hydrolysis of this body by heating it for 18 hours on a boiling water-bath with 3 per cent. hydrochloric acid. The colour of the solution gradually disappeared in the course of the treatment, with the separation of a brownish-red precipitate. This insoluble body amounted to about 30 per cent. of the original caramelan, and has not been studied further. There remained in the colourless solution a body of the nature of a reducing sugar. It could not be induced to crystallise, but the solid mass precipitated by ether from its alcoholic solution, showed a tendency to bi-rotation, and gave solutions which were dextro-rotatory. The osazone was prepared, and was found to melt at 197° C., and, on analysis, numbers were obtained corresponding to the osazone of a hexose. This osazone is insoluble in cold and boiling water, also in dilute alcohol, but dissolves in boiling 96 per cent. alcohol, glacial acetic acid, and acetone, and crystallises as yellow needles in spherical aggregates; its solution in acetic acid is laevo-rotatory.—J. F. B.

**Molasses Residues; Acids Soluble in Ether, derived from —.** A. Herzfeld. Zeits. Vereins Deutsch. Zucker-Ind. 1901, 51, [547], 720—745.

THE molasses residue principally examined, was obtained from Dessau, but for the sake of comparison some from Mochern was also used, and proved to be of approximately the same composition. The Dessau residue was of sp. gr. 1.41335, and contained 22.48 per cent. of water and 28.62 per cent. of ash.

The residue was acidified with dilute sulphuric acid, and shaken out with ether eight times, until the weight of the extract, after evaporating off the ether, became constant. Afterwards, 50 kilos. of the acidified residue were extracted with double the volume of ether, and the extract was subjected to fractional distillation, esterification, and crystallisation of some of the salts of the acids.

The Dessau molasses residue contains, per 100 of dry solids, 20.6 per cent. of compounds soluble in ether. These consist of 4.92 per cent. of formic acid, 20.88 per cent. of acetic acid, 20.9 per cent. of lactic acid, and about 3 per cent. of propionic, butyric, and valeric acids. Succinic acid was apparently present, but could not be identified with certainty.—L. J. de W.

**Saccharimeter; A New —.** P. Horsin-Déon.

See under XXIII., page 1141.

**Alkalinity; Indicators for —. Alterability of Stored Raw Sugars.** Köhler.

See under XXIII., page 1147.

**Honey-Dextrin; Nature of —.** E. Beckmann.

See under XVIII. A., page 1131.

## PATENTS.

**Glucose; Process and Apparatus for the Manufacture of —, by Saccharifying Mucedines.** L. C. A. Calmette, Lille, France. Eng. Pat. 11,622, Oct. 18, 1900.

CRUSHED maize or decorticated rice is introduced into a boiler with twice its weight of water, and hydrochloric acid equal to 0.5 per cent. of the weight of the grain, and the mixture is heated to boiling in three stages of one hour each at 100°, 110°, and 120° C., the acid being preferably added in small doses during the boiling. When the starch is converted into dextrose, maltose, and dextrin, the charge is concentrated to contain about 25 kilos. of grain per hectolitre, and discharged into a suitable apparatus, in which complete transformation of the maltose and dextrin into dextrose is effected by saccharifying mucedines, added in the form of young mycelium, without sporules.

Several species of mucedines, *mucors* or *aspergillus*, can be employed; the most suitable are those of the *aspergillus* group.

The mixture in the saccharifying vessel is emulsified by the action of filtered compressed air, sterilised by steam. A small dose of mucedines is added during the emulsifying action, which goes on for about 36 hours, or the time may be abridged to 24 hours by adding a leaven of mucedines previously prepared. The must is then rapidly cooled to 10°—15° C., or it may be heated to 55°, to check mucedine development, and filter-pressed. The residue forms an excellent food for animals, while from the filtered liquor syrups of various densities, or crystallised glucose can be obtained. When first introduced into the saccharifying vessel, sodium carbonate is added until the acidity is 0.25 per cent., reckoned as H<sub>2</sub>SO<sub>4</sub>: When the transformation into glucose is complete, the material is again neutralised with sodium carbonate until only very feebly acid.

The principal parts of the apparatus consist of an air-filter arranged for sterilisation by steam, an emulsifier in which the compressed air bubbles through the must in pipes, so contrived as to cause a circulation in the saccharifying vessel, which has the necessary pipes for charging, discharging, and for inlet and outlet of air, also means of cooling by water externally applied.—L. J. de W.

**Starch; Method of Manufacturing Thin Boiling or Modified —.** C. B. Duryea, Manhattan, New York, U.S.A. Eng. Pat. 11,442, June 4, 1901.

FOR some purposes, particularly for steam laundry use, it is highly desirable to have starches yielding pastes of a less viscous and colloidal character than ordinary starches yield when boiled with water.

The method here employed for making a thin boiling starch consists in suspending the material in warm water with 1 per cent. of acid, preferably sulphuric acid, and treating it at about 55° C., but not exceeding 59° C. When the proper point of conversion is reached, the heating is discontinued and the charge washed, and neutralised with some suitable alkali.—L. J. de W.

## XVII.—BREWING, WINES, SPIRITS, Etc.

**Mycoderma Cerevisiæ.** H. Van Laer. J. Fed. Inst. of Brewing, 1901, 7, 337—356.

THE author reviews at some length the investigations which have been carried out on this class of organism. The species most likely to be obtained when any beer or yeast is taken at random possesses the general morphological and physiological characters of the *mycoderma cerevisiæ* described by Hansen (Meddel. fra Carlsberg. Labor. 1888, 2, [5]). It is a non-sporulating variety, and its cells have for the most part the appearance of *Pastorianus*. The morphological appearance is, however, dependent on age and conditions of culture. The membrane is impregnated with fatty matters soluble in alcohol [? ether], consisting of olein and solid glycerides. The cell contains three or four vacuoles, the refractive index of which differs but little from that of the protoplasm. When cultivated in beer wort at 25° C., there appear in the protoplasm shining, fatty corpuscles varying in number from one to three. Should the



corpuscles be well formed, the vacuoles are very sharply defined, especially if the specimen be taken from the bottom of the culture flask. Glycogen appears in the young organism either at the centre of the cell as fine granulations or in the vacuoles as small spheres which give an intense reddish-brown iodine reaction. In old cultures the vacuoles sometimes contain crystalloids. The film has at first the appearance of a spider's web. Later on it becomes thicker and folds form in it, whilst packets of cells are continually being detached and form a deposit on the bottom of the culture vessel.

The depth attained by the colonies of a gelatin stab culture show that the organism is capable to a very great extent of existing in the absence of air. *Mycoderma* do not liquefy gelatin. With the exception of *Saccharomyces mycodermiquus* the commoner forms of this group have no other means of multiplication than that of budding (compare B. Fischer, Centralb. f. Bakteriologie, 1893, 14, 633; Brebeck, Zur Morphologie, Bakteriologie der Kahmpilze, etc., Jena, Gustav Fischer, 1894; Will, Zeits. f. das. Gesamte Brauwesen, 1897, 127).

The highest temperature of film formation is about 30° C., the maximum for most of the media examined by the author being about 42° C. Death occurs when the cells are heated to 50° C. or to 55° C. in wort or acid liquids. The cells preserve their vitality at 5° C., but there is no film formed on wort at this temperature, although this occurs on beer after 46 days.

The best mineral foods for *mycoderma cerevisiae* are the same as for beer yeast. Phosphoric acid and potash are the mineral substances used mostly by this organism, magnesia occupying a second place. *Mycoderma* can make use of the nitrogen of ammonium salts for the synthesis of its nitrogenous constituents, provided that a suitable carbonaceous material is present; and, in a similar way very varied carbonaceous food can be utilised if the nitrogenous matter be appropriately chosen.

A large number of ternary substances can be utilised by *mycoderma cerevisiae*. Among these are ethyl alcohol (Pasteur, Comptes Rend. 1862, 64, 265), but methyl, propyl, and butyl alcohol are unattacked, as also is any monohydric alcohol richer in carbon than these. Pasteur (*loc. cit.*) found that *mycoderma* destroyed the sugars. Beyerinck and Seyfert have been able to confirm the disappearance of dextrose and levulose, whilst maltose, cane sugar, and lactose remained unchanged. Arabinose and xylose are readily destroyed by *mycoderma*.

All the experiments made to discover invertase in the condition under which yeasts show an inverting action have failed.

The author discusses the question of the influence of *mycoderma cerevisiae* when present in pitching yeast. Hansen in 1878, found that all the beers of the lager cellars of the old Carlsberg Brewery were more or less contaminated with this organism without, however, exhibiting any indication of disease. In many cases the flavour was improved. Bělohoubek (Vortrag, Böhm. Bierbrauer, Prague, 1885) was the first to show that *mycoderma* is capable of causing considerable trouble in brewing. The author considers that the injurious action of this fungus depends on the vigour of the yeast and on its greater or less abundance. A beer which is free from yeast is very rapidly spoiled by *mycoderma* and soon develops a strong smell of mould. He brings forward experimental evidence showing that the presence of *mycoderma* in pitching yeast retards the fermentation somewhat, part of the extract being left undecomposed. This retardation is particularly noticeable in the case of Logos yeast. —J. L. B.

*Yeast; Process for the Artificial Preparation of —, without Lactic Acid Fermentation.* M. Bücheler. Ger. Pat. 123,437, June 22, 1900.

THE acidification of the mash in which the mother-yeast is prepared, is effected sometimes by a preliminary lactic acid fermentation, or the addition of commercial lactic acid, and sometimes by the substitution of a mineral acid for the lactic acid. According to the process here described, the acidification is brought about, making use of the salts of organic acids naturally present in the potato mash, by the

addition of a small quantity of sulphuric or phosphoric acid in such proportions that no excess of free mineral acid remains. The maximum point is determined by the behaviour to Methyl Violet as an indicator. Then the acidity of the medium is due entirely to organic acids, and all the advantages of a lactic acid mash are obtained with a minimum of cost and time. The mash is made in the usual way, the necessary quantity of mineral acid is added, the whole is cooled down to the fermentation temperature, and the mother-yeast is sown. After 18 or 20 hours the yeast is ripe, and the acidity of the mash is just the same as before it was set up, being about 50 per cent. lower than is generally the case with lactic acid mash. The avoidance of the lactic fermentation effected by this process saves 24 hours, and the sugar usually consumed in that operation is made available for alcohol production; moreover this process is uniform and certain.

—J. F. B.

*Yeast; Increase of the Efficiency of Bottom-fermentation* — A. Krause. Woch. für. Brau. 1901, 18, [41], 520—521.

THE author criticises the so-called "normal" system of fermentation in vogue in Germany, which consists in pitching the wort at a temperature of 5° C., and using only half a litre of yeast per hectolitre. The whole process of fermentation requires from 8 to 10 days in the vats, and, owing to the low temperature and small quantity of yeast, there are no visible signs of working for the first 15—30 hours. The wort lies exposed to any infection that may be about, and is very liable to absorb bad odours from the air, since it is absolutely without any protection. When fermentation does start, the liquid becomes saturated with carbon dioxide, which still further retards the activity of the yeast. Not until the stage of "Hochkrausen" is reached, with a rise of temperature to 9°—10° C., is the yeast in a position to afford protection to the wort, and even then it is retarded by the presence of the carbon dioxide and deficiency of air.

The "normal" system apparently only depends upon old custom, when the wort had to be started cold, to avoid excessive rise of temperature later on. At the present time, when such perfect arrangements are available for cooling the wort in the vats during fermentation, this system is out of date, and the low temperature, tends to assist the development of wild yeasts.

A great economy of cooling power and vat space would be effected, if fermentation were started at a higher temperature and carried through more rapidly.

The author has been working most satisfactorily for several months according to the following system:—The wort is pitched at a temperature of 9°—10° C., and the quantity of pitching yeast is doubled, being 1 litre of yeast per hectolitre of wort. Visible fermentation starts almost at once; moreover, 6 hours after pitching the wort is aerated with filtered air for 18 hours. Thus the yeast multiplies very rapidly, and soon attains its maximum activity, protection being afforded to the wort almost from the very first; the carbon dioxide is also removed by the aeration. The temperature is never allowed to rise above 10° C., the wort being cooled by water circulation. The beer is ready for drawing off in about 3½ days, as against 7½ days by the old system. The vat space necessary for working the brewery is thus halved. The beer ripens more quickly in the cellar; it also clears better and keeps better. The attenuation may be regulated as desired, according to the length of time during which the wort is aerated.

—J. F. B.

*Grain Yeast [Top-fermentation] and Bottom-fermentation Beer Yeast; Differentiation of —, by Determination of the Fermentative Power at Different Temperatures.* C. J. Lintner. Woch. für Brau. 1901, 18, [36], 446—447.

IT is a well-known fact that pressed bottom-fermentation beer yeast is unsuitable for baker's purposes; when it is used the dough rises rapidly to the proper height, but falls down during the baking, and the bread lacks the light consistency of that prepared with grain yeast. Hayduck's method for the determination of fermentative power, at



30° C., fails to account for this, since the beer yeasts generally show a higher fermentative power than the grain yeasts at this temperature. Experiments made by Barth also prove that the difference is not to be attributed to any proteolysis of the gluten by the enzymes of the bottom-fermentation yeasts.

In the baking of bread, the temperature of the interior of the dough does not exceed  $104^{\circ}\text{C}.$ ; this temperature is reached gradually, and the efficiency of the yeast depends upon the relative resistance of its fermentative power to rise of temperature. The author has therefore carried out determinations of the fermentative power by Hayduck's method at temperatures of  $30^{\circ}$ ,  $45^{\circ}$ , and  $50^{\circ}\text{C}.$  The following table shows the uncorrected volume of gas evolved by 10 grms. of pressed yeast from 400 c.c. of a 10 per cent. sugar solution:—

		30° C.	45° C.	50° C.
i Beer yeast (bottom-fermentation)....		c.c. 360	c.c. 248	c.c. 107
2        "                  "                  "                  "                  " .....	"     "     "	475	232	72
3        "                  "                  "                  "                  " .....	"     "     "	342	210	63
4        "                  "                  "                  "                  " .....	"     "     "	367	162	28
5        "                  "                  "                  "                  " .....	"     "     "	350	170	25
6 Grain yeast (top-fermentation).....		547	380	279
7        "                  "                  "                  "                  " .....	"     "     "	290	428	231
8        "                  "                  "                  "                  " .....	"     "     "	113	203	118
9        "                  "                  "                  "                  " .....	"     "     "	275	354	175
10      "                  "                  "                  "                  " .....	"     "     "	298	388	161
11      "                  "                  "                  "                  " .....	"     "     "	290	428	136
12 Weissbier yeast                          " .....	"     "     "	210	255	180

Here, then, is the explanation of the superiority of grain yeast over beer yeast for bread making:—whilst the volume of gas evolved by beer yeast is considerably less at 45° C. than at 30° C., that evolved by grain yeast is greater at 45° C. than at 30° C. The latter is also still considerably active at 50° C., which is a great advantage in bread making.

This difference in the behaviour of the fermentative powers of the two types of yeast at 30° C. and 45° C., combined with the raffinose test of Bau, is likely to prove of considerable utility in the analysis of pressed yeasts. In this way yeast No. 5 in the above table, which was fraudulently sold as grain yeast, was proved to be a pressed beer yeast.

The determination of the fermentative power at 45° and especially at 50° C. affords a measure of the relative value of grain yeasts for baker's purposes, which is not shown by the determination at 30° C. The greater the volume of gas evolved at 50° C. the greater is the value of the yeast from a bread making point of view, although the results obtained at 30° C. may have been in quite another direction.

The author suggests that, since the value of bakers' yeast depends upon such a purely physiological factor, it may be possible to acclimatise yeasts, hitherto unsuitable, so as to be more resistant to high temperatures.—J. F. B.

*Yeast; Agglutination of* —. H. P. Barendrecht.  
Centr.-Bl. f. Bakter. und Parasitenk.; **7**, [2], 623--627.  
Chem. Centr. 1901, **2**, [14], 818.

IF suspensions of yeast be introduced into increasing quantities of N/10 sulphuric acid, agglutination is observed, the time being dependent on the amount of acid present. Using 15 c.c. of a yeast suspension, no action was observed with 0.2—0.3 c.c. of N/10 acid. Experiments with hydrochloric, phosphoric, formic, acetic, propionic, and valeric acids, showed that the extent of the action is not proportional to the degree of acidity, but that the weaker the acid, the greater is the quantity necessary to produce the most rapid agglutination. The phenomenon is apparently due not alone to acidity, but also to the action of the hydrogen ions. The yeast cells showed no perceptible alteration under the microscope after agglutination. The process is stated to be a physiological one, and to be reversible, as the particles after complete agglutination by sulphuric acid can be restored to their original condition by agitation. The agglutination is connected with the life

of the cells. Yeast cultivated in wort was not capable of agglutination, but acquired that property if ammonium salts were added to the wort. The concentration of the yeast suspension has little influence on the action.

The formation of flocks observed in the manufacture of yeast by the aeration process is quite a different phenomenon; here the acidity has no direct influence. The active agent in this case is a bacterium, which the author has isolated, and to which he has given the name *Leukonostoc agglutinans*. This micro-organism produced lactic acid in wort, and on wort-gelatin containing cane sugar, formed a mucilaginous drop, but not in presence of dextrose, levulose, maltose, raffinose, and lactose. The agglomeration of yeast in the factory is probably due, therefore, to the agglutination of the cells by means of the mucilage produced by *Leukonostoc agglutinans*.—A. S.

*Enzyme Action.* A. Brown. Paper read before the British Association (Section B), Glasgow Meeting, 1901.

THE action of invertase on cane sugar, as examined by the usual methods, does not appear to follow the law of mass action, but resembles that of fermentation. It is probable, however, that in the inversion of cane sugar, the sugar enters into molecular combination with invertase previous to change, and, if such be the case, the time factor, which under the conditions would be of some magnitude, would limit the effect of mass action in inversion changes as observed in solutions of ordinary concentration. The author found by direct experiment, that at a certain point of dilution (with a 1 per cent. solution of cane sugar), invertase acting in sugar solution exhibits an order of change in conformity with mass action. Since the character of the action of fermentation has been shown to resemble that of inversion, the author considers it very probable that in this enzyme change, also, the action is limited by the time factor of molecular change, and that the influence may be exerted in all enzyme changes.—A. S.

*Barley and Malt; Pentosan Content of, —, with Special Reference to the Increase of Pentosans during Germination.* W. Windisch and R. Hasse. *Woch. für Brau.* 1901, **18**, [40], 493—495.

THE work of Tollens and his collaborators has proved that there is a small but distinct increase of the pentosans during the germination of the barley corn (see this Journal, 1901, 268). But it was not shown whether this increase was general, or localised in any particular part of the corn.

The authors have germinated barley and separated the coombes from the malt after kilning. The pentosans were then estimated as furfural-phloroglucide. The statistics are all referred to 100 parts of barley dry-substance.

	1. (Laboratory.)	2a. (Laboratory.)	2b. (Large Scale.)
Yield :—	Per Cent.	Per Cent.	Per Cent.
Malt dry substance.....	91·80	93·12	85·28
Coombes dry substance.....	3·19	2·25	5·31
Loss in steeping and by respiration.	5·01	4·63	9·41
Pentosans :—			
In barley dry substance.....	8·16	8·27	8·27
„ malt „ „ .....	9·41	9·49	10·60
„ coombes „ „ .....	11·51	11·06	17·07

Hence, calculating to 100 parts of barley dry-substance,—

	Parts.	Parts.	Parts.
Pentosans:—			
In malt .....	8·64	8·75	9·04
„  coombes .....	0·37	0·25	0·61
Total .....	9·01	9·00	9·65
Total increase during germination .....	0·85	0·73	1·68
Increase in malt .....	0·48	0·48	0·77
„  coombes ..	0·37	0·25	0·91



The distribution of the increase was found to occur proportionally more in the malt-corn or in the coombes, according as the growth was short or long.

The authors then studied the question of the distribution of the pentosans in the parts of the dressed malt. For this purpose the embryos were excised from the sample of malt (2b) prepared on the large scale, and the proportion of their weight to 100 parts of original barley was determined. The pentosans were then estimated in the residual malt-endosperms. There were found 9.28 per cent. of pentosans in the endosperms, equal to 7.17 per cent. calculated on the original barley dry substance. This represents a loss of 1.1 per cent., the whole of which would be accounted for by the pentosans in the embryos of the barley. Hence, the authors conclude that the pentosans formed during germination, are localised exclusively in the vegetative organs (acrospires and rootlets), and that the material from which these newly-formed pentosans are produced is not furnished by the pentosans of the original barley-corn, but is derived from the starch or saccharine constituents of the growing corn, and that the pentosans, consequently, have no functions as reserve materials.

—J. F. B.

*Sucrase or Invertase in Industrial Fermentations.* G. Dejonghe. Bull. de l'Assoc. des Chem. de Sucr. et de Dist. 1901, 19, [1 and 2], 77–81.

In his "Traité de la fabrication de l'alcool" the author, referring to the fact that in the fermentation of molasses the action ceases before the whole of the sugar is converted into alcohol and carbon dioxide, states that such cessation is not due to lack of invert sugar, since the sucrase secreted by the yeast remains active after the death of the yeast, and the inverting action of the sucrase appears to be unlimited; further there is no fear of the action of the sucrase being affected by the antiseptics employed in the fermentation since the soluble ferments are much more resistant to the action of antiseptics than are the living ferments. These views the author now upholds against objections raised by Verbièse and Effront, who consider that fermentation stops owing to want of invert sugar. He points out that the enfeeblement must take place in the yeast and not in the sucrase, since the latter is not an organised body but only organic material of a complex nature. Reference is made to the investigations of various authors on the great resisting power of sucrase to the action of antiseptics.—T. H. P.

*Molasses; Fermentation of* —. E. Barbet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1901, 19, [1 and 2], 81–86.

In the fermentation of molasses, it is necessary to facilitate the multiplication of the yeast cells so that sucrase may be secreted in quantity sufficient to invert enough sugar for the fermentation. This is done by supplying the yeast with suitable food, more especially with peptones and phosphoric acid, which are conveniently added in the form of maltopeptone or of saccharified maize. The author's process which makes use of a cheaper material, viz., the dead yeast cells from a previous fermentation, is carried out as follows. The mouth of the pipe by which the fermenting vat is emptied, is surrounded by a copper collar about 7 or 8 cms. high, and after the main bulk of the liquid has flowed away, the muddy residue consisting of dead yeast cells is led by another pipe into a copper autoclave. The spirit is then distilled off, after which a quantity of sulphuric acid equal to that necessary for each fermenting vat is added to the autoclave, which is then closed and subjected to a pressure of 2 atm. By this means the whole of the yeast cells are dissolved, and the liquid is pumped to the molasses requiring denitration, which is effected by the sulphuric acid present in the peptonised mixture. The method has been tested practically, and it is found that the time occupied by fermentation is diminished from 30 to about 21–23 hours from the filling of the vat; also during the filling the attenuation is more rapid. A disadvantage attending the use of this process is the rise of temperature brought about by the vigorous fermentation which takes place. This the author obviates by using less

than the usual quantity of molasses in the vat and adding to it a proportion of the fermented liquor from a previous operation.—T. H. P.

*Cleansing Material; New* —, for Brewery Vessels. A. Ljöö and V. Törnell, Stockholm. Austr. Pat. 4122; through Woch. für Brau. 1901, 18, [36], 447.

For cleaning and removing residues of organic matter from pipes, vats, &c. in breweries, a mixture of alkali hypochlorite, and caustic soda is recommended. A sodium hypochlorite solution containing 0.32 per cent. of active chlorine will remove the slime of foul yeast from the sides of glass beakers in 5 hours, whilst a 7 per cent. caustic soda requires a day and a 5 and 2.5 per cent. soda lye does not remove it completely in 3 days. The best mixture is 1 part of sodium hypochlorite with 0.5–1 part of caustic. The strongly oxidising action of this mixture kills all organisms, whereas calcium bisulphite only attacks the outer layers.—J. F. B.

*Wine; Plastering of* —. P. Carles. Ann. Chim. anal. appl. 6, 321–327. Chem. Centr. 1901, 2, [15], 874.

The influence of an addition of gypsum in the wine-press is three-fold, physical, chemical, and physiological. The first depends upon the fact that gypsum reduces the solubility of the tartar, and on the formation of alcohol, gives rise to the separation of a crystalline powder, which acts as a filter. With vegetable albuminoids, gypsum gives precipitates, which exert a clarifying action similar to that of tannin. The chemical action of gypsum is due to the conversion of tartar into calcium tartrate, potassium sulphate, and free tartaric acid, the completeness of the reaction depending upon the total quantity of tartar present, the degree of dilution, the temperature and the percentage of alcohol. In consequence of the influence of these varying conditions, it is almost impossible to calculate the amount of gypsum which should be added, in order that not more sulphuric acid should remain than is equivalent to 2 grms. of potassium sulphate per litre. The physiological action is a consequence of the chemical one. Free tartaric acid has a favourable influence on the growth of normal wine yeast, but retards that of injurious organisms. The great disadvantage of gypsum is the production of the deleterious potassium sulphate.—A. S.

*Brandies from Damsons and Grape Marc ("Trestersbranntwein")*; Examination of —. A. Zega. Chem.-Zeit. 25, [75], 793–794.

The detection of the origin of brandies is difficult, and for the purpose the author makes use of the numbers obtained from the following determinations:—1. Estimation of free acid. 2. Estimation of alcohol in the form of ethers. 3. Estimation of alcohol, extract, ash, and sometime fusel-oil. 4. The furfural reaction, aldehyde reaction, and behaviour of prussic acid to damson or plum brandy. 5. Behaviour of the distilled brandy to N/100 permanganate solution.

*Estimation of Ether.*—To 100 c.c. of the brandy, normal potash solution is added until 1 c.c. remains in excess after neutralisation. It is then distilled to half its volume with a small flame. The flame is raised and about 30 c.c. collected. The distillate when made up to 100 c.c. serves for the estimation of alcohol and for the different reactions. The residue left in the flask is cooled, neutralised with N/10 sulphuric acid, and an excess of 10 c.c. of acid added; it is then allowed to stand for a short time and made up to 100 c.c. 20 c.c. are neutralised with N/10 potash, using phenolphthalein as an indicator and another 20 c.c. with Methyl Orange. The difference between the two titrations shows the quantity of organic acid neutralised with the N/10 potash. If the quantity of alkali used for neutralising the free acid be subtracted from this, the amount of N/10 potash is left, which is necessary for the neutralisation of the combined organic acid (ether). With grape marc brandy the relation of combined to total acid varies from 1:2.5 to 1:5.3, and the author is of opinion that the ratio should not be lower than 1:2. For damson brandy he finds a



minimum relation of 1:4 and a maximum of 1:8·4. Experimental evidence is adduced showing that the amount of free acid is always greater than combined acid in natural brandies.

The treatment of the distilled brandy with N/100 permanganate is carried out as follows:—1 c.c. of the distillate is run into a stoppered glass cylinder together with 1 c.c. of N/100 permanganate, well shaken, and allowed to stand. A similar experiment is carried out with a mixture of pure alcohol and water, and the progress of the oxidation compared. If the brandy distillate become brownish yellow the cylinder is again shaken and the odour noticed. With grape marc brandy, a smell of aldehyde is noticed; with damson brandy the characteristic smell of plum stones. More permanganate is added to the cylinders, and if the brandy be derived from grape marc, a characteristic oenanthic ethereal odour is detected, whilst the smell of the damson brandy remains unaltered. The cylinder containing the mixture of pure ethyl alcohol and water is oxidised to acetic acid.—J. L. B.

*Malt Extracts; Examination of Commercial —.*

W. J. Sykes and C. A. Mitchell.

See under XXIII., page 1148.

*Fusel Oil in Alcoholic Liquids; Determination of —.*

E. Beckmann.

See under XXIII., page 1148.

*Saccharin in Wine and Beer; Detention of —.*

T. Wirthle.

See under XXIII., page 1146.

PATENTS.

*Yeast; Treatment of —.* G. C. Marks. London.

Eng. Pat. 21,153. Nov. 22, 1900.

THE protoplasm of yeast is continuously extracted by mixing compressed or moist yeast in the cold with gum arabic, chloride of sodium, carbonate of soda or other substance possessing the property of liquefying compressed yeast. When the fermentation of the mixture is well established, fresh quantities of yeast are added from time to time, the whole being well mixed, and after each addition sufficient time allowed for the mass to ferment again. In this manner the product does not contain large traces of the substance employed for producing liquefaction.—J. L. B.

*Beer Wort; Mash Tuns for use in the Preparation of —.*

H. Debeil, Belgium. Eng. Pat. 21,355, Nov. 26, 1900.

THE mash tun has a conical bottom with a central opening for the admission of water, this opening being provided with a cover which serves as a central support for the perforated false bottom; it has feet resting on the base of the mash tun between which water can flow and spread in a sheet over the whole surface of the bottom. The outflow openings are situated in a channel formed around the conical bottom.—J. L. B.

*Beer; Production of —.* H. A. Hobson, London.

Eng. Pat. 20,819, Nov. 17, 1900.

THE production of a hopped wort is claimed, by first digesting hops in water, separating the infusion from the hops and then mashing the malt, or malt and grain using the hop extract as mashing liquor. The second claim is for removing a certain proportion of the lupulin from the hops, then making an aqueous extract and fixing the tannic acid of the hops. The abstracted lupulin is then restored to the wort or finished beer.—J. L. B.

*Fermentation Industries; Cleansing in —.* A. Sjöö and

V. Törnell, Stockholm, Sweden. Eng. Pat. 21,911, Dec. 3, 1900.

THE patentees claim the use in the fermentation industries of a mixture of 1 part alkali hypochlorite with  $\frac{1}{2}$  to 1 part of alkali hydrate.—J. L. B.

## XVIII.—FOODS; SANITATION; WATER PURIFICATION, & DISINFECTANTS.

### (A.)—FOODS.

*Bakers' Yeast; Utilisation of Brewers' Yeast and Treatment for making —.* W. Sarnighausen. Woch. für Brau. 1901, 18, [39] 485-486.

ATTENTION, of late years, has been turned to the utilisation of beer-yeast which was formerly a waste product of the brewery. The most important of these uses is for the manufacture of vegetable "meat extracts"; its richness in albumin also makes it eminently suitable for cattle food. After suitable purification, beer yeast may be employed as a substitute for distillery [top-fermentation] yeast in bread making. The impurities which must first be dealt with, consist mainly of precipitated albuminoids, hop resins, dead cells, &c.; the bitter flavour must be extracted and the yeast must be turned out in a preserved state suitable for competition with distillery pressed yeast. The washing and extraction of the bitter principles may be carried out conveniently in the same vessel. This may be improvised with a large wooden tub having a stirring shaft with a crank at the upper end and paddles at the bottom and held in position by passing through two boards fixed across the tub. The lowest paddle should just clear the bottom. A solution of ammonium carbonate is placed in the tub and the yeast, previously suspended in water is added through a hair-sieve. The vessel is then filled up with water, the colder the better, and the stirrer is run for five minutes each time at intervals. After a time, the yeast is allowed to settle, and the supernatant water is run off. For this purpose the tub is pierced with a vertical series of holes closed with pegs. The treatment with water is repeated two or three times until the yeast is clean. For the extraction of a hectolitre of thick yeast sludge, 1 lb. of ammonium carbonate is generally sufficient.

The extracted yeast after thorough washing is drained and pressed, and may then be preserved by the admixture of some harmless water-absorbent substance.—J. F. B.

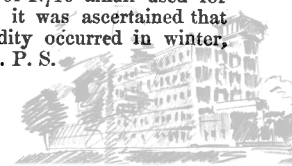
*Honey-Dextrin; Nature of —.* E. Beckman. Zeits. angew. Chem. 1901, 14, [38], 951.

THE author has examined the dextrin of honey by various methods, such as conversion into dextrinates and treatment with methyl alcohol in the presence of baryta water. It was found that basic lead acetate was also applicable as a reagent. For the determination of the molecular weight, the dextrin was converted into the benzoyl ester as in Schotten-Baumann's method. This ester can readily be obtained free from ash, and on its saponification the dextrin is converted into hemicellulose. The difficulty of determining the benzoyl group was overcome by using benzoyl sulphonic chloride in the place of benzoyl chloride. From his experiments the author concludes that honey dextrin must be assigned to the bioses.—C. A. M.

*Milk; Acidity of —.* P. Vieth and M. Siegfeld. Milch-Zeit. 1900, 29, 593-597; through Zeits. Untersuch. Nahr. und Genussm. 1901, 4, [19], 890-891.

THE authors have systematically examined a number of samples of milk from three English herds. The milk was tested after a 5 hours railway journey, and from 10 to 15 hours after milking. The results showed that in the hottest months the acidity was lowest, the maximum being reached during the first four months of the year.

The milk from other herds of cows at Hameln was also tested. The titrations were carried out with N/10 soda and N/10 baryta solutions. The acidity, as determined by the baryta solution was always about 3° higher than when titrated with soda solution. It was found that the acidity lay between 13° and 19° (using soda solution), the degrees representing the number of c.c.s. of N/10 alkali used for 100 c.c. of milk. In this instance it was ascertained that for three herds the maximum acidity occurred in winter, and for two herds, in summer.—W. P. S.



*Cherry Syrup in Raspberry Syrup ; Detection of —.*  
K. Windisch.

See under XXIII., page 1146.

*Formaldehyde in Milk ; Modification of the Sulphuric Acid Test for —.* A. G. Luebert.

See under XXIII., page 1146.

*Albuminoids ; Determination of the Decomposition Products of —.* E. Hart.

See under XXIII., page 1149.

#### PATENTS.

*Foods ; Process of Preparing Concentrated —.* C. M. Aikman, Glasgow. Eng. Pat. 17,416, Oct. 2, 1900.

SEPARATED milk is sterilised by heat, then mixed with sugar, salt, potassium bicarbonate or other alkali, and malt extract, and concentrated *in vacuo*, at a temperature not exceeding 140° F., to a volume of one-fifth to one-sixth. Either corn flour, wheat flour, malt extract, cocoa, sugar, or meat extract, and flavouring materials are then added, and the whole is carefully dried and ground to a fine powder.—L. A.

*Butter ; Manufacture of —.* M. Poppe, Bielefeld, Germany. Eng. Pat. 22,458, December 10, 1900.

BUTTER made from sterilized cream is flavoured so as to resemble butter made from sour cream, by mixing it with the volatile fatty acids obtained by saponifying genuine butter fat, and distilling the acidified soap.—L. A.

#### (B).—SANITATION ; WATER PURIFICATION.

*Sewage ; Chemical and Biological Changes occurring during the Treatment of —, by the so-called Bacteria Beds.* E. A. Letts and R. F. Blake. Paper read before the British Association (Section B), Glasgow Meeting, 1901.

THE explanation usually accepted as to the action of the so-called bacteria beds is, that the latter act as oxidising agencies, absorbing oxygen from the air during their periods of rest, and subsequently transferring it to the constituents of the sewage when the beds are filled, the transfer being mainly effected by nitrifying micro-organisms. It is found, however, that comparatively small amounts of nitrate and nitrite are produced in relation to the unoxidised nitrogen, which disappears during the treatment. For instance, the following results have been observed :—

	Manchester.	Sutton.	Leeds.
Nitrogen disappearing as "free" and "albuminoid" ammonia (grains per gallon).	1·634	7·185	1·528
Nitrogen found in the effluent as nitrate and nitrite after double contact with bacteria beds (grains per gallon).	0·636	1·100	0·11
Percentage on nitrogen disappearing.	39	15	7

In order to ascertain whether part of the unoxidised nitrogen which disappears, is lost in the gaseous state as free nitrogen, the authors made analyses of the dissolved gases present, both in the original sewage, and in the effluent from both beds of a double-contact system. They found that :—(1) Practically no oxygen was present, either in the sewage or effluents. (2) The effluent from first contact always contained considerably more carbonic anhydride than the original sewage, and with two exceptions, the effluent from second contact also contained an excess of that gas. (3) In 11 out of 12 series of analyses, the quantity of nitrogen in the effluent was in excess of that present in the original sewage, and, generally speaking it was in larger excess in the effluent from double contact than in that from single contact. On the average, the excess of nitrogen in the effluent from second contact over

that present in the sewage amounted to 0·272 part by weight per 100,000, whilst the loss of unoxidised nitrogen which had occurred in the sewage (by Kjeldahl's method) amounted to 2·2 parts, or, 12 per cent. of the nitrogen lost from the sewage during purification was thus accounted for, whilst in one particular case it amounted to 31 per cent. It is probable, too, that only a fraction of the free nitrogen actually evolved, would be retained by the effluent, the remainder escaping into the air.

The authors consider, also, that there can be no doubt that nitrogen passes into the tissues of animals and vegetables, the former of which may escape from the bacteria beds, and the latter (and possibly the former also) may remain permanently in the beds ; they have not, however, formed any estimate of the quantity so removed.—A. S.

*Bacterial Treatment of Sewage ; Humus and the So-called Irreducible Residue in the —.* S. Rideal. Paper read before the British Association (Section B), Glasgow Meeting, 1901.

AFTER referring to previous work relating to the subject, the author gives the results of the examination of a number of the humus residues from the bacterial treatment of sewage. The peaty deposit from a septic tank at Exeter had the following composition :—

	No. 1.	No. 2.	No. 3.
Distance from bottom, in inches .....	3·00	9·00	13·00
Organic matter, per cent. ....	32·35	32·40	31·31
Ash, per cent. ....	67·65	67·60	68·69
Nitrogen, per cent. ....	2·38	2·34	2·45
Percentage of N in organic matter....	7·36	7·22	7·82
Ratio of carbon to one of nitrogen ....	6·8	6·9	6·4

The author concludes that if sewage has undergone proper bacterial fermentation, the small quantity of "irreducible residue" is of the nature of humus, and practically inoffensive. Kenwood and Butler state that at Finchley in 1900, the deposit from open septic tanks could be removed with little offence, and has been spread, without nuisance, over small areas of land in the neighbourhood. It has the agricultural value of humus, and like peaty matters generally, it has a function in encouraging, when in small quantity, the nitrifying actions in the final oxidation, and itself undergoes slow oxidation to nitrates and carbon dioxide.—A. S.

#### PATENTS.

*Furnaces for the Destruction and Incineration of Ash Bin and other Refuse ; Apparatus for consuming Smoke in —.* F. Ball, Southwold, Suffolk. Eng. Pat. 17,674, Oct. 5, 1900.

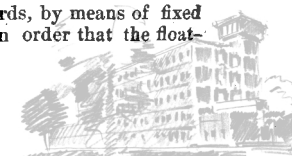
THE smoke from the furnace into which the refuse is fed passes through an arrangement of corrugated metal plates, described as a radiator or consumer, placed at the end of the grate. The radiator is at a high temperature, and is said to render the smoke innocuous. After traversing the radiator the smoke passes on to the chimney. A dust chamber and screen are provided. The air supplied to the furnace passes through flues at the side of the furnace, and is thus slightly heated, whilst the fire bars are deep and corrugated in order to heat the air, which passes between them. A high temperature is said to be thus secured in the furnace.—J. A. B.

*Refuse Destructors.* G. Watson, Leeds. Eng. Pat. 18,840, Oct. 22, 1900.

THE destructor is mounted on wheels to render it portable, and comprises a furnace supplied with air, through passages at its side, preferably by means of a forced draught produced by a boiler in which steam is generated by the products of combustion from the furnace. A smoke box and dust separator are affixed.—J. A. B.

*Sewage Works ; Impts. in —.* D. Cameron, F. J. Commis, and A. J. Martin, Exeter. Eng. Pat. 19,264, Oct. 27, 1900.

GRIT chambers are contracted upwards, by means of fixed or movable guide boards or plates, in order that the float-





ing solids may be confined within the stream of the sewage, and not be left behind in the chamber. Valves are also provided for drawing off the supernatant liquid when the grit is to be removed from the chambers, these valves being placed at the bottom of vertical pipes or wells, communicating with the chambers through openings at different levels, or through narrow vertical slots, in order that the valves may not become choked with grit.

In order to distribute the sewage effluent uniformly over the surface of large filter beds, it is conveyed through channels below the beds to vertical pipes, which rise through the material of the bed and terminate in flared castings or funnels, from which the liquid overflows on to the filter bed.—L. A.

*Liquid [Impure Water]; Purifying Apparatus.* L. Gathmann, Washington, Columbia, U.S.A. Eng. Pat. 9982, May 14, 1901.

THE object is to purify water from suspended impurities. The water is caused to pass into and through a vessel of such internal shape and construction that a vortical motion is imparted to the body of liquid, causing those impurities which are of greater specific gravity than the water or liquid to be carried inwards towards the centre of rotation, and downwards by gravitation, whilst the lighter impurities are also driven towards the centre, and caused to rise. Secondary outlets are provided at the top and bottom of the vessel for carrying away the separated impurities. Impurities having the same specific gravity as the liquid coalesce, and are also driven towards the centre of rotation, and upwards or downwards.—L. A.

#### (C).—DISINFECTANTS.

*Sulphuric Acid as a Typhoid Disinfectant.* S. Rideal. Paper read before the British Association (Section B), Glasgow Meeting, 1901.

THE author recommends sulphuric acid as a cheap and effective agent for sterilising drainage waters from an area infected with typhoid. 750 c.c. of tap water were infected with one drop of a 24-hour broth culture at 37° C. of *Bacillus typhosus*, and agar plate cultures were made with 1 c.c. of the mixture. It was found that an addition of 33 c.c. of N/10 sulphuric acid per pint was sufficient to destroy the bacilli. Allowing for the alkalinity of the sewage, an addition of 4 grms. of sulphuric acid per gallon would be sufficient to ensure the death of typhoid organisms in an infected drainage water. The author states that the free acidity produced would be speedily neutralised when the treated water became mixed with any fresh sewage which had not been similarly treated, and, as the infected sewage must always represent a very small proportion of the total drainage to be dealt with, this method of ensuring the absence of typhoid organisms in drainage waters would be satisfactory in practice.—A. S.

*Phenol, Salicylic Acid, and Salol; Determination of —, in Dressings.* F. Telle.

See under XXIII., page 1149.

#### PATENTS.

*Wash or Dip for Sheep and other Animals.* J. J. C. Roxburgh, Edinburgh, and C. F. H. Scott, St. Boswells, Roxburgh. Eng. Pat. 17,537, Oct. 3, 1900.

"SOLUBLE mineral oil" (8 galls.), strong solution of ammonia ( $\frac{1}{2}$  gall.), and heavy coal-tar oil ( $\frac{1}{2}$  gall.) are mixed together, with or without the addition of from 0.1 to 5 per cent. of arsenic trioxide.—L. A.

*Insect Life or Mildew; Compositions or Compounds for Use in Agriculture or Horticulture, for Eradicating —.* G. H. Richards, London. Eng. Pat. 20,194, Nov. 9, 1900.

SALICYLIC acid or tartaric acid are dissolved in nicotine, and the crystalline product is mixed with camphor and compressed into cakes or tablets, which are used for fumigating.—L. A.

*"Antiseptic or Detergent."* C. Pilling, Liverpool. Eng. Pat. 10,549, May 21, 1901.

POTASSIUM-SODIUM-AMMONIUM metaborate is prepared by dissolving one equivalent, or 112 parts, of potassium hydroxide in 150–200 parts of "boiling water," stirring in one equivalent, or 382 parts, of crystallised borax, and, when dissolved, adding 30 parts of ammonia solution (0.880). The solution is allowed to crystallise. The addition of ammonia is said to prevent the potassium and sodium metaborate from absorbing atmospheric carbon dioxide.—L. A.

#### XIX.—PAPER, PASTEBOARD, Etc.

*Cellulose; The Mixed Esters of —, and the Reactions of Cellulose with Nitrating Acid.* C. F. Cross, E. J. Bevan, and R. L. Jenks. Ber. 1901, 34, [12], 2496.

THE authors have investigated a number of mixed esters of cellulose, such as acetobenzoates, nitrobenzoyl nitrates, and conclude from the result that, under treatment with mixtures of nitric and sulphuric acids, the cellulose hydroxyls would react with both negative radicles yielding nitrosulphuric esters. This has been verified. The maximum fixation of sulphuric acid so far obtained, was 4 to 6 parts with bleached cotton, and 1.9 to 4.2 parts with unbleached cotton. These percentages are calculated to the weight of the original cellulose. The essential condition, most favourable to the retention of the sulphuric residues in combination, was found to be short duration of exposure to the action of the mixed acids. The products were variously and exhaustively purified, and the removal of the residual acids was carefully followed as a necessary precaution against possible error.

The presence of the mixed esters in the products purified by exhaustive washing was verified by treatment with diluted acetone, in which these products are soluble.

The investigations, of which this is a preliminary account, are being prosecuted in the direction of isolating the mixed esters in a form available for analysis.—C. F. C.

*Hydrocellulose; Production of —.* (Ger. Pat. 123,121, March 2, 1900), R. Sthamer, Hamburg. Zeits. angew. Chem. 1901, [36], 905.

A DIFFERENT hydrocellulose from that obtained by the process described in the abstract following is produced by immersing the crude cellulose in glacial acetic acid containing free chlorine, and warming the mixture to a temperature of 60°–70° C. with stirring. The product furnishes acetyl derivatives, distinguishable from the known similar compounds by their greater solubility in water and in alcohol.

—C. S.

*Hydrocellulose; Method of Producing —.* (Ger. Pat. 123,122, March 2, 1900), R. Sthamer, Hamburg. Zeits. angew. Chem. 1901, [36], 905.

CELLULOSE is treated with hydrochloric acid, at a temperature of 60°–70° C., in presence of a quantity of potassium chlorate insufficient to produce conversion into oxycellulose.

—C. S.

#### PATENTS.

*Paper, Paper Board, or like Cellulose or Fibrous Material; Preparation or Treatment of —, for Embossing and other purposes.* A. H. Martin, R. E. Tinker, and T. D. Ryan, all of Buffalo, U.S.A. Eng. Pat. 8643, April 26, 1901.

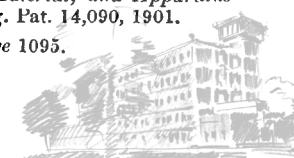
THE process claimed, consists in treating paper or similar material with a mixture of glue, sugar, or glucose, white or red lead, a filler (talc) and glycerin, drying, heating in order to make the material pliable, and then subjecting to pressure in order to produce the desired effects.—A. C. W.

*Celluloid Compounds and High Explosives; Manufacture of —.* J. E. Blomén. Eng. Pat. 9984, May 14, 1901.

See under XXII., page 1140.

*Drying of Fibrous and other Material, and Apparatus therefor.* H. H. Lake. Eng. Pat. 14,090, 1901.

See under I., page 1095.



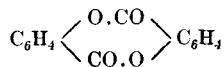
## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

*Bismuth Salicylate; a New Crystalline* —. P. Thibault. Bull. Soc. Chim. **25**, [16–17], 794–796.

THE author states that previous methods of obtaining bismuth salicylate, viz., the precipitation of aqueous or glycerin solutions of bismuth nitrate with sodium salicylate, yield products which are not uniform and contain nitric acid in the proportion of about 6 per cent. Causse's method, in which freshly precipitated bismuth oxychloride is treated with an alkaline solution of sodium salicylate, yields a product which contains a considerable proportion of chlorides, even after much washing, whilst on continued washing, its crystalline character disappears, and salicylic acid is lost. The author recommends the following method for obtaining a pure crystalline product:—15 grms. of crystallised bismuth nitrate in nitric acid solution are precipitated with excess of caustic soda or potash. On boiling, all the hydrated oxide is converted into the yellow crystalline anhydrous oxide. This is washed completely, and 10 grms. of salicylic acid, mixed with 200 c.c. of water, is added and left in a water bath until the reaction is complete, decanted whilst warm, washed with cold alcohol, and dried on the water-oven. A greyish red crystalline powder results, formed of small transparent prisms. Water decomposes it slowly in the cold, more rapidly when warm; water saturated with salicylic acid is without action, also ether and cold alcohol; but when treated with warm alcohol salicylic acid is liberated. It commences to decompose at 140° C. with production of phenol and blackening. Cold and hot acids separate salicylic acid, and give the corresponding bismuth salt; alkalis separate salicylic acid, and leave bismuth oxide. The salt corresponds to the formula  $(C_7H_5O_3)_3Bi_2O_3$ .—C. T. T.

*Disalicylide*. A. Einhorn and H. Pfeiffer. Ber. **34**, [12], 2951–2953.

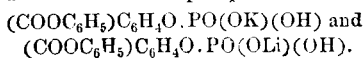
On attempting to obtain the  $\beta$ -lactone of salicylic acid by the action of phosgene gas on that acid in solution in pyridine the authors isolated, instead, disalicylide.



which is formed to the extent of about 12 per cent. with a mixture of amorphous anhydrides of salicylic acid. From these it is separated by recrystallisation, first, from glacial acetic acid, and, finally, from chloroform. It is thus obtained in prismatic needles, melting at 200°–201° C. It distils without decomposition, and is but slightly soluble in alcohol, more soluble in benzene, chloroform, and glacial acetic acid. It is not soluble in dilute soda solution, and only slightly dissolved by prolonged contact with strong solution of caustic soda. Heated with a molecular proportion of phenol to 200° C. for a few hours, it yields salol; when treated in a similar manner with  $\beta$ -naphthol, or with guaiacol,  $\beta$ -naphthol-salol and guaiacol-salol are formed, the latter separating from solution in absolute alcohol in small, brilliant crystals, melting at 70° C. The same method applied to other *o*-hydroxy-acids of the aromatic series, and to alcohol-acids, has been found by Mettler to yield other dimolecular anhydrides. Thus, from cresotinic acid the *o*-, *m*-, and *p*-cresotides melting at 231.5°, 207.5°, and 245.5° C., respectively, were obtained; and from  $\alpha$ -hydroxy-isobutyric acid, mandelic acid, and benzoic acid corresponding dimolecular anhydrides were isolated, which will ultimately be described.—J. O. B.

*Salvosol-Potash and Salvosol-Lithium*. Pharm. Zeit. **46**, [70], 694.

THESE two medicinal preparations are combinations of the respective alkalis with salol-*o*-phosphinic acid—



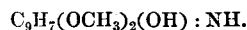
They are obtained by the action of the alkali on salol-*o*-phosphinate. Both are crystalline. The potassium compound is soluble to the extent of 1:20 in cold water. It is decomposed by heat, salol and salicylic acid being liberated. Salol-*o*-phosphinic acid is liberated on warming with dilute mineral acids. On heating with strong alkali, salicylic acid, phosphoric acid, and phenol result. The lithium salt is more soluble, and, on heating, evolves the odour of phenol without precipitating.—J. O. B.

*Cactus Alkaloids*.—IV. A. Heffter. Ber. 1901, **34**, [12], 3004–3015. (See also this Journal, 1895, 384; 1896, 554, 827; 1898, 688.)

FOUR alkaloids—mezcaline, anhalonidine, anhalonine and lophophorine—have hitherto been found in the “mescal button,” *Anhalonium Lewinii*. Kauder has also isolated from the drug anhalamine and pelletine, the latter previously found by the author in *Anhalonium Williamsii*. The author now confirms Kauder's results, but does not regard it as proved that pelletine is a constituent of *A. Lewinii*. *A. Williamsii*, the dry plant of which contains 3.5 per cent. of the alkaloid, is so similar to *A. Lewinii*, that the presence of pelletine equivalent to 0.2 per cent. of the hydrochloride would be easily explained by the presence of the former plants among the mescal buttons.

*Anhalamine*,  $C_{11}H_{13}NO_3$ , is obtained by adding ammonia, after crystallising out the bulk of the mezcaline sulphate, and shaking with chloroform. At the first extraction the solvent is found to contain spherical jelly-like lumps, which are filtered off, washed with a little chloroform, and pressed between paper. Though this appearance does not again occur, many extractions are required to remove the whole of the alkaloid. It is purified by repeated recrystallisation from hot absolute alcohol, when it forms spherical aggregates of microscopic needles, melting at 185.5° C. It is soluble in hot water, from which it crystallises in needles. The hydrochloride crystallises by slow evaporation from water in thin shining leaflets containing 2H<sub>2</sub>O. The chloroplatinate forms flat yellow needles, little soluble in cold water. The aqueous solutions of the salts give a blue coloration with ferric chloride, which turns green on heating and then disappears. Pelletine and anhalonidine give the same reaction, but not mezcaline, anhalonine, and lophophorine, which contain no hydroxyl group.

Anhalamine contains two methoxyl groups. When dissolved in caustic soda it gives, by the action of benzoyl chloride, benzoylanhalamine benzoyl ester,  $C_{11}H_{13}O_3N(COC_6H_5)_2$ , and benzoylanhalamine,  $C_{11}H_{13}O_3N.CO.C_6H_5$ . The alcoholic solution of the latter gives a blue coloration with ferric chloride, and the compound is soluble in caustic soda; the benzoyl group has therefore apparently taken the place of an imide hydrogen atom. Thus anhalamine appears to be a secondary base of the formula—



*Mezcaline*.—It is now found that the base is an oily liquid, which very readily absorbs carbon dioxide, forming a crystalline carbonate. By oxidation of a 1 per cent. solution of the sulphate by potassium permanganate, the trimethyl ether of gallic acid is produced. The methyl iodide compound of the base, when shaken with chloroform, gives a crystalline base, methylmezcaline, which again unites with methyl iodide. A solution of mezcaline in caustic soda, when shaken with benzoyl chloride, gives at once benzoylmezcaline,  $C_{11}H_{16}O_3N.CO.C_6H_5$ . Mezcaline is thus a secondary base; its constitution is represented by the formula  $CH_3.NH.CH_2.C_6H_4.(OCH_3)_3$  [1:3:4:5]. Dibromomezcaline was obtained by the action of bromine water on mezcaline or its hydrobromide suspended in water.

*Anhalonidine*.—When treated in alkaline solution with much benzoyl chloride, two benzoyl compounds were obtained similar to those produced by anhalamine. Methyl-anhalonidine hydriodide,  $C_{13}H_{17}O_3N.HI$ , is obtained by warming the base with 1 mol. of methyl iodide. When 2 mols. of methyl iodide are used, methylanhalonidine methyl iodide is also formed. Thus anhalonidine is a



secondary base; no nitroso compound could be obtained, since the alkaloid is decomposed by sodium nitrite in acid solution. The formula is thus  $(\text{CH}_3\text{O})_2(\text{OH})\text{C}_{10}\text{H}_7:\text{NH}$ . Oxidation and bromination experiments gave no useful result.—A. C. W.

*Mandragora Root; Alkaloids of* — O. Hesse. J. prakt. Chem. 1901, **64**, [15, 16, 17, 18], 274—286.

WENTZEL had stated that the base accompanying hyoscyamine in the mandragora root was a hyoscyne of the formula  $\text{C}_{17}\text{H}_{23}\text{NO}_3$  (Dissertation, 1900). This statement was corrected by Thoms and Wentzel (this Journal, 1901, 605). The author has experimentally examined Wentzel's research: 20 kilos. of dry Trieste mandragora root were moistened with soda solution (only the alkaloids separated by soda were in question, hyoscyne in particular), and extracted by ether under the reflux condenser. The ethereal solution was repeatedly extracted with dilute sulphuric acid, the acid solution made alkaline with sodium bicarbonate and extracted with chloroform, which took up some hyoscyamine. The bases were therefore again dissolved in sulphuric acid, and the solution treated with sodium bicarbonate and chloroform as before. The hyoscyamine now remained in the aqueous solution. The syrupy bases from the chloroform solution were neutralised with hydrobromic acid, and the filtered solution brought to crystallisation. The hydrobromides were extracted with acetone, which left the pure hyoscyne salt,  $\text{C}_{17}\text{H}_{21}\text{NO}_4 \cdot \text{HBr}$ , melting at  $194^\circ \text{C}$ . (Wentzel gave  $197^\circ \text{C}$ ). The author did not observe the rise in the melting point of the gold salt, from  $198^\circ$  to  $206^\circ \text{C}$ ., given by Wentzel. The base is decomposed by baryta into tropic acid and oscine,  $\text{C}_8\text{H}_{13}\text{NO}_2$ .

The mother liquors of the hyoscyne hydrobromide yielded pseudohyoscyamine on the addition of sodium bicarbonate and extraction with chloroform; the solution then, after addition of normal sodium carbonate, yields a new base, mandragorine, which forms the gold salt,  $\text{C}_{15}\text{H}_{19}\text{NO}_2 \cdot \text{HAuCl}_4$ . Mandragorine is decomposed by baryta into atropic acid and (probably) tropine.

The yields of the different alkaloids were: hyoscyamine 0.36, hyoscyne 0.04, pseudohyoscyamine 0.01 per cent., and a still smaller quantity of mandragorine.—A. C. W.

*Acetyltropic Acid.* O. Hesse. J. prakt. Chem. 1901, **64**, [15, 16, 17, 18], 286—288.

ATROPINE and hyoscyamine are converted into apo-atropine by the action of acetic anhydride, the tropic acid radical being transformed into the atropic acid radical. Acetyl chloride has no action on these bases. Acetic anhydride and acetyl chloride convert hyoscyne into an acetyl compound. The author now finds that tropic acid gives an acetyl compound,  $\text{C}_9\text{H}_9\text{O}_4(\text{C}_2\text{H}_5\text{O})$ , by the action of acetic anhydride. Acetyltropic acid forms white leaflets, which melt at  $80^\circ \text{C}$ .; it is readily soluble in ether, alcohol, acetone, and chloroform, less soluble in benzene. It is almost insoluble in cold water, and little soluble in hot, from which it separates as an oil, which crystallises only after many days. A solution of acetyltropic acid in ammonia gives with silver nitrate a turbidity which soon disappears.—A. C. W.

*Tropidine; Conversion into Tropine.* R. Willstaetter. Ber. **34**, [12], 3164—3165.

By heating tropidine hydrobromide to  $200^\circ$ — $210^\circ \text{C}$ . for three hours in a sealed tube with six times its weight of 10 per cent. sulphuric acid, the author has obtained a mixture of  $\psi$ -tropine and tropidine, which, on distillation and recrystallising from ether or from benzene, gave 24 per cent. of the theoretical amount of  $\psi$ -tropine boiling at  $240^\circ$ — $241^\circ \text{C}$ . (corr.), and melting at  $108^\circ$ — $108.5^\circ \text{C}$ . (corr.). Since this alkaline can be converted by oxidation into tropinone, and this ketone yields on reduction, geometrically isomeric tropine, the last step in the synthesis of the solanaceous alkaloids, atropine, atropamine, belladonnine, and the coca alkaloids, tropacocaine and racemic cocaine, has been accomplished.—J. O. B.

*Purine Derivatives; Comparative Experiments on the Pharmacological Action of* — O. Schmiedeberg. Ber. **34**, [12], 2550—2559.

THE pharmacological action of pure purine and some of its derivatives are compared in this paper with the results obtained with caffeine and theobromine.—J. O. B.

*Morphenol; Preparation of* — E. Vongerichten. Ber. 1901, **34**, [12], 2722—2723.

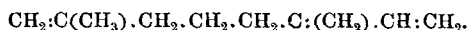
A SIMPLER method than that described in this Journal, 1900, 283, for the preparation of morphenol, consists in heating methylmorphimethine methyl iodide with alcoholic potash, at  $160^\circ \text{C}$ . The product of the reaction is poured into dilute sulphuric acid, extracted with ether, the ethereal extract taken up with dilute caustic soda, the solution filtered, and morphenol precipitated by sulphuric acid in grey crystalline flocks. The yield is 70 per cent. of the theoretical.

When treated in a similar manner dihydromethylmorphimethine methyl iodide gives trimethylamine and a compound free from nitrogen. By the same reaction Skraup and Wiegmann obtained ethylmethylamine from morphine and codeine, and ethyldimethylamine from codeine methyl iodide.—A. C. W.

*Terpene Series; Reduction in the* — Myrcene and other Olefinic Compounds. F. W. Semmler. Ber. 1901, **34**, [12], 3122—3130.

IN the reduction of unsaturated terpene ketones by alcohol and sodium, a double bond is also reduced only if it is in a neighbouring position to the ketone group. Thus carvone gives dihydrocarveol; carvenone and carvotanacetone give tetrahydrocarveol; isothujone gives thujamenthol and pulegone gives menthol. No cyclic terpene is reduced by alcohol and sodium, whence the author concludes that no cyclic terpene hitherto known contains the two double bonds in neighbouring positions.

Myrcene is an olefinic terpene, discovered by Power and Kleber in Bay oil. It is reduced by ethyl alcohol and sodium to dihydromyrcene, hence it contains two double bonds in neighbouring positions. Both myrcene and dihydromyrcene add four atoms of bromine. By the action of glacial acetic and sulphuric acids, dihydromyrcene is converted into cyclo-dihydromyrcene, which is very similar to cyclo-linaloolene. The cyclic compound only forms a dibromide. Myrcene yields succinic acid on oxidation, whilst dihydromyrcene forms levulinic acid and a ketoglycol,  $\text{C}_8\text{H}_{16}\text{O}_3$ , which is further oxidised to a  $\delta$ -diketone,  $\text{C}_7\text{H}_{12}\text{O}_3$ . Acetone is also formed. The most probable formula for myrcene appears to be—



Power and Kleber treated myrcene by Bertram's method in order to convert it into an ester and thence into an alcohol,  $\text{C}_{10}\text{H}_{18}\text{O}$ . Barbier has recently obtained an alcohol of this formula. The author has repeated these researches, and finds that the products are not individual substances: the myrcene is partially inverted.—A. C. W.

*Vanillin; Preparation of* —, from Protocatechuic Aldehyde. (Ger. Pat. 122,851, May 22, 1900.) R. Sommer, Vienna. Zeits. angew. Chem. 1901, [36], 905.

THE characteristic feature of this method is the employment of dimethyl sulphate as the methylating agent, with or without the presence of alkalis.—C. S.

*Ethoxy - Isoeugenol (Propene - Pyrocatechin - Oxyethyl Ether); Preparation of* — C. Pommeranz. D.R.P. 122,701, Jan. 3, 1900. Through Oesterr. Zeits. für Pharmacie, **55**, [34], 818.

ONE hundred grms. of safrol are heated to  $150^\circ \text{C}$ . for 20 hours with 400 grms. of 25 per cent. ether-alcoholic solution of caustic potash, the temperature being carefully maintained between  $140^\circ$  and  $150^\circ \text{C}$ . during the whole period. The reaction product is then diluted with four times its volume of water, shaken out with ether, the ethereal solution removed, the aqueous layer rendered faintly acid with  $\text{H}_2\text{SO}_4$  and the liberated ethoxy-isoeugenol removed by



again shaking out with ether. The ethereal solution is dried with anhydrous sodium sulphate, the ether distilled off, and the residue fractionated *in vacuo*. Thus obtained, ethoxy-isoeugenol is a colourless, almost odourless liquid, boiling at about 172° C. at 22 mm. pressure. Its alcoholic solutions are coloured green with ferric chloride. It is applicable as a medicinal agent, and for the preparation of [synthetic] perfumes.—J. O. B.

*Citronellal; Constitution of* —. C. Harries and O. Schauwecker. Ber. 1901, **34**, [12], 2981—2991. (See also this Journal, 1900, 769.)

CITRONELLAL dimethylacetal, by oxidation with permanganate in acetone solution, gives 80 per cent. of a glycol, which, on further oxidation with chromic acid, gives a dialdehyde,  $C_{10}H_{18}O_2$ , and a ketoaldehyde,  $C_9H_{16}O_2$ . The latter must be formed by further oxidation of the former. It then follows that citronellal has the formula—



—A. C. W.

*Storax; Oriental* —. A. Tschirch and L. van Itallie. Archiv **239**, [7], 506—532.

THE constituents of liquid oriental storax (derived from *Liquidambar orientalis*) are: Free cinnamic acid, vanillin, styrol, styracin, ethyl cinnamate, and storesinol, partly free and partly as ester. No benzoic acid was detected. Storesinol,  $C_{16}H_{26}O_2$ , occurs as a white, odourless, aggregating powder, melting at 156°—161° C. It is insoluble in light petroleum spirit, but dissolved by most other solvents, and by dilute alkali. It is an isomer of benzoiresinol, but has a lower melting point than that substance. It forms a crystalline compound with potassium hydrate, separating in acicular crystals. When fused with alkali, acetic and salicylic acids are formed. On distillation with zinc dust it yields phenol, toluene, and benzene; dry distillation alone gives these bodies as well as cresol, and apparently phenylacetylene. By the action of bromine, brominated amorphous derivatives are obtained. Storesinol does not acetylate, nor does it form a benzoyl compound, nor give any nitrogenous compound with hydroxylamine and phenylhydrazine. Oxidised with nitric acid, it gives picric and oxalic acids. The existence of methoxyl or ethoxyl groups could not be proved.—J. O. B.

*Storax, American*; —. A. Tschirch and L. van Itallie. Archiv **239**, [7], 532—540.

THE constituents of American storax (derived from *Liquidambar styraciflua*) are very similar to those of Oriental storax, consisting of cinnamic acid, vanillin, styrol, styracin, cinnamic phenyl-propyl-ester, and styresinol, partly free and partly in the form of the cinnamic ester. No ethyl cinnamate was, however, detected. Styresinol is apparently an isomeride of storesinol, having the same formula,  $C_{16}H_{26}O_2$ , and the same melting point, 261°—162° C. but differing in optical activity, its specific rotation being +13° 39' as compared with +52° for storesinol. In all other respects, both in its combinations and decomposition products, it is identical with storesinol.—J. O. B.

*Lemon Oil; New Aldehydes of* —. H. von Soden and W. Rojahn. Ber. 1901, **34**, [12], 2809—2811.

THE appearance of the paper by Burgess (this Journal, 1901, 745) compels the authors to publish their work whilst still unfinished. Lemon oil was freed from citral by shaking with sodium monosulphite, and sodium bicarbonate (Tiemann, this Journal, 1899, 169). The unattacked oil was distilled *in vacuo* and shaken with acid bisulphite solution. The product after washing with petroleum spirit, gave a mixture of aldehydes on adding soda and distilling with steam. The high boiling compounds were separated by distilling *in vacuo*; the distillate was then fractionated. The fraction boiling at 185°—190° C. appears to be a mixture of octyl and nonyl aldehydes. The latter, which is more abundant, gives a semicarbazone melting at 85° C.; the octyl aldehyde semicarbazone melted at 72° C. From 10 kilos. of lemon oil 10—15 grms. of aldehyde were obtained. Burgess apparently regards the aldehyde, which he found in lemon oil, as identical with the aldehyde of orange oil. The latter is, however, normal decyl aldehyde, according to Stephan (this Journal, 1901, 275).

The fluorescent substance found by Burgess, which is doubtless identical with the compound previously found by Crismer and by Theulier, is not the only fluorescent compound in lemon oil. On shaking with bicarbonate solution, acidifying and extracting with ether, small quantities of acids volatile and non-volatile with steam are obtained, of which the latter are fluorescent. The higher boiling fractions of lemon oil, containing oxygen compounds, also give a slight blue fluorescence, which may be due to methyl anthranilate.—A. C. W.

*Rose Oils; Occurrence of Phenylethyl Alcohol in* —. III. H. von Soden and W. Rojahn. Ber. 1901, **34**, [12], 2803—2809. (See also this Journal, 1901, 65; 1900, 768, 1036, 1037.)

THE authors have utilised this year's harvest of roses at Schladebach to determine the amount of phenylethyl alcohol in the flowers and the proportion of this constituent to the total volatile oil; also to compare the yields from petals and calyx. The principal result may be at once stated: phenylethyl alcohol is, in quantity, the chief constituent of the perfume of the rose; the amount obtained by distillation is 5—6 times as great as the quantity of the commercial pure rose oil.

Experiments 1—3 were made with flowers plucked on June 20; experiment 4 was performed 2½ weeks later. In the distillation, the fresh material was mixed with its own weight of water in the apparatus and about twice its weight of distillate collected (in the case of fresh calyces, 4—5 times the weight). In distilling the dried petals and calyces, the water lost in drying was also added. The total crude oil was extracted from the distillate by ether, it contains a large proportion of phenylethyl alcohol and since this has only a weak odour, the value of this oil is less than that of the ordinary commercial oil. The chief quantity of oil is contained in the petals, but the oil from the calyx is of some importance to the aroma of the product. The yields and properties of the crude oils are given in the table below. The yields of oil are somewhat too low, since the volatile oil was still present in the contents of the still.

	Experiment 1. 50 kilos. of Fresh Flowers.	Experiment 2. 50 kilos. of Fresh Flowers gave		Experiment 3. 50 kilos. of Fresh Flowers gave when dried		Experiment 4. 50 kilos. of Fresh Flowers gave	
		a. 41.5 kilos. of Petals.	b. 8.5 kilos. of Calyces.	a. 9.5 kilos. of Dry Petals.	b. 2.5 kilos. of Dry Calyces.	a. 40.5 kilos. of Petals.	b. 9.5 kilos. of Calyces.
Extracted crude oil .....	37.50 grms.	28.00 grms.	2.00 grms.	9.50 grms.	2.50 grms.	23.50 grms.	..
Sp. gr. at 30° C. ....	0.944	0.946	0.897	..	0.894	0.946	..
Rotation in 100 mm. tube .....	0° 17' at 31° C.	0° 40' at 34° C.	..	..	..	0° 8' at 30° C.	..
Solidified at .....	28° C.	29° C.	..	..	..	25° C.	..
Acid value .....	3.4	3.0	..	..	..	5.5	..
Ester value .....	19	16	..	..	..	17	..

The crude oils were separated into their constituents by adding water and distilling with steam. When no more "essential oil" was observed to pass over, the receiver was

changed, and distillation continued until the condensed water was almost odourless. The residue consisted almost entirely of paraffins. The whole of the distilled water was



mixed with 10 per cent. of 35 per cent. caustic soda lye, allowed to stand, filtered, and shaken with ether. After evaporation of the solvent, almost pure phenylethyl alcohol remained. The acid constituents (phenols, acids, lactones, &c.) were extracted from the alkaline liquor after acidifying. The "essential oils" obtained directly by distillation of the crude oils were similar in properties to ordinary rose oils (the oil from dried petals had, however, a certain

unpleasantness of odour). The oils from experiments 1 and 4a still contained phenylethyl alcohol, which was removed by distilling in steam, adding caustic soda, &c., as before. This method for isolating the alcohol is more convenient than that previously described.

The properties and approximate composition of the crude oils are given in the following table:—

	Experiment 1.	Experiment 2a.	Experiment 3a.	Experiment 4a.
"Essential oil," per cent.....	10	10	20	27
Sp. gr. at 30° C.....	0·845	0·826	0·837	0·866
Solidifying point.....	23° C.	27° C.	26° C.	18°—19° C.
Phenylethyl alcohol, per cent.....	60	57	50	54
Acid constituents, per cent.....	7·5	8	15	3
Residue (mainly paraffin), per cent.....	17·5	15	10	3
Loss per cent.....	5	0	5	13

The yields of "essential oil" were very different in experiments 2a and 4a, which are comparable; it is well known that the yield of rose oil during the same harvest depends largely on conditions of weather, moistness of soil, &c.

The author calls attention to the enormous loss of phenylethyl alcohol in the distilled water, in Bulgaria alone 2,000—4,000 kilos. per annum. Possibly a portion is used in the form of rose water.

Benzyl alcohol has been found in the aqueous distillate produced in preparing ylang-ylang oil in Manila.

—A. C. W.

*Jasmine Flowers; Essential Oil of* — VI. A. Hesse. Ber. 1901, 34, [12], 2916—2932. (See also this Journal, 1899, 296, 396, 513, 1159; 1900, 770; 1901, 275, 289.)

The author first replies to E. Erdmann (this Journal, 1901, 930). The sample of "jasmine pur" previously examined was again tested; not a trace of methyl anthranilate was found. A new sample from the same harvest was also examined, and it was found from this and other experiments that the ester (methyl anthranilate) was not present in appreciable quantity in pure extracts.

In regard to the production of oil during *enfleurage*, the solvent property of fats for the oil hardly comes into question, but rather their property of absorbing perfumes. The difficult solubility of benzyl alcohol in petroleum spirit is unimportant; jasmine oil and benzyl acetate, its principal constituent, are very readily soluble, and, in any case, 10,000—15,000 parts of solvent are used for one part of the oil in the blossoms. It is thus inconceivable that only 10 per cent. of the oil is extracted (see also the distillation experiments below). Erdmann proposes comparative experiments by *enfleurage* and maceration; in the former process the fat is treated with the blossoms 20—30 times on as many days; the macerations would have to be conducted on the same days with the same material. For technical reasons the experiment is practically out of question, and less oil would be obtained than by extraction with petroleum spirit, since, under these conditions, the fat is a worse solvent.

It is somewhat generally supposed that jasmine flowers yield no essential oil on distillation, the perfume being assumed to be destroyed. In fact, certain constituents of essential oils (esters of terpene alcohols) may be decomposed, but the constituents of the jasmine oils obtained by extraction and *enfleurage* are so stable that they cannot be appreciably decomposed in distillation. At the time the pomade previously examined (this Journal, 1901, 275) was made, 650 kilos. of fresh blossoms were distilled, producing a few grammes of oil and 620 kilos. of water. The latter gave, after addition of common salt and extraction with ether, 152 grms. of a brown oil which, on distillation with steam, yielded 127 grms. of volatile oil (inclusive of oil extracted from condensed water). The yield is thus 0·0194 per cent., by far the greater quantity being extracted from the condensed water. The oil was examined for methyl anthranilate and indole in the manner previously described; the proper fraction was also examined for the latter constituent; certainly far less than 0·2 per cent. is present.

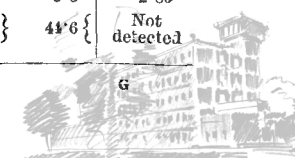
Less than 1 per cent. of indole is difficult to detect: 5 drops of the oil are mixed with the least possible excess of picric acid, and the picric acid or the picrate dissolved by means of a few drops of alcohol. On evaporation of the alcohol the small red needles of the picrate separate distinctly. The benzyl acetate was estimated as before (this Journal, 1899, 513). The saponification value of the acetylated oil showed that 8—10 per cent. of other free alcohols must be present, together with about 12 per cent. of free benzyl alcohol.

The distilled oil contains the high proportion of 1·5 per cent. of methyl anthranilate, whilst the oil extracted from fresh flowers is free from this substance, and that obtained by *enfleurage* contains 0·24—0·3 per cent. On the other hand, both distilled and extracted oil contain no indole (or a very small quantity), whilst the ten times greater quantity of oil produced by *enfleurage* contains 2·5 per cent. In order to ascertain whether storage in an open space would increase the proportion of the oil or alter the quantities of methyl anthranilate and indole, 80 kilos. of fresh blossoms were kept 24 hours and then distilled with steam; the oil was then extracted from the condensed water as described above. The yield was 0·0162 per cent. of rectified oil, containing 41·3 per cent. of benzyl acetate. The oil contained no indole; the quantity was insufficient to test for methyl anthranilate.

In *enfleurage* the glass plates of the "châssis" are covered on both sides with a thin layer of fat, upon which the jasmine flowers are spread. The plates are then placed one upon the other, thus forming a number of spaces with layers of fat above and below, by which the expired perfume of the flowers is absorbed. After 24 hours the flowers are removed from the layer of fat; they are then used as manure. 150 kilos. of these used flowers, corresponding to 500—600 kilos. of fresh blossoms, were distilled with steam, and the total oil isolated as before. Indole could not be detected in it. It was found that about as much oil was contained in the used flowers as in the corresponding quantity of fresh flowers. Thus the previous statement as to the production of oil during *enfleurage* is confirmed.

The following table contains the quantities of oil contained in 1,000 kilos. of flowers, as shown by extraction and distillation, and produced by the same quantity in *enfleurage*, together with the composition of the different oils, so far as it was determined:—

Process.	Extraction.	Distillation.	Enfleurage.	
			Absorbed by the Fat.	Remaining in the Flowers.
	Grms.	Grms.	Grms.	Grms.
Yield, containing	178	194	1,784	195
Jasmine.....	5·7	6·2	53·5	6·2
Benzyl acetate..	35·6	44·2	1,248·8	68·2
alcohol.....	21·3	23·2	107·0	34·3
Methyl an- thranilate....	Not detected	2·87	5·3	2·86
Indole.....	Not detected	Not detected	44·6	Not detected



If the indole were a product of the decomposition of the vegetable albumin, it would be found in the stored flowers and in the used flowers from *enfleurage*. It may thus be a constituent of the expired perfume alone; experiments are in process to collect the perfumes expired by living and by plucked flowers.

Methyl anthranilate is obtained by distillation and by *enfleurage*, but not by extraction; it is apparently produced during the two former processes by the decomposition of some more complicated compound.

In view of the different oils obtained by different processes from the same flower, the author proposes to call the oil contained in jasmine flowers and produced by distillation or extraction—"essential oil of jasmine," and that developed by the flowers in *enfleurage*—"essential oil of jasmine flowers."—A. C. W.

*Orange Flowers; Essential Oil of* —. I. A. Hesse and O. Zeitschel. *J. prakt. Chem.* 1901, **64**, [15, 16, 17, 18], 245–260.

ACCORDING to Passy (*Comptes Rend.* **124**, 783; this *Journal*, 1897, 631) blossoms from which perfumes are obtained may be divided into two classes—flowers containing no essential oil, but producing perfume so long as they live (jasmine, tuberoses), and flowers which contain perfumes stored in oil cells (roses, orange blossom). A. Hesse has shown that jasmine flowers produce very large quantities of oil during life, but that they do contain small amounts of oil (see preceding abstract). Thus *enfleurage* is the only rational method of obtaining jasmine oil.

Orange flowers contain the oil in cells, which may be perceived by the naked eye, thus the various processes by which the oil is extracted give comparative results, if properly carried out. The authors have compared these processes. The crude oils were rectified in steam in order to remove resins, paraffins, and other non-volatile substances. The oil floating on the distilled water, termed "separated oil," was removed and dried over sodium sulphate; the oil dissolved and suspended in the water, termed "water oil," was collected by salting out and extracting with ether, the

ethereal solution was then dried over sodium sulphate, the solvent distilled off and the last traces removed by means of a current of air *in vacuo* at 40° C. The "separated oil" and "water oil" mixed in the proper proportions give the rectified "mixed oil" of the corresponding crude oil.

*Distillation*—either with steam or with water over an open fire—is the most usual process. The products are Néroli oil and orange flower water. The latter reproduces the odour of the flowers much better than the former. The results of distillations conducted by Pillet Frères at Grasse in May 1900, working on fresh flowers and blossoms which had been kept 24 hours, are given in the table. It appears that about one third (containing the most valuable constituents) of the perfume in the blossoms passes into the orange-flower water.

*Maceration* consists in extracting the blossoms with hot fat at 60°–70° C. For the purpose of this investigation about 50 kilos. of pomade were prepared from 510 kilos. of blossoms, without any other addition. The essential oil was isolated by the method previously given (this *Journal*, 1899, 396). The pressed flowers, after the maceration, are generally used as manure; the authors find, however, that they still contain large quantities of the valuable odoriferous substances; quantitative results are not yet available.

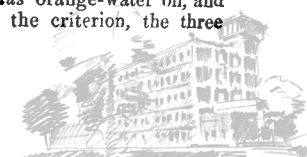
*Extraction* by means of a volatile solvent gives very good results. The authors examined a sample obtained from Grasse with every guarantee of purity. The commercial product, the first extract of the orange blossoms, contains about 50 per cent. of non-volatile, odourless substances (paraffins, vegetable wax, &c.). In order to study the behaviour of orange blossoms in the *enfleurage* process, 86 kilos. were subjected to *enfleurage à froid*; 8.5 grms. of oil were obtained.

The table gives the yields and properties of the various oils. The percentage of esters deduced from the saponification value is given in column 8 calculated as linalyl acetate, and in column 9 as methyl anthranilate. Column 10 gives the percentage of methyl anthranilate directly determined.

Description of Oil.			Yield from 1,000 Kilos. of Flowers.	Specific Gravity at 15° C.	sp.	Saponifica- tion Value.	Calculated as Linalyl Acetate.	Calculated as Methyl Anthrani- late.	Methyl Anthrani- late, directly determined.
1.	2.	3.							
Distilla- tion.	A.		Grms. about						
	Néroli oil .....	.....	800	0.875	+ 4.40	38.8	Per Cent. 13.6	Per Cent. 10.5	Per Cent. 0.61
	B.								
	Water oil from {	Separated oil....	233	0.948	+ 2.7	67.2	23.5	18.1	..
	fresh flowers.. {	Water oil .....	133	0.963	+ 1.47	81.2	28.4	21.9	..
Extraction.		Mixed oil .....	366	0.950	+ 2.0	72.0	25.2	19.4	16.0
	C.								
	Water oil from {	Separated oil....	276	0.912	+ 1.20	41.7	14.6	11.3	..
	kept flowers... {	Water oil .....	194	0.968	+ 2.30	82.9	29.0	22.3	..
		Mixed oil .....	470	0.933	+ 1.30	60.2	21.1	16.3	11.1
Macera- tion.	D.								
	Volatile oil from {	Separated oil....	396	0.891	..	41.8	15.7	12.1	..
	pure extract .. {	Water oil .....	215	0.933	..	84.0	29.4	22.7	..
		Mixed oil .....	611	0.907	..	55.5	19.3	14.9	7.6
	E.								
Enfleur- age.	Volatile oil from {	Mixed oil .....	388	0.913	- 5.0	78.1	27.3	21.8	9.2
	pomade .....								
	F.								
	Volatile oil from {	Separated oil....	..	0.882	+ 3.40	71.8	25.2	19.4	0.35
	press cakes.... {	Mixed oil .....	..	0.930	+ 2.0	42.0	14.7	11.3	8.85
Enfleur- age.	G.								
	Enfleurage oil....	Mixed oil .....	89	0.909	+ 8.34	58.2	20.4	15.7	5.2

As regards the quality of the oil obtained by the different processes, the true odour of the orange flower is given only

by the mixed oils, e.g., néroli oil *plus* orange-water oil, and if the odour of the total oil be the criterion, the three





processes of distillation, maceration, and extraction give equal results.

Jeancaud and Satie explain the difference between oils obtained by distillation and extraction of pomades by assuming a saponification of esters in the distillation (this *Journal*, 1900, 847). The authors consider that the pomade extract contained vaseline and non-odoriferous extracted matter. (Compare A. Hesse, this *Journal*, 1901, 275). The acidity of the condensed water cannot be used to calculate the amount of acetic esters saponified, since the water contains higher fatty acids and phenols. Pure linalyl acetate was found to be saponified to the extent of 20 per cent. by boiling with water for three hours; a mixture of linalool and linalyl acetate, containing 25.7 per cent. of the latter, lost about 2 per cent. of the ester when distilled in steam. Methyl anthranilate is not decomposed by boiling with water. Assuming that 20 per cent. of the linalyl acetate present is decomposed in the distillation, it may be calculated that the mixture of the oils A. and B. in the above table would contain *before* distillation about 19 per cent. of linalyl acetate, which is the amount found in the mixed oil obtained by extraction.

The high percentage of methyl anthranilate in the oil extracted from orange flower water is noteworthy; after removal of this ester and saponification, geraniol and phenylethyl alcohol were detected; the alkaline liquor contained phenylacetic acid.

In regard to the estimation of methyl anthranilate (this *Journal*, 1901, 289), it has now been found that the sulphate of the ester partially dissociates, especially in the presence of resinous substances, which are precipitated with it. Therefore the solution of the sulphate should not be filtered; any insoluble matter is brought into solution by alcohol, and the titration and saponification then conducted.

The authors give a table of the boiling points of linalyl and benzyl acetates and alcohols, carefully determined:—

—	762 mm.	25 mm.	10 mm.
Linalyl alcohol	195°—199° C.	98°—98.5°	81°—81.5°
Benzyl alcohol	205.5°	107°	89°—89.5°
Benzyl acetate	216°	110°—111°	92.5°—93°
Linalyl acetate	About 220° C. with decomposition.	115°—116°	96.5°—97°

—A. C. W.

*Saccharin; New Reactions for the Detection of* —. M. Spica.

See under XXII., page 1146.

*Morphine in Opium; Determination of* —, by means of Ammoniacal Silver Chloride. C. Reichard.

See under XXIII., page 1149.

*Aconitine, Belladonna, and Henbane; Determination of the Alkaloids in Extracts of* —. Stoeder.

See under XXIII., page 1146.

*Ipecacuanha Root; Determination of Alkaloids in* —. Stoeder.

See under XXIII., page 1147.

*Morphine; Characteristic Reaction for* —. C. Fleury.

See under XXIII., page 1146.

*Pomegranate Bark, and Extract of same; Alkaloidal Determination of* —. N. Stoeder.

See under XXIII., page 1150.

## PATENTS.

*Chloral; Improved Process and Apparatus for Continuously Producing and Rectifying* —. J. A. Besson, Caen, France. Eng. Pat. 17,202, Sept. 27, 1900.

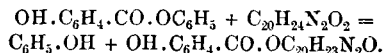
CHLORAL and its hydrate are the most volatile products of the action of chlorine on strong alcohol, and may therefore be removed by continuous distillation. The patentee describes and claims a suitable apparatus; chlorine and chlorinated alcohol are introduced into a vessel heated in a water bath, from which the vapours pass into a second, thence into a third heated vessel. The residual chlorine is absorbed by alcohol in a cylinder provided with baffle plates. The rectifier, which also acts continuously, is provided with baffle plates; it is fed at the middle with chloral, and at the top with sulphuric acid (66° B.). The final purification is effected by rectifying over chalk or sodium bicarbonate in the usual manner. The same apparatus may be used for converting chloral into chloroform.—A. C. W.

*Cyanamide and its Compounds; Manufacture of* —. G. W. Johnson, London. From the Deutsche Gold- und Silber Scheide-Anstalt vormals Rössler, Frankfurt-on-Main, Germany. Eng. Pat. 21,819, Dec. 1, 1900.

SODIUM cyanide dissolves in molten sodamide; if the temperature be raised, a violent reaction takes place at about 440° C., with production of disodium cyanamide:  $\text{NaNH}_2 + \text{NaCN} = \text{CN} \cdot \text{N} \cdot \text{Na}_2 + \text{H}_2$ . In the processes claimed, solid or molten sodamide is slowly introduced into the molten cyanide, or metallic sodium is added to the molten cyanide and ammonia passed through, the nascent amide then acting on the cyanide.—A. C. W.

*Pharmaceutical Compounds; Manufacture or Production of New* —. H. E. Newton, London. From Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 8165, April 20, 1901.

WHEN quinine is heated with phenyl salicylate (salol) in an oil bath at 170°—190° C., phenol distils and the quinine ester of salicylic acid remains. The product is dissolved in chloroform and shaken with 1 per cent. acetic acid in order to remove unchanged quinine—



The compound is a white crystalline powder melting at 140° C.; it is soluble in chloroform, hot alcohol, and benzene, little soluble in ether and cold alcohol. Ferric chloride colours the alcoholic solution reddish brown. The corresponding cinchonidine compound melts at 65°—70° C.; its acid sulphate forms white needles which melt at 165° C. The claims are for the manufacture of these salicylic esters, the compounds as new articles of manufacture and their use for therapeutic purposes.—A. C. W.

*Phthalic Acid and Benzoic Acids; Impts. in the Manufacture of* —. O. Imray, London. From The Basle Chemical Works, Basle, Switzerland. Eng. Pat. 15,527, July 31, 1901.

By heating the naphthols in presence of alkali with metallic oxides or peroxides such as copper or iron oxide, barium, lead, or manganese peroxide, to a temperature above 200° C., they yield phthalic and benzoic acid, together with a few intermediate products. For instance, 10 kilos. of  $\alpha$ - or  $\beta$ -naphthol, 90 kilos. of caustic soda, and 90 kilos. of cupric oxide, are stirred together with a little water and heated at 240°—270° C. until the cupric oxide becomes reduced to cuprous oxide. The excess of alkali is removed by lixiviation with a small quantity of water, the acids are then dissolved in water and decanted from the cuprous oxide. This solution is saturated with carbon dioxide and filtered from unaltered naphthol. The filtrate is decomposed with sulphuric acid and evaporated, the precipitated acids being purified by distillation. The distillate consists of phthalic and benzoic acids. In place of using a large excess of alkali, a smaller quantity may be taken and the operation conducted under pressure. The naphthols may

also be replaced by other naphthalene derivatives such as nitronaphthalene, the naphthylamines, naphthalene sulphonic acids or by nitroso-, and nitro- or sulpho-naphthols.

—T. A. L.

## XXI.—PHOTOGRAPHY.

*Gelatin ; Indirect Action of Sulphite upon* —. Lüppo-Cramer. Eder's Jarhb. Phot. 1901, 15, 44; through Chem.-Zeit. Rep. 1901, 25, 272.

THE author has found that the oxidation-products of several organic developers, especially those of "adurol," *p*-aminophenol, and "metol" exert a tanning influence upon the gelatin of a photographic plate; this action being more or less weakened, or even prevented, by sulphite. But as the above-mentioned three substances are made more powerful developers by increasing additions of sulphite, it seems possible that the tanning and the developing influence of the sulphite are not independent phenomena, but that one is the consequence of the other, e.g., that development proceeds more slowly where the film has been tanned. Adurol and quinol are stronger developers of collodion-bromide in the absence of sulphite. Thus it appears that the practical developing value of a substance cannot be predicted from its purely chemical reducing properties, since the substitution of an indifferent body like collodion by gelatin alters the action altogether.—F. H. L.

*Developers ; Substitution in* —. Lüppo-Cramer. Eder's Jarhb. Phot. 1901, 15, 63; through Chem.-Zeit. Rep. 1901, 25, 272.

AMONG the halogen derivatives of developers, "adurol" has recently attained considerable importance owing to its excellent qualities. From his experiments with this body the author has thought that the dicyanoquinol lately obtained by Thiele and Meisenheimer by the action of nascent hydrocyanic acid upon quinone might prove a useful developer, but the idea was erroneous. As this compound is not readily attacked by alkalis, it cannot be imagined that one or both of the nitrile groups are displaced by carboxyls when dicyanoquinol is treated with an alkali carbonate. The explanation of the fact that dicyanoquinol does not develop must be rather that the cyanogen group, like the carboxyl group in substances which would otherwise develop, destroys the power of reducing the latent photographic image.—F. H. L.

*Mercury Intensification ; The Chemical Process in* —. F. Novak. Chem.-Zeit. Rep. 1901, 25, [76], 280.

ACCORDING to Chapman Jones (this Journal, 1893, 983) when finely divided metallic silver is treated with mercuric chloride the double compound  $\text{AgClHgCl}$  is formed. By the action of ammonia on this, a black residue is obtained which contains compounds having the formulae  $\text{NH}_2\text{AgHgCl}$  and  $\text{NHAgHg}_2\text{Cl}$ . This observation is confirmed by Leteur's experiments (Comptes Rend. 1900, 130, [5]; this Journal, 1900, 275). When ammonia acts on a mixture containing little silver chloride and much mercurous chloride, the former is not completely dissolved, some of it always remaining in the black residue. According to Leteur, after three digestions with ammonia, the proportion of silver chloride to the total weight of the chlorides taken was 0.213, whilst Chapman Jones gives  $142.5 : 614.5 = 0.233$  as the proportion of silver chloride to the total chlorides in the product of the reaction of ammonia on the silver mercurous chloride which approximates very closely to Leteur's result.—T. A. L.

*Photographic Reducers ; Ammonium Persulphate and other* —. Brit. Jour. Phot. 1901, 48, [2159], 594—595. See also Chem.-Zeit. Rep. 1901, 25, [76], 280.

THE following reducers in conjunction with various developers have been comparatively tested by Messrs. Lumière:—(a) ammonium persulphate, 4 per cent. solution; (b) potassium permanganate, 0.5 grm., sulphuric acid, 1 c.c. water 1 litre; (c) Cerium sulphate, 6 per cent. solution; (d) Potassium ferricyanide, 5 grms., sodium thiosulphate 50 grms., water, 1 litre; (e) Potassium bichromate 1 grm.; sulphuric acid, 1 c.c.; water 1 litre. Ammonium

persulphate (a) preferably attacks the denser deposit and also preserves all six steps of gradation of the test negatives developed by the different developers; after development with diaminiophenol two steps were lost by each of the other reducers, and on the hydramine, diamino-resorcinol, hydroquinone, oxalate of iron, and pyro-acetone-developed negatives, one step was lost; with the *p*-aminophenol-developed negative there seemed to be no difference between the action of (a) and the other reducers, in fact the persulphate appeared to reverse its usual behaviour. Persulphate (1) acts more quickly upon a wet than a dry negative. (2) unlike the other reducers, it does not begin to reduce until some time after immersion and proceeds steadily; (3) continues to act after the negative is taken from the bath until stopped by a solution of alkali sulphite or bisulphite; (4) in strong or weak solution gives the same result, the time of action only being affected; (5) unlike other reducers it does not remove any veil due to over-exposure or over-development.

—J. W. H.

## PATENT.

*Photographic Copying of Line Drawings and the like ; Process for Producing Paper for the* —. H. Haucke, Wevelinghoven, Germany. Eng. Pat. 15,673, Aug. 2, 1901.

IT is stated that the method now in use for producing positive copies of line drawings by photographic means consists in applying to paper a "sensitive mixture of ferric oxides, tartaric acid, and gelatin," which paper is exposed to the action of light under the drawing to be copied. Where the light passes, the ferric oxide is reduced to ferrous oxide and the gelatin again becomes soluble. In order to render possible the use of ordinary drawing or printing paper the patentee claims a process for applying a coating insoluble in water (gelatin, chrome alum, and aluminium acetate; varnishes; casein, &c.) and then a coating soluble in water (dextrin, &c.), upon which the sensitive layer is applied. The first coating prevents the penetration of the sensitive layer into the paper and the second permits of the removal of the ferrous oxide.

—A. C. W.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### PATENTS.

*Primer for Producing Ignition by Electricity ; Manufacture of* —. R. Kändler, Dresden. Eng. Pat. 20,800, Nov. 17, 1900.

THE composition claimed is said to possess great conductivity, and can be ignited with certainty and in places at a great distance, even by a weak induction spark. It is composed of the following finely powdered ingredients:—Potassium chlorate, 5 parts; antimony sulphide, 3 parts; lead ferrocyanide, 2 parts; and gas carbon, 1 part. These are intimately mixed, with water added to prevent ignition by friction, and the mass is finally dried and powdered by grinding upon wood.—C. A. M.

*Explosive ; a New* —. O. Imray, London. From J. L. Norris, Washington City, U.S.A. Eng. Pat. 8365, April 23, 1901.

THE explosive consists of a mixture in about the following proportions:—Nitroglycerin, 70; oil of mirbane, 15; and empyreumatic oil, 15 per cent. by weight. The empyreumatic oil is the lighter oil of wood tar (*oleum picis liquida*). Magnesia, granulated charcoal, wood pulp, or other substances may also be introduced to give consistency when a granulated or moulded explosive is required.—C. A. M.

*High Explosives and Celluloid Compounds ; Manufacture of* —. J. E. Blomén, Pompton Township, New Jersey, U.S.A. Eng. Pat. 9984, May 14, 1901.

ACCORDING to this patent any two or more of the following substances are nitrated together:—viz., cellulose; an aromatic hydrocarbon, (e.g., benzene, naphthalene); and a hydroxy-derivative of a hydrocarbon, such as phenol or cresol. The base formed is suitable for the manufacture of celluloid. A second claim is for the addition to this explosive base



of an oxygen-conveying salt, such as the nitrates or chlorates of various metals. A neutral substance like paraffin may be added to check too rapid combustion.

—C. A. M.

**Smokeless Powder Charges for Guns.** H. Maxim, Brooklyn, New York, U.S.A. Eng. Pat. 13,620, July 4, 1901.

THIS invention has the object of obtaining uniform acceleration of the combustion of the charge and a simultaneous completion of the combustion throughout the entire mass; of securing the same ends in an explosive charge capable of maximum density of loading; and of enabling the charge itself to support the projectile without the use of a cartridge shell.

The principle of the invention lies in the construction of the charge of one or more units of explosive material, preferably in the form of rods or bars, with longitudinal and lateral perforations, so that explosion occurs from surface combustion in each of the different sections. The various forms in which the charge may be constructed are enumerated in different claims. Thus, it may consist of concentric tubes of smokeless powder, each of which has a number of transverse perforations. Or it may be a cylindrical mass divided by radial planes into longitudinal sections, each of which is perforated laterally. Or a solid rod may be perforated longitudinally and transversely at uniform distances. Or there may be a central longitudinal perforation with other longitudinal perforations to which a number of transverse perforations form vents; and so on.

—C. A. M.

**Explosives; Improvements in** — J. W. Mackenzie. London. From The Robin Hood Powder Company, Swanton, Vermont, U.S.A. Eng. Pat. 14,525, July 16, 1901.

THE explosive for which claim is made, is stated to be smokeless, and to subject the gun to very little strain. It is prepared by mixing in approximately the proportions mentioned, the following ingredients:—Ammonium picrate, 33.5; potassium picrate, 15.5; barium nitrate, 39.5; wood flour, 9.5; wheat flour, 1.25; lamp-black, 0.25; and potassium ferrocyanide, 0.50 per cent. The mass is kneaded into a dough with a paste of gum arabic or gum tragacanth, then granulated, and finally rendered non-absorbent by coating it with petroleum, previously subjected to the action of nitric acid, adding to the supernatant liquid after settling, ammonium hydroxide, which eventually sinks to the bottom, leaving the purified petroleum ready for use.

—C. A. M.

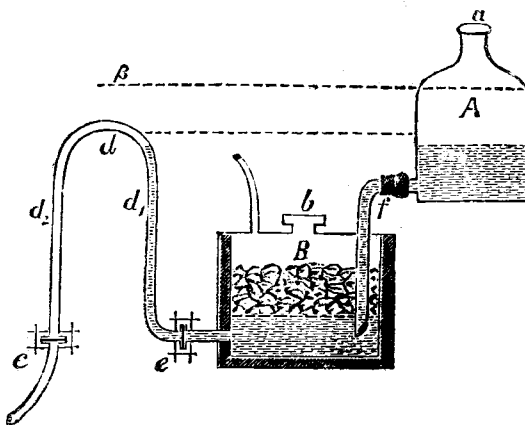
## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

**Hydrogen Sulphide Apparatus for Continuous Working.** H. Koch. Chem.-Zeit. 1901, 25, [81], 873.

THE apparatus shown in the figure consists of a glass acid vessel A of about 15 litres capacity, having its side tubulure connected by means of stout rubber tubing with the leaden tube *f*. The evolution vessel B is composed of lead and is about 32 cm. in length. It is fitted into a wooden box, and has its outlet tube connected with the  $\Omega$ -shaped leaden tube *d*, which has a diameter of 40 mm. This tube is so arranged that its top is parallel with the centre of that portion of the vessel A occupied by the acid. The connections of the  $\Omega$ -tube at *c* and *e* are constructed on the flange principle, and are covered with iron rings. In using the apparatus, about 15 kilos. of iron sulphide are introduced into B, and from 8 to 10 litres of sulphuric acid of sp. gr. of 1.5° B into A. If the pressure of gas drives the acid back into A, the liquid in the tube *d* is also driven to the same level. When a fresh supply of acid is necessary, it is introduced at *a*, and a corresponding quantity of spent liquid is forced from B through the tube *d*. This apparatus will work for months with a minimum of attention, and when the iron sulphide requires

renewing, the apparatus can be washed out by passing a current of water into A, and eventually removing the water by means of a pump. To obviate all unpleasantness



the outlet of the tube *c* is connected directly with the drain.—C. A. M.

**Saccharimeter; A New** — P. Horsin-Déon. Bull. de l'Assoc. des Chim. de Sucr. et de Dist., 1901, 19, [1 and 2], 101—104.

THE author describes a new form of saccharimeter constructed by Messrs. J. and J. Eric, of Prague. The instrument is a half-shadow one for use with white light, and is graduated for the French normal weight, so that it registers 100 with a quartz plate, 1 mm. in thickness. The optical arrangement is as follows: The polariser is a nicol, one-half being cut like a Jellett prism into two parts having an equal inclination, one to the right and the other to the left; this gives the double image. Next comes a fixed dextro-rotatory quartz disc, rather more than 4 mm. thick, which gives a dextro-rotation to each of the two rays emerging from the polariser; as these two rays have different planes of polarisation, they are affected in varying degrees by the quartz, the right hand rays suffering a retardation in relation to those on the left. Hence, on observing these rays through the analyser, the left is coloured blue whilst the right side is very pale yellow. The compensator, a non-achromatised lævo-rotatory quartz wedge 45 mm. in length, tends to bring the right-hand rays back into their original condition, that is, to give equality of tint. Next is placed a small disc of lævo-rotatory quartz which produces a final tint rather darker than Soleil's sensitive tint, and finally the analyser. Starting with a uniformly-tinted field and displacing the compensator to the right, for example, the left-hand rays, being polarised in a plane forming with the vertical an angle greater than that formed by the right-hand rays, are deviated to a still greater extent, so that they appear violet whilst those on the right are still only light yellow. The difference between the angles is chosen sufficiently small to allow of one of the sides passing to the blue and violet, while the other only traverses the scale from pale orange to pale yellow. Hence one-half of the field always appears almost colourless and the other passes through the darker tints of the spectrum; but when the zero position is approached, the light side begins to become tinted before the other loses its colour. The instrument also admits of the application of a system of double compensation by which the readings are controlled. The mechanical arrangements are very carefully executed, the polariser being kept quite out of reach of dust, the same being the case with the compensator, quartz plates, and vernier, which are enclosed in a massive box. The vernier is engraved on glass, which is less subject to variations of temperature than copper, and less sensible to external actions than ivory, besides being free from danger of oxidation.—T. H. P.



### INORGANIC CHEMISTRY.— QUANTITATIVE.

*Mineral Poisons, especially Arsenic and Antimony; A Method for destroying Organic Matter, applicable in Searching for —.* G. Denigès. J. Pharm. Chim. 1901, 14, [6], 241–246.

By the method described below portions of the cadaver may be destroyed within a few hours. Even the cacodyl molecule is oxidised. 200 grms. of the substance in coarse fragments are heated with 200 c.c. of nitric acid (sp. gr. 1·39), and 5 c.c. of a 2 per cent. solution of potassium permanganate. Disintegration is complete, and the frothing will have ceased after a period of 15–30 minutes, which varies according to the nature of the organ to be destroyed. The mass is then transferred to a porcelain basin of 1 litre capacity, and the first basin rinsed with 100 c.c. of nitric acid at 50–60° C. and 100 c.c. of warm water. The dish is covered by a large funnel, which fits just below the spout, and from which the neck has been removed. The mixture is kept in gentle ebullition for at least two hours; it is better to heat more gently for 4–5 hours. In any case evaporation must not be pushed to such an extent that the liquid blackens; this will not be the case if 70–80 c.c. remain. If the mixture should blacken, heating must be stopped and 10–15 c.c. of nitric acid added. After evaporation to the desired volume, the funnel is removed, and, without cooling, 100 c.c. of pure sulphuric acid added (if there is much fat, more acid may be required; the mass must remain fluid even after the action of the acid and intense blackening). Nitrous fumes are evolved, which then disappear and the liquid begins to turn black. After about two minutes from the appearance of the black coloration, 5 c.c. of nitric acid are added from a pipette in a thin stream over the centre and round the mass. This addition of nitric acid is four times repeated; the mixture is then rapidly heated for 5–6 minutes, so that the sulphuric acid strongly attacks the supernatant fat, the burner is removed and three quantities of 5 c.c. each of nitric acid added in the same manner as before at intervals of two minutes. The funnel is then replaced, and the sulphuric acid brought to the boil. Nitric acid is then added, 50–60 drops at intervals of 2–3 minutes, at the rate of a drop per second, by means of a funnel with capillary tube inserted in the orifice of the large funnel. The residual liquid at last changes to reddish yellow, then to pale yellow; it is now evaporated down to 10–15 c.c., 50–60 drops of nitric acid being added in four or five portions during the evaporation. After cooling, 100 c.c. of water are added, the liquid boiled to remove nitrous fumes, and sufficient water added to make a 10 per cent. solution (by volume) of the residual acid. The colourless liquid so obtained contains the whole of the arsenic and antimony in the original matter.

In destroying wool and hair, a rapid effervescence commences in the cold and proceeds without heating; solution is complete in 15–20 minutes. Double the ordinary quantity of nitric acid should be used. In the case of bone, after attacking with nitric acid and evaporating to 70–80 c.c., the liquid is filtered through cotton wool or gun cotton washed with nitric acid; these retain the fat, which is carefully washed. The filtrate is largely diluted, precipitated by sulphuric acid and filtered, the new filtrate is evaporated to 70–80 c.c., the fatty matter added, and then the liquid treated with sulphuric acid as described before.

In order to destroy the cacodyl molecule, after evaporation to the final 10–15 c.c., 5–6 grms. of potassium nitrate are added, and the mixture heated until sulphuric acid nearly ceases to be evolved. The liquid is then cooled and diluted.—A. C. W.

*Indicators; Employment of certain —, with Artificial Light.* A. Kufferath. Zeits. angew. Chem. 1901, [37], 916–918.

THE following indicators, suitable for ammonia titrations, were examined:—Methyl Orange, Fluorescein, C. chineal, Coralline, *p*-nitrophenol, Alizarin Green B, Resazurine,

and Luteol, both by daylight and that of a 16-candle electric glow lamp, a 13·5-candle Auer lamp, and a 12—12½-candle acetylene lamp.

The conclusions arrived at by the author, from the tests detailed in the paper, are that the acetylene light is to be recommended in the case of indicators giving a change to a colour of different kind to the original such as:—Methyl Orange, Cochineal, Coralline, Alizarin Green B and Resazurine, whereas the choice of a light is immaterial when the change is of another character, such as from colourless to pale yellow (*p*-nitrophenol and Luteol), or from pale yellow to yellow-green fluorescence (Fluorescein).

—C. S.

*Alkalis and Acids; Microchemical Detection of —. Ozone and Water; Detection of Small Quantities of —.* F. Emich. Monatsh. für Chem. 1901, 22, [7], 670–678.

*Microchemical Detection of Alkalis and Acids by means of Litmus-Silk.*—In order to bring litmus into a condition suitable for use in microchemical analysis, the author fixes it on silk in the following manner. Commercial litmus is boiled with somewhat less than an equal weight of water, the first impure extract being rejected, while the residue is again treated several times with small quantities of hot water and the solution filtered; the boiling filtrate is supersaturated with sulphuric acid, and used for dyeing silk which is left in the hot solution for half-an-hour and is then washed in running water, by which means it assumes a reddish violet tint; after drying, such "red litmus-silk" is kept in the dark. For preparing "blue litmus-silk," the red form is placed in a little water, to which extremely dilute alkali solution is carefully added, until the change in colour occurs, after which the silk is washed quickly once with distilled water, pressed between paper and dried. Under the microscope, using a condenser and a magnification of about 200, both indicators show strongly coloured threads. The author employs these threads in the following way. A single coloured cocoon thread is fastened to a piece of wax and cut with sharp scissors, so that there remains free about a centimeter of the thread which is drawn through a drop of alcohol to clean it, and then examined under the microscope to see that the cut end is uninjured in any way. A drop weighing about 0·05 mgrm. of the liquid to be examined is arranged on a suitable support, so that the end of the silk can be dipped in it and allowed to remain there during the concentration of the drop by evaporation, and when all the liquid has dried up, the tip of the thread is again examined under the microscope, after which it is cut off and the silk got ready for the next test. In the author's experiments the small quantities of liquids used were measured by means of loops of platinum wire and the evaporation of the drops was carried out on cover-glasses of rock crystal, by which means the disturbing influence of the alkali in glass was avoided. When red litmus-silk is employed, the smallest amounts of the different alkalis which can be distinctly detected are given. Barium and calcium carbonates distinctly show alkalinity when unweighable quantities of them are rubbed up with water and tested with the red silk. Except in the case of sodium compounds, the above quantities are smaller than are required to give the faintest trace of a flame coloration. 1/4000 mgrm. of ammonia can be detected in this way. The alkaline reaction of strychnine, morphine, and of guanidine carbonate can be observed in quantities of a few hundred-thousandths of a mgrm. The decomposition of glass by water may be shown by triturating 0·1 mgrm. of glass with 1 mgrm. of water and testing the liquid with litmus-silk; the reaction shows even with the more infusible glasses, but not with porcelain.

*Microchemical Detection of Ozone.*—Since, in the oxidising actions of ozone, acid or alkaline substances are frequently formed, litmus-silk may be used for the detection of small quantities of ozone. Thus with the red silk, potassium iodide or ferrocyanide may be used, or with the blue silk, sulphur or potassium or calcium sulphocyanide; this last salt is converted by ozone into a paste of gypsum needles.



**Detection of Small Quantities of Water.**—The hygroscopic properties of very small quantities of a salt can be studied by bringing the salt between two small electrodes, the deliquescence of the salt being evidenced by its greatly increased conductivity.—T. H. P.

**Ferric Salts; Note on the Reduction of** — G. T. Morgan. Analyst, 1901, 26, 225—227.

It is well known that zinc is not altogether a satisfactory reducing agent for ferric salts. In the following process, which admits of the use of pure zinc, either granulated or in sheets, the efficiency of the metal is considerably increased by employing it in the form of the zinc-copper couple.

A weighed portion (4 to 6 grms.) of ferric ammonium sulphate was dissolved in 100 c.c. of dilute sulphuric acid (3 per cent.) and the cold solution poured on to a zinc-copper couple, produced by immersing 8 grms. of granulated zinc for two or three minutes in 200 c.c. of a 10 per cent. solution of copper sulphate; a vigorous action set in, and after 10 minutes a drop of the solution no longer gave the thiocyanate colouration. The reduced solution, decanted from the couple and rapidly filtered to remove any floating particles of metal was made up to 250 c.c. with recently boiled dilute sulphuric acid, and aliquot portions were then withdrawn and titrated with standard permanganate solution. The percentage of iron in the specimen of ferric alum employed was 11.50, whilst two volumetric estimations, carried out in the above manner, gave 11.34 and 11.44. Under these experimental conditions the amount of zinc required to reduce the quantities of ferric salt usually taken for volumetric estimations is small, and the couple may be used for several determinations.

The zinc-copper couple may be employed for reducing ferric salts in hydrochloric acid solution, the action being far more rapid than that of zinc or ammonium hydrogen sulphite; it is therefore applicable for the volumetric analysis of iron ores with standard bichromate solutions. The author gives numbers to support this statement.

When the zinc-copper couple or granulated zinc is added to a neutral solution of ferric alum, a portion of the iron is precipitated as basic sulphate. The whole of the iron thus rendered insoluble is in the ferric condition, whereas the portion remaining dissolved is entirely converted by the reducing agent into the ferrous state. The brown precipitate, which rapidly forms when the couple is employed, readily dissolves in dilute acid, and the reduction then follows the normal course.—J. L. B.

**Calcium, Strontium and Barium; Determination of** —, as Oxalates. C. A. Peters. Amer. J. Science, Silliman, 12, [4], 216—224. Chem. Centr. 1901, 2, [15], 869.

IN the determination of calcium by titration of the oxalate with permanganate, accurate results are obtained if hydrochloric acid (with a manganese salt) be used as solvent. Strontium salts can be precipitated with sufficient accuracy by ammonium oxalate, if the solution contains one-fifth of its volume of 85 per cent. alcohol. The titration with permanganate follows satisfactorily if the strontium oxalate is decomposed either by sulphuric acid or by hydrochloric acid (with a manganese salt). Barium salts can be precipitated by ammonium oxalate with sufficient completeness if the solution consists to the extent of one-third of 85 per cent. alcohol. The barium oxalate can be dissolved in hydrochloric acid, after addition of a manganese salt, and then titrated with permanganate.—A. S.

**Silicate Analyses.** M. Stoermer. Chem.-Zeit. 1901, 25, [76], 809—811.

THE chemical analysis of a clay by the usual methods only determines the constituents, but gives no indication as to how the various elements are combined. For this purpose, Seger and Aron propose and employ a rational analysis, which consists in decomposing the clay with concentrated sulphuric acid, leaving quartz and feldspar undissolved. About 3—5 grms. of the dry finely-powdered unburnt substance are treated in a platinum dish with 100 c.c. of water and 50 c.c. of concentrated sulphuric acid. The dish is covered with a watch-glass and gently heated

until the sulphuric acid begins to fume strongly. After cooling and dilution with water, about 100 c.c. of hydrochloric acid are added, and the whole is boiled. The precipitate formed is then extracted several times with soda lye and hydrochloric acid, and the residue is finally heated, and weighed in a platinum crucible. This gives the amount of quartz and feldspar and, by difference, the remainder of the clay. The residue of the quartz and feldspar is heated with hydrofluoric acid and a drop of sulphuric acid. Silica is removed, leaving alumina and alkali as sulphates. The alumina is precipitated from the hydrochloric acid solution of these by ammonia and, on weighing, gives the alumina present in the feldspar,  $K_2OAl_2O_3 \cdot 6SiO_2$ . Multiplied by 5.41 this number gives the amount of feldspar, which, subtracted from the weight of quartz and feldspar, gives the quartz. The rational analysis is especially valuable in the porcelain and pottery industries. The mixtures are calculated according to the results of the rational analysis by which the raw materials are examined.—T. A. L.

**Cement; Detection of Powdered Slag in** —. W. Frcsenius. Zeits. angew. Chem. 1901, 14, [38], 951.

THE detection of this adulterant is based on the treatment of the powdered cement in a centrifugal machine with a liquid of definite specific gravity, such as a mixture of methylene iodide and benzene. The sulphur in the slag is also determined by treating the substance with a definite quantity of sulphuric acid and arsenic trioxide, filtering from the arsenic trisulphide, and titrating the excess of arsenic trioxide in the filtrate in the usual way.—C. A. M.

**Sulphur in Iron and Steel; Determination of** —. W. A. Noyes and L. L. Helmer. J. Amer. Chem. Soc. 1901, 23, 675—679.

THE authors have examined the different methods for the determination of sulphur in iron, and find that the best solvents for the iron are either bromine and water or dilute nitric acid with a little potassium bromide and an excess of potassium chlorate. These two methods of dissolving the metal are carried out as follows:—(1) A mixture of 5 grms. of the iron and 7 grms. of potassium chlorate is added in portions to 120 c.c. of dilute nitric acid (sp. gr. 1.20 or 1.2 by volume) and 1 gm. of potassium bromide contained in a flask, which is cooled if necessary. When the metal is dissolved, the liquid is transferred to a flat porcelain dish and evaporated to dryness in such a way that contact with the products of combustion of the gas is avoided. The dry mass is treated with concentrated hydrochloric acid, the liquid diluted and filtered, the filtrate and residue being treated as described below. (2) When dissolving the iron in bromine, the latter must be free from sulphur, which may be removed by distilling the bromine with a little sodium carbonate. 5 grms. of the iron is then added in portions to a mixture of 8 c.c. of bromine with 200 c.c. of water, cooling after each addition. Solution takes place readily, and when it is complete, the liquid is boiled for a short time to expel excess of bromine, filtered, and the residue washed.

The insoluble residue obtained by either of these methods is mixed with sodium carbonate in a platinum crucible into which the filter is also burned by means of a spirit lamp, the oxidation being completed by the addition of potassium nitrate and heating. The mass is then dissolved in water, the liquid filtered, acidified with hydrochloric acid, mixed with 5 c.c. of barium chloride solution, and digested until the precipitated barium sulphate settles rapidly, when it is filtered off and weighed as usual. Three-fifths of the weight of the barium sulphate thus obtained are added to that yielded by the filtrate from (1) or (2) which is treated as follows:—The whole of the filtrate and washings are added to 130 c.c. of 10 per cent. ammonia in a 500 c.c. flask and the liquid made up to volume. After mixing thoroughly, the solution is filtered through a dry filter and 300 c.c. of the filtrate, which must be strongly alkaline, evaporated in a wide beaker to 100 c.c., the products of combustion of the gas being meanwhile kept out of contact with the liquid. A drop of dilute hydrochloric acid (sp. gr. 1.12) and 10 c.c. of barium chloride solution are then added, and the liquid digested hot till the precipitated barium



sulphate settles quickly after stirring, when it is filtered, washed, and weighed in the ordinary way. The weight of the barium sulphate, increased by three-fifths of that obtained from the insoluble residue as above described, represents the sulphur in 3 grms. of the iron.

The authors find that with iron of a given character, the proportion of sulphur in the residue is probably nearly uniform, so that the original solution can be at once precipitated with ammonia without previous filtration, and a constant correction applied for the sulphur of the residue, thus considerably shortening the determination.—T. H. P.

#### Cyanide Solutions containing Zinc; Testing —.

L. M. Green. Inst. of Min. and Metall., Oct. 17, 1901.

AFTER briefly reviewing the drawbacks of the existing methods for estimating the various working constituents of cyanide solution used for gold extraction, the author gives a plan and methods for analysis, of which the following summary shows the essential points:—

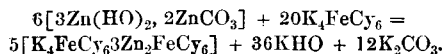
1. *Total Cyanide*.—To 50 c.c. of the solution are added excess of sodium hydrate and a little potassium iodide solution, after which the liquid is titrated with silver nitrate until a distinct permanent yellow cloudiness is obtained, any white cloudiness near the end point, due to zinc ferrocyanide, being disregarded. Result = T c.c.

2. *Protective Alkali (alkali and alkaline earth hydrates + half the monocarbonates)*.—Excess of potassium ferrocyanide, to precipitate zinc, is added to 50 c.c., and 2 T c.c. of silver nitrate solution run in to precipitate the cyanide, after which phenolphthalein is added and the solution titrated with decinormal nitric acid till colourless. Result = p c.c.

3. *Alkali Hydrates*.—An excess of barium chloride to precipitate sulphates and carbonates is added to 50 c.c., 2 T c.c. of silver nitrate run in, and the solution titrated with nitric acid till colourless, as in 2. Result = h c.c.

4. *Cyanides, Chlorides, &c. precipitated by Nitrate of Silver*.—To 50 c.c. are added 2 T c.c. of silver nitrate, then p c.c. of nitric acid, one drop of potassium chromate and the titration with silver nitrate continued until permanent red indication. This test is unimportant in itself, but necessary for the subsequent zinc determination. Result (total  $\text{AgNO}_3$  added) = N c.c.

5. *Zinc, less the Ferrocyanide*.—To 50 c.c. are added about 10 c.c. of roughly decinormal sodium carbonate to precipitate basic zinc carbonate and zinc ferrocyanide, and 2 T c.c. silver nitrate and the whole well shaken. The solution, after adding phenolphthalein, is rendered colourless with decinormal nitric acid with occasional shakings, during which operation the basic zinc carbonate assumes its normal composition. It is preferable then to add another c.c. of acid, shake and neutralise back with sodium carbonate to a pink shade. An excess of potassium ferrocyanide is added to react with the basic zinc carbonate according to the following equation:—



The alkalinity is then determined with nitric acid. Result = S c.c.

6. *Total Zinc*.—To 50 c.c. are added about 10 c.c. of sodium carbonate and N c.c. of silver nitrate, sufficient, that is, to decompose the zinc ferrocyanide. After shaking well, phenolphthalein is added and the solution neutralised as in test 5, excess of potassium ferrocyanide is then added and the alkalinity again estimated. Result = Z c.c.

Provided the nitric acid solution be decinormal and the silver nitrate contain 13.05 grms. per litre, the following are the factors for the various results:—

Total cyanides (estimated as $\text{KC}_y$ )	.....	$T \times 0.02\%$
"Protective alkali" (estimated as KHO)	.....	$p \times 0.0112\%$
Alkali hydrates (as KHO)	.....	$h \times 0.0112\%$
Alkali carbonate (as $\text{K}_2\text{CO}_3$ )	.....	$(p-h) \times 0.0276\%$
Ferrocyanide (as $\text{K}_4\text{FeCy}_6, 3\text{H}_2\text{O}$ )	.....	$(Z-S) \times 0.0351\%$
Zinc	.....	$Z \times 0.0081\%$

—A. W.

#### Red Lead; Determination of Foreign Impurities in —.

A. Jousser. J. Pharm. d'Anvers, 57, 231. Pharm. J. 1901, 67, [1631], 385.

2.5 GRMS. of the sample are stirred, in the cold, with 20 c.c. of nitric acid (1:4), and when the whole of the red lead is converted into  $\text{PbO}_2$ , hydrogen peroxide solution is added gradually to dissolve the latter; a few drops are usually sufficient. Foreign impurities, such as colcothar, powdered brick, sand, barium sulphate, &c., remain undissolved, and can be collected, washed, dried, and weighed.—A. S.

#### Perchlorates in Chili Saltpetre; Determination of —.

Grimm. Chem. Ind. 1901, 24, [17–18], 476.]

FOR this purpose it is usual to determine the chloride in the sample as such, and again, after conversion of the perchlorate into chloride by ignition, with or without the addition of manganese peroxide. The difference between the two results is a measure of the perchlorate present. In order not to lose chlorine, overheating must be avoided, and, as this is not easy when operating over a naked flame, the author recommends the following plan as very satisfactory:—The dish (platinum, nickel, or iron) containing the mixture of nitre and manganese dioxide (20 grms. of nitre + 3 grms. of dioxide) is covered over with another somewhat larger dish, inverted, both resting on a thin asbestos card, the latter being heated from below by a good Bunsen burner. The temperature is raised gradually to the maximum, and maintained for one hour. In presence of magnesium chloride it is unnecessary to add an alkali before ignition, with a view to preventing a loss of chlorine. The excess of sodium nitrate suffices to fix the halogen.

—H. T. P.

#### Bismuth; Determination of —.

C. A. Grabill. Eng. and Mining J. 1901, 72, [12], 354.

THE author has examined the method for the determination of bismuth recommended by Warwick and Kyle (this Journal, 1901, 620). He finds that the basic bismuth oxalate is slightly soluble in the cold, and more so in the hot solutions prescribed; it is possible, whilst adhering to the conditions laid down, to lose 2.8 per cent. of bismuth when working with 1 grm. of material. The permanganate should be standardised by means of an ore similar to the one under examination and containing a known proportion of bismuth. It is also pointed out that copper nitrate in presence of excess of oxalic acid, forms sparingly soluble copper oxalate; therefore, if copper be present in large amount, it must be separated by another method.—A. S.

#### Uranium; Quantitative Separation and Determination of —.

E. F. Kern. J. Amer. Chem. Soc. 23, [10], 685—726.

THIS work was undertaken with the object of finding out the most accurate and satisfactory method for the technical estimation of uranium. The usual quantitative methods for estimation of uranium are reviewed and their values checked by experiment, and new methods and modifications are suggested as the result of experimental comparison. After an historical introduction the following subjects are dealt with:—Separation of uranium from members of the fifth and sixth groups; separation of vanadium from uranium; separation of uranium from members of the third and fourth groups, particularly iron; separation of uranium from cobalt, nickel, and zinc; separation of uranium from the alkalis and the alkaline earth metals; separation of uranium from the alkali and alkaline earth metals by electrolysis of acetate solution; separation of uranium from phosphoric acid; determination of uranium as oxide; determination of uranium as phosphate; precipitation of uranium by ammonium dihydrogen phosphate; the volumetric estimation of uranium; titration of uranium solutions with potassium permanganate, including reduction of uranyl solutions by metallic zinc, metallic aluminium, metallic magnesium, and by passing uranyl solutions through reductor; reduction of uranyl solutions by stannous chloride; estimation of uranium in pitchblende by the various methods and by ether extraction. The conclusions drawn from this investigation, briefly stated, are as follows:—





(1) In order to separate uranium and the other members of group 4 from the metals of groups 5 and 6, the solution should contain not more than one part of concentrated acid (either hydrochloric or nitric acid) in 50 parts of solution.

(2) The separation of uranium from the metals of groups 3 and 4 is best accomplished by means of either a saturated solution of sodium carbonate, or else by ether followed by a saturated solution of ammonium carbonate. The latter method is preferable when the introduction of fixed alkalis and silica is undesirable.

(3) The ether extraction method for the separation of uranium from iron depends on the fact that ferric chloride is extracted from an aqueous hydrochloric acid solution, whereas the uranyl chloride is retained in the aqueous solution. For this separation it is necessary that the hydrochloric acid used for the solution be of 1.10 specific gravity and that three ether extractions be made. The ether used should be free from alcohol, and also previously shaken up with hydrochloric acid (sp. gr. 1.10).

(4) The separation of uranium from iron by means of sodium carbonate is complete, provided a large excess of a saturated solution of sodium carbonate be used, and the solution boiled for at least 15 minutes after the precipitation. The boiling is necessary in order to get all the uranium into solution. By such treatment no uranium remains with the iron, which is completely precipitated as ferric hydroxide in a form readily filtered and washed.

(5) The separation of uranium from the alkalis and alkaline earths by means of electrolysis is complete, easily accomplished, and gives accurate results.

(6) The separation of uranium from the alkalis and alkaline earths is accomplished by precipitating the uranium three times from a hot solution with ammonia in the presence of ammonium chloride.

(7) The separation of uranium from the alkalis and alkaline earths by means of an excess of ammonium phosphate in the presence of ammonium acetate is complete. The precipitations should be made from a hot solution and the boiling continued for at least 15 minutes.

(8) The yellow slimy amorphous precipitate of an ammonium uranate, formed by precipitating uranium with ammonia in the presence of an ammonium salt, is converted into a darker crystalline form by boiling it for about 20 minutes, and then allowing it to settle in the cold.

(9) The separation of the filter-paper from the precipitate of ammonium uranate for the purpose of igniting to  $\text{UO}_2$  or  $\text{U}_3\text{O}_8$  is unnecessary.

(10) The complete oxidation of uranium to  $\text{U}_3\text{O}_8$  is accomplished by igniting ammonium uranate, in either a platinum or porcelain crucible, over a blast-lamp. This is done by having the crucible in a slanting position and igniting intensely over a blast-lamp for about ten minutes, after which the crucible is allowed to cool in a slowly decreasing Bunsen flame.

(11) The reduction of  $\text{U}_3\text{O}_8$  to  $\text{UO}_2$ , as recommended by Rose for the purpose of control, was found unreliable.

(12) The estimation of uranium as phosphate is easily and accurately done when the precipitant used is ammonium phosphate, in the presence of ammonium acetate. The precipitate of  $\text{UO}_2\cdot\text{NH}_4\text{PO}_4$  on boiling becomes crystalline, and is easily filtered and washed. The ignited precipitate previous to weighing should be moistened with nitric acid (sp. gr. 1.42), dried and re-ignited at low redness in a porcelain crucible. Above this temperature, and especially so in platinum, a reduction of the  $(\text{UO}_2)_2\text{P}_2\text{O}_7$  always occurs. Whenever this happens it may be reoxidized to  $(\text{UO}_2)_2\text{P}_2\text{O}_7$  by moistening the greenish mass with nitric acid (sp. gr. 1.42) and re-igniting at low redness. The ignitions should be done in porcelain.

(13) The most rapid determination of uranium is accomplished by reducing a sulphate solution by means of pure metallic zinc and titrating it with standard potassium permanganate solution in an atmosphere of carbon dioxide. The reductions, whether made by means of metallic zinc, aluminium, magnesium, or in a long Jones reductor, were in all cases complete, and the results obtained were concordant with those obtained gravimetrically.

(14) When hydrochloric acid solutions of uranium are reduced by means of metallic zinc, aluminium, or magnesium, the reduction goes lower than  $\text{UCl}_2$ . It approached and in several cases reached the subchloride  $\text{UCl}_3$ . When stannous chloride is used, the results are utterly unreliable; so no reduction of uranium in an hydrochloric acid solution can be used for the estimation of uranium.—C. T. T.

*Tellurium; New Gravimetric Method of Determining* — A. Gutbier. Ber. 1901, 34, [12], 2724–2726.

This method depends on the fact that hydrazine hydrate and its salts separate the tellurium quantitatively from all tellurium compounds; acid solutions are precipitated either with hydrazine hydrate itself or its hydrochloride, whilst, with alkaline solutions, hydrazine hydrochloride is employed. The author describes the method of procedure in the case of telluric acid, and from this the application of the process to other tellurium compounds can be readily derived. In a roomy platinum or porcelain basin provided with a spout, and with a platinum or glass cover, a weighed quantity of telluric acid is dissolved in warm water, and to the solution a 10–20 per cent. solution of hydrazine hydrate is added by means of a pipette inserted in the spout, the dish being meanwhile kept covered to avoid loss by spurling. Action at once sets in, the solution assuming a dark blue to black coloration, and, after heating to boiling for a short time, metallic tellurium separates in flocks, while the supernatant liquid again becomes colourless. A further quantity of hydrazine hydrate is added, this procedure being continued until the reagent causes no further coloration in the liquid. The dish must be kept covered and, before each addition of the hydrazine hydrate, the solution must be heated to completely precipitate the tellurium. When all the tellurium is separated, the liquid is allowed to cool somewhat, and is then filtered through a filter which has been dried at  $105^\circ$  until constant in weight, the tellurium being washed repeatedly with hot water, and finally brought on to the filter. After thorough washing on the filter, the precipitate and paper are dried at  $105^\circ$  carefully and quickly, in order to avoid oxidation of the tellurium.—T. H. P.

*Molybdenum; Electrolytic Determination of* — L. G. Kollock and E. F. Smith. J. Amer. Chem. Soc. 1901, 23, 669–671.

On electrolysing at  $75^\circ\text{C}$ . a solution of sodium molybdate, containing 0.1302 gm. of molybdenum trioxide and 2 drops of concentrated sulphuric acid in 125 c.c. with a current of 0.1 ampère at a pressure of 4 volts, it was found that the whole of the molybdenum was deposited on the cathode as a black deposit of hydrated sesquioxide. When the latter is dissolved in nitric acid and the solution evaporated to dryness, it yields white molybdic acid, the weight of which agrees well with that of the sodium molybdate taken. This method may be applied to the determination of molybdenum in molybdenite which is first fused with a mixture of sodium carbonate and nitrate, the cooled mass being then extracted with water and the liquid filtered, acidified with sulphuric acid, and electrolysed. In case the sulphur in the mineral is to be determined, the filtered aqueous extract is acidified with acetic acid, then boiled to expel carbon dioxide, and electrolysed. The sulphur is then precipitated as barium sulphate from the liquid remaining after electrolysis.

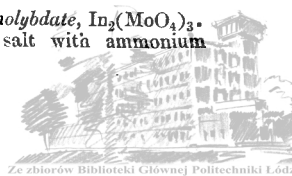
—T. H. P.

*Indium*. C. Renz. Ber. 34, [12], 2763–2765.

INDIUM can be determined in its salts by precipitating their hot solutions with dimethylamine. After washing and drying, the indium hydroxide is removed as completely as possible from the filter, and, after the latter has been separately burnt, is ignited and weighed as  $\text{In}_2\text{O}_3$ .

Indium hydroxide behaves towards bases as a weak acid. By carefully drying the precipitated hydroxide at  $100^\circ\text{C}$ ., the meta-acid,  $\text{InO}\cdot\text{OH}$  is obtained. The corresponding *magnesium indate*,  $(\text{InO})_2\cdot\text{O}_2\text{Mg}\cdot 3\text{H}_2\text{O}$ , is obtained by heating to boiling a solution of indium chloride with magnesium chloride, as a white powder, insoluble in water.

The author has prepared *indium molybdate*,  $\text{In}_2(\text{MoO}_4)_3\cdot 2\text{H}_2\text{O}$ , by precipitating an indium salt with ammonium



molybdate. It is a bulky white precipitate which becomes horny on drying; its formation can be used as a means of separating indium from zinc. *Indium platinocyanide*,  $\text{In}_2(\text{PtCy})_3$ , is obtained by the reaction of indium sulphate on barium platinocyanide. It forms white hygroscopic crystals, easily decomposed on heating.—J. T. D.

### ORGANIC CHEMISTRY.—QUALITATIVE.

*Cherry Syrup in-Raspberry Syrup; Detection of* —. K. Windisch. Zeits. Untersuch. Nahr. Genussm. 1901, 4, [18], 817—825.

The author states that both the fruit and stones of cherries contain amygdalin, which forms hydrocyanic acid on fermentation. This fact is used for detecting the admixture of cherry juice to other fruit juices. The method recommended is as follows: 20—30 c.c. of the syrup are distilled until about 2 c.c. have come over. To the latter, one drop each of tincture of guaiacum and very dilute copper sulphate solution are added. The formation of a blue colour indicates the presence of hydrocyanic acid. The colour is fugitive. Should the coloration be faint, a little chloroform may be added. On shaking, the colour dissolves in the chloroform, and becomes more apparent. As some cherries contain very little amygdalin, negative results by the above test do not prove the entire absence of cherry syrup.

The method may also be used for the detection of cherry wine in red wines.—W. P. S.

*Saccharin in Wine and Beer; Detection of* —.

F. Wirthle. Chem.-Zeit. 1901, 25, [77], 816.

The following improved method allows of the detection of 0.25 mgrm. of saccharin per 100 c.c. of wine, which corresponds in taste with about 0.12 per cent. of sugar. 200 c.c. of the wine are evaporated in a basin to about 20 c.c., which is introduced into a separating funnel together with the residue in the basin after stirring up with a few drops of sodium hydroxide and water, the whole being then strongly acidified with hydrochloric acid and shaken up with three separate quantities of 50 c.c. of ether. The ethereal solution is filtered into an Erlenmeyer flask, mixed with a few drops of concentrated sodium hydroxide solution and about 10 c.c. of water, well shaken, and the ether distilled off. The liquid is introduced into a small porcelain basin, to which the rinsings of the flask with a few drops of caustic soda and some water are also added; a small piece (about 1 grm.) of caustic soda is then placed in the liquid, which is slowly heated in an air-bath to  $215^\circ$ , and the temperature kept for a quarter of an hour at  $215^\circ$ — $220^\circ$ , the thermometer being placed in the air-bath so that the stem from  $37^\circ$  up is above the cork. The cold mass is extracted with warm water, carefully acidified with hydrochloric acid, and shaken up with ether and light petroleum spirit. The ethereal liquid is evaporated, the residue dissolved in a few c.c. of water; to the solution thus obtained are then added a few drops of ferric chloride solution of such a strength that a layer 1 cm. thick has a pale yellow tint. If only very small quantities of saccharin be present, i.e., less than 1 mgrm. per 100 c.c. of the wine, a dirty brown colour is obtained on the addition of ferric chloride. In this case the liquid is mixed with a little water, acidified, and shaken up with ether and light petroleum spirit, the ethereal solution being purified by shaking three times with 20 c.c. of water, carefully evaporated, and the residue dissolved in a few c.c. of water. With this liquid, very dilute ferric chloride solution gives a fine violet coloration.—T. H. P.

*Saccharin; New Reactions for the Detection of* —.

M. Spica. Gaz. chim. ital. 31, [2], 41—46. Chem. Centr. 1901, 2, [15], 871.

The methods proposed by the author are based (1) upon the conversion of the imino group of the saccharin into nitric acid, and (2) upon the oxidation of the saccharin to sulphaminobenzoic acid, and detection of the latter by means of the diazo reaction. The method of working is as follows:—The liquid (wine, syrup, &c.) to be examined is acidified with pure sulphuric acid (with a solid, the acid aqueous extract is taken), shaken with ether, or a mixture of ether and light petroleum spirit, and the extract divided

into three portions, and evaporated to dryness on the water-bath. One portion is examined by the method previously described by the author (Gaz. chim. ital. 25, [1], 207), for the presence of salicylic acid, the latter, if present, being converted into picric acid. The second portion is treated with a few drops of pure sulphuric acid and a small crystal of potassium permanganate, and heated gently; the excess of permanganate is removed by oxalic or sulphurous acid, and the mixture diluted with a few c.c. of distilled water. A few drops of a solution of diphenylamine hydrochloride are then added, and finally, by means of a pipette, which reaches to the bottom of the test-tube, pure sulphuric acid is run in, without allowing the liquids to mix. In presence of saccharin, the well-known nitrate reaction is observed—a blue ring at the zone of contact between the two liquids. The test is stated to be sensitive to the extent of 0.0007 per cent. of saccharin in wine. The third portion is mixed with a little lime, and heated, with shaking, to incipient charring. A few c.c. of water are added, the mixture heated to boiling, and, after standing some time, the liquid decanted into another tube, and treated with a few drops of pure hydrochloric acid, and a small piece of zinc. After about twenty minutes the reduced liquid is poured off, and a few drops of dilute alkali nitrite solution, and 5—6 drops of a solution of  $\alpha$ -naphthylamine hydrochloride added. Immediately, or after a few minutes, or even, when only a trace of saccharin is present, in the course of some hours, a crimson-red coloration is produced, if saccharin be present.

—A. S.

*Formaldehyde in Milk; Modification of the Sulphuric Acid*

Test for —. A. G. Luebert. J. Amer. Chem. Soc. 1901, 23, 682—683.

The following is an improved method for the detection of formaldehyde in milk. Five grms. of coarsely powdered potassium sulphate are placed in a 100 c.c. flask, 5 c.c. of the milk distributed over it by means of a pipette, and 10 c.c. of sulphuric acid (sp. gr. 1.84) carefully poured down the side of the flask, which is now allowed to stand quietly until the colour develops. If formaldehyde is present, the violet coloration of the potassium sulphate takes place in a few minutes, the colour gradually dispersing through the entire liquid. If no formaldehyde be present, the liquid will at once assume a brown colour, rapidly changing to black. Milk which has stood for several hours previous to testing gives the reaction even more rapidly than a fresh sample. The test is sensitive to a dilution of at least one part of formaldehyde in 250,000 of milk.—T. H. P.

*Morphine; Characteristic Reaction for* —. G. Fleary.

Chem.-Zeit. Rep. 1901, 25, [76], 276.

A FRAGMENT of the suspected substance is dissolved in a porcelain dish in a drop of  $\frac{1}{20}$  sulphuric acid in the cold; lead peroxide is then added, and the mixture is stirred for 6—8 minutes. After standing 3—4 minutes, the clear liquid is decanted and treated with a drop of ammonia, when a brown coloration is formed, due to the presence of protocathechuic acid, a decomposition product of the morphine.—T. A. L.

### ORGANIC CHEMISTRY.—QUANTITATIVE.

*Belladonna and Henbane; Differentiation of the Extracts of* —. Stoeder. J. Pharm. Chim. 1901, 14, [7], 297.

A SOLUTION of 0.1 grm. of the extract in 2 c.c. of water is shaken with 10 c.c. of ether, which is then shaken with 5 c.c. of water and two drops of ammonia. In the case of extract of belladonna, a blue fluorescence is observed in the ammoniacal liquid.—A. C. W.

*Aconitine, Belladonna, and Henbane; Determination of the Alkaloids in Extracts of* —. Stoeder. J. Pharm. Chim. 1901, 14, [7], 297.

SIX grms. of the extract are dissolved in 30 c.c. of water; 60 c.c. of chloroform and 3 c.c. of ammonia are added, and the mixture frequently shaken during three hours; 50 c.c. of the chloroform solution are filtered off and evaporated. The residue is taken up in 10 c.c. of N/10 sulphuric acid, and the excess of acid titrated as below (see following abstract).—A. C. W.



*Ipecacuanha Root; Determination of Alkaloids in —.*  
Stoeder. J. Pharm. Chim. 1901, 14, [7], 296—297.

THE ipecacuanha powder (12.5 grms.) is shaken with 125 c.c. of chloroform and 10 c.c. of ammonia at intervals during 12 hours; 5 c.c. of water are added to make the powder agglomerate. The chloroform solution is filtered, and 100 c.c. distilled. The residue is dissolved in 20 c.c. of N/10 sulphuric acid, the solution filtered, hæmatoxylin added, and the excess of acid titrated by N/10 alkali. Nux vomica may be determined in the same manner.

—A. C. W.

*Carbon Bisulphide in Benzol; Detection of —.* E. Votoček and R. Potměšil. Chem.-Zeit. Rep. 1901, 25, [76], 275.

THE method employed depends upon Hofmann's reaction,  $\text{CS}_2 + 2\text{C}_6\text{H}_5\text{NH}_2 = \text{CS}(\text{NHC}_6\text{H}_5)_2 + \text{H}_2\text{S}$ , which, especially in alkaline solution, proceeds very rapidly. The alkaline sulphide formed is detected by the nitroprusside reaction. The method is a speedy one, and only requires about 15 minutes for its completion.—T. A. L.

*Indigo; A New Hydro-Compound of —. Its Uses and Quantitative Determination.* W. Vaubel. Zeits. angew. Chem. 1901, [36], 892—893.

IN the course of an investigation of Indigo Blue and Indigo Red, the author found that, in presence of zinc dust, indigo dissolves to a deep red solution in alcoholic alkali of any kind. The same result ensues when the zinc is replaced by iron, ferrous sulphate, ferro-ammonium sulphate, aluminium, tin, or palladium; but not with silver, mercury, copper, or platinum.

The method adopted for preparing this new product was to shake up 5 grms. of Indigo Blue with 30 c.c. of 10—12 per cent. caustic soda, 100 c.c. of alcohol, and 2 grms. of zinc dust in a closed vessel, leaving the whole to settle for 12 hours, and then filtering off as quickly as possible from the residual indigo and zinc dust. On coming in contact with air, the product is reconverted into Indigo Blue, and if zinc dust be added, complete decolorisation and formation of Indigo White occurs. An admission of air then results in the reappearance of the red intermediate compound, which finally changes back into Indigo Blue.

Though at first the compound was regarded as probably one containing zinc and sodium, a more complete examination led to its being considered a hydro compound, or at the most an alkali compound, and the author has given it the name dihydroindigo blue, in conformity with its assumed composition.

Further experience has shown that the following proportions are the best for the preparation of the product:—Indigo, 1 gm.; zinc dust, 0.8—1.2 grms.; caustic potash (15 per cent.), 20 c.c. (or a corresponding amount of NaHO); alcohol (98 per cent.), 50 c.c. An excess of alkali considerably hinders the reaction, which, on the other hand, does not develop satisfactorily when the alkali is less than the quantity stated.

The author recommends this reaction as a quantitative colorimetric test for indigo; in any case, the rapidity with which the reaction begins will afford an indication as to the dyeing capacity of the indigo.—C. S.

*Silk; Method of detecting certain Weighting Matters in —.* A. Romann. Bull. Soc. Ind. Mulhouse, 1901, 328—329.

A SAMPLE of the silk to be examined is rinsed in hot water, and is then boiled with Alizarin, with the addition of chalk. It is afterwards washed and soaped. Pure silk thus treated takes a pale pink tint, while silk weighted with tin oxide is dyed an orange colour, which, if alumina be also contained in the silk, is more or less red, according to the amount of the latter present. By comparison with samples of known composition, it is possible, approximately, to determine the quantities of tin oxide and of alumina present, from the nature and intensity of the colour produced. Tannic acid may be similarly detected by treating the silk with a ferric salt, the various tannin matters giving characteristic colours.—E. B.

*Fats in Animal Fats; Detection of Vegetable —, by the Phytosterol Acetate Method.* Bömer. Zeits. angew. Chem. 1901, 14, [38], 951.

ACCORDING to the author, the addition of 1 per cent. of vegetable fat to an animal fat raises the melting point of the cholesterol or phytosterol acetate by 4°—6°. From 50—100 grms. of the fat are saponified and extracted with ether. The crude phytosterol is recrystallised from a little alcohol, and then converted into the acetic ester by heating with acetic anhydride. This ester only dissolves with difficulty in alcohol.—C. A. M.

*Cholesterol and Phytosterol; Quantitative Extraction of — from Fats.* E. Ritter. Chem.-Zeit. 1901, 25, [81], 872.

THE following simple method is stated to yield higher results than those obtainable by any of the other published methods. About 50 grms. of the fat are heated on the water-bath in a porcelain basin of about 1½ litre capacity, with about 100 c.c. of alcohol. When the alcohol boils, a hot solution of 8 grms. of sodium in 160 c.c. of 99 per cent. alcohol is introduced, and the heating continued until the alcohol has evaporated, after which 75 grms. of sodium chloride and sufficient water to dissolve nearly the whole of the contents of the basin are added. The liquid is continually stirred and evaporated to dryness, the drying being eventually completed in the oven at 80° C. The mass is then pulverised, and the fine powder left in the hot-water oven, and finally for some time in an exsiccator over sulphuric acid. It is next extracted for about nine hours in a Soxhlet apparatus with ether, and the ethereal extract transferred into an Erlenmeyer flask, leaving most of the small quantities of soap and glycerin adhering to the sides of the extraction flask. The latter is washed out with ether, the combined extract and washings evaporated to dryness on the water-bath, and the residue dissolved in as little alcohol as possible. The flask, which holds about a litre, is then nearly filled with water, little by little, with constant shaking. The precipitate is collected on a filter, washed with water, and dried on the paper at about 60° C. When dry it is transferred, with the aid of ether, into a weighed flask, and, after evaporation of the ether, dried at 100°—120° C. and weighed. The action of the sodium chloride is more than one of disintegration, for it cannot be replaced by, e.g., paper or sand.—C. A. M.

*Sulphur in Oils; Determination of —.* F. Jean. Ann. Chim. anal. appl. 6, 321. Chem. Centr. 1901, 2, [15], 867.

THE so-called solidified ("gefrorene") oils are produced by the incorporation of varying quantities of sulphur. For the determination of the sulphur, 5 grms. of the oil are heated in an enamelled iron crucible to 150° C., 4 c.c. of caustic soda of 36° B. and 2 c.c. of alcohol added, and the whole evaporated to dryness with constant stirring. The soap is dissolved in warm water, precipitated by adding excess of sodium chloride, filtered, and washed with salt solution. In the filtrate the sulphur compounds formed are titrated with N/10 iodine solution, after the addition of sodium carbonate and starch solution. 1 c.c. of N/10 iodine = 0.0016 gm. of sulphur. The author found in colza oil, 0.58 to 0.70 per cent. of sulphur; in olive oil, 0.031 per cent.; and in rape oil, 0.044 per cent. of sulphur.—A. S.

*Alkalinity; Indicators for —. Alterability of Stored Raw Sugars.* Köhler. Zeits. Vereins deutschen Zuckerind. 1901, 51, [546], 663—684.

THE regulations which came into force on January 1st for the determination of the alkalinity of raw sugar have caused great dissatisfaction, part of the sugar made in the earlier part of the season being found acid, and therefore subject to a deduction, only alkaline and neutral sugars being considered fit for delivery. Phenolphthalein is objected to by the raw sugar manufacturers as unsuitable, but the refiners refused to accept litmus as indicator. Consequently the Silesian Section of the Union appointed



a committee to study the action of various indicators, and to determine the alteration of alkalinity in raw sugars during storage.

They found that Herzfeld's method was satisfactory for discriminating between alkaline and acid sugars, but required supplementing as regards the examination of neutral sugars. One of the eight samples examined was pronounced by part of the commission to be alkaline by Herzfeld's test; by the others, acid. The sugar, however, was quite neutral; in fact, to litmus and turmeric it was alkaline. For when 5 c.c. of centinormal acid were added to the 10 per cent. sugar solution, no noticeable change of colour could be observed, but on adding 1 to 2 c.c. of centinormal alkali a distinct red colour was visible. A literal interpretation of Herzfeld's instructions would make this sugar not as not-alkaline, but as acid.

The committee therefore propose a provisional modification that in the case of neutral sugars, or those which give no red colour with neutral phenolphthalein water, one of the three following methods be used:—(1) To the 10 per cent. sugar solution 1 to 3 c.c. of centinormal alkali is added; if a red coloration appears the sugar is to be pronounced neutral or not acid; (2) a portion of the sugar is to be placed on litmus paper and sprinkled with water; if the paper is coloured blue, the sugar is to be pronounced not acid; (3) the spot method with turmeric is to be applied. Further, it is suggested that the term neutral sugar, or not-acid sugar, be also introduced.

The spot method of H. Steffens is made by means of a thin tube drawn out to a capillary, with the end cut off sharp at right angles. The sugar solution is applied with this tube to a paper coloured with sensitised turmeric. Alkalinities of 1 in 160,000 have thus been distinctly shown.

Herzfeld maintains that it is unscientific to speak of a neutral sugar, that phenolphthalein is the best indicator for certain substances which are present in raw juice, act the part of acids, and give the juice the power of self-inversion when heated. Such bodies are pectin substances, which are converted into parapeptic acid on heating; asparagin and glutamin, which at higher temperatures show increased acid properties; the latter are only imperfectly removed on defecation.

A sugar which had been found to be neutral, but alkaline to turmeric, was heated in solution for 16 hours at 95° C., and showed 0.26 per cent. of invert sugar. Another, that was alkaline to phenolphthalein, heated for the same time, showed under 0.05 per cent.

Ostwald regards the inversion of sugar as the true test of acidity, so that indicators are only a secondary, and not always a reliable sign. Sugars also which are not alkaline to phenolphthalein frequently keep well in store, no doubt, but those which are alkaline keep better, and it must ultimately be of great advantage to manufacturers to produce only such sugars.—L. J. de W.

**Malt Extracts; Examination of Commercial.**—W. J. Sykes and C. A. Mitchell. Analyst, 1901, 26, 227—231.

For the determination of total solids, the specific gravity of a 10 per cent. solution of the extract was taken and the number of grms. per 100 c.c. found by referring to Schultze's table. The phosphoric acid was precipitated as phosphomolybdate, weighed as the ammonium compound, and the result divided by 28.5.

The statement of Bömer, that zinc sulphate can replace ammonium sulphate for salting out "albumose nitrogen" in malt extracts, has not been confirmed; the authors cite numbers which show that both sulphates give practically the same results. In some cases determinations were made of the "nitrogen" precipitated by bromine in the filtrate from the zinc sulphate precipitations by Allen's modifications of Rideal and Stewart's method. It was found that the amount precipitated in this way closely approximated to that of the albumose nitrogen and to that precipitated by phosphotungstic acid.

The specific rotatory power was determined in the 10 per cent. solution, and calculated on the total solids. Kieselguhr was found to aid the clarification.

In the estimation of the diastatic power the authors decided to dilute the sample under examination to a specific gravity of 1013—that is, to the same specific gravity as the malt extract used in determining the diastatic power of a malt, and to calculate the result in the same way as if examining a malt. This will give an estimate of the amount of diastatic power which has been lost during the manufacture of the extract. For example, a commercial malt extract was diluted to a specific gravity of 1013; 1 c.c. of this was added to 100 c.c. of a 2 per cent. solution of soluble starch, and after the prescribed treatment the reduced copper weighed (this Journal, 1896, 560). A correction was made for the reducing substances present in the malt extract.

The following table gives the complete results of the analyses of a series of malt extracts; also of several substances which might be used as adulterants:—

	Water.	Total Solids.	Ash.	Phosphoric Acid.	Total Nitrogen.	Albumose Nitrogen.	Nitrogen precipitated by Phosphotungstic Acid.	Ammoniated Nitrogen.	Reducing Substances as Maltose.	Specific Rotation of Solids.	Diastatic Capacity.
1. Wort evaporated on water-bath.	18.80	81.20	1.60	0.67	0.60	0.09	..	0.05	..	97.2	..
2. Medicinal.	19.90	80.1	1.04	0.49	0.70	0.15	0.103	0.09	59.5	100.8	21.6
3. French.	19.90	80.1	1.62	0.50	0.42	0.098	0.21	0.021	51.04	117.3	12.0
4. " "	18.20	81.8	1.62	0.52	0.79	..	..	0.032	52.3	110.3	10.0
5. Scotch	23.4	76.6	1.90	0.76	0.53	..	..	..	54.0	97.9	13.0
6. English medicinal	21.1	78.9	1.32	0.53	0.77	0.19	..	..	56.0	111.5	5.8
7. " "	21.3	78.7	1.75	0.77	1.52	0.17	0.19	..	56.2	63.5	100.0
8. " "	19.9	80.1	2.20	0.66	1.03	..	..	..	56.1	87.3	25.7
9. " "	17.5	82.50	1.60	0.75	0.68	..	..	..	51.4	115.1	22.0
10. Bakers'.	24.4	75.6	2.10	0.79	0.98	0.14	..	..	52.56	85.9	24.0
11. "Diastase"	21.1	78.9	3.00	1.15	2.25	..	..	..	45.80	38.1	115.0
12. Glucose	15.8	84.2	0.55	0.065	0.20	..	..	..	62.8	128.2	..
13. Maltose dextrin, "non-fermentable."	24.9	75.1	0.55	0.23	0.19	..	..	..	50.3	153.1	..
14. Dextrin syrup.	18.0	82.0	1.0	0.24	0.30	..	..	..	55.63	134.1	..

—J. L. B.

#### Fusel Oil in Alcoholic Liquids; Determination of

E. Beckmann. Zeits. angew. Chem. 1901, 14, [38] 951.

The author's method is based upon the fact that the alcohols of fusel oil readily form esters with nitrous acid. The alcoholic liquid under examination is treated with calcium chloride and then extracted by shaking with carbon tetrachloride. The fusel oil in the extract is converted into

nitrous esters by treatment with sodium nitrite and potassium bisulphate, and the excess of nitrous acid removed by treatment with sodium bicarbonate. The liquid is then shaken with water, and the combined nitrous acid titrated with permanganate in a sulphuric acid solution which is kept cool in ice-water. Any aldehydes which may be present in the carbon tetrachloride solution are previously removed by means of sodium bisulphite.—C. A. M.



**Albuminoids; Determination of the Decomposition Products of —.** E. Hart. *Zeits. physiol. Chem.* **33**, 347—362. *Chem. Centr.* 1901, **2**, [15], 854—856.

THE results obtained by Haslam (see this Journal, 1901, 494), as to the amount of hexone bases given by heteroalbumose on decomposition with boiling dilute sulphuric acid, differed from those obtained by Pick (*Zeits. physiol. Chem.* **28**, 219), who used boiling concentrated hydrochloric acid. In an attempt to discover an explanation of this difference, the author prepared two samples of heteroalbumose from syntonin, (1) by a coagulation process, and (2) by Pick's alcohol-precipitation method. Both preparations contained about 16.1 per cent. of nitrogen (on the ash-free substance), but the product obtained by precipitation with alcohol yielded much more ash (consisting essentially of sodium chloride) than that obtained by coagulation. From the filtrates from the heteroalbumose, protalbumose was also prepared by Folin's method (*Zeits. physiol. Chem.* **25**, 152). The different substances were then decomposed with sulphuric acid. The following results were obtained:—

	Syntonin.	Hetero- albumose (by Coagu- lation).	Hetero- albumose (by Pre- cipitation with Alcohol).	Prot- albumose.
Percentage of Total Nitrogen.				
Histidin.....	4.53	1.92	0.64	5.76
Arginin.....	10.29	17.46	17.36	9.30
Lysin.....	3.98	3.76	8.11	3.76
Ammonia.....	4.32	2.92	4.95	4.01
Humin sub- stances.	8.34	11.65	6.89	9.17
Sum.....	31.46	37.71	37.86	32.00

The figures show that the heteroalbumose obtained by precipitation with alcohol yields less humin-nitrogen (and correspondingly more lysin-nitrogen and ammonia-nitrogen) than that obtained by coagulation. This diminution of the yield of humin-nitrogen is undoubtedly due to the presence of a fairly large quantity of sodium chloride, and by further experiments, the author found that by decomposition with sulphuric acid in presence of sodium chloride (or sodium sulphate, and, to a less extent, of dilute hydrochloric acid), albumin can in some cases give non-nitrogenous humin substances.—A. S.

**Formaldehyde; Determination of —.** A. G. Craig. *J. Amer. Chem. Soc.* 1901, **23**, 638—643.

VARIOUS methods for the determination of formaldehyde or trioxymethylene have been examined by the author, with the following results. The acid permanganate method yields concordant numbers. The Grützner method of heating with potassium chlorate, a measured volume of standard silver nitrate solution and nitric acid, and weighing the silver chloride formed on titrating the remaining silver nitrate, seems to give unreliable results. In Blank and Finkenbeiner's method, 1 gram. of trioxymethylene or 2 c.c. of formaldehyde is mixed with 25 c.c. of double normal soda solution in a flask, and 50 c.c. of 2.5 or 3 per cent. hydrogen peroxide added cautiously, the addition lasting three minutes; after standing for 10 minutes, the liquid is titrated with sulphuric acid using litmus as indicator. As thus carried out, the method gives good results. The Legler method which depends on the formation of hexamethylene tetramine by the action of ammonia on formaldehyde yields accurate results.—T. H. P.

**Phenol, Salicylic Acid, and Salol; Determination of —, in Dressings.** F. Telle. *J. Pharm. Chim.* 1901, **14**, [7], 289—291.

IN place of using bromine water in titrating phenol, salicylic acid and salol, the author proposes to add standardised sodium hypochlorite solution to an acid solution containing potassium bromide. The hypochlorite solution is prepared by diluting 35 c.c. of the commercial solution (containing about 10 per cent. of available chlorine) to 1 litre. One

mol. of salicylic acid is equivalent to 4 Br., 1 mol. of phenol to 6 Br., 1 mol. of salol to 10 Br.

The process is applied to dressings as follows:—2 grms. of the dressing, cut or torn into pieces, are moistened with alcohol in a 200 c.c. flask, and 2 c.c. of caustic soda (33 per cent.) added. After shaking, 40—50 c.c. of water are added, the liquid boiled, cooled, made up to the mark, and 1.5 c.c. of water added to compensate for the volume occupied by the cotton. The flask is well shaken and the liquid filtered; 50 c.c. are taken for an estimation of salicylic acid, 25 c.c. for phenol or salol. To this are added 5 c.c. of potassium bromide and 15 drops of hydrochloric acid, the hypochlorite is then run in until the liquid appears yellow. In the case of salicylic acid, 5 c.c. each of chloroform and alcohol are added; at the end of the titration the former is seen to be yellow.—A. C. W.

**Caffeine; Determination of —.** Beitter. *Chem.-Zeit.* 1901, **25**, [81], 869.

THE author classifies the methods discussed by Juckenack and Hilger (this Journal, 1897, 567). As regards the methods in which water is used as the extracting solvent, he considers these liable to give erroneous results. He has been unable to avoid loss in using the method of Brunner-Leins (this Journal, 1898, 965). Of organic solvents, chloroform is, in his opinion, the most satisfactory, and he has obtained very concordant results by Keller's method of extraction (this Journal, 1897, 568). On the other hand, the method of purification used by Keller is stated to be liable to cause loss, so the following modification is recommended:—The chloroform extract is freed from chloroform, and the residue taken up with 15 c.c. and subsequently with three successive portions of 5 c.c. of water on the hot water bath, and filtered. The aqueous extract is then extracted with chloroform, and the residue left on evaporation of the chloroform dried at 85° C., and weighed. The caffeine thus obtained is white and crystalline, and only in the case of Paraguay tea is it slightly coloured. The method was found to give accurate results in test experiments.—C. A. M.

**Morphine in Opium; Determination of — by means of Ammoniacal Silver Chloride.** C. Reichard. *Chem.-Zeit.* 1901, **25**, [77], 816—818.

THE author finds that the use of ammoniacal silver chloride has advantages over that of silver nitrate for the determination of morphine (see this Journal, 1901, 160). In the case of the first reagent the presence of chloro-compounds exerts no disturbing influence on the determination; the reaction also takes place more quickly than with silver nitrate. The procedure is as follows:—(1) If the opium is not in the solid state, but, for instance, in alcoholic solution, the latter is mixed with three times its volume of water and evaporated on the water bath to one-third of the total volume. (2) For solid opium, a weighed quantity of the dry powder is treated with 10 to 20 times its weight of boiling water and the solution frequently agitated for the space of an hour, by which time all the morphine is in solution. The solution obtained in either of these ways is filtered from the insoluble residue, which is washed several times with small quantities of boiling water. To the total filtrate is added a slight excess of ammoniacal silver chloride, the liquid being then warmed to accelerate the reduction. The brownish tint of the liquid due to the precipitation of the alkaloids of the opium by ammonia gradually changes to the black of the reduced silver. After several hours the precipitated silver and mixture of alkaloids are filtered off and washed with hot water until the wash water becomes clear and gives no black coloration with ammonium sulphide. After drying at 130° C., the filter paper is burned and heated with the precipitate in a weighed porcelain crucible until the latter becomes constant in weight. Two atoms of silver correspond with one molecule of crystallised morphine ( $C_{17}H_{19}O_3N + H_2O$ ). In none of the samples of opium examined by the author could the presence be detected of compounds such as sugar, which like morphine, have the property, under certain conditions, of reducing ammoniacal silver chloride. Should such substances be suspected, the



alkaloids should be precipitated by means of ammonia and removed by filtration, the filtrate being then treated with ammoniacal silver chloride; any reduction of the latter will be thus made evident.—T. H. P.

*Pomegranate Bark and Extract of Pomegranate Bark; Alkaloid Determination of —.* N. Stoeder. Pharm. Centralh. 42, [36], 547.

*Pomegranate Bark.*—20 grms. of the dry powder are treated with 100 c.c. of chloroform and 5 c.c. of solution of ammonia, and allowed to stand for 12 hours with frequent agitation; 20 c.c. of water, or sufficient to cause the aggregation of the powder, is then added, and after thorough shaking, the mixture is allowed to subside. Seventy-five c.c. of the chloroform solution (= 15 grms. of powder) are then withdrawn, filtered, and the filter washed with more chloroform until a drop of the filtrate when evaporated leaves a residue which gives no precipitate with Mayer's reagent. Two-thirds of the chloroform is then distilled off, the residue transferred to a separator, the distilling flask being washed out with two successive 5 c.c. of chloroform. The chloroformic extract is then shaken out with 10 c.c. of N/10 HCl solution, and the acid alkaloidal solution filtered. The chloroform extract is then washed with successive portions of 5 c.c. each, of water, which are then passed through the same filter, until the filtrate is free from alkaloidal and acid reaction. Three drops of 1 per cent. alcoholic solution of hæmatoxylin are then added, and the amount of free acid titrated back with N/10 alkali. The mean molecular weight of the total pomegranate alkaloids is taken as being 147.6. From 0.4 to 0.5 per cent. of alkaloid should be present in bark of European origin, or from 1.7 to 2 per cent. in East Indian bark.

*Extract of Pomegranate Bark.*—Dry extract of European pomegranate should yield from 1.25 to 1.5 per cent. of total alkaloids when treated as follows:—Six grms. of the extract are thoroughly rubbed down with a mixture of 10 c.c. of solution of ammonia and 25 c.c. of water, and allowed to stand for three hours, with frequent shaking. 120 c.c. of chloroform are then added, and the mixture left for another 12 hours with frequent agitation. It is then allowed to separate and 1 c.c. of the clear aqueous liquid is withdrawn, shaken out with 3 c.c. of ether, and the ether evaporated; the residue, when treated with 4 drops of water, should give no alkaloidal reaction with Mayer's solution. 80 c.c. of the chloroformic solution (= 4 grms. of the extract) are then withdrawn, and treated by the method prescribed above for pomegranate bark. Extract of East Indian pomegranate bark, should contain from 4 to 5 per cent. of alkaloids; of this, therefore, only 3 grms. should be taken for the alkaloidal determination.—J. O. B.

*Quebracho Extracts, Soluble in the Cold, and their Chemical Analysis.* J. Paessler and W. Appeluis.

See under XIV., page 1124.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Radio-active Lead.* K. A. Hofmann and E. Strauss. Ber. 34, [12], 3033—3039.

THE authors have again prepared radio-active lead sulphate from a large number of minerals containing uranium, and have confirmed the results already published (this Journal, 1901, 76, 290, and 625). The principal steps in the process of preparation were—evaporation with sulphuric acid; conversion of the sulphates into sulphides by treatment with ammonium sulphide; evaporation of the sulphides with hydrochloric acid; extraction of the chloride mixture with NaCl solution (in which radio-lead chloride dissolves readily, leaving ordinary lead chloride mostly insoluble); precipitation with  $H_2S$ ; conversion of the sulphide into sulphate; and heating the latter to  $450^\circ$  for several hours.

"Radio-lead" resembles ordinary lead in the formation of insoluble sulphate and chromate; precipitation from acid

solution by  $H_2S$ ; solubility of its salts in KOH solution; solubility of the chloride and nitrate in water; precipitation by ammonium carbonate; and in the insolubility of the neutral chloride in alcohol (96 per cent.). It differs from ordinary lead in the ready solubility of its chloride in NaCl solutions; it may be separated from lead by the addition of pure NaCl, whereby the lead is mostly precipitated and the active substance remains mostly in solution; or dilute HCl (3 per cent.) may be poured over the chloride and  $H_2S$  then passed, when the new substance will go into solution, the free acid preventing formation of its sulphide. Another point of difference is that its chromate is not converted into sulphate by repeated warming with dilute sulphuric acid; it must first be digested with ammonium sulphide and the residue then evaporated with HCl and  $H_2SO_4$ . If the sulphides prepared from the sulphates by means of ammonium sulphide are treated with fuming hydrochloric acid at the room temperature, ordinary lead sulphide remains brownish-grey for a considerable time, whereas radio-lead sulphide becomes white at once. Ordinary  $PbSO_4$  gives a blue luminescence, under the cathode discharge; whilst the radio-active sulphate gives a bright blue radiation and phosphoresces in addition. The active salts sometimes become rose-coloured, but the authors do not know the conditions which give rise to this colour.

The radio-activity of the sulphate cannot be due to the admixture of or induction by radium, polonium, or actinium, as mixtures prepared from these bodies and ordinary  $PbSO_4$  are completely decomposed by the authors' method of isolation and yield inactive  $PbSO_4$ .

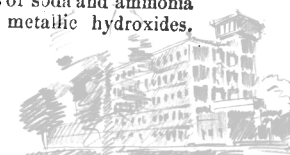
In causing the discharge of the electroscope, all the salts of radio-active lead examined are nearly equally powerful. Only the sulphate, however, affects the photographic plate through aluminium and through glass. The sulphide, chloride, chromate, &c., prepared from strongly active sulphate were photographically inactive, but on re-converting them into the sulphate, the activity was restored. The activity of radium and polonium, on the other hand, is practically the same in all their compounds. If the radio-active sulphate be evaporated with nitric and sulphuric acids, or if it is heated for 15 hours in the air at  $450^\circ C$ , the photographic activity becomes especially powerful; the activity disappears after about six months, but can be restored by exposing the sulphate to cathode rays. If the sulphate is only partly converted into sulphide, by heating it with sulphur, the activity is destroyed, but returns on allowing the substance to oxidise back to sulphate in the air. The effect of the sulphate on the gelatin-bromide plate excels that of bismuth oxychloride (polonium) prepared simultaneously from the same pitchblende, and is as great as that of the barium sulphate (radium) prepared from the same mineral. On the other hand polonium preparations discharge the electroscope much more rapidly than radio lead sulphate; therefore, the photographically demonstrable radiation is not identical with that which causes the electrical discharge.—H. B.

*Bromine on Metallic Silver; Action of — in the Light and in the Dark.* V. v. Cordier. Monatsh. für Chem. 1901, 22, [7], 707—716.

THE author has made experiments on the action of bromine on silver similar to those already described on the action of chlorine. His results show that the behaviour of bromine towards silver is quite different from that of chlorine, for whilst light favours the formation of silver chloride, it exerts a retarding influence on the production of the bromide. When a roll of silver gauze, which has been submitted to the action of bromine either in the light or in the dark, is placed in a current of carbon dioxide, no loss of bromine can be observed.—T. H. P.

*Salicylates of Sodium and Ammonium; Solubility of Metallic Hydroxides in —.* J. Wolff. Bull. de l'Assoc. des Chim. de Sucre et de Dist. 1901.

CONCENTRATED solutions of salicylates of soda and ammonia dissolve certain freshly precipitated metallic hydroxides,





The proportion of oxide dissolved varies with the concentration of the liquids and the nature of the oxide.

Cupric hydroxide dissolves instantaneously in the cold, its solubility being very great. The colour of the solution is a fine emerald green.

Ferric hydroxide and alumina only dissolve with heat at about 80° C., and are much less soluble. The ferric solution is an intense red; the alumina, colourless. Chromium hydroxide is soluble with difficulty to a green-blue solution.

The ferric solution is precipitated by concentrated soda, but not by ammonia.

The copper solutions are changed to blue without formation of precipitate, and alumina is likewise not precipitated by soda or ammonia.

If a current of  $\text{SH}_2$  gas is passed through a solution of oxide of iron and alumina in salicylate of ammonia, the iron is precipitated completely as sulphide; the alumina remains in solution. The method can only be used quantitatively when small quantities of the oxides are present.—L. J. de W.

*Egg Albumin; Formation of  $\alpha$ -Pyrrolidinedicarboxylic Acid and Phenylalanine by the Hydrolysis of*—E. Fischer. Zeits. physiol. Chem. 33, 412—416. Chem. Centr. 1901, 2, [15], 856.

AMONG the amino-acids formed on the hydrolysis of egg-albumin with hydrochloric acid, the author has isolated, by distilling the ethyl esters under diminished pressure and collecting in fractions, (1) active  $\alpha$ -pyrrolidine carboxylic acid, (2) racemic  $\alpha$ -pyrrolidine carboxylic acid, and (3) phenylalanine.—A. S.

*"Active" Oxygen. VII. Auto-oxidation of Unsaturated Hydrocarbons.* C. Engler and W. Frankenstein. Ber. 1901, 34, [12], 2933—2941.

FULVENE was discovered by Thiele (Ber. 33, 666). The fulvenes quickly absorb oxygen from the air and resinify. Dimethyl fulvene was now prepared by condensation of cyclopentadien with acetone, and was used in the form of 5—7 per cent. benzene solutions.

When a benzene solution of dimethylfulvene is shaken up with air, it gradually grows turbid and then deposits a voluminous translucent precipitate which later becomes dense, and when separated and washed represents a white granular precipitate. The time taken to deposit this compound is much shortened if the liquid is exposed to the action of light and heat. It is found that the dimethylfulvene has taken up two molecules of oxygen per molecule, the resulting compound being hence a diperoxide of the composition  $\text{C}_5\text{H}_{10}\text{O}_4$ . It explodes at 130°, and when a small quantity of it, in a melting point tube, is heated in a beaker of sulphuric acid, it explodes and generally bores a sharply defined round hole in the bottom of the beaker. The diperoxide is insoluble in benzene and its homologues and in alcohol, ether, petroleum etc.; it is soluble in, but decomposed by, hot nitrobenzene or acetic acid. It will not keep but quickly becomes yellow, especially under the action of heat and light, and loses its explosive properties. As an oxidising agent, in the ordinary sense of the word, the diperoxide has however small power; thus with titanous acid it gives a yellow coloration only after long shaking, vanadic acid being reddened under similar conditions. Methylthylfulvene behaves similarly to the dimethyl compound, whilst methylphenylfulvene takes up oxygen much more slowly, giving a peroxide of the composition  $\text{C}_{13}\text{H}_{12}\text{O}_4$ . Cyclopentadiene absorbs only a small quantity of oxygen, a resinous body being formed. By allowing (1) hexylene and (2) dimethylfulvene to absorb oxygen in presence of indigo-sulphuric acid solution it is found that half the total oxygen at first absorbed is in the "active" condition. The various possible formulae for dimethylfulvene diperoxide are discussed.—T. H. P.

*Glucose; Isomeric Acetohalogen Derivatives of*—, and *Synthesis of Glucosides.* E. Fischer and E. F. Armstrong. Ber. 34, [12], 285—2900.

THE utility of Colley's acetochloroglucose in the synthesis of the glucosides has been greatly curtailed on account of

the great difficulty in obtaining it in a pure crystalline form. Königs and Knorr (this Journal, 1901, 626) have recently prepared  $\beta$ -acetobromoglucose in the pure state, and have obtained several important derivatives, showing that the pentacetylglucose melting at 134° C., belongs to the  $\beta$  series. The authors have now succeeded in preparing both the  $\alpha$ - and  $\beta$ -acetohalogen compounds of glucose from the corresponding pentacetylglucoses. The pentacetylglucose was treated in sealed tubes at the ordinary temperature with dry, liquefied hydrogen chloride or bromide (obtained by means of liquid air). The products were:—

From $\alpha$ -pentacetylglucose,	{	$\alpha$ -Acetochloroglucose, m.p. 63°
melting at 112° C.		—64° C.
	{	$\alpha$ -Acetobromoglucose, m.p. 79°
		—80° C.
From $\beta$ -pentacetylglucose,	{	$\beta$ -Acetochloroglucose, m.p. 73°
melting at 134° C.		—74° C.
	{	$\beta$ -Acetobromoglucose, m.p. 88°
		—89° C.

The authors have also applied their method to the preparation of  $\beta$ -acetochlorogalactose, m.p. 74°—75° C., but only one pentacetylgalactose, the  $\beta$ -derivative, is as yet available. From octacetylmaltose they have obtained heptacetylchloromaltose, which yields methylmaltoside on treatment with methyl alcohol and silver acetate. From its behaviour towards enzymes, this maltoside is recognised as a  $\beta$ -derivative. Emulsin splits it up into methyl alcohol and maltose whilst yeast enzymes attack the maltose molecule, forming glucose and  $\beta$  methylglucoside, the latter reaction being exactly analogous to the hydrolysis of amygdalin by yeast enzymes.

Lastly, the authors have improved upon Michael's method for the synthesis of phenolglucoside from acetochloroglucose.  $\beta$ -Acetochloroglucose is treated with phenol and sodium ethylate in alcoholic solution for 24 hours at the ordinary temperature, and then saponified with baryta water, the yield being 35 per cent. of the theoretical. A yield of 60 per cent. was obtained by treating the acetochloroglucose in ethereal solution with dry sodium phenolate. Evidence was here obtained of the formation of a double compound, and two molecules of sodium phenolate were required.

The observations made, afford evidence in confirmation of Fischer's previous theory that the isomerism of the  $\alpha$  and  $\beta$  groups of glucosides is stereochemical, as regards the terminal carbon atom, and not structural, since all mobile hydrogen atoms are here replaced by acetyl groups.

—J. F. B.

## New Books.

NOTES ON ESSENTIAL OILS, with special reference to their Use, Composition, Chemistry, and Analysis. By T. H. W. IDRIS. With Table of Constants of the more commonly occurring Oils. 2nd Edition. Idris and Co., Pratt Street, Camden Town, London. 1900. Price 5s.

SMALL 8vo volume, containing preface, table of contents, and 223 pages of subject-matter, with alphabetical index. The subject-matter is classified as follows:—CONSTITUENTS OF ESSENTIAL OILS: I. Terpene and Camphor Groups. II. Benzene Derivatives. III. Methane Derivatives: (a) Alcohols; (b) Aldehydes; (c) Acids; (d) Ketone. IV. Geraniol Series. ESSENTIAL OILS.

CONDENSATION. Ein Lehr- und Handbuch über Kondensation und alle damit zusammenhängenden Fragen, auch einschliesslich der Wasserückkühlung; für Studierende des Maschinenbaues, Ingenieure, Leiter grösserer Dampfbetriebe, Chemiker und Zuckertechniker. Von F. J. WEISS, Civilingenieur in Basel. Julius Springer, Berlin. 1901. Price M. 10.

LARGE 8vo volume, contains preface, table of contents and 384 pages of subject-matter, illustrated with 96 engravings. There is no alphabetical index, but the table of contents is well arranged and clear. Its subdivisions are as follows:—CLASSIFICATION OF CONDENSORS: A. Mixed Condensation. B. Surface Condensation. C. Time for the



first Exhaustion of the Condensing Space. D. Power Requirements. E. Uses of Condensation. F. Calculation of a large Central Condensing Plant. G. Evaporation Connections. H. Valve Gearing and Distribution of Steam with Condensing Engines, &c.

**JAHRBUCH DER CHEMIE.** Bericht über die wichtigsten Fortschritte der reinen und angewandten Chemie, unter Mitwirkung von den Herren H. Beckurts, C. A. Bischoff, E. F. Dürre, J. M. Eder, P. Friedländer, C. Häussermann, F. W. Küster, J. Lewkowitsch, M. Märcker, W. Muthmann und F. Röhmman. Herausgegeben von RICHARD MEYER. X. Jahrgang. 1900. Friedrich Vieweg und Sohn, Braunschweig. 1901. Unbound, M. 14. Bound, M. 15.

THIS Jahrbuch contains preface, list of contributors, table of contents, and subject-matter filling 532 pages, and followed by indexes of subject-matter and authors. The subjects represented, with contributors, are as follows:— I. Physical Chemistry. F. W. Küster. II. Inorganic Chemistry. W. Muthmann. III. Organic Chemistry. C. A. Bischoff. IV. Physiological Chemistry. F. Röhmman. V. Pharmaceutical Chemistry. H. Beckurts. VI. Chemistry of Foodstuffs. H. Beckurts. VII. Agricultural Chemistry. M. Märcker and W. Naumann. VIII. Metallurgy. E. F. Dürre and Fr. v. Kügelgen. IX. Fuels and Explosives; Inorganic Chemical Technology. C. Häussermann. X. Technology of the Carbohydrates and the Fermentation Industry. M. Märcker, W. Naumann, and E. H. Schultze. XI. Technology of Fats, Oils, and Petroleum. J. Lewkowitsch. XII. Chemistry of Coal-tar and Dyestuffs. Richard Meyer. XIII. Chemical Technology of Textile Fibres. P. Friedländer. Photography. J. M. Eder and E. Valenta.

**A DICTIONARY OF DYES, MORDANTS, AND OTHER COMPOUNDS USED IN DYEING AND CALICO PRINTING,** by CHRISTOPHER RAWSON, WALTER M. GARDNER, and W. F. LAYCOCK, Ph.D. Chas. Griffin & Co., Ltd., Exeter Street, Strand, London. 1901. Price 16s. nett.

8vo volume, containing preface, list of firms referred to and subject-matter filling 372 pages,

In this dictionary each substance receives first a general description as regards origin, preparation, and general properties. Then follows a paragraph under the head of *Properties*, next *Application*, then *Examination and Analysis*, and, finally, *Impurities*.

**PHARMACOPEDIA.** A Commentary on the British Pharmacopœia, 1898. By EDMUND WHITE, B.Sc., and JOHN HUMPHREY. Henry Kimpton, 13, Fumival Street, London, E.C. 1901. Price 14s. nett.

CROWN quarto volume, containing 576 pages of subject-matter and 46 full-page plates of illustrations. At the end is an alphabetical index of subjects. The preface gives a full definition of the general purpose of the work, the works of reference consulted, &c. Then follows the subject-matter, arranged in alphabetical form, each substance being first generally described, and then treated under the headings of "CHARACTERS AND TESTS," and finally, under "NOTES."

**DIE PHYSIKALISCHEN UND CHEMISCHEN METHODEN DER QUANTITATIVEN BESTIMMUNG ORGANISCHER VERBINDUNGEN.** Von Dr. WILHELM VAUBEL. Julius Springer, Berlin. 1902. 2 volumes. Unbound, M. 24; bound, M. 26.40.

THE first volume contains 593 pages of subject-matter, and is illustrated with 74 wood engravings. It commences with a preface, and this is followed by a table of contents. This first volume is devoted to the following subjects:— I. Method of Determining the Melting- and Solidifying Points. II. Boiling Points and Vapour Pressures. III. Specific Gravities. IV. Method of Solution and Extraction. V. Method of Precipitation. VI. Of Capillary Analysis. VII. Determining Viscosity. VIII. Determining the Electrical Conductivity. IX. Estimating the Refractive

Indices. X. Calorimetric Methods. XI. Spectro-Colorimetric Method. XII. Method of Dye Testing or Dye Trials. XIII. Determining the Optical Activity. XIV. Estimating the Heat of Combustion. XV. Heat of Reaction. XVI. Flash-Point and Temperature of Ignition.

VOLUME II. contains 518 pages of subject-matter, and 21 wood engravings. It is devoted to the following subjects, arranged as chapter titles. I. Method of Determining Carbon and Hydrogen. II. Nitrogen. III. Halogens. IV. Sulphur and Phosphorus. V. Method of Evaporation and Ash Determination. VI. Acidimetry. VII. Alkalimetry. VIII. Method of Saponification. IX. Acetylisng. X. Benzoylising. XI. Method of Bromination. XII. Iodising. XIII. Diazotising. XIV. Method of Formation of Azo-Dyestuffs. XV. Methods of Determination by Condensation with Phenylhydrazine. XVI. Aldehydes with Phenols. XVII. Aldehydes with Amines. XVIII. Decomposing with Acids. XIX. Treatment with Nitrous Acid. XX. Action of Nitric Acid. XXI. Oxidising with Arsenic Acid. XXII. Oxidation with Concentrated Sulphuric Acid. XXIII. Method of Oxidation with Halogens and their Oxy-compounds. XXIV. With Chromic Acid. XXV. With Permanganate. XXVI. Methods of Oxidation with Fehling's Solution or other Cupric Compounds. XXVII. With Mercuric Compounds. XXVIII. Method of Determining with Silver Nitrate. XXIX. Oxidation with Compounds of the Gold-Platinum Group. XXX. Reduction with Sulphurous or Hydrosulphurous Acid. XXXI. Reduction with Stannous Chloride. XXXII. Estimation by means of Enzyme or Fermentative Action. XXXIII. Determining the Antitoxins.

At the close of this Vol. II. is the alphabetical index of subjects covering both volumes.

## Trade Report.

### I.—GENERAL.

#### TRADE OF EGYPT.

*Ch. of Comm. J., Nov. 1901.*

Small increases occurred, during 1900, in imports of hides and leather goods, stone and earthenware, and glass. The export of gum, of which large stocks are warehoused at Kordofan, but cannot be moved, from lack of means of transport, amounted to 25,075 qrs., of the value of 111,211 £E. The export of earth-nut kernels also showed a large reduction. Of cane sugar, 37,811 qrs., and 906,451 cantars of 50 kilos., were exported.

#### IMPORTS OF TANGIER (MOROCCO).

*Foreign Office Annual Series, No. 2723.*

Candles came almost entirely from the United Kingdom, but 132 cases were brought from France, and 506 from Belgium, in 1900, which is an increase on former figures.

The principal chemicals were potash, 564l., and soda, 380l.

The United Kingdom sent 1,075l. worth of matches, against 2,125l. from Italy, and 1,695l. from France.

The oils imported were cotton-seed oil, 3,967l., of which 2,286l. came from the United Kingdom; linseed oil, 72l.; olive oil, 574l., from Spain.

#### CHEMICAL FIRE ENGINES FOR HAYTI.

*Bd. of Trade J., Nov. 7, 1901.*

The United States Minister at Port-au-Prince calls the attention of manufacturers of chemical fire engines to the market in Hayti. He says: "In this capital, as well as in all of the principal cities of the Republic, the houses are chiefly constructed of wood, which, in this climate, soon becomes like tinder, needing but a match to start a large and destructive conflagration that destroys half a city before the flames are mastered. The cities have good engines, which are practically useless, on account of the limited supply of water."



## TARIFF MODIFICATIONS IN MEXICO.

*Bd. of Trade J., Nov. 7, 1901.*

With regard to the modifications introduced into the Mexican Customs Tariff from Nov. 1st, the following is a list of articles affected, the new rates of duty being compared with those hitherto in force. Surtaxes, amounting in the aggregate to  $10\frac{1}{2}$  per cent. of the duties, are leviable on the new, as they were on the old, rates:—

[NOTE.—Additions to the Tariff are printed in italic type.]

Tariff No.	Tariff Classification.	New Rates of Duty.	Former Rates of Duty.
50	Fish oil, in glass bottles .....	Kilo.— \$ c.	Kilo.— \$ c.
50a	" in tins or wooden vessels.	(legal) 0 12 } (legal) 0 10	
64	Cod-liver oil, in glass bottles.	" 0 12 } " 0 10	
64a	" in tins or wooden vessels.	(gross) 0 10 }	
66	Vaccine virus and serums for hypodermic injections.	Free.	Free.
173a	Potato starch .....	Kilo.— (gross) 0 03	Kilo.— (net) 0 04
	Cotton-seed oil—		
	Purified—	(net) 0 10	
182	In tank-car or tank-ship.	(gross) 0 09	" 0 10
182a	In barrels, drums, or tins.		
	Crude—	(net) 0 05	
182b	In tank-car or tank-ship.	100 kilos.— (gross) 4 50	" 0 05
182c	In barrels, drums, or tins.		
	Cocoanut oil, and crude or boiled linseed oil—	Kilo.— (net) 0 10	(net) 0 10
183	In tank-car or tank-ship.	(gross) 0 09	(gross) 0 10
183a	In barrels, drums, or tins.	" 0 06	(gross) 0 05
188	Vegetable tar .....	" 0 05	
295	Dyewoods and barks, whether pulverised or not.	Free.	" 0 01
260	Crucibles of platinum .....		
291	Tin, in bars or in matte .....	Kilo.— (gross) 0 12	(legal) 0 12
294	Lead, in bars, pigs, or ingots.	" 0 03	(gross) 0 05
297	Lead, manufactures of, not elsewhere specified.	(legal) 0 03	(legal) 0 07
300	Lead in sheets, or in tubes or pipes; also lead for window panes.	(gross) 0 05	(gross) 0 05
321a	Ferro-manganese, containing 25 per cent. or more of manganese.	" 0 05	Not specially tariffed.
360	Magnesium, barium, or strontium carbonates.	" 0 03	Kilo.— (legal) 0 03
564	Emery .....	" 0 01	" 0 01
565	Spar .....	" 0 08	" 0 08
568	Ochres .....	" 0 07	" 0 08
574	Plumbago .....	" 0 05	" 0 08
596	Paraffin candles .....	" 0 18	(gross) 0 20
415	Tiles, ridges, and ventilators of earthenware, for roofs, and earthenware tubes for drainage.	Thousand 2 50	Thousand 2 50
417	Insulators, of glass or china, for electrical conduction.	Free.	Free.
659	Sizing for woven stuffs .....	Kilo.— (gross) 0 04	(legal) 0 05
664	Natural or artificial madders (alizarins).	" 0 07	(gross) 0 05
676	Bicarbonate of potash or soda	(legal) 0 08	(legal) 0 03
676a	Carbonate of potash or soda ..	(gross) 0 02	" 0 03
679	Colours in powder or in crystals.	" 0 07	(gross) 0 08
679a	Colours prepared .....	" 0 12	" 0 08
683	Chloride of lime, soda, or potash.	" 0 01	(legal) 0 01
693	Dye-woods, extracts of .....	" 0 07	(gross) 0 05
698	Medicinal soaps .....	(legal) 0 75	(legal) 0 40
703	Orchil .....	(gross) 0 07	(gross) 0 05
714	Salts of strontium and of barium, not otherwise specified.	(legal) 0 03	(legal) 0 03
722	Sulpho-olein, alkaline .....	(gross) 0 05	(gross) 0 05
734	Beer, cider, and refreshing beverages, in bottles.	(net) 0 20	(net) 0 20
735	Beer and cider, in barrels ..	(gross) 0 08	(gross) 0 08
743	White or cream-laid paper, satined or not, suitable for printing.	(legal) 0 07	Not specially tariffed.
745	White or coloured paper, satined or not, suitable for writing.	" 0 20	" "
746	Straw and other wrapping paper, not elsewhere specified.	" 0 06	Kilo.— (legal) 0 05

Tariff No.	Tariff Classification.	New Rates of Duty.	Former Rates of Duty.
753	Paper, painted, glazed, embossed, or enamelled; including that known as "couché."	Kilo.— \$ c. (legal) 0 10	Kilo.— \$ c. (legal) 0 10
854	Lubricating oils .....	(gross) 0 05	" 0 05
898a	Scented soap .....	(legal) 0 75	" 1 00

## IMPORTS OF REVAL (RUSSIA).

*Foreign Office Annual Series, No. 2719.*

Reval took, during 1900, from the United Kingdom, drugs, 104,515 poods; colours and dyestuffs, 5,142 poods; and gums and resins, 57,885 poods. The principal imports from Germany were drugs, 136,093 poods; colours and dyestuffs, 34,248 poods; and gums and resins, 22,874 poods.

## CUSTOMS DECISIONS OF VICTORIA.

*Bd. of Trade J., Oct. 31, 1901.*

Description of Articles.	Tariff No.	Rate of Duty.
Casein, used in photographic process..	..	Free.
Paint—		
Nubian, as varnish .....	577	2s. per gall.
Removing compound, being a solution of caustic alkali.	..	Free.
Paper—		
Peltine, chemically prepared, viz.—		
Uncut .....	433	6s. per cwt.
Cut .....	431	2d. per lb.
Varnish—		
Thin litho and extra strong litho....	577	2s. per gall.
Vinegar—		
Tarragon .....	579	6d. per gall.

## COMPARISON OF BRITISH AND AMERICAN TRADE FOR THE FIRST SEVEN MONTHS OF 1901.

*Comm. Intelligence, Oct. 26, 1901.*

**Candles.**—British exports of candles were 249,977l., the American exports being 31,000l.

**Cement.**—British exports of cement were 375,297l., against the American total of 76,825l.

**Chemicals, Drugs, Dyes, and Medicines.**—British exports were as follows during the seven months:—

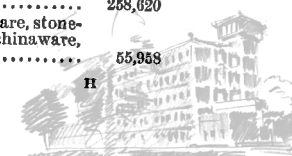
Bleaching materials—	£	£
To United States .....	123,880	
Other countries .....	51,566	
		175,446
Sulphate of copper .....		804,696
Medicines, &c. ....		789,137
Soda compounds .....		618,370
Other articles .....		1,610,905
Total .....		3,998,554

## American exports were as follows:—

	£
Acids .....	23,350
Ash .....	7,453
Baking powder .....	34,764
Sulphate of copper .....	439,916
Dyes .....	46,237
Ginseng .....	57,416
Acetate of lime .....	123,159
Medicines .....	388,615
All other .....	635,280
	1,776,190

## Glass and Earthenware:—

British exports of—	£	American exports of—	£
Glass and glass-ware .....	621,610	Glass and glass-ware .....	258,620
Earthenware, stone-ware, chinaware, &c. ....	1,187,669	Earthenware, stone-ware, chinaware, &c. ....	55,953



**Rubber :—**

Manufactures of caoutchouc.....	648,859	India-rubber manufactures and scrap	386,000
Caoutchouc shoes..	89,392	India-rubber shoes..	58,271

**Chemical Manures.**—American returns place these goods under the heading "Fertilisers," and divide their exports into "crude phosphates," 399,308 tons, and "all other," 7,853 tons.

British chemical manures exported to—	£	American fertilisers exported to—	£
Germany.....	171,267	Germany.....	218,120
Belgium.....	67,782	United Kingdom...	71,724
France.....	95,936	France.....	57,240
Spain.....	335,426	Other European Countries.....	275,375
British West Indies and Guiana.....	117,196	West Indies and Bermuda.....	7,456
Other countries.....	582,242	Other countries.....	49,309
<b>Total .....</b>	<b>1,369,849</b>	<b>Total .....</b>	<b>679,224</b>

**Hides and Skins :—**

British exports of skins and furs of all sorts to—

France.....	202,150
United States.....	478,436
Other countries.....	198,164
<b>British total .....</b>	<b>878,780</b>

American exports of furs and fur skins to—

United Kingdom.....	331,741
France.....	13,135
Germany.....	93,010
British North America.....	100,084
Other countries.....	7,329
<b>American total .....</b>	<b>545,359</b>

American exports of hides and skins to—

United Kingdom.....	2,400
Germany.....	47,254
British North America.....	41,153
Other countries.....	15,110
<b>American total .....</b>	<b>105,917</b>
<b>American total .....</b>	<b>651,276</b>

**Soap and Starch :—**

British exports of—	£	American exports of—	£
Soap.....	668,147	Soap.....	207,109
		Starch.....	218,418
<b>Sugar :—</b>			
Sugar, refined, and candy.....	240,709	Molasses.....	34,562
		Syrup.....	306,515
		Brown sugar.....	875
		Refined.....	50,805
		Candy and confectionery.....	66,484
<b>British total ....</b>	<b>240,709</b>	<b>American total ..</b>	<b>459,041</b>

**Tin :—**

Unwrought tin.....	70,651	Manufactures of tin ..	64,999
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**Zinc :—**

Zinc and spelter:		Zinc ore.....	159,016
Wrought and unwrought.....	99,502	Pigs, bars, plates, and sheets.....	42,753
		All other manufactures.....	10,633
<b>British total.....</b>	<b>99,502</b>	<b>American total ..</b>	<b>212,472</b>

**RUSSIAN CUSTOMS REGULATIONS.****Foreign Office Miscellaneous Series, No. 569.**

This is a translation, by Mr. A. A. Wotzel, of the Board of Trade, of the Russian regulations concerning the receipt of goods into the jurisdiction of the customs establishments, the inspection thereof, payment of duty thereon, and release thereof.

**CHEMICAL IMPORTS OF SICILY (ITALY).****Foreign Office Annual Series, No. 2716.**

Caustic soda comes chiefly from the United Kingdom.

Carbonates comprise carbonate of lead, 2,000 kilos., in 1900; carbonate of potash, 53,000 kilos., in 1900, and 67,000 kilos., in 1901; carbonate of soda, 16,000 kilos., in 1900, and 14,000 kilos., in 1901. Of the total imports of carbonates the United Kingdom supplied 134,000 kilos., in 1900, to 69,000 kilos., supplied by Germany.

Chlorides consist of chloride of lime, of which 87,000 kilos. were imported in 1900, and 39,000 kilos., during 1901, and potassium chloride, imported to the extent of 139,000 kilos., in 1900, and 59,000 kilos., during 1901. Of the total amount during 1900, 85,000 kilos. came from Germany alone.

During 1900, Germany provided 140,000 kilos. of nitrate of soda; the United Kingdom only supplied 14,000 kilos.

Sulphates consisted principally of sulphate of ammonia, to the extent of 697,000 kilos., in 1900, and 674,000 kilos., in 1901, and sulphate of copper 173,000 kilos., in 1900, and 301,000 kilos., in 1901; the greater part of the latter article came from the United Kingdom.

**TRADE OF ZANZIBAR.****Foreign Office Annual Series, No. 2718.**

**Imports.**—During 1900, hides showed an increase of 17,234l.

**Exports.**—Copa showed a decrease of 26,644l.; gum copal, 16,574l.; rubber, 16,012l.; and ivory, 11,958l. Hides showed an increase of 15,173l.

**IMPORTS OF JAPAN.****Chem. and Druggist, Oct. 26, 1901.**

The following table gives the values in yen\* of the principal drug staples imported for 10 years :—

Year.	Mercury.	Soda-ash.	Caustic Soda.	Chlorate of Potash.	Amorphous Phosphorus.	Paraffin Oil.	Bicarbonate of Soda.	Nitrate of Soda.	Glycerin.
1891	105,660	..	255,308	207,907	80,873	143,609	43,152	..	21,608
1892	95,546	..	173,010	309,917	86,086	225,974	60,218	..	26,807
1893	115,082	..	192,426	742,317	166,637	150,611	72,565	..	26,855
1894	127,541	..	205,467	840,640	178,232	363,015	80,754	..	41,291
1895	141,302	126,149	234,000	419,053	260,823	266,340	102,917	..	59,004
1896	139,302	146,698	84,263	429,042	174,898	192,553	119,113	288,696	66,235
1897	156,527	184,060	229,593	497,651	280,636	327,916	115,616	140,443	90,912
1898	176,303	373,370	422,714	632,060	298,253	270,349	143,335	117,320	268,786
1899	219,013	482,877	521,852	418,884	216,124	286,807	157,710	231,488	272,867
1900	258,698	..	929,326	679,312	244,979	511,559	153,564	41,114	89,294

\* 1 yen = 2s. 0½d.

**IMPORTS AND EXPORTS OF BOSNIA AND HERZEGOVINA (AUSTRIA).****Foreign Office Annual Series, No. 2715.**

**Imports** which showed an increase, during 1900, were minerals, 1,064 tons, and drugs, perfumery, and colours, 878 tons.

There was a falling-off in sugar, 448 tons, and bricks, earthenware, and china, 2,569 tons.

**Exports** which showed an increase were minerals, 9,680 tons; dyestuffs, 956 tons; and chemicals, 818 tons.

Those showing a decrease were sugar, 319 tons, and mineral oils, 3,937 tons.



## CHEMICAL PREPARATIONS IN HUNGARY.

U.S. Cons. Reps., Nov. 1901.

Chemical preparations (apart from gunpowder, which forms a monopoly of the Hungarian Government) can be freely imported, with the following exceptions, mentioned in the customs administrative ordinance of the Minister of Finance, issued in 1882:—

White face paint may be imported only with special license of local authority where consignee resides.

Explosives and inflammables, under the existing regulations as to safety—collodion cotton up to 1 kilo. (2·2046 lb.) net, with license of local authorities.

Any other substances concerning which special orders may be issued, e.g., liquors, foods, and other objects injurious to the public health.

## THE AUSTRALIAN TARIFF.

Chem. and Druggist, Nov. 16, 1901; and

Bd. of Trade J., Nov. 21, 1901.

Subjoined are particulars of the Australian tariff which came into operation on October 8 last, from the paper published for the Government of the Commonwealth of Australia. The tariff is divided into 16 divisions, namely:—(1) Stimulants, (2) narcotics, (3) sugar, (4) agricultural products and groceries, (5) apparel and textiles, (6) metals and machinery, (7) oils, paints, and varnishes, (8) earthenware, cement, china, glass, and stone, (9) drugs and chemicals, (10) wood, wicker, and cane, (11) jewellery and fancy goods, (12) leather and rubber (13) paper and stationery, (14) vehicles, (15) musical instruments, and (16) miscellaneous. The letters "n.e.i." mean "not elsewhere included."

## Spirits, viz.:—

Spirits and spirituous compounds, n.e.i., when not exceeding the strength of proof.....	per gall.	14s.
When exceeding the strength of proof...	per proof gall.	14s.
Amyl alcohol and fusel oil.....	per gall.	14s.
Methyl alcohol and collodion.....	"	3s.
Perfumed and bay rum .....	"	25s.

Wine, medicinal or medicated and Vermouth (in bottle) .....

Glucose..... per cwt. 8s.

## Sugar, viz.:—

The produce of sugar-cane, including molasses, golden syrup, and syrups, n.e.i. ....

Blue, laundry .....

Candles, tapers, and night-lights; solid spirit-heaters, including the weight of the immediate containing package; stearin, paraffin wax, beeswax, and Japanese or vegetable wax; also lard and refined animal fats .....

Limejuice and other fruit-juices and fruit-syrups (non-spirituous).....

Linseed cake and oil-cake.....

## Soap, viz.:—

Perfumed, toilet, and medicated (including the weight of wrappers) .....

N.E.I., including polishing .....

Starch .....

Manures .....

Metals and manufactures of metals .....

Table-waters, aerated or mineral, and preparations packed for household use for the production thereof, including sparklets; also ale, porter, and other beer, cider, and perry containing less than 2 per cent. of proof spirit ....

(Special exemptions from metal duties: Crucibles; capsules, metallic. Electrical materials—viz., accumulators or storage batteries, except glass jars, cable (covered), carbons, incandescent lamps. Scales—viz., chemical, analytical, and assay.)

Blackings, including dressings, soaps, oils, inks, pastes, polishes, stains, and varnishes for leather; Berlin and Brunswick blacks, furniture oil-paste and polish, and bronzing and metal liquids .....

Greases, axle, and thickened or solidified oils; solid or viscous compounds for lubricating, and tallow (unrefined) .....

## Oils, viz.:—

Cotton-seed, in bulk or otherwise .....	per gall.	2s.
(Including castor), in vessels not exceeding 1 gall.:—		
½ pints and smaller sizes .....	per doz.	6d.
¾-pints and over ½-pints .....	"	1s.
Pints and over ¾-pints .....	"	2s.
Quarts and over pints.....	"	4s.
Over a quart.....	per gall.	1s. 4d.

## In vessels, exceeding 1 gall., viz.:—

Olive .....	"	1s. 4d.
Castor, china, colza, linseed, gasoline, mineral, spirit oils, n.e.i., and cotton-seed when methylated pursuant to departmental by-laws .....	"	6d.
Lubricating (mineral), mineral n.e.i., and kerosene .....	"	3d.
N.E.I. ....	"	6d.
(Special exemptions: Oil—viz., fish, including cod (unrefined), seal, whale, penguin, petroleum (crude), degrass, sod, naphtha, benzine, mirbane, and turpentine.)		

## Paints and colours, viz.:—

Ground, in liquid, partly or wholly prepared for use.....	per cwt. and 15 % ad val.	1s.
Colours (dry), dry white-lead, and patent driers and putty .....	per cwt.	1s.
Varnishes, varnish-stains, lacquers, enamels, Japans, liquid sizes, patent knotting, oil and wood finishes, petrifying liquids, damp-wall compositions and lithographic varnish .....	per gal. (and 15 % ad val.)	1s.

(Special exemptions: Colours, artists', dyes (dry), not packed for retail sale; lamp, ivory, bone, and vegetable blacks; London purple and Paris Green; sulphate of copper; ultramarine blue; whitening.)

Cement, plaster of Paris, &c. .... per cwt. 1s.

Filters of all kinds, fire and glazed bricks, fire-lumps, fire-clay manufacturers, n.e.i., asphalt and roofing tiles .....

Glass, n.e.i.; also Seltzogenes and Accessories .....

and Siphon-bottles .....

Glassware, n.e.i. ....

(Special exemptions: Glass—viz., lenses, unmounted; locket, brooch, and watch-glasses; bottles, empty, of not more than 6 fl.-dr. capacity. Scientific instruments and Apparatus—viz., instruments for measuring the density of liquids. Scientific Apparatus (glass)—viz., beakers, flasks, test-tubes, vacuum-tubes, burettes, pipettes, weighing-bottles and tubes, eudiometers, nitrometers, radiometers, fat-extraction tubes, filter-pumps, gas-washing, reduction, and absorption bulbs and tubes, test-measures in centimetres and grms.; also carbonic acid, sulphuretted hydrogen, decomposing-water, and bacteriological apparatus of glass.)

Glue, not liquid, and gelatin, sheet..... per lb. 2d.

Glue, gelatin, and cements, n.e.i., including mucilage and printers' roller-composition ....

(Special exemptions: Dry gums, shellac, dextrin, sandarach, and mastic. From stone duty: Pestles and mortars—agate.)

Acetic acid, extract or essence of vinegar, and vinegar:—

Containing not more than 6 per cent. of absolute acid .....

Containing more than 6 per cent, but not more than 30 per cent. ....

For every extra 10 per cent. or part of 10 per cent. ....

Acids, viz.:—

Hydrochloric, nitric, and sulphuric. ....

Ammonium carbonate and calcium carbide .....

Drugs and chemicals, viz.:—

Salicylic and boric acids; bisulphites of potassium, sodium, calcium, and magnesium, foaming powders and liquids, and malt and hops substitutes .....

Insecticides, sheep washes, and disinfectants, n.e.i. ....

Medicines, viz.:—

Patent and proprietary medicines, and other medicinal compounds; non-spirituous medicinal extracts, essences, juices, infusions, solutions, and syrups; medicinal waters and oils, n.e.i.; and medicines for animals...

(Special exemptions: Essential oils (non-spirituous), bacteriological products and serum).



Perfumery, including perfumed ammonia, camphor in blocks or tablets, toilet preparations (non-spirituous) perfumed or not; lanoline, glycerin, vaseline, and petroleum jelly not medicated.....	ad val.	20 %
Saccharin.....	per lb.	20s.
Soda crystals.....	per cwt.	1s.
Stationery, manufactured, including inks and ink powders; wax, sealing and bottling.....	ad val.	25 %
(Special exemptions: Paper—viz., emery and flint-paper and cloth, filter paper, litmus paper.)		
Explosives, viz.—		
Ammunition and fireworks.....	ad val.	20 %
Powder, sporting.....	per lb.	4d.
" n.e.i.....		1d.
" blasting.....		Free
Photographic dry-plates, films, and sensitised paper.....		15 %
(Special exemptions: Scientific instruments and apparatus imported by governing bodies for use in universities, colleges, schools, or public hospitals.)		
India-rubber manufactures.....		Free

### U.S. IMPORTS OF CHEMICAL RAW MATERIALS FOR FIRST NINE MONTHS OF 1901.

*Bd. of Trade J., Nov. 14, 1901.*

The following raw materials for use in chemical manufactures were imported into the United States during the nine months ended 30th September, 1900 and 1901, respectively:—

Articles.	Nine Months ended 30th September.	
	1900.	1901.
	Lbs.	Lbs.
Iron ore.....	1,584,345,280	1,625,274,240
Sulphur, crude.....	261,842,560	267,637,440
Hides and skins.....	234,668,656	229,751,187
Lead ore.....	168,788,085	172,027,087
Copper ore.....	67,399,360	147,943,040
Soda, caustic; sodium nitrate, &c.....	363,066,394	377,734,324
Potash.....	108,210,849	121,871,190
Dyewoods.....	105,400,960	72,380,320
Tin in bars, &c.....	14,847,417	56,908,901
Copper pigs.....	54,443,378	56,239,624
Gums.....	58,277,583	49,968,085
India-rubber, crude.....	34,493,337	40,181,040

The following table shows the value of the principal groups of manufacturers' materials imported in the nine months of 1900 and 1901:—

Articles.	Nine Months ended 30th September.	
	1900.	1901.
	Dolls.	Dolls.
Chemicals, drugs, &c.....	39,479,137	40,910,420
Hides and skins.....	39,655,802	41,168,929
India-rubber and gutta-percha.....	21,551,804	21,021,442
Tin.....	15,281,577	14,679,768

NOTE.—Dollar = 4s. 2d.

### III.—TAR PRODUCTS, PETROLEUM, Etc.

#### DRAWBACK ON TAR AND PURE AMMONIA IN UNITED STATES.

*Chem. Trade J., Oct. 19, 1901.*

The provisions of Treasury Decision 22,332, dated July 5th, 1900, establishing a rate for allowance of drawback on coke manufactured by the New England Gas and Coke Company, of Boston, Mass., from imported slack coal, are, by a circular of the United States Treasury Department, extended, as far as applicable, to cover tar and pure ammonia (the latter being combined with sulphuric acid as ammonium sulphate), manufactured by the said company wholly from the same imported material, and exported. In the liquidation of entries, the rate of drawback which shall be allowed on the tar shall be 20 c. per

long ton, and for each such ton exported, 26·32 long tons of coal shall be charged against the record of importation. The rate of drawback which shall be allowed on the ammonium sulphate shall equal  $\frac{1}{3}$  c. per pound on the pure ammonia contained therein, determined by official analysis of samples to be taken as ordered by the collector; provided, that the quantity shall not exceed 560 lb. for each long ton of ammonium sulphate exported, and that for each ton so exported, 112 long tons of coal shall be charged against the record of importation.

#### ASPHALT AT PALERMO (SICILY).

*Foreign Office Annual Series, No. 2716.*

The asphalt exported from the port of Palermo, during 1900, was 494 tons.

The total shipment from Sicily amounted to 74,130 tons, the greater part of which went to Germany, the United States, and France.

#### PARAFFIN WAX IMPORTS OF SICILY (ITALY).

*Foreign Office Annual Series, No. 2716.*

Paraffin wax is imported chiefly from the United Kingdom.

### IV.—COLOURING MATTERS, Etc.

#### INDIGO IN INDIA.

*Chem. Trade J., Nov. 9, 1901.*

The following report has been received at the India Office, giving the final forecast of the indigo crop of 1901:—

**Area.**—Last year a slight improvement took place in the cultivation of this crop, but during the present year there has been a decline, in consequence of the continued fall in prices. The total area returned, in December 1900, amounted to 262,175 acres; the area this year is estimated at 160,897 acres, a decrease of about 39 per cent. Compared with the average areas of the preceding five and ten years, the decline amounts to 50 per cent. in each case.

**Condition.**—In the forecast, issued on July 11th last, it was reported that damage was done to the indigo crop by locusts and grasshoppers; whilst the late commencement of the rains stunted the growth of the plants. The prospects of the crop continued unsatisfactory till the end of August; but the fine, dry weather during September was generally favourable for manufacture, and the product of dye is reported to have been better than was originally anticipated. Assuming 100 to represent a normal crop, the average condition of the present year's crop is now reported to vary from 50 to 85 per cent.

**Output.**—The total estimated production of indigo dye this year works out to 28,352 factory maunds (Doab, 16,913 maunds; Benares, 11,439 maunds), or 40 per cent. below last year's estimate, and the average estimated yield of the preceding five and ten years. The exports of indigo from October 1st, 1900, to March 31st, 1901, amounted to 37,791 standard maunds, of which 23,575 maunds went to Calcutta, 12,138 maunds to the Punjab, and the rest to other parts of the country.

### VII.—ACIDS, ALKALIS, AND SALTS.

#### SULPHUR IN JAPAN.

*Chem. and Druggist, Oct. 26, 1901.*

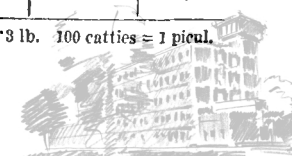
Prices opened at 2·25 yen\* per picul†, and closed at 2·28 yen per picul. Exports tend to increase considerably owing to improving demand abroad.

The values and exports during 10 years were as follows:—

Year.	Catties.	Yen.	Year.	Catties.	Yen.
1891	31,663,045	284,832	1896	20,507,630	308,588
1892	21,844,290	230,963	1897	15,322,307	321,341
1893	17,373,205	238,832	1898	21,045,468	477,014
1894	21,103,646	244,542	1899	27,866,719	574,868
1895	26,445,913	296,136	1900	29,726,967	698,224

\* 1 yen = 2s. 0½d.

† 1 picul = 133·3 lb. 100 catties = 1 picul.





## SULPHURIC ACID PLANT AND SULPHUR IN SICILY.

*Foreign Office Annual Series, No. 2716.*

A British firm is erecting plant for the manufacture of sulphuric acid on a large scale.

The total exports of sulphur, for 1899, were 494,000 tons, and for 1900, 562,000 tons.

Sulphur exports from Catania, during 1900, amounted to 175,998 tons, of which 114,258 tons went to foreign countries, as against 98,092 tons, in 1899.

The Anglo-Sicilian Sulphur Company's contract with the mine owners expires next July, and the company will probably exercise their option of a renewal for a further five years, the result having been of mutual benefit. The average price for best thirds brimstone, during 1900, was 3*l.* 13*s.* 6*d.* per ton f.o.b. Catania.

Rich sulphur beds were discovered in the Comitini district, in the neighbourhood of the Cannarella mine, near Castro-Giovanni, and also in the Agira district, and satisfactory prospecting operations were carried through at Cametrice (Piazza Armerini district), at Linosella (Agira District), at Cavalcator, and in other places.

## MAGNESITE: U.S. CUSTOMS DECISION.

*Eng. and Mining J., Nov. 2, 1901.*

Merchandise invoiced as magnesite and returned by the local appraiser as "ground magnesite, not perfect; as magnesite cement," was assessed at 20 per cent. *ad valorem*, under paragraph 89, Act of 1897. The importers claimed the article to be free from duty under paragraph 605, as "magnesite crude or calcined, not purified." The protest was sustained and the article is to be admitted free from duty.

## VIII.—GLASS, POTTERY, AND ENAMELS.

## GLASS IMPORTS OF SICILY (ITALY).

*Foreign Office Annual Series, No. 2716.*

Window and plate glass come mostly from Belgium, table glass from Germany and Austria-Hungary.

## TILES, EARTHENWARE, AND GLASSWARE IMPORTS OF BRAZIL.

*Foreign Office Annual Series, No. 2724.*

Encaustic tiles come chiefly from Belgium, and roofing tiles from France.

In earthenware and glassware, the United Kingdom supplies a fair portion of the granite table-ware, but Belgium is the largest supplier, whilst for porcelain ware, Germany heads the list, followed by France, Portugal, Belgium, and then the United Kingdom.

For common kinds of glassware, the leading suppliers are Germany, Belgium, and France. The United Kingdom participates in the trade of better quality goods. The United Kingdom also has a share in the window-glass trade, but the bulk comes from Belgium.

## IX.—BUILDING MATERIALS, Etc.

## PORTLAND CEMENT EXPORTS OF GERMANY.

*U.S. Cons. Reps., Nov. 1901.*

Germany's exports of Portland cement have considerably increased during the last few years; even in 1900, notwithstanding the unfavourable business situation, a further increase has been noted. In 1894, the exports of Portland cement from Germany amounted to 362,000,000 kilos. (355,535 tons); in 1900, to 543,990,000 kilos. (534,276 tons), or 15,315,000 kilos. (15,041 tons) more than in 1899.

From Stettin, the exports of Portland cement have decreased; these amounted to 80,632,000 kilos. (79,192 tons) in 1900, against 88,689,000 kilos. (87,105 tons) in 1899. On the other hand, Hamburg has taken the leading place, and trade has further developed in 1900, although the increase was not so considerable as in the preceding year. In 1900, the exports of Portland cement from Hamburg amounted to 306,571,800 kilos. (301,097 tons), valued at about 3,000,000 dols.; in 1899, to 293,483,900 kilos. (288,243 tons), valued at about 2,950,000 dols.

The principal countries importing it from Hamburg are the United States, which alone took 115,381,400 kilos. (113,321 tons); Australia, with 32,100,000 kilos. (31,527 tons); Brazil, with 19,300,000 kilos. (18,955 tons); and Cape Colony, with 17,200,000 kilos. (16,893 tons). Since 1896, the exports to Cape Colony have been quintupled. Among other countries importing Portland cement may be mentioned the Argentine Republic, Chile, and British North America. The chief exports to European countries were made to Great Britain, Norway, Spain, and Portugal.

The exports of Portland cement *via* Bremen showed a slight decrease last year, amounting to 49,123,400 kilos. (48,246 tons), compared with 52,332,400 kilos. (51,398 tons) in 1899.

## CEMENT TRADE OF NATAL.

*Bd. of Trade J., Oct. 31, 1901.*

*Importation.*—The cement consumed in Natal is at present imported. One company has recently been formed there to manufacture cement, and the initial results are satisfactory. The following table shows the quantity and value of cement imported into Natal, distinguishing the countries of origin, in each of the years 1898, 1899, and 1900, and also in the first six months of 1901:—

Country.	1898.		1899.		1900.		First Six Months of 1901.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Barrels.	£	Barrels.	£	Barrels.	£	Lbs.	£
United Kingdom .....	8,556	2,731	13,947	4,366	67,996*	20,297*	17,220,752*	13,723*
Belgium .....	25,887	7,433	43,824	14,264				
Germany .....	28,576	8,882	39,208	11,085				
Other countries .....	6,013	1,839	10,617	3,162				
Total .....	69,132	20,885	112,596	32,877	67,996	20,297	17,220,752	13,728
In transit .....	13,967	..	7,964	1,279				

\* Detailed statistics have not yet been published.

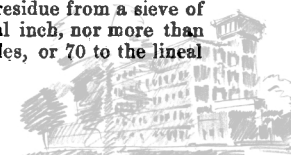
Several brands of Belgian cement are, according to the Belgian Consul, greatly appreciated. German cements are invading the South African market more and more.

The following are the conditions of contract for the town of Durban:—

*Testing.*—For testing purposes, blocks are made and exposed to the air for a day to allow them to set. They are then placed in moulds and plunged into water for six days, immediately after which they are tested. If the

average block is unable to withstand a pressure of 743 lb. per 2½ square inches, i.e., 330 lb. per square inch, the cement is refused.

The cement must be of a bluish colour and weigh not more than 112 lb. per bushel. It must be finely ground and give not more than 7 per cent. residue from a sieve of 2,500 holes, or 50 holes to the lineal inch, nor more than 25 per cent. from a sieve of 4,900 holes, or 70 to the lineal inch.



Samples for testing will be taken from three barrels chosen at hazard from each lot of 100 barrels. In cases where the trials of each of these lots of 100 barrels do not give satisfaction, the entire lot would be refused and the contractor required to remove it immediately at his own expense, the administration reserving the right to annul the rest of the contract.

**Price.**—The gross price varies from 9s. 6d. to 11s. 1d. per barrel delivered on the quay. The retail price varies from 14s. to 16s., according to the brand. English cement is the dearest.

**Import Duty.**—According to the tariff of the South African Customs Union, the import duty on cement is at the rate of 6d. per 100 lb. (English).

#### CEMENT TRADE OF CONSTANTINOPLE (TURKEY).

Constantinople consumes annually 10,000 tons of cement, imported chiefly from France (70 per cent.), followed by Germany and England. The two latter countries are increasing their exports, the quality of the cement being considered superior to that of the French product. It is stated that English cement can no longer compete with German cement. As regards French cement, custom will protect the import for some time to come, facilitated as it is by the low rate of transport, *viz.*, about 6 francs (4s. 10d.) per ton.

The annual import of cement into Constantinople is divided as follows:—

	Tons.	At per Ton.
		£ s. d.
Roquefort .....	3,000	1 5 7
Natural Portland .....	2,500	1 7 2
Artificial cement (2nd quality) ...	1,250	1 12 0
(1st quality) ...	1,000	1 17 7
Belgian, Italian, German, and Austrian cements.	500	1 16 0
English cement .....	500	2 8 0

NOTE.—Actual prices, but the quantities are only approximate.

German cement is sold c.i.f. Constantinople; French and English cement free on board at the port of shipment. The import duty, which is borne by the buyer, is nominally 8 per cent. *ad valorem*, but in reality it amounts to 12 per cent., and even to 14 per cent.

#### PUMICE STONE BRICKS IN GERMANY.

*Eng. and Mining J.*, Oct. 19, 1901.

In the Neuwied district, between Coblenz and Andernach, there is a large industry in the manufacture of bricks from volcanic sand and pumice stone, which are found along the base of the Eifel. The surface soil is stripped off, and the sand piled up, to be shovelled into the mixing bed of water and lime close at hand. Before moulding, the pumice is screened, the larger pieces being broken to pass through a sieve having  $\frac{1}{2}$  in. meshes. These screened pieces are then covered with a thin coating of cement, and the brick is moulded. The cement is not mixed with the pumice so as to form solid cement blocks, but by the fragments being coated first. The brick is then obtained by these coatings adhering to one another. For the manufacture of 20,000 bricks about  $4\frac{1}{2}$  tons of lime are required. After the mixing process the substance is poured into iron moulds provided with bottoms of detachable boards, which, after the removal of the moulds, act as driers. The bricks are ready for shipment when they have been exposed to the air for a very short time.

#### X.—METALLURGY.

##### ALUMINIUM PLANT IN CANADA.

*Eng. and Mining J.*, Oct. 12, 1901.

The new Canadian plant of the Pittsburgh Reduction Company is ready for operation, and the company will try to secure an export trade with Great Britain. At first the crude aluminium will be exported, but later a rolling mill similar to that at New Kensington, Pa., will be built near the new works. The total cost of the present plant has

been about 1,000,000 dols. The company now has about 10,000 H.P. installed and will manufacture about 10 tons of aluminium daily.

##### ALUMINIUM CONVENTION.

*Chem. and Druggist*, Oct. 26, 1901.

A convention has recently been concluded between the producers of aluminium comprising the Pittsburgh Aluminium Reduction Company, which is believed to control the trade in the United States; the Neuhausen Aluminium Company of Neuhausen, Switzerland, together with the Baden works owned by the company at Rheinfelden; the Lend works in Austria; and the British Aluminium Company in England. The object of the convention is to arrange prices on a common basis, to seek new markets for the sale of aluminium, and to support the industries already utilising this metal.

##### ANTIMONY DEPOSITS IN NOVA SCOTIA.

*Imp. Inst. J.*, Nov. 1901.

These deposits are situated in Hants County, Nova Scotia. They were discovered in 1882, and worked till 1892, when the mines were closed, and remained so until 1899.

The mineral occurs as stibnite in two veins, one of which has been traced upon the surface for a distance of over 1,200 ft. The thickness of the ore has varied from a few inches to 7 ft., this being the greatest thickness attained, in which case the ore was solid stibnite. When the ore is exposed, it oxidises to hermesite or valentinite. It is found in places associated with pyrites, mispickel, and galena. More or less gold is always found in the ore, and the amount varies with the percentage of antimony. Up to 1892, 3,121 tons were shipped, containing over 50 per cent. of antimony. The St. Helens Metal Recovery Company, after proving that they can successfully treat the mineral, have erected works to treat 600 tons per month, and have agreed to buy ore containing antimony as low as 12 per cent., and to pay for 75 per cent. of the gold content.

Samples of ore taken from different parts of the vein give the following analyses:—

Thickness of Ore.	Percentage of Antimony.	Value of Gold (per 2,000 lb.).
Ins.		Dols.
12	5.50	26.00
6	10.50	30.00
6	4.95	3.60
24	41.13	185.00
6	33.00	35.40

The average thickness of ore left in the vein is estimated at 6 ins., containing 12 per cent. of antimony and 23 dols. of gold per ton of 2,240 lb. At a greater depth (below the old working) high grade ore gave on analysis 60.29 per cent. of antimony, and 2.66 oz. of gold per ton of 2,000 lb., and a second grade ore 5.27 per cent. of antimony and 1.38 oz. of gold per ton. There are probably other veins in the district.

##### ZINC ORES IN NEW SOUTH WALES.

*U.S. Cons. Reps.*, Nov. 1901.

Zinc ores are widely distributed over New South Wales. Specimens of ore from ninety-six different localities have been received at the Department of Mines in Sydney. There are several combinations of this ore among these specimens, such as red oxide of zinc, which contains 80.3 per cent. of zinc. The carbonate of zinc contains 52 per cent. of zinc, in combination with silver and zinc blende. Sulphide of zinc, containing 67 per cent. of zinc when pure, also often contains iron, and sometimes cadmium. This is the most common ore of zinc, and is found associated with galena in most of the silver mines of New South Wales. No attempt has been made to work this mineral specially for the production of zinc, but its occurrence is regarded as detrimental to the ores of silver and lead with which it is nearly always associated.



## WOLFRAM AND MOLYBDENITE IN QUEENSLAND.

*Bd. of Trade J., Oct. 24, 1901.*

Of the 189½ tons which formed the year's output of wolfram, 188 tons came from Wolfram Camp, on the Hodgkinson field. The mineral appears to be plentiful, but the fall in price has latterly checked production. At a shallow depth the mineral occurs in a clean state, and requires little labour to fit it for the market. In the deeper ground it is found disseminated through quartz, from which it is separated by dollying and sluicing. Associated with wolfram is molybdenite. Twelve months ago very little was known about molybdenite, but latterly a market has been established, and 60*l.* a ton has been obtained for the mineral on the field. Bismuth is also found, the miners receiving about 3*s.* per pound. As the clean surface deposits become rarer, the erection of concentrating machinery to supersede the present tedious process of hand dressing has been suggested, and it is said that a suitable plant can be set up at a cost of about 1,500*l.* But it would be rash to predict any assured permanency for the field. Wolfram generally occurs in patches, and rarely is there any defined lode. The demand for all these rare minerals is limited, and a small increase in production floods the market and brings down the price. Cornwall, Saxony, Spain, Portugal, and North and South America all produce wolfram, so that there does not seem to be much room for expansion.

## MANGANESE IN BRAZIL.

*Foreign Office Annual Series, No. 2724.*

The influence of high exchange is depressing in connection with manganese mining.

Experts calculate that this industry cannot be carried on profitably at an exchange over 12*d.* per milreis. But even with the rate about 10*d.*, it would seem that there is little profit in the business, and several of the workings have been closed. No doubt the lower value in consuming centres is also partly responsible for this state of things. Shipments continue to be made, and some 34,000 tons of manganese ore left this port, during the first eight months of 1901, for the United Kingdom, the United States, and Belgium, and notwithstanding all drawbacks, there is still considerable activity in mining affairs, several prospecting parties being in the field, among which Belgian enterprise is particularly well represented.

## MINERAL PRODUCTION OF JAPAN.

*Eng. and Mining J., Nov. 9, 1901.*

Mr. I. Hosoi, of the Imperial Bureau of Mines at Tokio, gives a statement of the mineral production of Japan for the year 1900. The figures given below are in metric tons, with the exception of those for gold and silver, which are in kilograms, and for petroleum, in gallons:—

<b>Metals:</b>		
Gold .....	kilos.	2,130
Silver .....	"	58,953
Antimony, crude .....	tons	81
Antimony, refined .....	"	349
Copper .....	"	25,304
Tin .....	"	12
Lead .....	"	1,877
Pig iron .....	"	21,299
<b>Oil:</b>		
Crude oil .....	galls.	22,175,825
Refined oil .....	"	2,077,223
<b>Other ores and minerals:</b>		
Graphite .....	tons	94
Manganese ore .....	"	15,228
Iron pyrites .....	"	16,146
Arsenic .....	"	5
Sulphur .....	"	14,435
<b>Chemical products:</b>		
Sulphuric acid .....	"	932

The important increases over 1899 were in copper and sulphur. Sulphur from Japan is being exported largely to the Western States of America.

## XII.—FATS, OILS, Etc.

## VEGETABLE WAX IN JAPAN.

*Chem. and Druggist, Oct. 26, 1901.*

Exports, during 1900, greatly decreased compared with 1899, owing to the high price of raw material, on account of poor harvest; stocks in foreign markets also contributed to the decline.

The quotation in January was 14·80 yen per picul, which continued until May, when the market became quieter, buyers offering 14 yen, whilst sellers complained of unremunerative prices, and it closed at 15·20 yen per picul, at the end of the year. The following is a comparative table of exports during ten years:—

Year.	Catties.*	Yen.†	Year.	Catties.	Yen.
1891	2,673,057	316,835	1896	2,148,344	371,701
1892	2,449,701	285,567	1897	4,205,343	730,576
1893	3,150,967	353,706	1898	3,789,791	609,760
1894	4,401,195	562,135	1899	4,569,613	642,219
1895	2,792,330	334,847	1900	3,702,087	561,435

\* 1 catty = 1½ lb. 1 picul = 100 catties. † 1 yen = 2*s.* 0½*d.*

## SOAP AND CANDLES IMPORTS OF BRAZIL.

*Foreign Office Annual Series, No. 2724.*

Soap and candles are made extensively within the country. Of the soap imported, during 1900, the greater part was supplied by the United States.

Stearin came chiefly from Belgium and France, and the latter country, together with Germany, supplied spermaceti. Wax is brought from Germany.

## OLIVE OIL IN SICILY (ITALY).

*Foreign Office Annual Series, No. 2716.*

The importation of some 278,000 kilos. of olive oil from Spain, during 1900, is remarkable for an olive-growing country. Most of the output goes to the United States. The exports of olive oil to the United Kingdom, during 1900, amounted to 42,000 kilos.; during the first half of 1901, this rose to 62,000 kilos.

The quantity of olive oil produced at Catania, in 1900, was about 1,980,000 gallons, of poor quality.

## STEARIC ACID IMPORTS OF SICILY (ITALY).

*Foreign Office Annual Series, No. 2716.*

During 1900 and 1901, the imports of stearic acid from the United Kingdom, amounted to 22,000 kilos., in each year; the imports from Germany being 30,000 and 10,000 kilos., for the two years, from Holland, 28,000 kilos., in 1900, and 11,000 kilos., in 1901; and from Belgium, 17,000 kilos., in 1900, and 13,000 kilos., in 1901.

## OIL DUTIES IN MEXICO.

*Bd. of Trade J., Oct. 24, 1901.*

The following paragraphs relating to oils have been altered, the new rates of duty being as stated:—

Tariff No.	Tariff Classification.	New Rate of Duty.	
182	Cotton-seed oil— Purified, in tank car or tank ship.	Kilo., net	Pes. Cts. 0 10
182a	Purified, in barrels, drums, or tins.	" gross	0 09
182b	Crude, in tank car or tank ship	" net	0 05
182c	" in barrels, drums, or tins	100 kilos., gross	4 50
183	Cocoa-nut oil, and crude or boiled linseed oil— In tank car or tank ship .....	Kilo., net	0 10
183a	In barrels, drums, or tins .....	" gross	0 09
854	Lubricating oils .....	" "	0 05
50	Fish oil— In glass bottles .....	Legal kilo.	0 12
50a	In wooden vessels .....	"	0 10
64	Cod liver oil— In glass bottles .....	"	0 12
64a	In wooden vessels .....	Kilo., gross	0 10



A decidedly protective policy is discernible, especially in the case of crude imported oils, which are clarified or prepared for market in Mexico. The increase of duty on such oils is levied on the prepared article.

#### SOAP IMPORTS OF ZANZIBAR.

*Foreign Office Annual Series, No. 2718.*

Soap of the common mottled variety comes from the United Kingdom and Germany, but German soap is in most request, being more malleable and less liable to break.

#### XIII. B.—RESINS, VARNISHES, Etc.

STICKLAC AND GUM-BENJAMIN (GUM-BENZOIN) AT CHIENGMAI (SIAM).

*Foreign Office Annual Series, No. 2717.*

The former brisk trade with Bangkok in gum-benjamin and sticklac has now practically ceased to exist.

Gum-benjamin now either finds its way down to the Tonquin markets, by way of the Red River, or to Bangkok, by way of Paklai, on the Mekong River, and Utaradit.

The "Ton Khamjan," or gum-benjamin tree, is found growing along the slopes of the hills around Chiengmai.

#### XIII. C.—INDIA-RUBBER, Etc.

INDIA-RUBBER AT ZANZIBAR.

*Foreign Office Annual Series, No. 2718.*

In the island of Pemba, especially at its northern end, the *Landolphia Kirkii* grows. It is found over scattered areas, and does not attain to a large size, but grows to a considerable length, and the latex is clean and white. With respect to the rubber trees that have been planted experimentally in the Government plantations, the Assam rubber, *Ficus elastica*, is possibly the most vigorous, but the Central American variety, *Castilloa elastica*, appears to be well suited to the climate; of the others the Pará and Ceara rubber trees grow rapidly, either from seed or cuttings, but their yield has so far been small. The so-called gutta-percha tree appears to be of doubtful economic value as the latex obtained from it loses its plastic character after a few months and becomes friable.

RUBBER EXPORTS OF THE BRITISH CENTRAL AFRICA PROTECTORATE.

*Foreign Office Annual Series, No. 2722.*

The amount of rubber exported was less in 1900 than in 1899, the value being 9,300*l.* as against 13,100*l.* A larger amount of rubber than during 1899 has come in transit through the Protectorate from outside countries.

RUBBER CULTURE IN PERAK (STRAITS SETTLEMENTS).

*U.S. Cons. Rep., Nov. 1901.*

"De Indische Mercuur" states that the director of the Government plantations has issued a report on the results of cultivation in the district of Perak. By moderate tapping of 82 Para rubber trees, averaging 14 years of age, 327 lb. of first-class and 33 lb. of second-class rubber were produced. The first class, equal to the best Para rubber, was sold in London at 3*s.* 10*d.* (93 cents.); the second class, at 2*s.* 6*d.* (61 cents.), per pound. Each tree yielded about 2 lb. of rubber.

#### XIV.—TANNING; LEATHER; GLUE, Etc.

SUMACH EXPORTS OF SICILY (ITALY).

*Foreign Office Annual Series, No. 2716.*

The sumach crop, in 1900 and 1901, was disposed of as shown in the table.

Of the ordinary variety, the exports, for 1900, were 9,611,496 kilos., and for 1901, 6,810,403 kilos.

Sumach can still be procured from other growers than those belonging to the ring, though it is a question whether it can be shipped at the same rates, especially by vessels calling here in the ordinary way.

To—	Quantity.	
	1900.	1901.
	Kilos.	Kilos.
United Kingdom .....	9,356,000	6,516,000
France .....	6,752,000	5,310,000
Germany .....	4,745,000	1,121,000
United States .....	2,882,000	3,617,000
Belgium .....	2,564,000	1,148,000
Holland .....	1,123,000	646,000
Austria-Hungary .....	812,000	476,000
Russia .....	728,000	489,000

#### XVI.—SUGAR, STARCH, Etc.

IMPORTATION OF SACCHARINE.—NOTICE TO IMPORTERS.

*Chem. Trade J., Nov. 2, 1901.*

The following notice has been issued by the Commissioners of Customs:—

*Saccharine and Substances of a like Nature or Use.*

Notice is hereby given that, under the regulations made by the Commissioners of Customs and the Commissioners of Inland Revenue, in pursuance of section 8 of the Finance Act, 1901, the importation into the United Kingdom of saccharine (including substances of a like nature or use) is prohibited, except in packages containing not less than 11 lb. net weight, and into the ports of London, Harwich, Dover, Folkestone, Newhaven, Southampton, Hull, Leith, Goole, Grimsby, West Hartlepool, and Grangemouth.

Any smaller quantity than that specified imported into the ports named, and any quantity whatever imported at other ports or through the medium of the Foreign and Colonial Parcel Post, will be forfeited, and the importer of the goods will render himself liable to a penalty of 50*l.* in respect of each offence committed in contravention of these regulations. (See also this Journal, 1901, 949.)

IMPORT DUTIES ON ARTICLES CONTAINING SUGAR IN UNITED KINGDOM.

*Bd. of Trade J., Nov. 21, 1901.*

A further amended schedule of the fixed rates of duty on articles containing sugar has been issued by the Commissioners of Customs, from which it appears that the following additions require to be made to the schedules already published:—

Milk powder:—	s.	d.
If declared by the importer not to contain any added sugar .....	Free.	
If declared by the importer not to contain more than 38 per cent of added sugar .....	cwt.	1 6
In all other instances, and in cases in which the importer wishes to dispense with sampling and test .....	cwt.	3 4
Importations entered as free will be delivered on deposit of duty at the 1 <i>s.</i> 6 <i>d.</i> rate pending analysis. Importations entered at the 1 <i>s.</i> 6 <i>d.</i> rate are liable to sampling at the discretion of the officers.		

Further, that "Soy, when containing molasses or other sweetening matter," formerly assessed for duty at the rate of 2*s.* per cwt., is now dutiable at the rate of 1*s.* per cwt. (See also this Journal, 1901, 515, 629, 948.)

1900-1 SUGAR CAMPAIGN IN AUSTRIA-HUNGARY.

*Bd. of Trade J., Nov. 7, 1901.*

No change in the number of sugar factories in the Dual Monarchy has taken place during the last year. Of the 213 existing factories, 128 are in Bohemia, 64 in Moravia and Silesia, and 21 in Bosnia.

*Beet Sugar.*—The area of beet cultivation increased during the last campaign four per cent., from 325,000 to 339,000 hectares. The total production of beet was 1,000,000 tons below that of the previous year, the two harvests in question having yielded 7,410,000 tons and 8,480,000 tons respectively, but last year's beet was two per



cent. richer in sugar than that of the previous year, so that the actual amount of sugar produced only fell below the previous year's yield by 10,000 tons, the figures being 1,080,000 and 1,090,000 tons respectively.

**Refined Sugar.**—The total exportation of refined sugar amounted, during the last campaign, to 544,600 tons, as compared with 517,560 tons, during the previous year. Half found its way to England and America *via* Hamburg, an increase over the previous year of 33,100 tons. The exportation of refined sugar to India also showed an increase from 44,100 tons, during 1899–1900, to 52,600 tons, during 1900–1901. The exportation to Switzerland, on the other hand, has decreased during the last two campaigns from 32,600 tons to 8,500 tons; whilst Italy and the Balkan States continue to lose their importance as markets for Austro-Hungarian sugar. Japan, also, has not fulfilled expectations, owing to the Excise regulations which encourage the importation of raw sugar. Whereas during the campaign of 1899–1900 23,100 tons of refined sugar were shipped from Austria-Hungary to Japan, during the last campaign, the total exportation only reached 20,000 tons. China took 800 tons last year as compared with 2,200 tons during the previous campaign. Portugal is a new and promising market, and imported from the Dual Monarchy 4,800 tons of refined sugar last year, as compared with 2,100 tons during the previous campaign. It appears, therefore, that whilst the production of refined sugar in Austria-Hungary increases year by year, the number of foreign markets for its disposal shows a distinct tendency to become smaller.

**Raw Sugar.**—During the campaign of 1900–1901, 89,700 tons of raw sugar were exported from Austria-Hungary as compared with 133,000 tons in the previous year. Of this quantity 58,900 tons went to England, 13,400 tons to America, and 4,100 tons to Japan.

#### SUGAR INDUSTRY IN ITALY.

*Bd. of Trade J., Oct. 24, 1901.*

The past two seasons have witnessed a remarkable development of the Italian beet-sugar industry. Previous to 1899, there were in the whole of Italy only four sugar factories, with a total annual capacity of about 8,000 tons. During the season of 1899–1900, the number of factories increased to 24, and the production of sugar quadrupled. At the end of the year 1900, there existed 28 raw-sugar factories and 10 refineries, whilst six factories and two refineries were in course of construction. The industry continued to develop during the season just closed, but such strides as had been generally anticipated were not made. This was chiefly due to the imposition of a new sugar tax—which, in a measure, checked the spirit of enterprise—as well as to the fact that the beet crop was both meagre in quantity and indifferent in quality.

Official data as to the yield of the season of 1900–1901 are not yet accessible, but the amount of raw sugar manufactured will probably reach 550,000 metric quintals (55,000 metric tons).

#### SUGAR MACHINERY IN CUBA.

*Bd. of Trade J., Oct. 31, 1901.*

The following is a complete list of articles included under the heading of "Machinery and apparatus for making sugar and brandy," dutiable, for a period of 12 months from the 1st October, at the rate of 5 per cent. *ad valorem*.

The following articles, whoever be the importer:—

Weighing machines (platforms) for weighing sugar cane.

Complete machines of every kind for grinding cane.

Cane shredders.

Sugar clarifiers.

Complete apparatus for diffusion.

Purifying apparatus.

Filters and filtering apparatus.

Apparatus called "*trenes jamaquinos*" complete.

Furnaces for making animal black.

Steam desiccators (*trampas*) and granulators.

Centrifugal machines, mixing and lifting (*subidores*) machines, with their motors.

Vessels called "*bombonas*" and "*cachimbos*."

Skimmers, distributors, and sugar moulds.

Apparatus or vessels (*tachos*) acting in vacuum, also their machines, pipes, and cocks, of copper or iron, polarimeters.

Skimming pails (*cachaceras*).

Sugar crushers.

Cars (*porta-templa*).

Sulphur dioxide apparatus.

Dumping apparatus for cars (*porta-templa*).

Cane-transporting apparatus.

Furnaces for burning bagasse.

Automatic circulator and regulator for vacuum pan apparatus.

Apparatus for compressing sugar.

#### SUGAR IN BRAZIL.

*Foreign Office Annual Series, No. 2724.*

This industry has attained considerable development in the State of Rio, particularly in the neighbourhood of Campos, but, as the yield has been over-stimulated, prices have dropped to about 80 reis per kilo., i.e., about 1½d. per lb. for refined (crystal) sugar.

Many of the factories have decided to ship their sugars, unrefined, to Liverpool.

#### XVIII. A.—FOODS.

##### FOOD ADULTERATION IN BRAZIL.

*Foreign Office Annual Series, No. 2724.*

The regulation prohibiting entry of alimentary preparations and liquids containing substances injurious to public health is still in strict operation, and the greater part of the goods re-exported during the year, valued at 27,400*l.*, consisted of imported articles of this nature. The admixtures which are the most frequent causes of condemnation are boric, salicylic, and sulphuric acids, and alcohol of bad quality.

#### XVIII. B.—SANITATION.

##### DISCHARGING CHEMICALS INTO SEWERS.

*Chem. and Druggist, Oct. 26, 1901.*

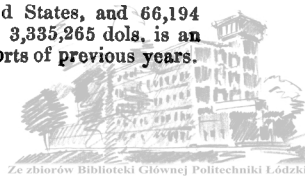
At Bow Street Police Court, on Oct. 23, a firm was charged with allowing chemicals or manufacturing refuse to fall into a public sewer. For the prosecution it was explained that action was taken under section 9 of the London County Council (General Powers) Act of 1894, and it was stated that, for some time, sewermen had been overcome by sulphurous fumes, and one had his hand and arm burned by coming into contact with acid. The nuisance was traced to defendants' premises, and samples were taken last month. It was found that one contained 201 grains, per gall., of sulphurous acid, others 318 and 295 grains. The fumes could be noticed half a mile away from the defendants' premises, and the sewers had been much corroded. If the acid had been treated with lime or soda, the discharge would have been rendered perfectly harmless. The counsel for the defence said this was the first prosecution of the kind under the Act. The defendants, and their predecessors of the same name, had been in business for about 30 years, and this was the first complaint made against them. They felt strongly that they should have had their attention called to the matter before these proceedings were taken. It was customary for them to mix two or more chemicals which together were harmless, but in the present case one of them appeared to have been separately discharged, and so caused the nuisance. A fine and costs were imposed.

#### XIX.—PAPER, PASTEBOARD, Etc.

##### WOOD PULP IN CANADA.

*Comm. Intelligence, Nov. 21, 1901.*

The Department of Customs has published a statement of the exports by Canada of pulp wood and wood pulp for the year ended June 30, 1901. The values thereof amount to 3,335,265 *dols.*, of which 966,920 *dols.* were sent to Great Britain, 2,302,215 *dols.* to the United States, and 66,194 *dols.* to other countries. The total of 3,335,265 *dols.* is an increase of 616,477 *dols.* over the exports of previous years.



To the United States, this is an increase of 244,321 dols., and to Great Britain, an increase of 366,372 dols. The exports of pulp wood showed an increase of 494,247 dols., the United States taking 500,744 dols. worth more in 1891 than in the previous year, and Great Britain, 6,172 dols. less. Of wood pulp, the increased value disposed of was 122,230 dols., the details being: increase to Great Britain, 372,544 dols.; increase to other countries, 6,109 dols.; decrease to the United States, 256,423 dols. Thus, while the United States increased their purchases of pulp wood from Canada by over half a million dollars, and decreased their purchases of the manufactured article by over a quarter of a million, Great Britain's decrease was in the pulp wood and that but small, while in the manufactured article, involving greater labour and the employment of a larger amount of capital, the mother country took from Canada an increased amount greater than the decrease of the United States by 116,121 dols.

It is estimated that the capital invested in pulp mills (as distinct from paper mills) in Canada, at the present time, is not less than 20,000,000 dols. The total annual product of the mills is estimated at 300,900 tons of mechanical pulp and 169,800 tons of chemical pulp, the former valued at 10 dols. and the latter at 30 dols. a ton, making in all a total value of over 8,000,000 dols. The amount of pulp wood consumed for the quantity of wood pulp stated is 970,000 cords a year. These calculations are based upon the stated capacity of the 44 Canadian mills which have sent returns to the Dominion statistician.

#### BARIUM-COATED PAPER FOR X-RAY SCREENS: U.S. CUSTOMS DECISION.

*Eng. and Mining J., Nov. 9, 1901.*

Paper covered with barium-platinum-cyanide crystals, used to show the shadows produced by the Roentgen machine, is not a manufacture of paper, but is dutiable as a surface-coated paper under paragraph 398, Act of July 24, 1897, at the rate of 2½ cents per lb., and 15 cents *ad valorem*. The addition of the chemical preparation does not alter the character of the article as paper. To amount to a manufacture there must be a new name, character, and use.

#### PAPER IMPORTS OF BRAZIL.

*Foreign Office Annual Series, No. 2724.*

In the paper and stationery trade, Great Britain supplies 13,000*l.* to Germany's 103,000*l.* France sends almost four times and Belgium three times as much as Great Britain.

#### XX.—FINE CHEMICALS, Etc.

##### TARTRATES IN ODESSA (RUSSIA).

*Chem. and Druggist, Oct. 26, 1901.*

There are two factories for the manufacture of cream of tartar in Odessa, and the value of the output last year was 49,770*l.*, an increase of 210*l.*, compared with 1899.

##### OTTO OF ROSE IN BULGARIA.

*Chem. and Druggist, Oct. 26, 1901.*

It has been suggested to the Bulgarian Customs that, in future, all flasks of otto of rose exported from the country should bear the Customs House seal. Hitherto the packages containing the flasks have been sealed, and the new suggestion is put forward with a view to suppress ottos which are sold as Bulgarian, but made elsewhere with a modicum of otto from the Balkans.

#### CITRIC ACID AND CITRATE OF LIME AT MESSINA (SICILY).

*Foreign Office Annual Series, No. 2716.*

The yield of citric acid has declined over 50 per cent., falling from 2,186 to 983 cwt. Messrs. Nascio, Aveline and Co., of Messina, manufacture this product.

The total production of citrate of lime, in 1899, amounted to 20,875 cwt., of which 9,246 cwt. were shipped to ports of the United Kingdom, as against 20,030 and 5,509 cwt., respectively, in 1900. The quantity exported to the United Kingdom has fallen off very conspicuously.

#### ESSENTIAL OIL AND CITRIC ACID EXPORTS OF SICILY (ITALY).

*Foreign Office Annual Series, No. 2716.*

Essences and volatile oils consisted of 35,000 kilos. of orange essence, in 1900, and 33,000 kilos., in 1901; 23,000 kilos. of lemon essence, in 1900, and 27,500 kilos., in 1901. Of the totals of both kinds, the United Kingdom took 35,000 kilos., in 1900, and 28,000 kilos., in 1901; whilst 10,000 kilos. went to the United States, in 1900, and 7,000 kilos., in 1901.

Half the production of citrate of lime and orange and lemon juice went to the United Kingdom. Orange juice amounted, in 1900, to 13,000 kilos.; crude lemon juice, in 1900, to 730,000 kilos., in 1901, to 461,000 kilos.; and concentrated lemon juice, in 1900, to 14,000 kilos. Of the total, France took 439,000 kilos., in 1900, and 404,000 kilos., in 1901; the United Kingdom, 275,000 kilos., in 1900, and 19,000 kilos., in 1901.

#### CHEMICAL AND DRUG IMPORTS OF BRAZIL.

*Foreign Office Annual Series, No. 2724.*

The United Kingdom takes the lead only in regard to potash, Germany and the United States being preferred for other chemicals and for drugs, of which France also sends an important proportion.

#### MONAZITE SANDS AT ESPIRITO SANTO (BRAZIL).

*Foreign Office Annual Series, No. 2724.*

Important finds have been made of monazite sands, which will prove very valuable to this State so soon as the question of foreshore rights has been settled with the Federal Government.

#### ERRATA.

*This Journal, Oct. 31, 1901.*

Page 1043, col. 2, line 19 from bottom, for "about" read "about"; line 18 from bottom, for "ground-nut" read "earth-nut"; lines 4 and 10 from bottom, for "ground-nut" and "ground-nuts" read "earth-nut" and "earth-nuts."

Page 1044, col. 1, in first two tables, for "ground-nut" read "earth-nut"; col. 2, first table, for "ground-nut" read "earth-nut."

Page 1045, col. 1, line 23 from top, instead of "It coagulates," &c. read "The milky fluid coagulates"; also for lines 24—26, read "sea salt alone, effects but slight coagulation until aided by the action of heat."

#### BOARD OF TRADE RETURNS.

##### SUMMARY OF IMPORTS.

Articles.	Month ending 31st Oct.	
	1900.	1901.
	£	£
Metals.....	3,044,719	2,621,791
Chemicals and dyestuffs.....	413,115	423,825
Oils.....	917,248	1,048,268
Raw materials for non-textile industries.....	6,850,815	5,353,345
Total value of all imports....	11,225,897	9,449,229

##### SUMMARY OF EXPORTS.

Articles.	Month ending 31st Oct.	
	1900.	1901.
	£	£
Metals (other than machinery)....	3,603,162	3,354,125
Chemicals and medicines.....	771,126	787,755
Miscellaneous articles.....	3,233,783	3,437,857
Total value of all exports...	7,608,071	7,579,737





IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Bleaching materials Cwt.	23,646	16,327	12,855	5,531
Soda compounds...	22,071	22,645	7,531	7,531
Borax, &c. ....	27,113	9,139	11,280	5,494
Brimstone .....	15,035	32,206	8,532	8,254
Nitrate of potash..	..	20,339	12,995	18,004
Chemicals, other Value £	2,396	..	122,688	141,852
Cutch and gambier. Tons	..	829	44,246	17,053
Dyes:—				
Alizarin..... Value £	..	..	13,394	15,325
Aniline and other ..	..	..	55,036	55,726
Indigo .....	142	1,120	2,282	16,998
Bark.....	20,370	30,293	6,590	10,294
Valonia..... Tons	2,438	2,356	27,732	25,744

IMPORTS OF RAW MATERIAL FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Bark, Peruvian .. Cwt.	1,625	3,666	4,770	11,623
Caoutchouc .....	37,345	33,909	459,800	382,108
Gum:—				
Arabic .....	15,918	8,347	25,615	15,089
Lac, &c. ....	5,776	3,733	16,948	15,221
Gutta-percha ....	16,718	9,606	220,640	160,501
Hides, raw:—				
Dry .....	56,894	17,367	141,333	53,911
Wet .....	52,491	68,603	116,894	161,952
Ivory .....	793	677	29,933	25,382
Manure:—				
Guano..... Tons	560	3,580	2,897	14,815
Bones.....	7,701	5,202	33,464	18,594
Paraffin..... Cwt.	81,370	122,936	119,690	122,521
Linen rags .....	1,017	1,908	8,896	14,366
Rapato .....	20,005	11,977	79,348	43,798
Pulp of wood.....	44,094	32,290	245,022	81,684
Rosin..... Cwt.	89,926	145,192	26,026	32,896
Tallow and stearin ..	159,859	113,595	205,050	159,697
Skins:—				
Goat .....	881,285	1,211,823	77,650	117,550
Sheep.....	1,428,280	1,197,340	154,131	115,936
Nitrate of soda..... Tons	10,340	6,022	88,099	67,357
Phosphate of lime ..	26,541	41,566	41,943	62,295

IMPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Cement..... Tons	11,071	29,669	20,712	48,628
China and earth- Cwt.	34,481	44,333	89,227	101,761
enware.				
Drugs..... Value £	..	..	111,757	113,857
Glass:—				
Sheet .....	56,538	129,364	85,900	72,006
Flint .....	12,334	36,011	15,277	38,971
Plate .....	42,613	58,390	76,844	105,585
Bottles .....	119,619	131,825	51,921	67,026
Other..... Cwt.	28,867	30,002	49,725	58,377
Glue and gelatin ..	14,616	21,680	33,778	49,924
Leather, unmanu- ..	133,955	120,413	825,932	787,784
factured.				
Oil-seed cake .... Tons	20,814	21,743	146,140	137,912
Paints and pig- Value £	..	..	106,753	119,375
ments.				
Paper, pasteboard Cwt.	499,606	541,291	351,916	382,778
Scientific instru- Value £	..	..	62,461	65,300
ments.				
Soap and soap Cwt.	13,487	23,060	18,654	27,054
powder.				
Zinc manufac- ..	29,198	35,945	36,716	41,296
tures.				

## IMPORTS OF OILS FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Cocoa-nut..... Cwt.	24,285	55,068	27,711	68,461
Olive..... Tons	632	787	24,912	29,905
Palm .....	75,527	116,859	85,748	131,148
Petroleum:—				
Illuminating.. Gall.	20,204,045	19,892,897	398,500	359,976
Lubricating... ..	3,064,102	3,761,760	91,561	112,718
Seed..... Tons	3,575	4,039	94,184	97,718
Train, &c. .... Tons	2,297	3,907	38,965	71,033
Turpentine .....	53,551	70,799	77,938	89,476

## IMPORTS OF METALS FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Copper:—				
Ore..... Tons	6,097	6,144	77,670	62,731
Regulus .....	8,340	10,945	359,693	419,224
Unwrought.....	5,514	5,043	400,154	343,481
Lead, pig and sheet ..	21,059	15,186	336,350	181,378
Pyrites .....	59,947	52,924	103,567	90,584
Quicksilver .....	76,710	15,112	9,980	1,619
Silver ore..... Value £	..	..	110,457	63,337
Tin..... Cwt.	75,697	66,503	472,721	374,582
Zinc..... Tons	4,668	6,471	93,018	109,128

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH  
ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Alkali..... Cwt.	334,253	..	106,271	£
Bleaching materials ..	132,458	142,471	38,076	47,188
Copper sulphate .. Tons	719	410	16,898	8,882
Chemical manures ..	36,244	42,273	206,209	216,815
Medicines..... Value £	..	..	117,792	118,685
Soda compounds:—				
Ash..... Cwt.	..	131,585	..	28,419
Caustic .....	..	116,731	..	58,370
Bicarbonate .....	..	30,993	..	10,816
Crystals .....	..	22,847	..	3,648
Sulphate .....	..	42,311	..	3,138
Other sorts .....	..	23,680	..	11,177

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 31ST OCTOBER.

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Gunpowder.... Lb.	775,000	Cwt. 5,882	14,731	17,365
Candles.....	1,944,500	2,525,300	33,630	42,174
Caoutchouc..... Value £	..	..	119,683	96,969
Cement..... Tons	20,326	25,503	38,777	47,393
Products of coal, Value £	..	..	174,081	85,841
Earthenware .....	..	..	169,062	162,985
Stoneware .....	..	..	17,168	16,676
Glass:—				
Plate..... Sq. Ft.	196,122	Cwt. 7,603	14,596	11,613
Flint .....	9,421	8,807	23,979	21,412
Bottles .....	81,042	61,952	41,038	33,502
Other kinds ... ..	26,839	19,715	26,206	21,278
Leather:—				
Unwrought....	12,738	10,141	125,098	102,773
Wrought..... Value £	..	..	57,591	57,189
Seed oil .....	2,380	3,366	83,583	94,838
Floorcloth..... Sq. Yds.	2,102,800	2,140,500	108,977	105,064
Painters' materials Val. £	..	..	172,783	166,453
Paper..... Cwt.	91,944	82,461	145,781	132,999
Rags..... Tons	3,909	5,406	23,304	30,048
Soap..... Cwt.	62,434	72,811	74,300	82,627



**EXPORTS OF METALS (OTHER THAN MACHINERY)  
FOR MONTH ENDING 31ST OCTOBER.**

Articles.	Quantities.		Value.	
	1900.	1901.	1900.	1901.
Brass..... Cwt.	11,534	10,483	£ 57,285	£ 54,374
Copper.....	64,873	101,570	250,462	371,773
Lead..... Tons	2,767	3,836	55,329	52,509
Plated wares... Value £	..	..	51,937	56,207
Tin..... Cwt.	9,575	12,441	65,393	73,352
Zinc..... "	15,111	14,409	15,233	11,679

## 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.

20,492. J. Lowden. Apparatus for revivifying spent, foul, or waste lime from gas works or chemical works. Oct. 14.

20,549. H. Balcke. Improvements in or relating to cooling or evaporating apparatus. Oct. 14.

20,757. D. Turk. An improved device for regulating and controlling the supply of gas and air to furnaces and the like. Complete Specification. Oct. 16.

20,998. E. Geille. Improvements in or relating to muffle furnaces. Oct. 19.

21,191. E. J. Hess. A method of and apparatus for production of liquid air, and for the production of motive power. Oct. 22.

21,282. D. B. Morison. Improvements in evaporating and condensing apparatus. Oct. 23.

21,727. A. F. Kécheur and W. A. B. Heerey. A new or improved carburetted apparatus. Complete Specification. Oct. 29.

22,102. G. R. Hislop. Improvements in regenerative settings of gas and other retorts. Nov. 2.

22,244. H. Devonport. Improvements in ovens or furnaces for annealing metallic articles, and for burning bricks and the like. Nov. 5.

22,274. T. Lowe and J. Whysall. An improved composition for removing and preventing incrustations in steam generators, and for cleaning liquor pumps and connections or the like. Nov. 5.

22,301. G. B. Schwerin. Improvements in and relating to apparatus for the extraction of water, and other fluid from mineral, vegetable, and animal substances. Nov. 5.

22,308. Per. W. Lindberg. Improvements in centrifugal separators. Nov. 5.

22,455. M. Blake and R. H. Smart. Improvements in or relating to vacuum pans, and other apparatus heated by steam worms. Complete Specification. Nov. 7.

22,542. The British Thomson-Houston Company, Ltd.—From Asa F. Batchelder, United States. Improvements in electric air-compressors. Complete Specification. Nov. 8.

22,677. E. K. Dutton and E. L. Dutton. Improvements in centrifugal machines for separating, clarifying, and discharging liquids and matters, and in apparatus connected therewith. Nov. 11.

22,891. J. A. Drake. Improvements in or connected with regenerative and other furnaces and kilns. Nov. 13.

23,043. G. A. Bell. Improvements in and relating to filtering apparatus. Nov. 14.

23,049. G. G. M. Hardingham.—From P. Meura, Belgium. Improvements in filter-presses. Nov. 14.

23,129. H. Herberz. Improvements relating to hot air drying apparatus. Complete Specification. Nov. 15.

23,235. T. Kirkland. Improvements in and relating to apparatus for heating liquids. Nov. 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

#### 1900.

15,315. W. T. Sugg. Apparatus for compressing gases. Nov. 6.

16,817. W. W. Hewitt. Apparatus for drying. Oct. 30.

17,054. P. Naef. Process and apparatus for treating materials with liquid and gas. Oct. 30.

18,928. E. L. D. H. Saragonet. Charging apparatus for Siemens-Martin and other furnaces. Oct. 30.

20,216. F. de Mare. Mercurial centrifugal pump. Nov. 20.

21,302. W. W. Harris. Refrigerating apparatus. Oct. 23.

21,431. G. T. Zohrab. See Class II.

22,354. G. W. Blackburn. Coolers, condensers, and heating apparatus. Nov. 20.

#### 1901.

15,989. A. F. P. Hayman. Stoppers for inclined retorts. Oct. 2.

16,071. E. Stauber. Apparatus for rapidly drying moist material. Nov. 20.

17,624. F. C. Crean. Apparatus for disintegrating and drying pulp. Nov. 20.

19,237. E. Füllner. Process of separating mechanical admixtures from liquids, and apparatus therefor. Nov. 6.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

20,726. C. R. Hinckesman. Improvements in connection with burners for incandescent gas lighting. Oct. 16.

20,747. A. J. Boulton.—From A. M. Plaissetty, France. Improvements in or relating to the manufacture of incandescent filaments and mantles. Oct. 16.

20,760. H. Praetorius. Improved process and apparatus for mixing liquid fuels of different specific gravities. Complete Specification. 16 Oct.

20,986. W. R. Harrison, A. Robb, and T. L. Bell. Improvements in or in connection with apparatus for the production of acetylene gas. Oct. 19.

21,082. A. C. Wells. Improvements in and relating to acetylene gas generators, or the like. Oct. 21.

21,120. A. H. Byron. Composition of matter for generating gas for motive power purposes, method of producing the gas, and apparatus to be used in connection therewith. Complete Specification. Oct. 22.

21,125. F. Crossley. Improvements in incandescent gas burners. Oct. 22.

21,354. H. Higgins. Improvements in or relating to the treatment of peat, and in apparatus therefor. Oct. 24.

21,356. G. H. Hughes. Improvements in or relating to the production of gas. Oct. 24.

21,376. J. Radcliffe. Improvements in the manufacture or production of combustible gas. Complete Specification. Oct. 24.



21,377. J. Radcliffe. Improvements in apparatus suitable for the manufacture or production of combustible gas for calcining or roasting ores and other substances, and for other purposes. Complete Specification. Oct. 24.

21,415. A. G. Browning and H. Musgrove. Improvements in the composition of artificial fuel and kindlers. Oct. 25.

21,534. C. Killing. Improvements in incandescent gas burners. Complete Specification. Oct. 26.

21,615. M. Koblenzer. Improvements relating to the manufacture of incandescent gas mantles. Oct. 28.

21,692. H. D. Fitzpatrick.—From F. H. Macpherson, Canada. Improvements in vapour generators for incandescent lighting. Complete Specification. Oct. 29.

21,720. H. A. Humphrey. Improvements in the purification of gases. Oct. 29.

21,756. E. F. Colborn. Improvements in the production of combustible gas from hydrocarbon oil. Complete Specification. Oct. 29.

21,757. J. Cayrol. Improvements in burners for lighting-gas and other combustible gases. Complete Specification. Oct. 29.

21,763. P. Hudson. Improvements in apparatus for producing and projecting light for signalling and other purposes. Oct. 29.

21,771. C. S. Burtwell. Improvements in apparatus for generating acetylene gas. Oct. 29.

21,837. A. L. Schubert. See Class IX.

21,926. R. Farrington. An invention for protecting incandescent gas mantles against concussion and breakage. Oct. 31.

21,982. W. A. Feurt and H. H. Martin. Improvements in acetylene gas generators. Complete Specification. Oct. 31.

22,016. W. J. Crossley and J. Atkinson. Improvements in gas producers. Nov. 1.

22,105. J. W. Bray. Improvements in and relating to acetylene gas burners. Complete Specification. Nov. 2.

22,131. H. A. Humphrey. Improvements in centrifugal apparatus for purifying gases. Nov. 2.

22,349. C. H. Schill. Improvements in the manufacture of gas, and in apparatus employed in such manufacture. Nov. 6.

22,486. J. F. Henderson and C. W. Clayton. An automatic carbonic acid gas generator and holder. Complete Specification. Nov. 7.

22,645. W. H. Westwood, E. T. Wright, and W. W. S. Westwood. Improvements in the discharging mouthpieces of inclined gas-making retorts. Nov. 9.

22,648. W. Doman and S. W. Bullas. Improvements in apparatus for purifying gases. Nov. 9.

22,766. F. Paul, jun., and A. F. Gundlack. Improvements in the generation of gas. Complete Specification. Nov. 11.

22,786. D. Irving, W. P. Gibbons, and G. B. A. Gibbons. Improvements in the construction of regenerative and other furnaces for gas retorts. Nov. 12.

22,931. The Simon - Carves Bye-Product Coke - Oven Construction and Working Company, Ltd.—From The Société Anonyme de Carbonisation, France. Improvements in or relating to coke ovens. Complete Specification. Nov. 13.

23,056. R. Fabry and F. Linard. Improvements in coke ovens. Complete Specification. Filed Nov. 14. Date applied for 29 June 1901, being date of application in Belgium.

23,206. W. P. Thompson.—From Compagnie Générale d'Incandescence par le Pétrole et l'Alcool, France. Improvements in and relating to incandescent burners. Complete Specification. Nov. 16.

23,212. J. De Brouwer. Improvements in apparatus for charging retorts in the manufacture of gas. Nov. 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

18,279. P. Naef. Treatment of fuel and the recovery of by-products. Nov. 20.

19,074. R. Marston. Manufacture and use of nitrogen and nitrous oxide from atmospheric air. Oct. 23.

19,087. P. R. De Fauchaux D'Humy. Manufacture of fuel. Oct. 30.

19,254. R. P. Pictet. Method of and apparatus for the separation of gases from their mixtures. Oct. 30.

19,377. W. J. Crossley and J. Atkinson. Gas producers. Oct. 30.

19,776. J. Mallof. Incandescent gas burners. Nov. 6.

20,484. T. S. Clapham. Apparatus for use in the purification of gas. Oct. 30.

20,588. F. Coe. Incandescent gas lighting and burners. Oct. 30.

20,893. H. Holm. Carbonisation of peat, and apparatus therefor. Oct. 30.

21,431. G. T. Zohrab. Drying and condensing of peat or other material, and apparatus therefor. Oct. 30.

21,641. A. Custodis. Manufacture of coke. Nov. 20.

22,644. C. Humfrey. Washes for producer or other gases. Oct. 30.

23,605. The Portable Gas Fountain Syndicate, Ltd.—From J. Thovert, France. Incandescent gas burners. Oct. 30.

23,662. A. J. Bouit.—From Desiderius Turk and The Actiengesellschaft "Lauchhammer," Germany. Process for the production of gases of high caloric value from low caloric fuel material. Oct. 30.

23,701. A. Rosenberg. Self-igniting incandescent gas lights. Oct. 30.

1901.

83. J. Bonnet and J. S. Muller. Machines for the manufacture of incandescent gas mantles. Oct. 30.

100. E. J. Duff and The United Alkali Company, Ltd. Means to be employed in the treatment of producer gases. Nov. 20.

2020. E. Gobbe. Apparatus for the utilisation of the heat of coke removed from retorts or ovens. Nov. 20.

3586. J. H. Darby. Apparatus for the manufacture of coke. Nov. 6.

4323. E. R. Besemfelder. Manufacture of cyanogen compounds from gas mixtures containing ammonia. Oct. 23.

5757. A. Hayes. Vaporising and burning hydrocarbon oils. Nov. 13.

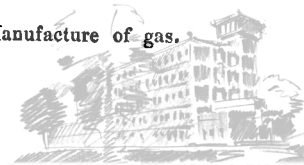
5948. R. W. B. Creeke. Apparatus for washing and cleansing gas from ammonia and other impurities. Oct. 30.

16,347. H. H. Lake. From P. Jebsen, Norway. Treatment of peat. Oct. 30.

16,356. P. A. Fichet and R. M. J. Heurtey. Gas generators. Nov. 6.

17,171. E. B. Cornell and W. C. Alderson. Process and apparatus for making a fixed gas for lighting, heating or any other purpose. Oct. 23.

17,510. F. W. C. Schniewind. Manufacture of gas. Nov. 13.



- 17,852. S. F. Pierce. *See* Class X.
- 17,863. F. Chailly. Manufacture of fuel briquettes, blocks, or pieces. Nov. 6.
- 17,975. J. O. O'Brien. — From M. Wagner, France. Acetylene lamps and generators. Nov. 20.
- 18,513. O. Ruppert. Manufacture of illuminating gas and coke, and apparatus therefor. Nov. 13.
- 19,247. G. Ihle. Bunsen burners for use in incandescent lighting. Nov. 6.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, PETROLEUM.

#### APPLICATIONS.

- 20,716. T. Rigby. Improvements in the treatment of furnace gases or producer gases, and connected with the recovery of ammonia and tar from the same, and in apparatus therefor. Oct. 16.
- 21,874. A. Nikiforoff. Improvements in the manufacture of the benzols and their homologues, and in apparatus therefor. Oct. 30.

### IV.—COLOURING MATTERS AND DYESTUFFS.

#### APPLICATIONS.

- 20,551. J. Y. Johnson. — From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of azo colouring matters and intermediate products relating thereto. Oct. 14.
- 20,552. J. Y. Johnson. — From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of derivatives of indigo, and of intermediate products relating thereto. Oct. 14.
- 20,553. J. Y. Johnson. — From The Badische Anilin und Soda Fabrik, Germany. The manufacture of a new colouring matter, of lakes therefrom, and of an intermediate product relating thereto. Oct. 14.
- 20,741. R. B. Ransford. — From L. Cassella and Co., Germany. The manufacture of purified sulphur dyestuffs from dialkylamidooxydiphenylamines and new intermediate products. Oct. 16.
- 21,077. O. Imray. — From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of phenylamidoacetonitrile and homologues thereof. Oct. 21.
- 21,773. R. B. Ransford. — From L. Cassella and Co., Germany. The manufacture of acridine dyestuffs. Oct. 29.
- 21,879. G. W. Johnson. — From Kalle and Co., Germany. Improvements in and connected with the manufacture and production of blank colouring matters containing sulphur. Oct. 30.
- 22,128. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture and production of new derivatives of the anthraquinone series. Nov. 2.
- 22,189. C. D. Abel. — From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of colouring matters belonging to the triphenylmethane series. Nov. 4.
- 22,222. G. W. Johnson. — From Kalle and Co., Germany. Improvements in the manufacture and production of brown dyes containing sulphur. Nov. 4.
- 22,306. R. B. Ransford. — From L. Cassella and Co., Germany. The manufacture of dyestuffs. Nov. 5.
- 22,385. C. D. Abel. — From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of sulphurised colouring matters directly dyeing cotton. Nov. 6.
- 22,583. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of anthraquinone derivatives. Nov. 8.

22,674. A. Smith. Improvements in the method of preparing indelible inks for marking linen and cotton textile materials. Nov. 11.

22,733. O. Imray. — From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Improved manufacture of phenylglycine, its homologues, and salts thereof. Nov. 11.

22,734. C. D. Abel. — From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of sulphurised colouring matters directly dyeing cotton. Nov. 11.

22,762. J. Y. Johnson. — From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of colouring matter of the anthracene series. Nov. 11.

22,838. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of derivatives of the anthracene series. Nov. 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

12,899. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of azo colouring matters, and of intermediate products for use therein. July 3.

19,202. G. B. Ellis. — From F. A. Pertsch, Switzerland. Manufacture of anthranilic acid and colouring matters. Oct. 30.

20,718. B. Willcox. — From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of new colouring matters of the anthracene series and of intermediate products related thereto. Oct. 23.

20,719. B. Willcox. — From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of colouring matters of the anthracene series. Oct. 30.

21,310. R. B. Ransford. — From L. Cassella and Co., Germany. Manufacture of a sulphide colour and of its leuco compound. Oct. 30.

21,365. G. B. Ellis. — From Société Chimique des Usines du Rhône, anciennement G. P. Monnet et Cartier, France. Manufacture of sulphonated aldehydes and colouring matters therefrom. Nov. 6.

21,821. B. Willcox. — From The Badische Anilin und Soda Fabrik, Germany. Manufacture of an initial material for the production of indigo. Nov. 13.

21,897. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of dyestuffs of the anthracene series. Nov. 6.

21,898. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of dyestuffs containing sulphur. Oct. 23.

22,214. G. B. Ellis. — From Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. Manufacture of substituted sulphinides. Oct. 30.

22,328. W. L. Wise. — From Knoll and Co., Germany. Manufacture of biacetate of anthrapurpuriae. Nov. 20.

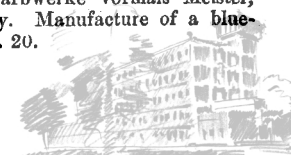
22,391. O. Imray. — From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of compounds of phenyldimethylpyrazolone and dimethylamidodimethylphenylpyrazolone with camphonic acid. Oct. 23.

22,758. O. Imray. — From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of phenylglycine-ortho-carboxylic acid. Oct. 23.

23,902. F. Kehrmann. Manufacture of colouring matters of the thiazine series and of intermediate products therefor. Nov. 6.

1901.

392. O. Imray. — From Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of a blue-grey dyestuff for cotton. Nov. 20.



913. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of dyestuffs capable of giving on fibre shades fast against washing. Nov. 13.

1787. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of colouring matters on the fibre, and of products for use therein. Nov. 20.

11,022. A. Rahtjen. Method of preparing monobrome-indigo and dibrome-indigo as well as monochlore-indigo and dichlore-indigo as well as monochloremono-brome-indigo. Nov. 13.

11,533. A. Thilmany. Manufacture of composite colouring matters or pigments. Nov. 20.

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

##### APPLICATIONS.

21,272. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements relating to printing with the aid of dyestuffs containing sulphur. Oct. 23.

21,595. T. Burnley and R. J. Midgley. Improvements in or connected with the method of and means for treating ramie or rhea fibre, china grass, or other similar vegetable fibre. Oct. 28.

21,628. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of derivatives of cellulose. Oct. 28.

21,645. P. Bourcart. An improved process and apparatus for the mercerisation of vegetable fibres. Complete Specification. Oct. 28.

21,881. H. L. Offermann. An improved process for washing wool. Oct. 30.

22,407. C. F. Topham. Improvements in or connected with the manufacture or treatment of fibres, or filaments, produced from solutions of cellulose. Nov. 6.

22,688. W. Rodger. An improved method of producing variegated or parti-coloured effects upon yarns or fabrics or other substances. Nov. 11.

22,689. W. Rodger. An improved method of producing variegated or parti-coloured effects upon woollen, worsted, and silk yarns or fabrics, or mixtures thereof. Nov. 11.

22,968. A. J. E. Hill. Improvements in the treatment of fabrics. Nov. 13.

22,996. J. T. Pearson. Improvements in mercerising, lustring, drying, and otherwise treating yarn and other similar fibrous substances and materials with liquids, air, and other gases. Nov. 14.

23,042. C. Bardy. Improvements in the manufacture of viscose silk. Nov. 14.

23,181. T. Pratt. Improvements in apparatus for mercerising cotton yarns in hank form, applicable also for dyeing, bleaching, drying, and otherwise treating such yarn or yarns of other fibrous material by analogous processes. Nov. 16.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

15,191. J. O. Obermair. Dyeing apparatus. Oct. 30.

21,072. F. B. M. Raabe. Manufacture of yarn from certain vegetable waste material. Nov. 13.

22,776. G. de Keukelaere. Machines for dyeing textile materials. Nov. 20.

22,947. R. Spitz. Manufacture of yarn, and cloth therefrom. Oct. 30.

23,110. A. J. Boulton.—From A. Gagedois, France. Bleaching of vegetable fibres and fabrics. Nov. 20.

23,157. C. F. Topham. Apparatus for use in the production of textile fibres or filaments from solutions of cellulose or of other material from which fibres or filaments can be formed. Nov. 13.

1901.

1285. C. D. Abel.—From Actiengesellschaft für Anilin Fabrikation, Germany. Dyeing. Oct. 30.

4303. J. Imray.—From E. Bronnert, M. Frémery, and J. Urban, Germany. Manufacture of thread from cellulose solutions. Oct. 23.

#### VII.—ACIDS, ALKALIS, AND SALTS.

##### APPLICATIONS.

21,314. A. Mason. Improvements in the use and utilisation of carbonate of lime obtained in the recovery of sulphur from alkali waste, and commonly called "Chance mud." Oct. 24.

22,470. R. Hodgson. Improvements in apparatus for the manufacture of broad salt from brine. Nov. 7.

22,629. C. Dreher. Process for the manufacture of faintly acid and neutral solutions and salts respectively of titanic acid and lactic acid. Complete Specification. Nov. 9.

22,889. G. F. Berry. 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. Nov. 12.

23,124. W. P. Thompson.—From Stassfurter Chemische Fabrik vormals Vorster und Grüneberg A. G. Germany. Improvements in the production of anhydrous cyanide of sodium from an aqueous solution. Complete Specification. Nov. 15.

23,125. W. P. Thompson.—From Stassfurter Chemische Fabrik vormals Vorster und Grüneberg, A. G., Germany. An improved process for making briquettes of a mixture of cyanide of potassium and cyanide of sodium. Complete Specification. Nov. 15.

23,188. C. Dreher. Process of manufacturing double salts of titanic acid and oxalic tartaric acid, and of oxalic lactic acid respectively, capable of neutralisation. Nov. 16.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

17,053. P. Naef. Process and apparatus for treating materials, such as bicarbonate of soda and other materials for drying, calcining, or distilling them. Oct. 30.

19,326. L. Alpe. Composition or flux for use as a substitute for borax. Nov. 6.

21,214. D. de Vulitch and J. d'Orlowsky. Process of and apparatus for the production of calcium carbide. Nov. 13.

23,811. J. G. Lorrain.—From G. Thomson, United States. Treatment of liquids containing copper and the like. Nov. 20.

1901.

276. G. M. Vis. Purification of brine. Nov. 13.

1385. O. Imray.—From Farbwerke vormals Meister Lucius und Brüning, Germany. Manufacture of sulphuric anhydride by the contact process. Nov. 13.



## VIII.—GLASS, POTTERY, AND ENAMELS.

## APPLICATIONS.

20,643. C. Berhenke. Process and apparatus for drying bricks and other earthenware, or ceramic products. Complete Specification. Oct. 15.

21,158. T. W. Simpson. Improvements in or connected with machines for manufacturing glass bottles and the like. Oct. 22.

21,846. L. Grote. Improvements in or relating to machines for making glass bottles. Oct. 30.

22,577. B. J. B. Mills.—From A. Bocuze, France. Mould for glass works. Nov. 8.

## COMPLETE SPECIFICATION ACCEPTED.

1900.

21,226. A. E. Brown. Drying of earthenware articles such as bricks, tiles, pipes, terra-cotta pottery, clay, or the like, or other articles, such as timber, &c. Oct. 30.

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

## APPLICATIONS.

20,891. S. Höyer-Ellefson. Improvements in and relating to fire-proof floors and the like. Complete Specification. Oct. 18.

21,669. A. Steel. Novel process for re-converting or utilising discarded, broken, or old plaster of Paris moulds, objects, or ornaments. Oct. 29.

21,837. A. L. Schubert. Improvements in process of producing lime or cement and combustible gases. Oct. 30.

22,314. C. von Forell. A process and apparatus for the preparation of Portland cement from blast-furnace slag or cinder. Complete Specification. Nov. 5.

22,601. J. S. Rigby. Maturing Portland cement. Nov. 9.

23,162. H. E. Mason. A new or improved refractory material for use in substitution for ordinary fire-clay and fire-clay compounds, and the like. Nov. 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

1900.

21,226. A. E. Brown. See Class VIII.

1901.

3101. F. Howett. Artificial stone, in situ pavements, floors, walls, ceilings, and the like. Nov. 6.

10,084. S. E. Boivie. Process for the manufacture of artificial stone. Nov. 6.

15,402. G. Feyerabendt. Method of preserving wood by treating it with sodium aluminate. Oct. 23.

16,640. O. Hertwig and E. Liebang. Manufacture of artificial marble and the like. Nov. 6.

17,661. P. A. Newton.—From The New Jersey Wire Cloth Company, United States. Fireproof constructions and method of making the same. Nov. 6.

## X.—METALLURGY.

## APPLICATIONS.

20,507. E. H. Hopkins. An improved method of condensing zinc. Oct. 14.

20,565. E. Knudsen. Process and apparatus for smelting and concentrating unroasted sulphide ores. Complete Specification. Filed Oct. 14. Date applied for March 14, 1901, being date of application in Norway.

20,657. H. Wachwitz, L. Sattler, and M. Dünkelsbühler. Improvements in or relating to the manufacture of plated metals. Complete Specification. Oct. 15.

20,703. R. F. Macfarlane. Process for recovering iron and copper from copper works slag. Oct. 16.

20,875. E. C. Furby and G. Franklin. Non-corrosive metal. Oct. 18.

21,017. W. M. Cranston.—From The American Copper Mining and Extraction Company, United States. Improved method or process for obtaining copper metal from copper-bearing ores. Oct. 19.

21,019. C. G. P. de Laval. Improvements in or appertaining to the distillation of zinc from material containing the same. Filed Oct. 19. Date applied for April 19, 1901, being date of application in Sweden.

21,097. C. Rogers and A. M. Oswald. Improved process for the extraction and recovery of zinc from sulphide ores or tailings. Complete Specification. Oct. 21.

21,176. H. H. Lake.—From Hawley Down Draft Furnace Company, United States. Method of and apparatus for melting and treating metals. Complete Specification. Oct. 22.

21,257. J. Baxeres de Alzugaray. Improvements in and connected with the manufacture of metallic alloys. Oct. 23.

21,377. J. Radcliffe. See Class II.

21,424. H. Brandenburg. A process for recovering metals and metallic compounds from materials containing metals, and particularly from residues. Complete Specification. Oct. 25.

21,445. O. J. Steinhart, J. L. F. Vogel, and C. F. Claus. Improvements in obtaining tin, antimony, and arsenic from complex sulphide ores. Oct. 25.

21,471. E. Young. Improvements in the utilisation of waste products for the manufacture of iron and steel. Oct. 25.

21,481. E. Meininghaus. Improvements in the process of making iron and steel, and in apparatus used therein. Complete Specification. Oct. 26.

21,738. J. F. Duke. Improvements in alloys. Oct. 29.

21,940. D. Reynolds. Process for the production of steel direct from the ore. Complete Specification. Oct. 31.

22,130. W. S. Rawson and R. D. Littlefield. Improvements in refining metals and apparatus for that purpose. Nov. 2.

22,233. P. M. Justice.—From C. Davis, United States. Improved method of treating iron and steel. Complete Specification. Nov. 4.

22,334. W. Stubblebine. Improvements relating to metallurgical furnaces. Complete Specification. Nov. 5.

22,367. F. Rey and S. Gregory.—From C. Wessell, United States. Improvements in or relating to the refining of spelter. Nov. 6.

22,549. W. L. Wise.—From R. McKnight, United States. Improvements in the extraction of gold from refractory ores. Nov. 8.

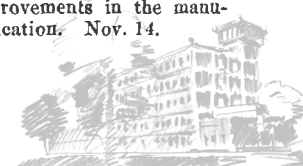
22,554. J. G. Lorrain.—From G. Thomson, United States. A new or improved process of making alloys, and products of the said process. Nov. 8.

22,730. H. Johnson and G. W. Frier. Improvements in the process of manufacturing steel. Nov. 11.

22,753. H. J. Haddan.—From the Meteorit Gesellschaft, G. m. b. H., Germany. A new or improved process for improving the quality of aluminium. Complete Specification. Nov. 11.

22,927. E. H. Hopkins. An improved method of condensing zinc. Nov. 13.

23,060. W. Pruszkowski. Improvements in the manufacture of steel. Complete Specification. Nov. 14.





## COMPLETE SPECIFICATIONS ACCEPTED.

1900.

14,291. E. L. Graham. Process for the disintegration, separation, and concentration of metals from their ores. Nov. 13.

15,166. H. F. Kirkpatrick-Picard. Treatment of slags and by-products containing zinc. Oct. 30.

18,669. G. W. Hinckley. Roasting iron compounds. Oct. 30.

19,273. T. Barton and T. B. McGhie. Process for the dezincing of zinc desilverised lead. Oct. 30.

19,326. L. Alpe. See Class VII.

19,592. E. M. Bradford. Process for the recovery of tin from slag, "hardhead," or other by-products of the smelting of tin ore or manufacture of tin. Nov. 6.

20,513. A. Simon. Manufacture of iron. Nov. 6.

20,702. T. A. Irvine. Extraction of copper by the wet method. Nov. 20.

20,894. H. Goldschmidt. Process for welding materials. Nov. 20.

21,552. A. J. Boulton. From E. Goldschmidt, Germany. Production of steel. Oct. 30.

22,554. E. H. Hopkins. Treatment of complex ores. Nov. 20.

23,315. A. Reynolds. Converters. Nov. 13.

1901.

788. W. W. Slater and J. Galloway. Economical treatment of auriferous matters. Nov. 20.

6328. A. J. Lustig, L. Kahn, and I. Lehman. Apparatus for use in casting ingots. Oct. 23.

10,705. A. G. Betts. Coating of aluminium or its alloys. Nov. 13.

13,867. M. Ruthenburg. Methods of and apparatus for agglomerating comminuted ores and concentrates. Oct. 23.

17,639. F. C. Crean. Manufacture of iron or iron alloys. Oct. 23.

17,852. S. F. Pierce. Heat-producing devices for the smelting of ores and the fusing of minerals in furnaces. Nov. 6.

19,150. J. Mactear. Machine for treating alluvial deposits for the extraction of metals and minerals therefrom. Oct. 30.

19,403. A. Manhardt. Process for producing an aluminium alloy. Nov. 6.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## APPLICATIONS.

20,845. R. B. Sangster. Improvements in electrical storage cells or accumulators. Oct. 18.

20,945. The Cowper-Coles Galvanising Syndicate, Ltd., and S. Cowper-Coles. Improvements in means to be employed in the electro deposition of metals. Oct. 19.

21,285. L. Bristol. Improvements in electric batteries. Oct. 23.

21,294. A. Tribelhorn. Improvements in electric accumulator batteries. Complete Specification. Oct. 23.

21,342. M. A. Codd. Improvements in dry cells. Oct. 24.

21,362. C. T. J. Oppermann. Improvements in secondary batteries. Oct. 24.

21,649. L. Roselle. Improvements in and in connection with accumulator plates. Oct. 28.

21,915. A. Philip. Improvements in the electro deposition of metallic copper. Oct. 31.

21,942. P. Nauhardt. Process for the electrolytic treatment of tin, and an apparatus therefor. Complete Specification. Oct. 31.

22,303. P. Marino and G. Marino. Improvements in and relating to secondary batteries. Complete Specification. Nov. 5.

22,482. J. Wetter.—From H. Bremer, Germany. Improved process or processes for coating or impregnating conducting materials, such as iron, with minute particles or other materials, such as carbon, by means of electric currents. Nov. 7.

22,767. A. E. Greville. Improvements in primary batteries. Nov. 11.

23,215. V. Cheval and J. Lindeman. Improvements in electrical accumulators. Complete Specification. Filed 16 Nov. Date applied for, 22 April 1901, being date of application in Belgium.

## COMPLETE SPECIFICATIONS ACCEPTED.

1900.

19,072. G. J. Gibbs. Electrical accumulators. Oct. 23.

23,310. The British Power Traction and Lighting Company, Ltd., and G. J. Gibbs. Electrical accumulators. Oct. 30.

1901.

294. M. M. Blair. Galvanic cells or batteries. Nov. 13.

2853. G. W. Johnson.—From Société Paul Chapuy and Co., France. Electric batteries. Nov. 20.

12,974. A. F. Madden. Apparatus for making secondary battery plates. Nov. 6.

13,048. A. Tribelhorn. Electrodes for accumulators. Nov. 13.

15,816. R. M. Lloyd. Storage batteries, and alarms therefor. Nov. 13.

15,819. R. M. Lloyd. Storage batteries, and ventilation thereof. Nov. 13.

16,128. A. Tribelhorn. Electric accumulator batteries. Nov. 13.

16,187. A. Lessing. Cells of primary batteries. Oct. 23.

19,068. W. S. Henneberg and E. C. H. Pape. Process for electrolytically precipitating and simultaneously amalgamating metals. Nov. 20.

## XII.—FATS, OILS, AND SOAP.

## APPLICATIONS.

20,803. S. H. H. Barratt and The United Asbestos Company, Ltd. An improved waste oil purifier. Oct. 17.

21,229. J. B. Scammell and E. A. Muskett. Improved process for solidifying and toughening oils. Oct. 23.

## COMPLETE SPECIFICATION ACCEPTED.

1901.

19,186. J. Widmer. See Class XVII.

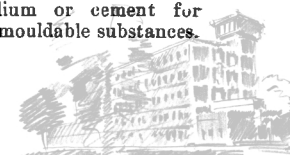
## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

## APPLICATIONS.

20,878. C. L. Boiron and H. Bourdoin. Improvements in the manufacture of india-rubber goods or articles. Oct. 18.

20,789. C. L. Boiron and H. Bourdoin. New or improved process for the treatment of waste or old india-rubber material. Oct. 18.

20,889. L. Grote. An improved process of manufacture of a non-hygroscopic cohesive medium or cement for binding pigments or other pliant or mouldable substances. Oct. 18.



21,526. E. G. Bertrand. Improved paint or protective composition. Oct. 26.

21,546. B. J. B. Mills.—From International Chemical Company, United States. Pigment and process of manufacturing same. Complete Specification. Oct. 26.

21,914. T. A. Cunningham. New protective composition paint. Complete Specification. Oct. 31.

21,976. F. Ellershausen. Improvements in the manufacture of cadmium and zinc pigments from substances containing these metals. Oct. 31.

22,201. H. H. Lake.—From Vereinigte-Gummiwaaren-Fabriken Harburg-wein vormals Meiner J. N. Reithoffer, Germany. Improvements relating to the manufacture of rubber-sponge. Complete Specification. Nov. 4.

22,295. C. A. Luck and A. G. Luck. Improvements in bottles or pots for spirit varnish, knotting, or the like. Nov. 5.

22,758. A. Combanaire and J. de la Fresnaye. Improved process and apparatus for the purification of gutta-percha. Complete Specification. Nov. 11.

23,228. J. C. Roberts. An improved composition or compound for use as cement, paint, or enamel. Nov. 16.

#### COMPLETE SPECIFICATION ACCEPTED.

1900.

21,061. W. Ward. Quick-drying liquid cement paint for the coating of bottoms of iron and steel vessels, and other submerged surfaces, for the prevention of corrosion and fouling. Nov. 20.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### APPLICATIONS.

20,535. E. J. A. Köster. Improvements in the production of chrome leather. Oct. 14.

21,863. M. Hönig. Improvements in the manufacture of tanning extracts. Complete Specification. Oct. 30.

22,062. W. H. Philippi. Improvements relating to the tanning of hides and skins. Nov. 1.

22,576. M. P. Hatschek. Improvements in the treatment of tanning liquors or extracts. Complete Specification. Nov. 8.

22,849. W. P. Thompson.—From A. Wunsch, the Firm of Herkommer und Bangerter, and the Firm of A. Oesinger Company, Germany. An improved chrome tanning process. Complete Specification. Nov. 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

20,612. C. Brandt. Process of quick tanning. Nov. 20.

1901.

19,542. R. Croasdale. Process for treating raw hide. Nov. 13.

#### XVI.—SUGAR, STARCH, AND GUM, ETC.

##### APPLICATIONS.

21,842. J. Robins. New or improved process and apparatus for cooling sugar-syrup. Oct. 30.

21,985. F. Hlavati. Improvements in the process of extracting sugar from beetroot and sugar-cane juices, and other fluids. Complete Specification. Oct. 31.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

18,958. M. Lambert. Crystallisation process, especially applicable for use in the manufacture of sugar. Oct. 30.

20,897. E. Casper. Centrifugal apparatus for treating raw sugar. Nov. 20.

20,898. E. Casper. Drums for drying sugar. Nov. 20.

20,901. E. Casper. Apparatus for treating raw sugar. Nov. 20.

23,508. J. C. F. Lafeuille. Apparatus for the moulding and centrifugal treatment of sugar. Nov. 20.

#### XVII.—BREWING, WINES, SPIRITS, ETC.

##### APPLICATIONS.

20,490. B. T. Howard. Improvements in and connected with presses used in the manufacture of cider, perry, and the like. Oct. 14.

21,824. A. J. Boulton.—From J. G. Dornig, Germany. A new or improved manufacture of alcohol. Oct. 30.

22,116. C. E. Brunt. Improvements in the manufacture of yeast. Nov. 2.

22,135. D. Wickham. Improved apparatus for simultaneously cooling or chilling and gasifying beer and other liquids or beverages. Nov. 2.

22,181. R. C. Wild. Improvements in the manufacture of oppodeldoc or solidified spirit. Nov. 4.

22,928. F. Wrede. Process of and apparatus for converting brewers' yeast into bakers' yeast. Nov. 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1900.

20,855. B. M. Drake and J. M. Gorham. Apparatus for cleansing casks. Nov. 20.

1901.

377. A. Méyer. Vessels for mashing and fermenting purposes. Nov. 13.

931. B. Fischer. Apparatus to promote the germination of malt. Nov. 20.

19,186. J. Widmer. Packing of solidified spirits, mineral oils, or their products of distillation. Nov. 20.

#### XVIII.—FOODS, SANITATION, ETC., AND DISINFECTANTS.

##### APPLICATIONS.

##### A.—Foods.

21,717. G. G. Wolff. A new or improved food product, and process for manufacturing the same. Oct. 29.

21,737. J. H. Hooker. Substances from milk. Oct. 29.

22,336. D. J. Edmonds and W. Weddel.—From D. R. S. Galbraith, New Zealand. Improvements relating to the treatment of milk, cream and butter. Nov. 5.

22,875. W. C. C. Pakes and W. H. Barnes. Apparatus for sterilising milk or other liquids. Nov. 12.

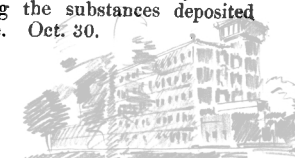
23,141. L. N. Schlarb. Improvements in processes for curing meat. Complete Specification. Filed 15 Nov. Date applied for 3 June 1901, being date of application in United States.

##### B.—Sanitation; Water Purification.

21,316. R. Malabar. Improvements in and connected with the purification of sewage, or other foul or impure liquids. Oct. 24.

21,746. J. E. C. I. Putzeys. Improved means of and apparatus for purifying water. Oct. 29.

21,856. W. P. Thompson.—From Maschinenbau Actiengesellschaft vormals Beck and Henkel, Germany. An improved process for treating the substances deposited from sewage water and the like. Oct. 30.



22,142. C. A. Day.—From Staubschutz Gesellschaft mit beschränkter Haftung, Germany. Improvements in the treatment of offals. Nov. 2.

22,151. A. J. Liversedge. Improvements in appliances for the destruction of refuse and the recovery of useful products therefrom. Nov. 4.

22,923. F. P. Candy. Improvements in apparatus for use in or in connection with the purification of sewage and polluted waters. Nov. 13.

#### C.—Disinfectants.

21,074. V. I. Feeny.—From La Société Anonyme des Etablissements Geneste Herscher et Cie., France. Apparatus for disinfection by the evaporation of solutions, such as formic aldehyde. Complete Specification. Oct. 21.

23,144. F. Reschke. An improved process for manufacturing an antiseptic. Nov. 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

##### A.—Foods.

1900.

22,761. J. A. Timmis. Manufacture of food. Nov. 20.

22,905. A. Pellerin. Manufacture of margarine. Oct. 30.

1901.

7326. F. C. Kullak. Preservation of foods and media therefor. Oct. 23.

##### B.—Sanitation; Water Purification.

1900.

19,315. H. Peck. Destructor for human excreta. Oct. 30.

1901.

497. R. Orchard and C. E. Fox. Purification or sterilisation of water. Nov. 13.

#### C.—Disinfectants.

1901.

508. A. Strandh. Disinfectant compound known as "Lettubrin." Oct. 30.

#### XIX.—PAPER, PASTEBOARD, ETC.

##### APPLICATION.

22,662. J. N. Goldsmith and The British Xylonite Company, Ltd. Improvements relating to the manufacture of celluloid. Nov. 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1901.

21,150. C. K. Graham and The British Uralite Co., Ltd., Manufacture of paper, cardboard, and the like, and apparatus or machinery therefor. Oct. 23.

23,379. D. N. Bertram and S. Milne. Strainers for treating pulp. Nov. 20.

1901.

17,624. F. C. Crean. See Class I.

#### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

##### APPLICATIONS.

20,550. G. W. Johnson.—From Kalle and Co., Germany. The manufacture and production of cinnamyl-quinine hydrochlorate. Oct. 14.

21,077. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. See Class IV.

21,295. J. Rother. A process for producing a powder from alsol sublimate, sozojodol, quicksilver, and veratrine. Complete Specification. Oct. 23.

21,635. G. W. Johnson.—From The Vereinigte Chininfabriken Zimmer and Co., Germany. The manufacture and production of carbonic acid ethers of the dicinchona alkaloids. Oct. 28.

21,758. O. Mierisch and O. Eberhard. Improvements in processes for making milk extracts resembling meat extracts. Complete Specification. Oct. 29.

21,768. F. J. Reichert and C. L. J. J. Heydemann. A method and apparatus for the preparation of an extract of coffee. Oct. 29.

21,838. A. C. McLaughlin. Improvement in the process of making ichthyol sulfonate of ammonium. Oct. 30.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1900

22,214. G. B. Ellis.—From Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. See Class IV.

22,328. W. L. Wise.—From Knoll and Co., Germany. See Class IV.

22,887. B. Willcox.—From The Badische Anilin und Soda Fabrik, Germany. Oxidation of aromatic hydrocarbons and their derivatives. Nov. 20.

23,507. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of neutral soluble silver compounds. Nov. 20.

#### XXI.—PHOTOGRAPHY.

##### APPLICATIONS

21,154. J. W. T. Cadett. Improvements in machinery for coating photographic papers and films with emulsion. Oct. 22.

21,245. W. A. C. Selke. An improved process of photo-sculpture. Complete Specification. Oct. 23.

22,390. W. N. L. Davidson. Improvements in cinematograph apparatus for production of pictures in natural colours. Nov. 6.

22,727. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture or production of photographic plates and papers. Nov. 11.

#### COMPLETE SPECIFICATION ACCEPTED.

1901.

14,510. L. Drefus. Method of photography, and apparatus therefor. Oct. 23.

#### XXII.—EXPLOSIVES, MATCHES, ETC.

##### APPLICATIONS.

20,755. J. Führer. An improved safety detonating composition as a substitute for preparations of fulminate of mercury. Complete Specification. Oct. 16.

21,517. W. Frost. Machines for dipping and heading matches. Oct. 26.

21,621. Safety Explosives, Ltd., and H. A. Thiersch. Improvements in explosives. Oct. 28.

21,660. W. J. Sollas. Composite safety match. Oct. 29.

21,708. M. Buckmaster. An improved powder. Oct. 29.

22,965. A. T. Cocking and Kynoch, Ltd. Improvements in and relating to explosives. Nov. 13.

22,966. A. T. Cocking and Kynoch, Ltd. Improvements in and relating to explosives. Nov. 13.



## COMPLETE SPECIFICATIONS ACCEPTED.

1900.

19,216. A. J. Fredrikson. Wax matches, and machinery for manufacturing the same. Oct. 30.

19,773. C. H. Curtis and G. G. André. Manufacture of gunpowder. Oct. 23.

20,799. R. Kändler. Process for the manufacture of a safety explosive resembling dynamite. Oct. 30.

1901.

984. C. H. Curtis, C. L. Watson Smith, D. J. Metcalfe, A. C. Pearey, and A. F. Hargreaves. Explosives. Nov. 6.

16,509. H. H. Lake.—From F. C. Dininny, jun., United States. Match-making machinery. Nov. 20.

## PATENTS UNCLASSIFIABLE.

## COMPLETE SPECIFICATIONS ACCEPTED.

1900.

18,188. P. Naef. Treatment of solid material with gas, especially applicable for catalytical reactions. Nov. 20.

18,191. P. Naef. Treatment of solid material with gas, especially applicable to catalytical reactions. Nov. 20.

1901.

14,420. J. Philipps, H. Lebel, and R. Grimoin-Sanson. Fabric or material. Oct. 30.

