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It has been felt for some time that it would tend to the advancement of applied chemistry if the Society were to encourage manufacturers to bring their problems and difficulties under the notice of men of skill and experience, such as may be found in works and technical laboratories as well as in universities and colleges.

The Council is now prepared to consider applications from manufacturers and industrial associations who desire to avail themselves of such assistance.

The order of procedure will be as follows:—

The manufacturer or his representative will read a paper at one of the Sectional Meetings setting forth clearly and fully the nature of the problem awaiting solution, and the conditions, chemical, physical, and economic by which it is surrounded. Sufficient statistical information will be supplied to give a fair general idea of the scope and magnitude of the problem. This paper would appear in the Society's Journal, and would serve as a guide to the competitors and a standard of reference to the prize committee which the Council would appoint to regulate the competition.

When the manufacturer or Industrial Association offers a sum of money for a prize or prizes, the Council will accept that sum in trust on behalf of the prize committee.

Problems may be dealt with in several ways, of which the following will serve as illustrations:—

(1.) Special investigations bearing on the subject, apart from the suggestion of practical applications of the facts discovered.

(2.) Practical solutions of problems. The competitors might patent these.

(3.) Essays on the work of others which might bear on the solution of problems.

These last would be a valuable training for students, while the essays themselves would serve as a starting point for new investigations, and would enhance the value of the Society's Journal.

The first prize competition under this scheme is now set forth on p. 1098 of the December number 1899. The Scottish Papermakers' Association offers prizes to the value of 100*l.* for solutions of certain problems connected with their industry. Mr. R. C. Menzies has described the nature of the problems, and the Prize Committee appointed by the Council has laid down the conditions of the competition, particulars of which may be obtained from Mr. G. Monro Thomson, W.S., 123, George Street, Edinburgh. Subsequent problems will be advertised from time to time under the Society's official list of notices in the Journal.

Collective Index.

The Collective Index (1881—1895) is now ready. The prices are as follows:—

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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,500 per month.

Annual General Meeting.

The Annual General Meeting will be held in London on July 18th, 19th, and 20th next. A draft programme is inserted in this number of the Journal with particulars of the Paris Excursion on July 21st.

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Liverpool Section.

Meeting held on Wednesday, February 28th, 1900.

MR. ALFRED SMETHAM IN THE CHAIR.

ESTIMATION OF PROPIONIC AND BUTYRIC ACIDS IN ACETIC ACID.

BY MAX MUSPRATT.

It is a matter of common knowledge that pyroligneous acid, and consequently, pyrolignate, or commercial acetate, of lime contain appreciable quantities of the homologues of acetic acid.

It follows therefore that all acetic acid contains these impurities, and for the majority of purposes, in small quantities, they are unobjectionable; for crystallising purposes, however, the yield and appearance of the crystals may be affected; and, as buyers are inclined to blame the acid if anything goes wrong in the crystallisation, it is desirable for manufacturers to be able to protect themselves by estimation.

A simple method of estimation has still to be discovered; but in the course of investigating various methods I drew conclusions which led me to a method of easy manipulation, and which can be carried out with the usual reagents of a works laboratory.

The only handbook on analysis in which the problem is dealt with is Allen's "Commercial Organic Analysis," and even there suggestions rather than details of methods are given.

It is pointed out that by partial neutralisation and subsequent distillation the higher homologues, such as butyric and propionic acids, can be separated from acetic and formic acids; but the problem still remains as to how much propionic or butyric has been left in the retort as a salt, or how much free acetic acid has distilled over. Indeed this method does not do more than focus the field of further operations, though it is of great utility for this purpose.

A more promising method when only two of the homologues are present is the formation, drying, and weighing of the barium salts. This weight gives with the acidity a simultaneous equation capable of simple solution.

The weak point in this method is that it is necessary to know beforehand that only two of the acids are present, and there is no way of confirming the presence or absence of the third.

It was to a method upon the same lines as this, but with a salt capable of easy confirmation, that I finally came; but before describing it I will continue my experiences of other methods.

An ingenious method of an entirely different character is that of Duclaux; this depends upon the fact that solutions of the acids in question, when distilled, come over in a constant proportion for each fraction of the distillate, the constant being different for each of the homologues.

Thus, if 110 c.c. of acetic acid are distilled, and each 10 c.c. tested, a certain proportion of the total acid is contained in the first fraction, and an increasing proportion in each of the subsequent fractions; with propionic and butyric acids the largest proportion is in the earlier fractions, but the proportion is different for the two.

The matter is made simpler to understand by giving extracts from Duclaux's tables:—

Fraction.	Percentage of Total Acetic.	Percentage of Total Propionic.	Percentage of Total Butyric.
1	5·9	10·5	16·4
2	6·2	10·6	14·7
3	6·7	10·4	13·2
4	6·9	9·9	11·8
5	7·3	9·9	10·1
6	7·6	9·3	9·1
7	8·2	8·9	7·6
8	9·2	8·5	6·3
9	9·8	7·8	4·8
10	12·1	7·2	3·5
11	20·2	7 0	2·5

I am strongly of opinion that this law can only be true within certain limits of concentration and rate of distillation, but Duclaux does not seem to make any reservations.

The application of this method to an unknown acid mixture is carried out by obtaining its fractional proportions and comparing with the tables. The relative quantity of the two unknown acids should be constant for each fraction.

I made a few experiments with this method. An acetic acid, believed to contain butyric acid and possibly propionic acid, was distilled, 1,100 c.c. being taken, and each 100 c.c. was titrated. The results from the titration were calculated out, first on the assumption that propionic was the other acid present, and then for butyric acid. The relative liquors were as given below, acetic acid being taken as 1, P. representing the calculations as propionic, B. as butyric:—

——	P.	B.
1st fraction	1·96	0·41
2nd „	1 37	0·43
3rd „	1·50	0·36
4th „	1·30	0·36
5th „	0·60	0·55
6th „	{ 0·60	1·00 }
7th „	{ 0·18	0·25 }
8th „	2·80	0·59
9th „	1·65	0·38
10th „	1·80	0·62
11th „	1·80	0·67

Fractions 6 and 7 may be rejected, as the titrations gave figures so close to those of the tables that very small errors of working made very large errors in calculation.

Considering the two series of figures, those for propionic are so entirely divergent that the presence of this acid to any extent is out of the question. Column B. is not entirely satisfactory, but, omitting the last two fractions, which are always unreliable, five out of seven fractions give practically constant figures and the ratio from the average of the seven, *viz.* 1 : 0·44, is approximately correct.

The original acid had an acidity of 8 c.c. normal caustic for 10 c.c. of acid; hence the acid contained 3·3 per cent. acetic acid and 1·9 per cent. butyric acid.

Further practice would doubtless have led to greater uniformity in results; but the time and attention required, combined with the large part played by the personal equation, led me to abandon the method.

The fractional crystallisation of the silver salts was next tried, but the large quantity of silver required, as well as the difficulty of boiling down the silver salt without decomposition, caused me to postpone this method until all others failed.

It finally occurred to me to investigate the sodium salts, as these are easily dehydrated, and after weighing can be decomposed into sodium carbonate and titrated, as a check on any loss in dehydrating. A further reason for following up this investigation was the hope that the differing solubilities of the sodium salts might offer a means of readily determining whether two or more acids were present.

Without difficulty I obtained some butyric acid, which I tested as follows:—After dilution a certain volume was titrated carefully with a normal solution of pure caustic soda, a similar quantity was exactly neutralised with sodium carbonate boiled down to dryness in a platinum dish, and weighed; the salt was finally decomposed at a red heat and titrated. The results were, taking the acidity as the mean between the first and last titration,—

Acidity, 42·0 c.c. .... 4·73 grms. sodium butyrate.
Actual weight of salt... 4·77 „ „

The calculated and found weights are thus sufficiently accurate.

The same figures for acetic acid were—

Acidity, 40·6 c.c. .... 3·43 grms. sodium acetate.
Weight found.......... 3·45 „ „

Having obtained these figures, I next tried a mixture of the two, which contained in 20 c.c.—

Acetic acid .... 31·4 c.c. normal NaOH.
Butyric acid.............. 15·0 „ „

After neutralising and dehydrating the mixed salt weighed 4·2 grms.

Calculating this out as if it were an unknown mixture, we obtain the following equations:—

From total acidity.................. $A + B = 46·4$
From sodium salt .... $0·082\,A + 0·11\,B = 4·2$

Solving these equations—

A = acetic acid .... 32·0 c.c. normal NaOH.
B = butyric acid .... 14·4 „ „

As compared with other methods of estimation these results may be considered thoroughly satisfactory. In adopting this method for unknown mixtures, however, the danger still remained that the presence of more than two of the homologues would make the results absolutely misleading, and I therefore looked for a simple method of checking which could be combined with the above estimation.

The solubilities in alcohol finally gave this necessary guide.

An excess of anhydrous sodium butyrate was treated with absolute alcohol for about 10 days, and as much as 30 parts in 100 of alcohol dissolved; but as this exposure was much too long for practical work, I repeated the estimation for an exposure overnight, when 5 grms. were treated with 20 c.c. of alcohol, and the amount dissolved corresponded to 17·8 grms. per 100 c.c. of alcohol.

Treating the anhydrous sodium acetate in the same way, only 2 grms. per 100 c.c. of alcohol were dissolved.

I applied this test to the mixture of butyric and acetic acids already referred to; the dehydrated sodium salts, weighing 4·2 grms., were treated with 20 c.c. alcohol, and 10 c.c. filtered off. The alcohol was evaporated, the salt weighed, and subsequently titrated after fusion, the results being:—

Acidity, 7·6 c.c. .... 0·836 grm. butyrate.
Weight of salt .... 0·840 „ „

Calculating on the total alcohol used, this gives an acidity of 15·2 c.c. normal NaOH, as against 15 c.c. in the solution.

As far as the butyric acid with which I had worked was concerned, these results proved the method reliable; some months afterwards I obtained butyric acid from a different source, and obtained absolutely divergent results as regards solubility in alcohol. Repetition of the experiments in the first sample confirmed their accuracy, and I then saw the necessity of distinguishing between normal and iso-butyric acid. Having first satisfied myself which was normal and which iso, I estimated the solubilities of their anhydrous salts of sodium, and at the same time those of propionic and acetic acids.

			Grms.
Acetic acid :	100 c.c. absolute	alcohol dissolves	1·8
Propionic acid :	„	„	2·9
Normal butyric acid :	„	„	4·0
Iso-butyric acid :	„	„	17·8

This oversight in my first estimation is not so important as might be expected—firstly, as far as commercial acetic acids are concerned, because the butyric acid occurring there is usually the iso form ; and, more generally, because the presence of even normal butyrate much reduces the solubility of both the propionate and acetate.

The general method of estimation to which any experiments have led may now be briefly summarised.

(1.) *Where it is known that only two of the homologues are present.*—The acidity must be measured, and, after exactly neutralising with sodium carbonate, the salt is dried and weighed. As a check on the acidity, the salt is decomposed to sodium carbonate and titrated.

(2.) *Where an Acetic Acid is to be tested for small quantities of Propionic and Butyric Acid.*—The acid is neutralised to about 5 per cent. of acidity and distilled from a retort of metal for preference. A convenient quantity, say sufficient to give 2 to 5 grms. sodium salt, of the distillate is carefully neutralised in the sodium carbonate and dehydrated in a platinum dish. From the weight of the salt and the acidity of the distillate, the results are calculated out as acetate and butyrate, and sufficient alcohol added to dissolve out the butyrate, even if it is normal butyrate. After filtering, the alcohol is driven off, the salt weighed, and then found and titrated. The butyrate calculated from this should agree with that found previously, unless propionic acid is present.

If, therefore, the results disagree, the presence of propionic acid is assured, and must be removed. The one method for this is that of Linnemann, and depends upon the lower solubility of basic lead propionate in hot than in cold solutions.

As described in Allen's "Organic Analysis," the mixed acids are boiled down to dryness with excess of litharge, treated with a little cold water, the propionate and some acetate being dissolved. After filtering, the solution is boiled, and the propionate thrown down suddenly and almost completely as a crystalline precipitate.

In two points my experiences do not agree with this description : in the first place, basic lead propionate has a solubility of only 1 in 10 in cold water, while basic lead acetate has about 1 in 2 ; it therefore seems improbable that the propionate would be dissolved out before the bulk of the acetate. I accordingly treated a mixture of the basic salts with successive quantities of water, and found that the earlier fractions were richer in acetate and poorer in propionate than the later ones. I also found that the deposit was a gradual one, and not a sudden one, and that if acetic acid was present in considerable excess in the original acid, the acetate commenced to come down before all the propionate had been thrown out of solution. The separation was, however, a good one, the deposit of each of the above fractions being checked by formation and weighing of the sodium salt. It is also simple to detect when the acetate commences to separate out, as it forms a light white scale on the surface of the liquor, easily distinguished from the crystalline precipitate of the propionate.

If isobutyric acid is present, the separation is not interfered with ; the basic lead salt of normal butyric acid is, however, very insoluble even in hot solution, and is likely to be partly precipitated with the propionate. The proportion of butyrate can, however, be easily calculated from the sodium salts.

It is of vital importance in this last process that all the acids be converted into their most basic salts, and the boiling down with litharge should be repeated once or even twice.

In conclusion, I must acknowledge the assistance of Mr. T. Brown in a large number of the manipulations.

DISCUSSION.

Dr. KOHN asked Mr. Muspratt whether he had tried steam distillation for the separation of the acids, and also whether in Duclaux's method of separation he had studied the influence of the rate of distillation on the result. In respect to the indirect estimation of the composition of a mixture of three homologous fatty acids by the analysis of their sodium salts, it must be borne in mind that the small differences in the percentages of sodium in the three salts made such a method considerably less accurate than the results from prepared mixtures would indicate. Such methods of analysis applied to compounds differing little in their molecular weight were naturally open to this objection, and he would be glad to hear from Mr. Muspratt whether mixtures of varying composition were tested by the method. The different solubilities of the sodium salts of the acids in alcohol were interesting. At what temperatures were these determined, and were any marked differences noted with a change in temperature ?

Mr. MAX MUSPRATT, in reply, said that distilling with steam was a good idea, but in a works laboratory they had not time to work out every process. Duclaux's process was exceedingly complicated, and would be entirely out of court for works purposes. As to the proportions of the mixtures in the experiments, one almost always aimed at getting proportions about balancing. Of course, the errors in estimation were considerable, but the method he had suggested for practical purposes was near enough for all manufacturing requirements. With regard to the solubilities in alcohol, he had not estimated these at various temperatures; most of the experiments had been carried out during the course of this winter, and therefore the temperature would be rather below the normal of 16½° C. ; but he had not given special attention to this matter of temperature. He would point out that they were industrial chemists, and when it came to a matter like the present one, results less accurate than those expected in, say, the analysis of soda ash served their purpose.

THE TESTING OF ACETONE.

BY JAMES T. CONROY, B.SC., PH.D.

THE manufacture of acetone is based on the direct decomposition into acetone and calcium carbonate which calcium acetate undergoes on heating.

In practice, the calcium acetate employed takes the form of "grey acetate of lime." This material is by no means pure; it contains, in addition to moisture and tarry substances, both free and combined, not only calcium acetate, but also calcium salts of the homologues of acetic acid—formic, propionic, butyric, and possibly still higher acids of the series.

In most of the samples which have come under my observation there was usually present, also, a small percentage of free acid.

Hence, on the dry distillation of grey acetate of lime, we get not only acetone, but also homologous ketones, and, arising from the formic acid, aldehydes together with free acid and tar. Thus from calcium acetate, propionate, and the two butyrates there is the possibility of 10 ketones arising, and there have actually been isolated from crude acetone, methyl ethylketone, methyl propyl, and methyl isoprotylketone, and methyl butylketone. From the calcium formate there might be produced one aldehyde for each acid present.

The crude acetone is purified by dilution with water which separates a considerable portion of the higher ketones and tarry substances ; and by neutralisation. The liquor purified thus far is then fractionated. With proper working the acetone should be obtained in a state of considerable purity.

One of the chief uses of acetone is as a solvent in the manufacture of cordite, the insoluble cellulose hexanitrate being by its aid worked into a homogeneous mixture with the nitroglycerine. For such use the acetone must be free from high boiling constituents, and especially from acid or acid-forming substances, which are injurious, owing, I believe, to their inducing a decomposition of the nitro bodies (esters) present in the explosive.

I have given this brief account of the manufacture and use of acetone, so that you may see what impurities are likely to occur and in what manner they may be harmful—in brief, to explain the form taken by the British Government specification for acetone. This is as follows :—

Specification for Acetone.

1. The acetone to be not more than 0·800 specific gravity at 60° F. When mixed with distilled water it must show no turbidity, and must leave no residue on evaporation at 212° F. On distillation, four-fifths by volume of the quantity taken must distil over at a temperature not exceeding 138° F. The residual matter left after this distillation must not contain, besides acetone, any ingredient that is not a by-product incidental to the manufacture of acetone.

2. One c.c. of 0·1 per cent. solution in distilled water of pure permanganate of potash, added to 100 c.c. of the acetone, must retain its distinctive colour for not less than 30 minutes.

3. The acetone, tested by the following method, must not show more than 0·005 per cent. of acid, calculated to acetic acid :—

To 50 c.c. of the sample diluted with 50 c.c. of distilled water, with 2 c.c. of phenolphthalein solution (1 grm. to 1,000 c.c. of 50 per cent. alcohol) added as an indicator, add from a burette N/100 sodium hydrate solution (1 c.c. 0·0006 grm. acetic acid), and calculate to acetic acid in the usual manner.

Until recently the specific gravity limit was 0·802, and the time limit in the permanganate test two minutes.

You will thus notice that no direct estimation of the acetone is made. In fact there does not appear to be any quick and accurate method for estimating acetone; the various processes proposed suffering from the disadvantage of including, along with the acetone, homologous ketones and other substances. The present method of testing is framed in such manner as to restrict the various impurities within certain limits. In the preceding specification, sections 1 and 3 call for but little comment.

I might, however, point out that in determining the acidity with such dilute alkali as N/100, and using an indicator sensitive to carbonic acid, care should be taken that the water used for dilution should be free from any dissolved CO_2, and its neutrality ascertained at the time of testing. Blowing through the pipette used for any of the solutions must likewise be avoided. I have found that such "blowing" may mean a consumption of very nearly 2·0 c.c. N/100 alkali, whilst the limit of acidity allowed only requires 3·3 c.c.

The remaining test, that with permanganate, calls, however, for much more remark.

What first drew my attention especially to it was a marked irregularity in the time results obtained with permanganate in a series of experiments I was making on the stability of commercial acetone to light. These irregularities caused me to look into this test more carefully, and with the following results.

In the first place I satisfied myself that under similar conditions constant results were obtainable. Thus, if two 100 c.c. portions of any sample were treated, side by side, with 1 c.c. of a 0·1 per cent. potassium permanganate solution, as directed, the duration of the coloration was invariably the same for each portion.

This constancy of result only held, however, so long as the conditions were similar. A slight variation in temperature entirely altered the result, as the following series of experiments shows.

These experiments were done on three samples of acetone of different degrees of purity :—

Sample a.—A specially pure acetone.

Sample b.—Acetone of good quality, rather above specification requirement.

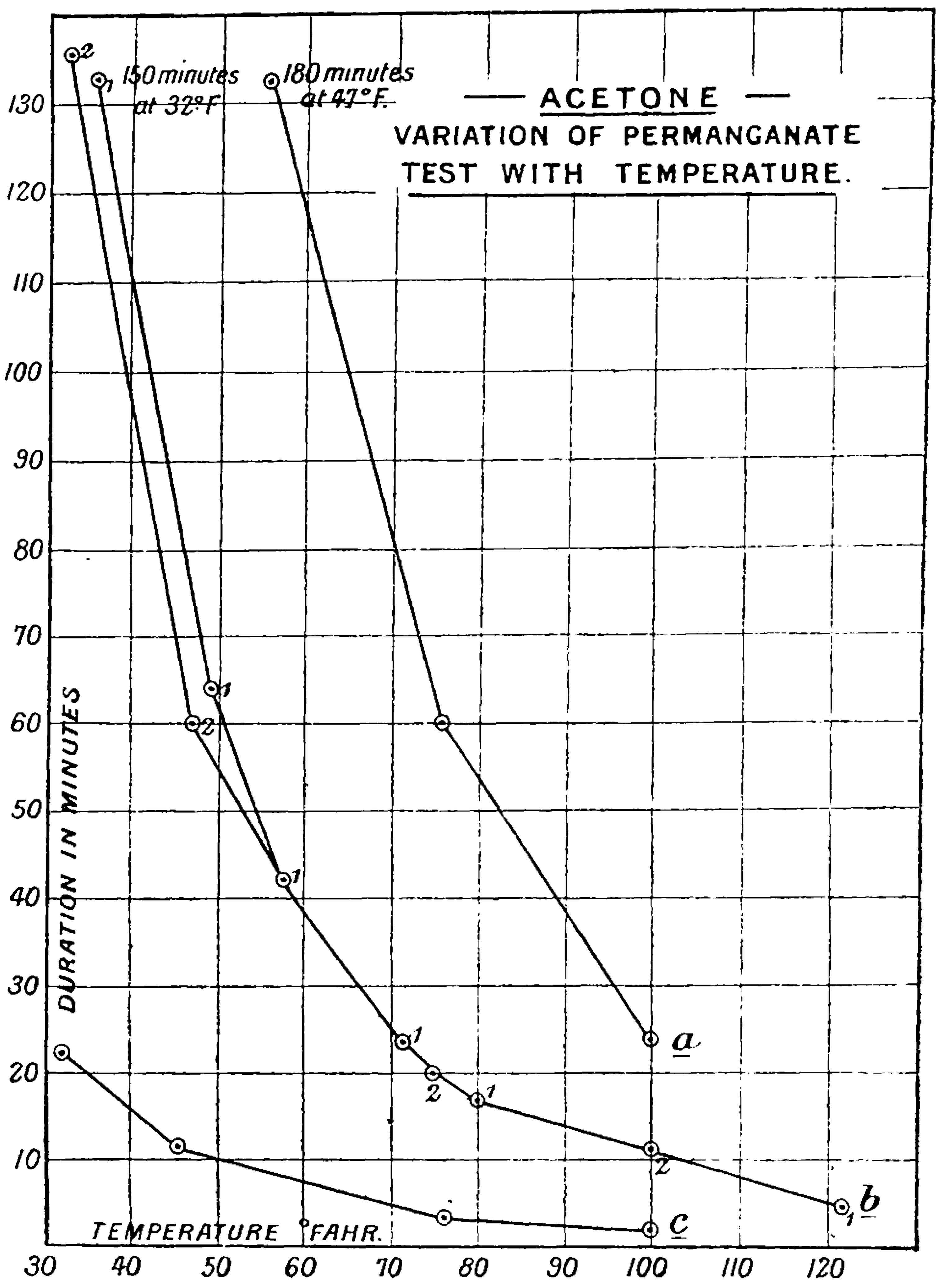
Sample c.—Acetone below specification requirement (in permanganate test only).

Two series of experiments were made on consecutive days with sample *b*; the values obtained in the two series I have distinguished on the curve by the figures 1 and 2.

In performing these experiments the acetone was first brought to the required temperature, and the 1 c.c. $KMnO_4$ solution then added, the whole being shaken to ensure uniform distribution of the permanganate. The time of addition was noted, and the temperature maintained constant until the permanganate colour had vanished. As vanishing point was taken the stage at which all pink had left the solution and the latter was just turning yellow.

The results are plotted in the accompanying curves :—

Temperature Curve.



The temperatures were taken on the Fahrenheit scale, since this scale is quoted in the specification.

From these curves (mean, in case of b_1 and 2) I have compiled the following Table I., in which the duration is given for every 5° C.

TABLE I.

Variation of Permanganate Test with Temperature.

Temp. ° C.	Sample *a*.		Sample *b*.		Sample *c*.	
	Duration.	Ratio.	Duration.	Ratio.	Duration.	Ratio.
	Minutes.		Minutes.		Minutes.	
0	22	2·4	140	2·4	..	..
5	14	1·9	100	2·6	..	..
10	9	1·9	57	2·1	..	..
15	7·5	2·5	38	2·0	117	2·0
20	5	2	27	1·8	85	1·8
25	3	..	19	1·6	58	1·8
30	2·5	..	15	1·6	46	..
35	..	..	12	1·6	32	..
40	..	..	9·5	1·9	..	..
45	..	..	7·5	..	..	..
50	..	..	5	..	..	..

Alongside the minutes column, I have given a column showing the ratio between the duration at any given temperature, and the duration at a temperature removed from this by 10° C.

This ratio is approximately constant.

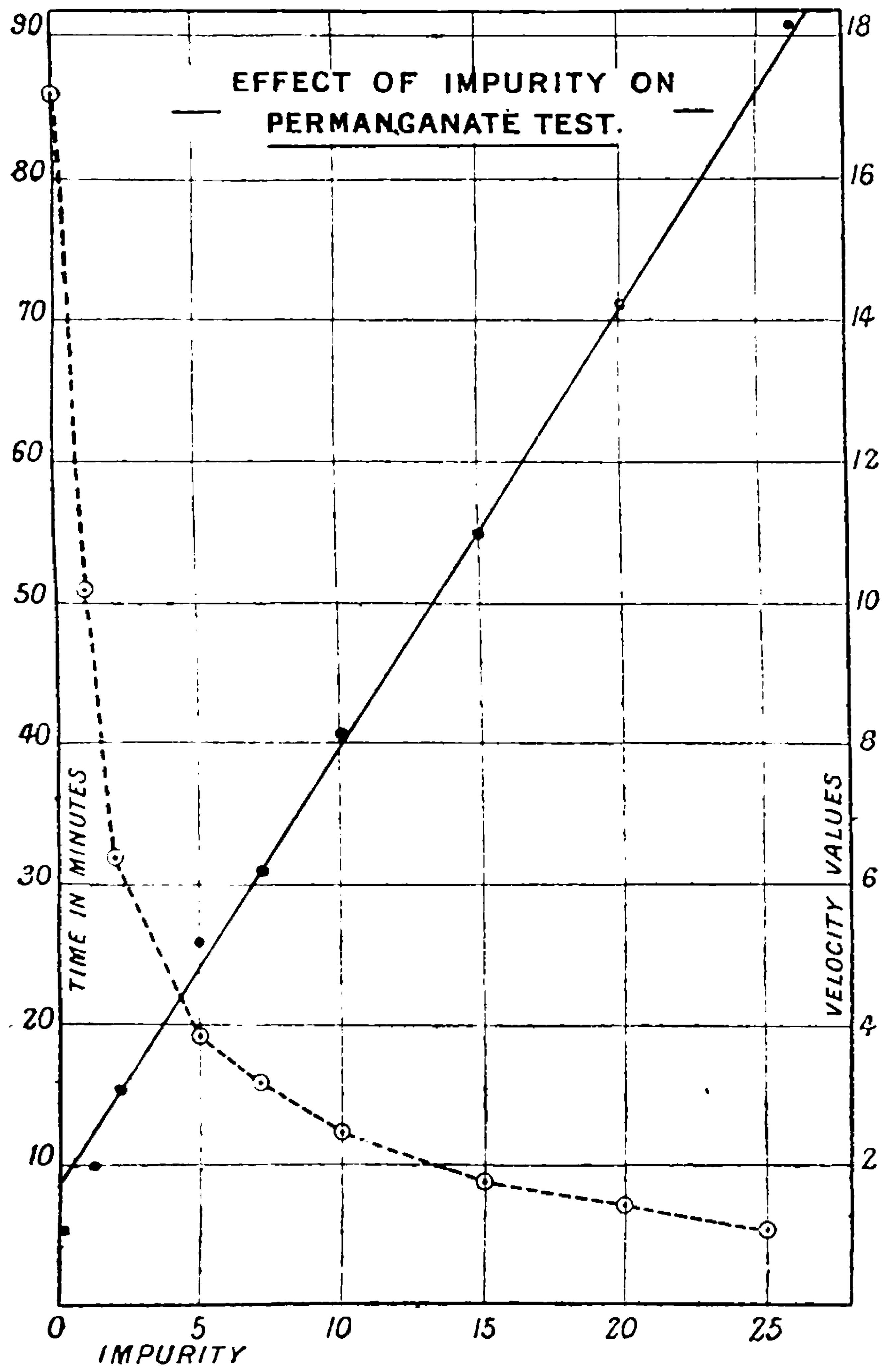
In other words, while the temperature form an arithmetical series, the "duration figures" form a geometrical series.

This is in accordance with the general law which has been found to exist between the "velocity of reaction," and temperature, and it is interesting to note that in this case, as in many others, the ratio of the velocity constants is approximately 2 for temperatures differing by 10° C.

Considering sample *b*, we find that at 15° and 25° C. the times during which the permanganate colour persists to be 38 and 19 minutes respectively.

Thus an acetone which, at approximately average temperature, is of excellent quality, would, in hot weather, unless precautions were taken, appear to be considerably below the required standard. It therefore appears to me to be advisable to specify some standard temperature at which this test is to be carried out. 60° F. would probably be the most convenient temperature, and would correspond with that at which the specific gravity is taken.

Impurity Curve.



Turning now to the quantitative aspect of this test, there is much, I am afraid, which remains obscure. The actual impurities, their number and their amount, are, so far as I am aware, unknown. Probably they are chiefly of the aldehyde class, admixed to some extent with the acetone homologues.

I have failed to find any connection between the quantity of permanganate immediately decolorised by various samples and the time required for the decolorisation of 1 c.c. The reaction seems to be essentially a time reaction.

Further, varying the quantity of permanganate within certain limits does not appear to affect the test. Thus 0·5, 1·0, and 2·0 c.c.s permanganate solution were decolorised in the same time by equal quantities of acetone. The varying quantities of permanganate only affected the depth of the pink tint.

I am, however, able to give some definite results which were obtained by contaminating a pure acetone with varying measured quantities of an impurity occurring in the manufacture of acetone.

The results obtained are shown in Table II., and in the accompanying curve.

Table II.

Effect of Quantity of Impurity on Permanganate Test at 60° F.

Quantity of Impurity.	Duration of Colour.	Velocity value. $\frac{1}{\text{duration}}$ (x 100).
	Minutes.	
0	86	1·17
1	51	1·96
2	32	3·13
5	19	5·26
7	16	6·25
10	12¼	8·17
15	8¾	11·15
20	7	14·30
25	5½	18·20

If, instead of plotting the time value, we substitute the reciprocals of these, in other words, values proportional to the velocity of the reaction between the impurity and the permanganate, we find that the time curve becomes a straight line.

That is (for a given impurity) the rate of decoloration is directly proportional to the quantity of impurity present. This, like the temperature curve, is also in accordance with a well-known law.

Stability to Light.

Chemically pure acetone should remain perfectly colourless on exposure to light. Further, it is not attacked by permanganate in the cold.

Most commercial acetones become on exposure to light more or less yellow, some rapidly others only slowly. This change is usually accompanied by an increase in the acidity and also in the quantity of reducing substances as shown by the permanganate test. This diminution in the purity even takes place in samples whose colour remains unaltered, it seems to be most marked in acetones containing initially reducing substances of low boiling point. Thus in a series of experiments made some months ago, the acidity increased during three months' exposure to daylight, to 10 times its original figure, whilst the permanganate test finally gave below five minutes instead of over 30 minutes as originally. This series of experiments was made before I had noticed the effect of temperature on the permanganate test. The following table (III.), from a series of determinations I am now making, will show the magnitude of the change occurring in a comparatively short time.

Table III.

Effect of Light on Acetone (Commercial).

—	Sample A.		Sample B.				Sample C.	
	$KMnO_4$ Test.	Acidity.	$KMnO_4$ Test.	Acidity.	(In dark) $KMnO_4$	(In dark) Acidity.	$KMnO_4$ Test.	Acidity.
	Minutes.	Per Cent.	Minutes.	Per Cent.	Minutes.	Per Cent.	Minutes.	Per Cent.
Start........................	115	0·0024	38	0·0021	38	0·0021	7½	0·004
In 10 days.....................	13	0·0038	2½	0·0034	36	0·0020	1½	0·005
In 16 days.....................	14	0·0048	2	0·006	37	0·0023	1½	0·0057

The permanganate test was in all cases made at 60° F.

Sample B, under the heading "in dark," was kept in a bottle which was "black varnished" on the outside. All four samples were placed close to a window having a northerly aspect, and in a room maintained at about 60° to 65° F.*

The prejudicial effect of light is thus shown to be both rapid and pronounced.

This brings my present notes to a close. The chief object in making this communication is to draw attention to the variation with temperature in the results obtained by the permanganate test, and I would like once more to emphasise the desirability of fixing some definite temperature, at which it should be carried out. A further point is that all light should be carefully excluded from samples of acetone intended for testing according to the present specification.

Finally, I would acknowledge my thanks to the United Alkali Company, and to Dr. Raschen, to whom I am indebted for permission to read this paper.

London Section.

Meeting held on Monday, March 5th, 1900.

MR. BOVERTON REDWOOD IN THE CHAIR.

THE LATE MR. WM. THORP.

The Chairman: Before we proceed to the formal business of the meeting I wish to make allusion to the loss which the Society has sustained since we last met in the death of our old friend William Thorp, a Vice-President of the Society and Past Chairman of this Section. Mr. Thorp was a man of singularly gentle manners, and at the same time one who was conspicuous for firmness and for the outspoken character of his utterances. He always appeared to me to be one of the few men who are capable of forming an independent judgment upon what I may term an abstract consideration of right and wrong without being in any way influenced by ulterior considerations. Expediency—that antidote for many an unquiet conscience—was a thing of which he never took account; and it followed consequently that his views (always tersely and clearly expressed) commanded the attention and respect even of those who did not share them. I do not say that he was invariably right in his conclusions, for this cannot be said of any strong man, but his highly developed common sense and logical method of thought generally led him in the right direction. Many a sunny disposition would have been soured and crabbed by the physical disabilities against which Mr. Thorp so manfully struggled for many years; and I feel sure it may be said that in the quiet, retired, unostentatious life he led there was as much true heroism as has been exhibited recently on the battle-fields of South Africa.

Mr. T. Tyrer: By your courtesy, sir, I also should like to testify to my sense of the high character and worth of my old friend William Thorp. Nothing could be more appropriate than the remarks you have made, and they anticipate what most of those who knew him best would have wished to say. I was chiefly struck by this characteristic of Mr. Thorp—his absolute truthfulness. I never knew him to prevaricate, and I never knew him to say anything about which he had a doubt without expressing that doubt. Such qualities are much too rare in our professional life. Though his intellect was of the keenest, there was nothing of the "smart" man about Mr. Thorp. I had such a regard myself for his honesty and ability that I could consult him on matters that I would never have mentioned to others; and in thus consulting him I was always struck with the unhesitating way in which he at once separated the "wheat from the tares" and pointed out what was morally the right course. He possessed the love and respect of all who knew him; his friendships were candid and sincere, and were never broken; his conscientiousness and sense of duty were profound.

Mr. David Howard: I find it extremely difficult to speak on such an occasion as this of one who had been my friend for nearly forty years, and of whom I never knew any evil. It is a lesson to all of us that we too should so show that pureness of heart that leaves no doubt that he has fulfilled the blessedness thereof.

Prof. F. Clowes: I should like to add a few words regarding my old schoolfellow William Thorp, who was not only my friend, but whom I had come to look upon as almost an elder brother. So close were our ties that I feel this evening as if I were one of his family, and that I may reasonably thank those gentlemen who have made such just and feeling remarks in appreciation of his character. His name does not occur very often in connection with original work in the proceedings of our Society; but it must be remembered that he was constantly engaged in carrying out research for confidential and private purposes. The results, however, could not be published in the scientific journals. He was not only a staunch friend and a man of the strictest integrity, but he was also a chemist of very great attainments, who accomplished a vast amount of most valuable literary and original work.

The Chairman said that there was one other matter, before proceeding to the business of the evening, to which he wished to make brief reference, *viz.*, the Annual Meeting of the Society, which was to be held this year in London. It would be satisfactory to the members to know that arrangements of a most promising character were well advanced, and that there was every reason to anticipate that the meeting would be a success. That it should be so was more than ordinarily desirable, because they had this year an American President, who would doubtless be supported at the meeting by a number of American members, and he confidently appealed to all present to do their best to help the Committee of the London Section in making the approaching gathering a record one in every sense.

NAPHTHALENE VAPOUR IN COAL GAS.

BY R. W. ALLEN, M.A.

(*Contribution from the Laboratory of University College, Auckland, New Zealand.*)

The discussion as to the amount of naphthalene vapour ordinarily present in coal gas, and the prevention of its deposition therefrom, having been somewhat revived of late, the result of some experiments made by me, about 18 months ago, may be of some interest.

The most plausible explanations of the deposition of naphthalene in gas pipes, being all based on the supposed saturation of the gas by the vapour of that solid at the ordinary temperature, and its consequent supersaturation with deposition of some of the naphthalene when the temperature falls, it seemed well first to ascertain whether this was really the case.

By first determining the amount of naphthalene required to saturate a given volume of such gases as air, hydrogen, and carbon dioxide, gases which, having no affinity for or action on naphthalene, may be designated as "indifferent gases," and then ascertaining the amount of naphthalene required to saturate the same volume of coal gas, carefully purified from any naphthalene it might already contain, at the same temperatures, one might ascertain whether coal gas really differed from an indifferent gas in its behaviour towards naphthalene.

Assuming such a difference, the question arose whether this was due to the presence of the higher hydrocarbon vapours in the coal gas, for these, when condensed being solvents for naphthalene, might be assumed to have some action on the vapour of that solid, and prevent it exerting its proper vapour pressure. If such were the case, then the presence of the vapours of such powerful solvents for naphthalene as ether, carbon bisulphide, benzene, and ethyl-alcohol in the gas ought to emphasise the difference. But

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* Sample A remained colourless after 16 days, as also did sample B when kept in the dark. Exposed to the light, sample B became yellow after 6 days. Sample C was yellow at starting.

if no difference could be detected, then, by comparing the amounts of naphthalene required to saturate equal volumes of air and coal gas at the same temperatures, the exact amount of that substance in the gas could be easily ascertained.

Accordingly, by means of two totally different methods, I determined the weight of naphthalene in grains required to saturate 100 cb. ft. of the indifferent gases, air, hydrogen, and carbon dioxide at various temperatures, between 32° F. and 266° F. The two methods employed were : (1) the direct "evaporation method," wherein a known volume of each gas was passed through U-tubes, containing dry powdered naphthalene, and the weight of the solid carried over estimated by direct weighing; (2) by first determining the vapour pressure by the ordinary barometric method, and calculating therefrom the weight required to saturate the given volume of the indifferent gas.

Without entering into an exact account of the method of working, and the precautions taken to ensure accuracy, let it suffice to say that the results obtained by the two methods were in entire accord. In Table I. is given the vapour pressure at each 9° F., from 32° F. to 212° F., and the corresponding weight of naphthalene in grains required to saturate 100 cubic feet of an indifferent gas.

Note.—Errors of experiment have been as far as possible eliminated by plotting out the observed vapour pressures on a diagram on which 20 divisions = 1 mm. of vapour pressure. The corresponding weights of naphthalene being plotted out on a new diagram.

Table I.

Temperature.	Vapour Pressure.	Weight of Naphthalene required to saturate 100 cb. ft. of Gas.
° F.	Mm.	Grains.
32	0·022	6·0
41	0·034	9·8
50	0·047	14·1
59	0·062	19·0
68	0·080	24·6
77	0·103	30·9
86	0·135	39·5
95	0·21	57·5
104	0·32	83·5
113	0·51	132·0
122	0·81	208·0
131	1·26	317·0
140	1·83	482·0
149	2·65	723·0
158	3·95	1050·0
167	5·43	1430·0
176	7·4	1898·0
185	9·8	2426·0
194	12·6	3035·0
203	15·5	3727·0
212	18·5	4420·0

It will be noticed how different the true maximum vapour pressure at 59° F., namely, ·058 mm. is from that generally accepted for 60·8° F., namely, 2 mm. And how the corresponding weight of naphthalene required to saturate 1,000 cubic feet of gas at 59° F., namely, 178 grains, agrees with the weight extracted from the same volume of coal gas by Dr. Bueb, by his washing process, namely, 200 grains at a temperature of about 60° F., 200 grains being the saturation value at 62° F. for an indifferent gas.

I now determined the amount of naphthalene required to saturate the same volume of coal gas, carefully freed from any trace of naphthalene, at the same temperatures; and obtained results identical with those for the indifferent gases, hence it would appear that the vapours of the heavier hydrocarbons present in coal gas exert no influence on the power of the gas for saturating itself with the naphthalene.

To confirm this, experiments were made with air containing the vapours of the most powerful solvents for naphthalene, namely, ether, carbon disulphide, chloroform, benzene, and ethyl alcohol; and others also with acetylene and marsh gas to see whether the hydrocarbon gases themselves were quite without influence. The following table (Table II.) gives the values at 140° F. obtained for the various gases, and for comparison's sake those at the same temperature found for air, hydrogen, and coal gas.

Table II.

Gas employed.	Temp. 140° F. Weight of Naphthalene required to saturate 100 cb. ft. of the Gas.
	Grains.
Air	537
Hydrogen	537
Coal gas	538
Acetylene	538
Marsh gas	536
Air saturated at 60° F. with ether	548
,, ,, carbon disulphide.	537
,, ,, chloroform	512
,, ,, benzene	547
,, ,, ethyl alcohol	549

From this it would appear that the presence of solvent vapours in quantities insufficient to saturate the gas exerts no influence on the amount of naphthalene vapour that the gas can hold in diffusion, for the slight differences observed are probably due entirely to the difficulty of calculating the exact volume of gas employed owing to the unequal absorption of the vapours by the water of the aspirator in which the gases were measured.

The gas was next saturated at the temperature of the experiment, namely, 140° F. with the vapours of benzene and ethyl alcohol, and passed over the naphthalene; so great was the increase in the amount of that body carried over that the cooler parts of the apparatus were almost immediately blocked up and the experiments stopped.

Having thus established the fact that coal gas could not, unless saturated with a solvent vapour, hold in diffusion more naphthalene vapour than the same volume of an "indifferent gas," and having found that the gas employed was not saturated with benzene or petroleum oil, it was clear that the amount of naphthalene actually present in the gas could be deduced by observing how much additional naphthalene was required to saturate a hundred cubic feet at a given temperature.

To diminish the errors of experiment as far as possible, the amount of naphthalene required to saturate the same volume of gas from which all traces of naphthalene had been first removed, was also simultaneously determined. The results obtained are shown in Table III.

Table III.

Gas employed.	Temp. 113° F. Weight of Naphthalene required to saturate 100 cb. ft. of the Gas.
	Grains.
Ordinary sample of coal gas	153
Gas from which all naphthalene had been removed by passage through tubes immersed in a freezing mixture.	152
Gas from which all naphthalene had been removed by passage through a freezing mixture, and then through bulbs containing hot sulphuric acid.	152

From this it would appear that the coal gas, which was supposed to be very nearly saturated with naphthalene and which actually did form bad deposits, in reality contained none of the vapour of that body. This was confirmed by a long series of similar experiments and also by the following fresh ones suggested by Professor F. D. Brown. A little air saturated with naphthalene at the atmospheric temperature by passage through a tube containing some of the powdered solid was passed through V tubes immersed in a freezing mixture of ice and salt; at this temperature practically the whole of the naphthalene was deposited out in the V tubes, the detection of a very small amount indeed being thus possible. A considerable volume of coal gas was now similarly treated, but no trace of naphthalene was to be seen, only a little benzene and oily substances. This

I took at the time to be conclusive proof of itself that the gas contained absolutely no naphthalene; in truth it did not quite do this, it only showed that the possible amount present dissolved in the benzene was very small. However taken with the previous series of experiments, it was fairly conclusive. Subsequently Prof. Brown experimented with large volumes of the gas at the works, but whether before it entered the mains or after I cannot say. He was unable to detect anything but a trace of naphthalene present, and yet the trouble from the deposits was very great in the pipes of that company.

The truth of these observations I have since confirmed in the following way:—Naphthalene in solution in benzene, ether, alcohol, or any oil may be detected by first converting it into nitro-naphthalene by digestion with acetic and nitric acids and then oxidising the nitro-naphthalene with chromic acid—on cooling crystals of nitro-phthalic acid separate out—benzene gives no such product. Hence by taking some of the mixed liquid obtained by passing the coal gas through V tubes immersed in a freezing mixture, acting on it with acetic and nitric acids and oxidising the product with chromic acid mixture, crystals of nitro-phthalic acid will be formed if any naphthalene be present in the gas; but no naphthalene could be found in the gas in this way.

It would thus appear that we have the anomaly of coal gas free from naphthalene, yet forming deposits of that body during its passage through the pipes. The only possible explanation is that under certain conditions naphthalene is formed in the pipes themselves, and condenses and deposits itself there. That this is always the case where naphthalene deposits are formed I do not wish for a moment to assert; I only say that some such explanation is necessary to account for gas, proved to contain no naphthalene, forming naphthalene deposits on its way through the pipes.

Moreover, it would satisfactorily account for the formation of deposits by gas containing less naphthalene than is sufficient to saturate it. The usual explanation—fall of temperature—will hardly suffice, for an ordinary sample of gas purposely saturated with naphthalene at 60° F. will form no deposit of the solid if cooled to 15° F., the amount of benzene and hydrocarbon oils simultaneously condensed being sufficient to dissolve the whole of the naphthalene deposited. I have repeatedly tried this with the London gas and found that it is so. Assuming the theory of the spontaneous formation and subsequent deposition of naphthalene in the pipes themselves to be correct the question arises as to the way in which this is brought about. Is there some unstable substance present in the gas or formed under certain conditions in the pipes, which, under new conditions, spontaneously decomposes or combines with the hydrocarbons to form naphthalene? This question I am unable at present to definitely answer. My belief—and I have certain grounds for this belief—is that such an unstable body is either already present in the gas or is formed by the gas as it traverses the mains and under suitable conditions combines with the hydrocarbon gases to form naphthalene. The temperatures at which this occurs being above that at which benzene or petroleum oil vapour is condensed out of the gas, the naphthalene will deposit itself as soon as the amount of naphthalene present begins to exceed that required for saturation.

In brief summary let me draw attention to the following points:—

I. The very small amount of naphthalene required to saturate a given volume of gas at the ordinary temperature.

II. The fact that the presence of benzene or hydrocarbon oils in quantities insufficient to saturate the gas does not affect the amount of naphthalene that a given volume of gas can hold in diffusion.

III. The easy detection of naphthalene in gas by the formation of nitro-phthalic acid.

IV. That gas containing less naphthalene as it leaves the gasometer than is sufficient to saturate it at the lowest temperature to which it is likely to be subjected, or to form solid deposits when account is taken of the solvent power of the benzene, &c. simultaneously condensed, will yet form such solid deposits in the distributing pipes.

V. The consequent necessity of looking elsewhere than to changes of temperature for the explanation of the formation of the deposits.

Discussion.

Dr. F. Clowes said that he was glad to have been asked to say a few words, as he considered himself mainly responsible for the paper having been brought forward. It was a paper of a somewhat startling character. The statement had been generally accepted, although he knew of no reason for its acceptation, that naphthalene vapour could be carried over in larger quantities by coal gas than by ordinary gases. Mr. Allen had given figures which proved that such a notion had no foundation in fact. He could not see how anyone could avoid the conclusion drawn by Mr. Allen that air and hydrogen carried over the vapour of naphthalene to the same extent as coal gas. The work brought before them that evening had been been carried out by a young chemist, who was fitted for his task by possessing great skill in manipulation and the greatest integrity. Those who remembered the character of Prof. F. D. Brown's work when he was in London and at Oxford would know that any work done in his laboratory, under his own direction, could be vouched for as regards its accuracy. Prof. Brown had taken an interest in the subject placed before them to-night, and he undoubtedly felt himself responsible for its general trustworthiness. He himself had not made any experiments on this subject, but he could not feel any doubt as to the spirit in which the experiments which Mr. Allen had brought forward should be received by the Society. He hoped, however, that some of those present would repeat this work, and he felt no doubt that they would arrive at a similar conclusion to that stated in the paper. He could not help feeling that Mr. Allen was correct in stating that naphthalene was, under certain conditions, formed in gas in which it did not pre-exist. He hoped that gentlemen connected with the gas industry would interest themselves in the fresh aspect now given to this subject, as they would all like to know more about the origin of this substance, and about the chemical conditions under which it was formed. He also hoped that Mr. Allen, who had shown himself a very ingenious experimentalist, would be induced to devote some of his leisure to further prosecution of this investigation, and would at a future meeting of the Society bring forward further information on this extremely important subject.

Mr. T. S. Lacey said that it seemed to him that the chief item of interest in the paper to gas manufacturers was that it appeared to give a method of estimating the amount of naphthalene in coal gas. The question of naphthalene deposits was a very difficult one; and what all who were interested in the gas industry would like to know, before attempting to form any opinion as to the causes of its deposit, was how to measure the amount of naphthalene in coal gas. It was important to be able to estimate the amount of naphthalene present before they could form even guesses as to the cause of its deposit or to make estimates of what was going on in the pipes. There was one circumstance in the distribution of coal gas that differed slightly from the experiments which Mr. Allen had made, and that was the presence of water vapour. He thought it quite possible that the presence of water vapour in the gas might considerably alter its behaviour; he assumed that the experiments had all been made with dry gases. It was well known that if coal gas were passed through a water-meter it was very frequently the case that deposits of naphthalene were found immediately afterwards. That would certainly appear to show that saturation of the gas with water vapour had some effect in causing a rapid deposit of the naphthalene; and he should be inclined to judge that experiments made on dry gases would not form a guide as to what might occur with gas charged with water vapour.

Mr. J. W. Helps said that the theory which Mr. Allen had brought forward was one which did not coincide with their general ideas, nor did it coincide with their experience. He had always held the idea that in works where naphthalene was produced and no special steps taken to remove it, gas did leave the works practically saturated with naphthalene at a particular temperature.

He should like to ask Mr. Allen how his theory would coincide with one or two difficulties and facts he had noticed. For instance, in the case where gas was passed at a temperature of 50° or 60° into the holders on a hot summer's day saturated with naphthalene. In the holders its temperature was very considerably raised, and the gas left the works unsaturated, but at a much higher temperature than it went into the holders. He found from experience that deposits were in such cases much greater than they were in winter. If the gas did not pick up the naphthalene at a high temperature and drop it at a lower one, he would like to know where it came from. His own works at Croydon were very near those managed by his brother; similar coal was used and very nearly the same treatment employed; but in the town supplied by his brother's works, *viz.* Redhill, naphthalene had never been seen, while in his own district it was seen far too frequently. Naphthalene was generally known as the gas manager's bugbear, and if Mr. Allen or some other chemist could find the means of ridding them of it, he would earn the thanks of the whole gas industry. It did seem as though gas managers had let the matter slide in a very absurd way, and he hoped Mr. Allen and others would go carefully into the matter, and try to rid them of it. As to the two gasworks of Redhill and Croydon to which he had referred, while otherwise the method of treatment was similar, in the case of Redhill, where no naphthalene was found, the gas and tar passed together through a long length of main, and it would appear as though the naphthalene were washed out.

Mr. Watson Smith wished to know whether Mr. Allen, in examining the gas, had also examined the coal tar deposited from the crude gas, because he had on several occasions examined both, and when the gas was charged with naphthalene he had always found that the coal tar was rich in naphthalene too. He knew that gas manufacturers who used Wigan cannel coal could obtain a gas free from naphthalene, and the coal tar also would be free from it, but that coal tar would contain anthracene so contaminated with solid paraffins that it would be either of much reduced value or valueless for the alizarin manufacturer. Hence gas manufacturers were obliged to use the cannel with caution and mix it judiciously with other less bituminous coals, so as to obtain a saleable coal tar. These less bituminous coals gave no trouble as regards solid paraffins, but naphthalene would now appear both in tar and gas. It was a choice of either of two evils. There was some relation between the coal, the carbonising temperature of the retorts, and the tar and gas produced. Naphthalene was notoriously a pyrogenetic substance, *i.e.*, it was *formed* in destructive distillations at temperatures at which many other bodies or groups were decomposed, being built up, as it were, from their remains. How, on the other hand, such remains were going to be preserved intact through scrubbers and even in the cool mains until certain districts or service pipes were reached, then electing to submit to union to form naphthalene, he could not comprehend. (See this Journal, 1889, 949—954.) He wished also to know how Mr. Allen managed to completely free coal gas from naphthalene without the benzene vapours being also removed. If the benzene were eliminated he should hardly consider the remainder coal gas, as it would be of non-illuminating character. But with benzene perforce also some toluene and other volatile impurities would be removed, for he knew as a fact that such condensed benzoles were very impure and crude.

Mr. W. J. A. Butterfield said that he was very interested and gratified to learn that Mr. Allen had weighed the quantity of naphthalene taken up by all gases and had found that the quantities were the same, as the statement had been made that coal gas containing large quantities of other hydrocarbons could not contain naphthalene to saturation. Perhaps Mr. Allen could give him some information as to the point in the distributing system or on the works at which he took the gas in which he found no naphthalene. He had stated, in support of his figures for the amount of naphthalene that gas could contain at various temperatures, that whereas he had found about 20 grains at 59° F., Dr. Bueb had by a washing process on a manufacturing scale actually removed the same amount. That seemed to prove Mr. Allen's figures. He believed that Dr. Bueb was working at a point after the condensers. At that point, therefore, the coal gas was saturated with naphthalene. Could Mr. Allen give them any idea where the naphthalene got out of the gas, if he found the gas free from naphthalene at some other point? It seemed to him that freezing the gas, as Mr. Allen had done, and thus getting a liquid condensation product would not enable one to say conclusively that thereafter the gas could contain no naphthalene, and that therefore no deposits could take place in the main subsequently, because, although in the first instance one obtained a liquid condensation product in which naphthalene was dissolved, after a time one had gas passing over which was not fully saturated with the liquid hydrocarbons. This gas would therefore take up the latter and leave the naphthalene behind. Manufacturers had to contend with the difficulty that at one time gas was more nearly saturated with hydrocarbons which were readily condensable as liquids, such as benzene, and at another time the gas produced would be far more nearly saturated with naphthalene and not so nearly saturated with these liquids. Under the latter conditions he thought it conceivable that naphthalene might be deposited in parts of the mains where there was not enough liquid deposited to carry it into the siphons. He had heard it stated that naphthalene deposits in the mains diminished the illuminating power of the gas. It seemed to him improbable that the small quantity of naphthalene in question would have much effect either way, for even if the gas were saturated it would only make a difference of about 0·15 candle. If the gas passed through mains containing a deposit of naphthalene one would expect it to arrive at the burners saturated. That a reduction of illuminating power occurred was, however, conceivable on the assumption that the naphthalene exerted a scrubbing action on the gas and removed some of the illuminating hydrocarbons, such as benzene. He had attempted to make some experiments in that direction that afternoon, but they were nullified by the variations in the illuminative value of the gas supply at the time.

Mr. Otto Hehner said that he could not help being struck with the fact that, whilst the author's experiments were evidently very carefully performed, there appeared to be no connection between them and the deposition of naphthalene in the pipes. No adequate proof had been forthcoming that the same gas which the author had shown to be free from naphthalene was at the same time depositing that hydrocarbon. The deposition of naphthalene in the pipes from the gas in question seemed to him to be based upon mere hearsay evidence. Important conclusions, such as those arrived at by Mr. Allen, required strict scientific proof, but such proof was entirely wanting. Knowing, as they did, the circumstances under which naphthalene was known to be formed, no chemist, he thought, would accept, without very convincing data, the suggestion that naphthalene could form in the cold whilst the gas was being conducted through the pipes. Some of the author's experiments seemed to him to be somewhat superfluous, for it might have been accepted that the tension of naphthalene vapour would be the same when mixed with air or other indifferent gases, the temperature being the same. No doubt Mr. Allen, in ordinary gas analysis, readily accepted the fact that the tension of water vapour, for instance, was the same for equal temperatures in hydrogen, nitrogen, or carbon dioxide. He considered Mr. Allen to have done valuable work in supplying them with a very simple means of detecting naphthalene in gas, but it should not be forgotten that similar work had lately been brought before another Section of the Society, in which the facility with which naphthalene combined with picric acid had been utilised as a basis of a method.

Mr. Forbes Carpenter hoped that Mr. Leather, of Burnley, who had had the most experience in this country in the processes for the removal of naphthalene from coal gas by washing with heavy oils saturated with light hydrocarbons, would, if present, contribute some information as to what he had done in that direction. It was not only in winter time that the deposit of naphthalene was troublesome; and he could quite understand how in certain conditions of atmosphere and temperature, with alternation of periods of gas supply, saturated and unsaturated, *quâ* the lighter hydrocarbons and *quâ* naphthalene, a previous deposit of naphthalene might be moved so as to block a pipe. If, as Mr. Hehner had said, there was a deposit of naphthalene in an old pipe, it might be moved so as to choke up a local service. If anyone present could say whether Mr. Leather had had a perfectly clean main to experiment on in a new district with washed coal gas, that would be a help towards understanding the paper from their point of view, and could be claimed by Mr. Allen in support of his theory.

Mr. H. Leicester Greville said that the tendency of naphthalene to accumulate in bends and angles of pipes was probably due to friction, though moisture probably had influence on the formation of deposits. The statement in the paper, that gas normally left the works free from naphthalene vapour, would require corroboration, considering that the presence of naphthalene in quantity was so evident at all points of the manufacture. That naphthalene—a substance always regarded as essentially a product of high temperature—could be absolutely produced in the gas at the ordinary temperature of the air, would also require a large amount of confirmation. If naphthalene could possibly be permanently retained in the gas instead of being deposited, it would be a great boon to gas companies on the ground of its great value as an illuminant.

Mr. R. W. Allen, in reply, said he had treated the matter purely from the scientific standpoint. For the statements he had made about the deposits in the mains he had the authority of Prof. F. D. Brown, Chairman of Directors of the local company, and also of the Chief Engineer. The work extended over a year, and that to a very great extent eliminated any variations that otherwise might have been supposed to exist in the amounts of naphthalene in the gas as it issued from the mains. Reference had been made to Mr. Leather. In a paper on "Naphthalene Deposits" read by him before the Gas Institute (see Journal of Gas Lighting, **73**, 1734) he had stated that gas which contained at the most only 14 grains of naphthalene per 100 cb. ft.—an amount insufficient, as he (Mr. Allen) had shown, to saturate the gas at a very much lower temperature than it was likely to be subjected to in the mains—yet deposited naphthalene. In reply to Mr. Watson Smith he might say that he made no effort to prevent the benzene being removed along with the naphthalene, but experimented afterwards with gas from which the naphthalene was carefully removed and the benzene reintroduced in a quantity insufficient to saturate it at the temperature of the bath. Mr. Hehner had asked why he did not convert the frozen-out naphthalene into picrate of naphthalene. It was because the benzene would also have been converted into solid picrate, whereas by first converting the mixture into nitrobenzene and nitronaphthalene and oxidising this with chromic acid, the nitronaphthalene formed nitrophthalic acid, which separated out on cooling, whereas the nitrobenzene did not give a similar compound. Mr. Hehner had also remarked that he (Mr. Allen) had worked with much too small a volume of gas to ensure accuracy. It was true that the volume of gas was comparatively small, but he had the utmost confidence that his weighings were correct in every instance to, say, a tenth of a milligramme, and the total errors were probably under 1 per cent. The weight carried over by the volume of gas he worked with was 0·0056 grm. at the lowest temperature employed, 8·0° C.—a perfectly appreciable quantity, capable of being weighed accurately, especially when account was taken of the great care he took to prevent the U-tubes of naphthalene absorbing moisture. The large number of experiments made at each temperature also tended to reduce errors to a minimum. He also stated that he never had had a doubt but that the weight of naphthalene required to saturate hydrogen, coal gas, carbon dioxide, air, &c., was exactly the same. This is not the point; the doubt—one held by many men of note—was whether all these gases would saturate themselves completely with naphthalene with equal rapidity, and what the speed of complete saturation was. In reply to Mr. Helps' question he regretted to say he could not throw any light on the point, as he was not a practical man. But it seemed to him that the statement made by Mr. Helps, that a hot summer's day increased the deposition of naphthalene, rather supported his own view. A hot day was just the time when the benzene would not condense out, and the conditions then might be those necessary for the formation of the naphthalene in the gas. Other references had been made to the way in which abrupt bends seemed to facilitate the formation of naphthalene. On the hypothesis that the gas and pipes were mutually electrified by the friction of the gas passing along, it was conceivable that the naphthalene molecules, being the heaviest ones present in the gas, might be attracted by the oppositely charged iron of the pipes, and held fast at the bends, where a sort of back eddy might be produced in the gas. He had endeavoured to test this by highly electrifying the gas as it passed along a tube presenting many obstacles to its passage, but could obtain no result, although both high alternating currents and large static charges were employed to electrify the gas.

The Chairman remarked that the subject was one of the highest importance to coal gas makers, and it only remained for him as Chairman, in tendering to Mr. Allen the thanks of the meeting, to endorse the suggestion which had been made by Prof. Clowes, that the author should continue his investigations and endeavour to obtain some experimental proof of the revolutionary view of the genesis of naphthalene in coal gas which had been submitted to them that evening.

THE IODINE VALUE OF OILS.

BY ARTHUR MARSHALL, F.C.S., A.I.C.

Although there is an extensive literature on the subject of the determination of the iodine value of oils, yet the exact nature of the reactions taking place in the various methods that have been proposed is still somewhat obscure. A great advance in the practical carrying out of the determination has been made by Wijs (Berichte, 1898, **31**, 750), who has proposed to employ a solution of iodine monochloride in acetic acid in the place of Hübl's solution of iodine and mercury bichloride in alcohol. Wijs's solution possesses the two great advantages that it is rapid in its action, and that it alters very little in titre. Lewkowitsch has shown that it gives the same iodine values as Hübl's solution, if the latter be properly applied (Analyst, 1899, 257).

In opposition to Ephraim (Zeit. angew. Chem. 1895, 254), and his own previous opinion (Zeit. anal. Chem. 1898, 277), Wijs considers that with Hübl's solution, iodine monochloride is not added on to the unsaturated acid radicles directly, but that the iodine chloride formed reacts with water, producing hypoiodous acid—

$$ICl + H_2O = HIO + HCl \qquad (1),$$

and that this is added on to the oleic acid or other unsaturated substance—

$$C_{17}H_{33}CO_2H + HIO = HOIC_{17}H_{33}CO_2H \qquad (2).$$

That this addition product then reacts with the hydrogen chloride formed according to the previous equation (1).

$$HOIC_{17}H_{33}CO_2H + HCl = ClIC_{17}H_{33}CO_2H + H_2O \quad (3).$$

In support of this view Wijs states that, if hydrogen chloride be added to Hübl's solution, it retards its action. Hydrogen chloride, he says, would increase the amount of iodine chloride in the solution, but would, to some extent, prevent the formation of hypoiodous acid, as shown in equation (1). It is true that it has this effect, for, if iodine and mercuric chloride be dissolved in alcohol in suitable proportions, and if then hydrochloric acid be added, the brown colour of iodine disappears and only the yellow colour of iodine chloride remains. It is, however, practically impossible to predict what effect the hydrogen chloride would have upon the reaction between iodine chloride and oleic acid, &c. Wijs then proceeds to show that freshly prepared hypoiodous acid is absorbed to the full extent by arachis oil practically instantaneously. He, however, omits to show that hypoiodous acid is present in Hübl's solution in any quantity. As it is an extremely unstable substance, there is probably never more than a very small quantity present. As soon as it is formed it must be converted into iodic acid. In order to prove that hypoiodous acid does not necessarily take any part in the action, it is only requisite to show that the addition takes place equally well, where there cannot possibly be any present. I prepared a solution of iodine monochloride in dry carbon tetrachloride. The solution was kept over fused calcium chloride to prevent the presence of any trace of moisture. The iodine values of some samples of oil were then determined by means of this solution, and also by means of a solution of iodine chloride in glacial acetic acid.

Time.	Oil.	Iodine Value.	
		ICl in CH_3COOH.	ICl in CCl_4.
Mins.			
10	Rape	103·0	103·8
10	,,	102·0	103·0
10	,,	102·0	103·2
10	,,	102·2	104·2
½	,,	..	102·8
30	,,	..	104·8
10	Linseed	178·0	178·0
10	,,	178·5	178·9
1	,,	..	178·0

It is evident from these figures that iodine chloride acts in exactly the same way in the absence of water as it does in the presence of it. If anything, it acts more energetically, for Wijs found that the action on linseed oil of a solution in acetic acid containing 5 per cent. water took about seven minutes, whereas, with carbon tetrachloride, it is apparently completed in one minute. I doubt, however, whether the addition ceases immediately potassium iodine solution is added. From the colour of the carbon tetrachloride beneath the aqueous layer, it is evident that it contains no free iodine, but a considerable quantity of iodine chloride, which no doubt acts further upon the oil whilst the titration is proceeding.

The colour of the carbon tetrachloride solution gives considerable information of what is proceeding in it, for, if it contain free iodine, it becomes brilliant purple, whereas iodine chloride colours it a pale brownish yellow. When substitution takes place, iodine is at once set free in accordance with the equations—

$$RH + ICl = RCl + HI \text{; and}$$
$$HI + ICl = HCl + 2I.$$

If, therefore, a considerable amount of substitution take place, it will become apparent to the eye. There is, however, a more delicate test for substitution, viz., the acidity of the solution. This can be very conveniently estimated iodometrically. After the iodine has been titrated with thiosulphate in the usual way, potassium iodate is added to the solution. When this is done every equivalent of acid present liberates an equivalent of iodine—

$$6HCl + KIO_3 + 5KI = 6KCl + 3H_2O + 6I \quad (1.)$$

The iodine is then titrated with thiosulphate. This was carried out with the oils above, but in no case did potassium iodate bring back the colour when proper precautions had been taken to exclude moisture.

A mere trace of water in the carbon tetrachloride solution rapidly produces the change—

$$3ICl + 3H_2O = HIO_3 + 3HCl + 2HI \quad (2.)$$

The hydriodic acid then reacts with more iodine chloride liberating iodine, as becomes evident to the eye—

$$2HI + 2ICl = 2HCl + 2I \quad (3.)$$

The solution at the same time becomes cloudy in consequence of the separation of iodic acid, which is insoluble in carbon tetrachloride. If the solution be dried over calcium chloride, it becomes clear once more.

From the above equations it will be seen that for every molecule of iodic acid formed there are also six equivalents of acid, which, it might be supposed, should be sufficient to convert the iodic acid into iodine again in accordance with equation (1). This action is, however, only complete when there is present a considerable excess of both iodide and iodate.

Wijs has stated that with Hübl's reagent the oil, after absorbing iodine and chlorine, splits off a certain amount of hydrogen chloride (Zeit. anal. Chem. 1898, 277), thus:—

$$CO_2HC_{17}H_{33}ICl = CO_2HC_{17}H_{32}I + HCl.$$

and the experiments he cites makes this appear very probable.

From what I have said above, it is evident that this does not take place in the carbon tetrachloride solution.

Hübl, in his original paper (Dingler's polyt. Journ. 1884, 281), mentions that with his reagent he obtained a product having the composition $C_{18}H_{34}O_2ICl$. Further evidence that iodine and chlorine are added on in these simple atomic proportions is given by the colour of the carbon tetrachloride solution, which remains unchanged during the addition. If, however, the mixture be allowed to remain sufficiently long for substitution to take place, the purple colour can be seen to develop.

I think that the above experiments make it clear that the so-called "iodine absorption" consists simply in the addition of iodine chloride directly to the double linking of the unsaturated acid, and that no other reaction takes place to any appreciable extent.

That the absorption is complete in the case of the ordinary unsaturated fatty acids is proved by the following facts:—

(1) That iodine absorptions demanded by theory have been obtained in the case of oleic, isoleic, brassidic, and isoerucic acid.

(2) That the same values are obtained with Hübl's solution, with Wijs's solution, with a solution of iodine chloride in alcohol (Ephraim, Zeit. angew. Chem. 1895, 254), and with iodine chloride in carbon tetrachloride.

(3) That in the case of Wijs's solution the iodine value obtained is unaffected by the excess of the reagent employed (Lewkowitsch, Analyst, 1900, 34).

McIlhiney in a recent paper having endeavoured to resuscitate the "bromine value" (Journ. Amer. Chem. Soc. 1899, 1084), I thought that it was of interest to determine the bromine value of the same sample of linseed oil, with which the above experiments were made. I made a solution of bromine in carbon tetrachloride of about N/5 strength (McIlhiney recommends N/3 strength) and proceeded in the usual way. Calculating the result as iodine, I obtained the following results:—In 10 mins., 140·2 per cent. of iodine; in 20 mins., 141·3 per cent. of iodine. The mean of the results with iodine chloride was 178·3. It is therefore evident that under these conditions the absorption is far from complete. There was no evidence of substitution. McIlhiney also obtained values lower than those given by Hübl's method. He states that substitution takes place, and that the addition is instantaneous.

I also tried the effect of iodine bromine. Iodine and bromine were dissolved in carbon tetrachloride and the operations carried out as usual. In ten minutes I obtained an iodine absorption of 167·0, *i.e.* 11·3 below the true value. It is evident from these experiments, that iodine chloride must not be replaced by either bromine or bromine iodide.

Both Wijs and Lewkowitsch have made statements as to the effect of time on the iodine chloride-acetic acid solution. Wijs (Berichte, 1898, 752) stated that a solution in 95 per cent. acetic acid lost only 0·3 per cent. of its titre in the first 96 hours. Lewkowitsch with acid of the same strength found a decrease of 4 per cent. in 80 hours, but with 99 per cent. acetic acid he found the solution unaltered after two months.

I have not found the titre to keep as constant as is stated by Lewkowitsch, although it does not vary sufficiently rapidly to cause any inconvenience. Even when new the titre does not alter so rapidly as to prevent use at once, but after months it still continues to decrease slowly.

In order to investigate this point I further purified my acid by recrystallising twice, and pouring off part of the acid each time. I then placed 25 c.c. in a dry stoppered bottle with 25 c.c. of my solution of iodine chloride in carbon tetrachloride. After 10 minutes potassium iodide was added, and the iodine was titrated in the usual way. It required only 90·6 c.c. of $\frac{N}{20}$ thiosulphate, whereas the iodine chloride used should have required 95·75 c.c. There was an absorption, therefore, equal to 5·38 per cent. of the original amount in 10 minutes. This experiment was repeated, allowing the action to proceed for 12 hours. There was then an absorption of 22·9 per cent. of the halogen present. In a similar experiment, in which a few drops of water were added, there was an absorption of 24·1 per cent. in 12 hours. In all these cases the bottles were kept in a dark cupboard during the action.

It is well known that chlorine acts upon acetic acid in the presence of iodine quite readily if the liquid be warmed, producing chloracetic acid. From the above experiments it is evident that the action takes place also in the cold, though slowly. This substitution must gradually weaken the solution under ordinary circumstances, though it is evident from Lewkowitsch's statement, that this does not always take place. In any case the action is too slow to seriously interfere with the value of the method.

The weakening of the solution must depend to a large extent upon the amount of chloracetic acid formed in the preparation of the reagent, and this may account for the differences observed by different observers.

In conclusion I wish to say that I do not propose to substitute carbon tetrachloride solution in the place of that proposed by Wijs for general use, but in some cases it will be found more useful. On the whole, acetic acid is more convenient to work with than carbon tetrachloride.

DISCUSSION.

Dr. LEWKOWITSCH said that he did not think that the paper added much to their knowledge. The literature on the subject of iodine values was so startling in its proportions that he had himself stated in that room that he congratulated any chemist who did not add to it. It was true he had himself lately committed a few ideas on the subject to print, but his excuse was that Wijs' beautiful method had appeared meanwhile, and this method had, in his opinion, completely cleared the air. Stability of Wijs' solution was demonstrated for at least five months, and no one could complain if alterations should occur after that time.

Mr. P. KAY asked what was the best way of preparing Wijs' solution. The ordinary method of finding the strength by titration was inconvenient, and he would like to know if there were any physical sign to guide one as to when the combination with chlorine was complete.

Mr. A. MARSHALL, replying to the last speaker first, said that it was quite easy to stop the passage of the chlorine at the correct time without titrating. The solution of iodine in acetic acid was dark brown in colour, whereas the solution of iodine monochloride was light yellow. The chlorine should therefore be passed in until this change of colour took place. The final change took place at quite a sharp point. He could not agree with Dr. Lewkowitsch as to the want of necessity for this investigation. No doubt Wijs' method for the determination of the iodine value would be generally adopted by chemists. There would consequently be a tendency to adopt Wijs' theory as to the nature of the reactions taking place. He thought it was a matter of importance that the exact meaning of the term "iodine value" should be known with certainty.

As to the keeping properties of Wijs' solution, he thought it was remarkable that the solution prepared by Dr. Lewkowitsch should retain its strength so much better than those prepared by Wijs or himself. He suggested that perhaps Dr. Lewkowitsch's solution had been prepared on a hot day, and that consequently a great part of the acetic acid had been converted into chloracetic acid during the preparation of the solution.

Dr. LEWKOWITSCH remarked that, on the contrary, it was a very cold day.

Mr. MARSHALL, continuing, said that possibly chlorination of the acetic acid would not occur if the solution were quite free from water. In consequence of the hygroscopic character of glacial acetic acid, it could not be kept anhydrous unless access of damp air were prevented.

Dr. LEWKOWITSCH said that perhaps Mr. Kay would like to know that a still more convenient way of preparing the solution was to accurately weigh off quantities of iodine and iodine trichloride.

Mr. MARSHALL added that iodine monochloride itself could be prepared without difficulty in a sufficiently pure state, and might even be bought so. He had prepared with it the solution of carbon tetrachloride with which the above experiments were performed.

The CHAIRMAN said that he thought that the Committee were justified in extending a hearing to papers of this description, which contained carefully recorded statements of actual results obtained in painstaking work.

Manchester Section.

Meeting held on Friday, March 2nd, 1900.

DR. J. GROSSMANN IN THE CHAIR.

THE NATURE OF INDIA-RUBBER.

BY DR. CARL OTTO WEBER.

THE great experimental difficulties encountered in the investigation of colloidal bodies are responsible for the slow development of the chemistry of these substances. Their physical constants are highly indeterminate; and instead of melting points, boiling points and solubility, they simply exhibit a gradual merging of one state into another. Thus the characterisation of the colloids and their derivatives, and their isolation and purification from reaction mixtures is generally a matter of the very greatest difficulty.

These difficulties are met with in a pronounced degree in the chemical investigation of india-rubber, which forms the most important raw material of a large and, though perhaps only in a mechanical sense, highly developed industry. Indeed, not even is the empirical formula of india-rubber established with certainty; the question as to whether it should be regarded as a practically uniform substance, a hydrocarbon, an oxygenated product, a mixture of isomeric or polymeric compounds still being open to discussion. It is thus not surprising that we should be entirely in the dark respecting the question of the cause and nature of the difference exhibited by the numerous varieties of india-rubber obtained from plants belonging to very different botanical families.

If unworked, crude rubber* be treated with chloroform or carbon bisulphide, partial solution gradually takes place; and it was very early observed that, by this treatment, the rubber was separated into two parts, the one being soluble, the other insoluble, and presenting a peculiar reticulated appearance under the microscope. Very divergent statements are to be found respecting the relative proportions in which these two constituents occur. In one case the amount of the insoluble body is given as varying from 30 to 70 per cent. (Ladenburg's Handwörterbuch, Bd. V., 479), but on the other hand Gladstone and Hibbert (Jour. Chem. Soc. Trans. 1888, 679) found in Para rubber 4 per cent. only. For this reason I have thought it desirable to re-examine this point.

Gladstone and Hibbert suggested that the insoluble "modification" of india-rubber was produced during the drying of the juice, because they observed that by heating the soluble part it was more or less changed, and less susceptible of subsequent solution. They found this heat effect to increase as the temperature was raised and also with the length of time during which the heat continued. For this reason I employed for my experiments, not washed and dried rubber, but the thin films into which the inner parts of a crude loaf of rubber can be split. These films contain a considerable proportion of water which was got rid of by often repeated treatment with acetone. During this operation the films, which were white at first, assumed

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* All the following statements refer to Para rubber, unless som other variety is expressly stated.

the colour of light leather. They were at last completely freed from all resinous and oily matter by extraction with acetone in a Soxhlet apparatus, and then, without drying, immediately transferred to chloroform. After a little over a fortnight, during which time the bottle containing the substance was frequently shaken, a solution was obtained consisting of two layers. The lower of these was a bright, clear solution, while the upper consisted of small clots or shreds of the insoluble part of the rubber. This upper layer was removed and a number of times treated with fresh quantities of chloroform, two or three days intervening between each of these changes of solvent. The quantity of the insoluble substance so obtained amounted to very nearly 6·5 per cent. of the dry weight of the india-rubber used. It swells to an enormous volume in such solvents as chloroform, ether, petroleum spirit, and carbon bisulphide, but even on long standing in these no appreciable amount passes into solution. The fibrous structure exhibited under the microscope by thin sections of this insoluble substance is not, as has been suggested, comparable with that of certain elementary animal or vegetable tissues, but appears to be the result of a peculiar process of shrinking of the drying substance, producing a reticulated appearance closely resembling some of the artificial colloidal structures described in Bütschli's[*] recent book on this subject.

This insoluble body, even when diluted by solvents, is entirely free from stickiness, and when dry, in the form of larger pieces, shows a remarkable degree of toughness, but only very moderate distensibility. The analysis of this body gave the following figures :—

0·1440 grm. substance.....	0·3174 grm. CO_2, 0·1407 grm. H_2O.
Calculated for $C_{30}H_{64}O_{10}$..	C 61·64, H 10·95.
Found....................	C 61·13, H 10·85.

The analysis of the soluble part without any further purification gave this result :—

0·1151 grm. substance.....	0·3651 grm. CO_2, 0·1201 grm. H_2O.
Calculated for $C_{10}H_{16}$.....	C 88·23, H 11·76.
Found....................	C 86·51, H 11·59.

It will be seen that the body representing the soluble part contains nearly 2 per cent. of oxygen, but I shall presently show that on further purification this percentage tends to approach a vanishing point. I desire, however, to call already in this case attention to the fact that the ratio C : H in the above analysis very accurately corresponds to a theoretical ratio 10:16.

The above analysis already enables us to recognise as erroneous the statement,[†] originally, I believe, due to Payen, and ever since quietly accepted by every writer upon the subject, that the soluble and the insoluble part of india-rubber are identical in their composition. Our data further allow us to correct the exaggerated figures quoted by various writers respecting the proportion of the insoluble substance in india-rubber. We further see that Gladstone and Hibbert's attempt to explain the presence of the insoluble part of india-rubber as due to the action of heat upon the latter can no longer be accepted, although the general effect of heat upon india-rubber, as observed by these authors, is no doubt true enough.

The most remarkable fact concerning this insoluble portion, is its high percentage of hydrogen, and the very large amount of oxygen it contains. Very suggestive in this relation appears the circumstance that on calculating from Spiller's[‡] analysis the formula of the resinous substance he isolated from oxidised india-rubber, we find it to correspond to a body $C_{30}H_{48}O_{10}$. The only difference between Spiller's resin, which, by the way, again shows the relation C : H = 10 : 16, and the insoluble constituent of india-rubber, consists simply in a considerable excess of hydrogen in the latter. Although it must be admitted that we possess neither physical, nor, directly convincing, chemical criteria respecting the purity and homogeneity of those two bodies, so that their analytical constants, and the formulæ deduced from them can only be held to be a general and statistic expression of their genetic relationship, it is nevertheless worth pointing out that both substances are obviously derived from the same hydrocarbon. This could scarcely be looked upon as a mere coincidence.

Spiller's resin, accepting the formula given above, would appear to be simply an oxygenated addition product of india-rubber. From this the insoluble constituent of india-rubber differs widely by its large excess of hydrogen, which is, indeed, so considerable, that the formula of this insoluble body very closely approximates that of a polyprene hydrate, $C_{30}H_{48}$, $10H_2O$. As a matter of fact this formula agrees equally as well with our analytical data as the formula at first deduced from them. Altogether, it appears to me that this highly interesting body cannot be looked upon as a transformation product of india-rubber, but is more likely a link between india-rubber and the carbohydrates, the celluloses in particular, which I conceive to be the raw material from which the plant produces all the terpenes, including india-rubber.

I have searched for similar insoluble constituents in other varieties of india-rubber, but failed to discover any trace of such in Congo ball, Lagos, Borneo, and Assam rubber. Considering, further, the very small amount of this constituent in Para rubber, I do not intend to go into further details at present respecting its nature and properties, but propose to deal now with the chief constituent of india-rubber, the soluble portion, or india-rubber proper.

The easily soluble part of india-rubber analysed above still contains about 2 per cent. of oxygen. If we add alcohol, drop by drop, to a very dilute solution (0·1 per cent.) of this india-rubber no precipitation occurs at first, but very soon a point is reached when the solution assumes a milky opalescence. Such a solution filters without a change, but on shaking it violently, it becomes perfectly clear, small stringy flakes being deposited. On filtering, a perfectly clear filtrate is obtained. From this the rubber is precipitated on adding an excess of alcohol, and the precipitated mass is dried in a current of hydrogen, or carbonic acid at a moderate temperature. In this manner the following product was obtained :—

0·1894 grm. substance.....	0·6108 grm. CO_2, 0·2024 H_2O.
Calculated for $C_{10}H_{16}$	C 88·23, H 11·76.
Found....................	C 87·91, H 11·86.

Although such a product cannot always be obtained, nor from every sample treated, preparations containing at most 0·4 per cent. of oxygen are always accessible.[*] Considering that the percentages are either within, or closely verging upon, the amount of the experimental error inherent to combustion analyses, no doubt can remain that, to within a few per cent., india-rubber consists of a soluble hydrocarbon of exactly the same empirical composition as the terpenes, $C_{10}H_{16}$.

Regarding the oxygen in india-rubber, the question arises as to the form or state in which the oxygen is present, whether simply in the form of molecular particles of the above discussed "insoluble constituent," or as an oxidation product of the rubber hydrocarbon (polyprene), or as an oxygen addition product of india-rubber. At the present time it is impossible to answer this question by direct experiment, owing to the entire lack of methods for disentangling colloidal mixtures. It seems to me, however, that the results of analysis throw considerable light upon this question. The combustion of a great number of different brands of india-rubber, all of which had been purified by extraction with acetone, gave figures

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[*] Bütschli, Ueber Strukturen, Leipzig, 1898. The study of this remarkable work is strongly recommended to all who are in any way interested in colloids and the nature of the colloidal state in general.

[†] Thus Seeligmann, in his recent book, "Le Caoutchouc et la Gutta Percha," Paris, 1896, says on page 113: "Le caoutchouc est en effet, formé de deux substances isomériques," and on page 178: "Ce que nous appelons caoutchouc n'est pas un hydrocarbure fixe et déterminé, mais un mélange d'au moins deux hydrocarbures polymères à équivalents élevés dérivant d'un carbure fondamental C_5H_8 et dont l'un possède au plus haut degré la nervosité, l'autre l'adhesivité."

[‡] Spiller's figures are: C 64·00, H 8·46, O 27·54 (see J. Chem. Soc., Feb. 1865, Vol. III. (New Series), 44).

[*] Gladstone and Hibbert (J. Chem. Soc. 1888, 630) found C 87·46, H 12·00.

differing more or less widely amongst themselves, and from those calculated for polyprene. Different brands of india-rubber, and even different qualities of the same brand, show great variations in the percentage of oxygen they contain. But on closer examination of the figures obtained, it was found that the relation C : H = 10 : 16 remained practically unaffected, no matter how much oxygen there might be present. Subjoined I give a summary table of the results obtained from a number of varieties of india-rubber.

Variety.	C.	H.	O.	C : H.
	Per Cent.	Per Cent.	100 per Cent. −(C + H).	
Para*...............	87·87	11·51	0·61	10 : 15 7
Congo Ball†.........	85·15	10·82	4·02	10 : 15·2
,, ,, *.........	86·41	11·07	2·51	10 : 15·3
Borneo†............	86·63	10·83	2·53	10 : 15·0
,, *.............	87·76	11·23	1·00	10 : 15·3
Ceara†.............	83·21	11·41	5·37	10 : 16·4
,, *.............	86·20	11·56	2·23	10 : 16·0
Lagos†.............	86·11	11·54	2·34	10 : 16·0
Sierra Leone†.......	86·38	11·34	2·27	10 : 15·7
Assam†.............	87·17	11·47	1·35	10 : 15·8
Mangabeira†........	87·08	11·39	1·52	10 : 15·7

* Extracted with acetone, dissolved, filtered, and precipitated.
† Only extracted with acetone.

These figures show plainly enough that all these varieties of india-rubber contain essentially the same hydrocarbon, $C_{10}H_{16}$, that their percentage of oxygen varies very considerably, but that this oxygen leaves the carbon hydrogen ratio practically unaffected. This, I contend, is possible only if the oxygenated bodies present are purely addition products of oxygen and ($C_{10}H_{16}$). That crude rubber, indeed, greatly inclines to take up oxygen from the air, may be gathered from the fact that a washed sheet of Para after drying in the usual hot air stove was found to contain 5·9 per cent. of oxygen, part of the same sheet dried in a vacuum pan contained only 1·7 per cent. of oxygen.

We may therefore state that india-rubber consists chiefly of a hydrocarbon $(C_{10}H_{16})_n$, readily soluble in benzene, chloroform and carbon bisulphide, that at least one variety (Para) contains a small quantity of a body insoluble in the above solvents, and possessing a composition widely different from that of india-rubber as above stated, and that as a further impurity, oxygenated addition products of the hydrocarbon $(C_{10}H_{16})_n$ occur in quantities varying considerably in different brands of india-rubber, but the solubility of which appears to be much the same as that of the india-rubber hydrocarbon itself.

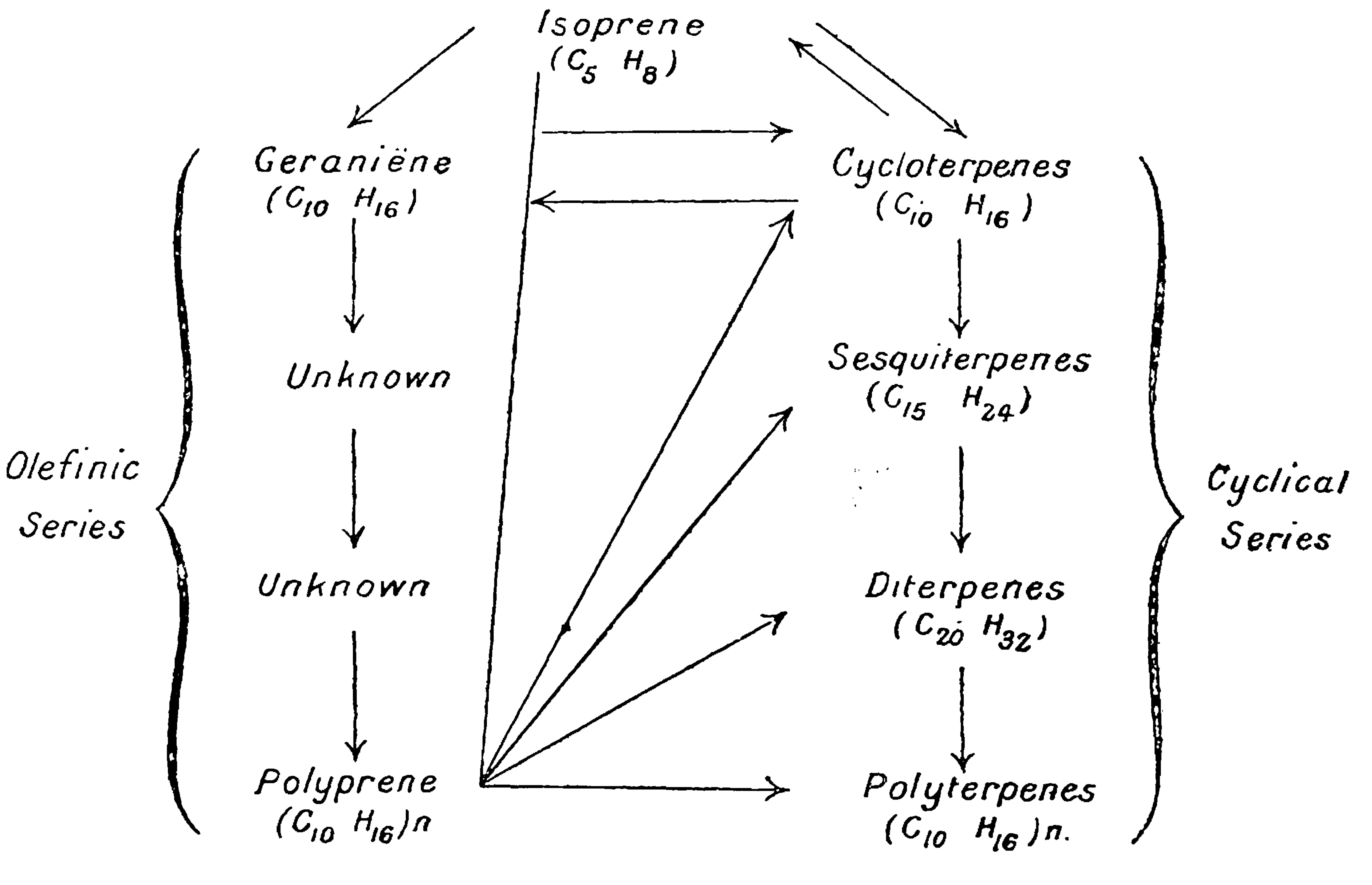
The oxygen addition products contained in india-rubber do not appear to sensibly affect the chemical behaviour of the whole. This applies as much to the vulcanisation process of india-rubber, which, as a chemical reaction, is rather badly defined, as to the polyprene derivatives hereafter to be mentioned. This, it seems to me, like many other indications, points to a very high molecular weight for india-rubber. On this subject very little is known at present.

That the colloids as a class are characterised by enormous molecular weights is generally admitted. It should, however, not be forgotten that this admission involves the assumption that the colloids, in the colloidal state,* possess finite and definite molecules. The evidence for this assumption is none too convincing.

Attempts to determine the molecular weights of colloids were made by Pfeffer, Lineburger, Sabanejew and Alexandrow, Vaubel, and Gladstone and Hibbert, and led to values ranging from 1,000 to 14,000. For india-rubber, Gladstone and Hibbert obtained the figure 6,504. Various observations suggest, however, that the supposed molecular weights of colloids, refer to extremely large molecular complexes, which, by changing the nature or state of the solvent, are capable of reduction by division. I am not in a position yet to furnish numerical data to prove this proposition, to which I was led by the observation† that by carefully adding precipitants to concentrated gelatinous solutions of colloids under certain conditions no precipitation takes place, but the colloidal jelly is completely liquefied. Jellies liquefied in this manner show not the slightest sign of turbidity and can be kept indefinitely in this state. Gelatinous solutions of cellulose sulphocarbonate (viscose), cellulose nitrates, cellulose tetracetate and india-rubber show this striking phenomenon very clearly.

The direct estimation of the molecular weight of india-rubber is a matter of very great difficulty, owing to the great viscosity, even of dilute solutions. Beckmann's boiling point method confronts us with the difficulty of heating colloidal solutions without producing coagulation deposits on the surface of the vessels. For this reason all my experiments with the bromide $(C_{10}H_{16}Br_4)_n$ and the chloride $(C_{10}H_{14}Cl_8)_n$ proved futile. These bodies possess, moreover, for the purpose in question, the drawback of an enormously increased molecular weight, owing to the accumulation of halogen in the already monstrously large polyprene molecule, and the now following investigations were carried out not in the least in the hope of meeting with a derivative of polyprene, which might lend itself to a determination of its molecular weight and so by deduction of that of polyprene itself, by one of the well known methods.

Action of chlorine: On passing chlorine into a chloroform solution of india-rubber, Gladstone and Hibbert obtained the body $C_{10}H_{14}Cl_8$, a dichlor-hexachloride. The composition of this compound is in perfect agreement with the conclusion arrived at by the same authors in the course of their optical examination of india-rubber, that this hydrocarbon has for $C_{10}H_{16}$ three pairs of doubly linked carbon atoms. Accordingly, india-rubber (polyprene) must be an olefinic and cannot be a cyclic compound, like the ordinary terpenes; in other words the constitution of polyprene would be that of an olefinic polyterpene, standing at the end of the olefinic terpene series, just as the ordinary polyterpenes stand at the end of the cycloterpenes, the hemiterpene C_5H_8—isoprene—representing the connecting link between the two series in this manner—



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* Many colloids are known to exist in both the colloidal and the crystalloidal state. In the latter they possess, of course, finite and definite molecules. The change of a substance from the crystalloidal to the colloidal state appears to be determined by an extensive aggregation of crystalloidal molecules, but in what this aggregation differs from that leading to the formation of crystalloid solids is only very imperfectly understood at present.

† Which finds very important practical application.

the arrows indicating the transformations, so far observed, of members of the one series into such of the other.

Action of Bromine: Gladstone and Hibbert described two bromine derivatives, $C_{10}H_{16}Br_4$ and $C_{10}H_{15}Br_5$; of these the former is obviously a direct addition product, while the latter owes its formation to the reaction $C_{10}H_{16}Br_6 = C_{10}H_{15}Br_5 + HBr$.

The hexabromide cannot be isolated, as, whenever its formation might be expected, the above formulated splitting off of hydrobromic acid takes place.

Of the two existing bromides, the tetrabromide, $C_{10}H_{16}Br_4$, is the most interesting. It is readily obtainable by the direct addition of bromine, to a solution of polyprene in chloroform. From this solution, Gladstone and Hibbert obtained the tetrabromide either by evaporating the solution, or by precipitation with ether, "but it always proved to be unstable on drying, with loss of hydrobromic acid." After years' experience with this substance, during which a very large number of preparations of it were made, I cannot altogether confirm the above statements of these authors. The isolation of the tetrabromide from the chloroform solution by evaporation is certainly hopeless, as partial decomposition is quite unavoidable. By running, however, the chloroform solution in a very fine jet into alcohol a snow white product is obtained, which, after washing with warm alcohol, can be dried at from 40 to 45° C., without the slightest degree of decomposition. The product thus obtained can be kept for an indefinite period without decomposing in the least.

Concentrated chloroform solutions (6 per cent.) of the tetrabromide are precipitated in the form of continuous threads, which, on stirring, form themselves into cocoon-shaped masses. Very dilute solutions (1 to 0·5 per cent.), on being run into alcohol yield no precipitate, but form a white milk-like emulsion, which can be preserved indefinitely in this state. On adding to such an emulsion a small quantity of ammonium sulphocyanide dissolved in alcohol, the tetrabromide is readily precipitated in form of a fine curdy mass. The bromide thus obtained gave the following figures on analysis:—

0·1786 grm. substance	0·2937 AgBr.
Calculated for $C_{10}H_{16}Br_4$	70·17 Br.
Found.........................	69·93 Br.

In a few isolated instances, the observation was made that the chloroform solution of the bromide, immediately after the preparation had been made, assumed the state of a very persistent jelly which could in no wise be brought into solution again. The dry bromide prepared from this in no way differed from the other preparations in composition, but was absolutely insoluble in all solvents. Eventually the curious fact was established, that this peculiar phenomenon always occurred when the preparation was attempted in a solution, the chloroform of which had been recovered from a previous operation. On the other hand, working exactly in the accustomed manner, in one case, a product was obtained which was even more soluble in benzene than in chloroform, which is the only solvent I know at present for polyprene bromide. These appear, however, to be merely extreme cases of series of physical changes of which polyprene bromide is capable, as I have found that also those preparations which yield apparently normal products, vary very considerably in their solubility in chloroform. We are here probably confronted by one of the many obscure phenomena exhibited by colloids, and for which at present we can suggest no explanation. Certain is this, that these differences are purely physical, as they find no expression in the chemical composition of the preparations in question, nor in their behaviour in chemical reactions.

As already stated, polyprene bromide is more or less readily soluble in chloroform. These solutions, are, however, of a highly colloidal nature, and all attempts to utilise them for the determination of the molecular weight of the bromide resulted in failure. In hydrocarbons, ether, acetic acid, acetic anhydride, and carbon bisulphide the bromide is quite insoluble. In aniline, pyridine, and quinoline it dissolves on moderate heating, in piperidine already in the cold, but the solution in every case seems to be accompanied by partial decomposition.

On heating the bromide to from 50° to 60° C., yellow discoloration sets in accompanied by evolution of hydrobromic acid. At higher temperatures, this decomposition proceeds very rapidly, leading to the formation of a dark brown substance. Even after very long heating to temperatures up to 150° C., this substance still retains a considerable proportion of bromine.

Considering this sensitiveness to higher temperatures the action of boiling alcoholic soda upon the bromide is surprisingly small. Sodium ethylate has a more pronounced action especially if heating under pressure be resorted to :—

0·6240 grm. $C_{10}H_{16}Br_4$ were in a sealed tube with sodium ethylate heated for six hours to 100° C.

			Per Cent.
Weight of insoluble white residue....	0·3623 grm.	=	58·06*
" " Br in residue....	0·1134 "	=	18·28
" " Br in solution ...	0·3210 "	=	51·44

0·6154 grm. $C_{10}H_{16}Br_4$ were in a sealed tube with sodium ethylate heated for 10 hours to 120° C.

			Per Cent.
Weight of insoluble white residue....	0·3438 grm.	=	55·87*
" " Br in residue	0·0823 "	=	13·38
" " Br in solution...	0·3478 "	=	56·51

The insoluble residue was not further examined, but the fact is worth calling attention to that the weight of this residue, plus the weight of the bromine in solution considerably exceeds the weight of the tetrabromide used. This is quite unintelligible unless we assume that in part at least ethoxyl is substituted for bromine, thus : —

$$C_{10}H_{16}Br_4 + 4C_2H_5O.Na = C_{10}H_{16}(O.C_2H_5)4 + 4NaBr.$$

Whether the reaction proceeds throughout in the sense of this equation, or whether bromine is at the same time split off in the form of hydrobromic acid I am at present not in a position to decide.

The tetrabromide is remarkably indifferent against inorganic acids. Even the action of fuming nitric acid in the cold is surprisingly slight. Boiling solutions of chromic acid, or chromic and sulphuric acid are without any appreciable effect.

It is evident therefore that polyprene tetrabromide, far from being the highly unstable body which Gladstone and Hibbert describe, is as a matter of fact a perfectly stable compound, and one exhibiting a remarkable degree of indifference against chemical changes.

Action of iodine:—Throughout the chemical literature on india-rubber we meet the statement that iodine is without action on it. Gladstone and Hibbert, in their paper, to which I have already repeatedly alluded and which is, beyond doubt, the most valuable contribution ever made to the chemistry of india-rubber, confirm this statement. Nevertheless, as I have found, polyprene is able to form with iodine an addition product containing a very large proportion of iodine. This iodide is readily obtained by uniting a 3 per cent. solution of india-rubber in carbon bisulphide with an excess of a concentrated solution of iodine in the same solvent, shaking the mixture well and allowing it to stand about 12 hours. After this time the whole solution is transformed into a jelly. This is forced through fine silk gauze and the resulting thin pulp transferred into alcohol. The whole is subsequently filtered, washed with alcohol until every trace of free iodine has disappeared, and the product on the filter is then dried at a temperature not exceeding 50° C. A yellowish olive-brown body is thus obtained, which was found to be insoluble in all ordinary solvents. It is soluble in warm aniline, better still in warm piperidine, but this solution appears to be accompanied by partial decomposition. On exposure to direct sunlight, very slowly in diffused daylight a faint evolution of iodine is noticeable, and the colour of the substance changes into a dark brown. The same change is produced by heating the substance to from 80° to 100° C. Its analysis gave the following figures :—

0·5470 grm. substance ..	0·7273 grm. AgI.
0·2008 " ..	0·1689 grm. CO_2, 0·0560 grm. H_2O.
Calculate for $C_{20}H_{32}I_6$ ..	C, 23·21; H, 3·09; I, 73·09.
Found....................	C, 22·93; H, 3·08; I, 72·03.

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* All these percentages are related to the weight of the tetrabromide used for the experiment.

Considering that the india-rubber used for this preparation contained 1·94 per cent. of oxygen the figures agree fairly well with the calculated formula. Whatever may be suggested regarding the purity or otherwise of the above preparation, the hitherto generally accepted notion of the indifference of polyprene against iodine renders the existence of a derivative containing over 72 per cent. of iodine a very interesting fact. The circumstance that for $C_{20}H_{32}$ only six atoms of iodine, against eight of bromine, are added may, no doubt, be taken as a further indication of a very high molecular weight of polyprene.

Haloid acids. The destructive action of hydrochloric acid upon india-rubber tubing and stoppers is universally recognised, but no experiments appear to have been made to elucidate the nature of this action.

The result of the action of hydrochloric acid upon polyprene dissolved in chloroform is, as would be expected, largely determined by the presence or absence of moisture during the reaction. Under the last-named condition I did not succeed in obtaining a compound of definite composition. Products were obtained at various times with a percentage of chlorine varying between 11 and 18 per cent. A polyprene hydrochloride, $C_{10}H_{17}Cl$, would require 20·22 per cent. of chlorine.

A different result is obtained on employing a current of moist hydrochloric acid. In this case absorption proceeds at first very rapidly, but gradually slackens, and after from 10 to 12 hours the reaction ceases completely. Towards the end a very marked change becomes noticeable in the solution which perceptibly darkens in colour, and at the same time becomes more limpid. This solution is run into alcohol, which precipitates a snow-white clotty substance. On standing in the alcohol, these clots gradually grow harder and brittle, and in this state the mere touch with a glass rod is sufficient to disintegrate them entirely into very small irregularly shaped particles. This product is washed with alcohol and dried in a desiccator. It forms, then, an easily friable white mass, fairly easily soluble in chloroform, but insoluble in all other commonly used solvents. It is soluble in warm aniline, pyridine, and piperidine, but apparently not without decomposition. The analysis of this product gave the following figures :—

0·3837 grm. substance	0·5223 grm. AgCl.
0·2188 grm. substance	0·4588 CO_2, 0·1615 grm. H_2O.
Calculate for $C_{10}H_{18}Cl_2$.......	57·41 C, 8·62 H, 33·97 Cl.
Found:......................	57·12 C, 8·13 H, 33·67 Cl.

These figures agree well with those of a polyprene dichlorhydrate. The india-rubber employed for this preparation contained 2·10 per cent. of oxygen. To obtain this compound care should be taken to keep the temperature during the operation below 10° C., and also to wash the precipitated product with cold alcohol in order to avoid loss of hydrochloric acid.

Already on heating to 40° C., polyprene dihydrochloride loses a considerable quantity of hydrochloric acid. The evolution of this gas ceases as soon as the percentage of hydrochloric acid in the residual body has fallen about 18 per cent., and even a considerable rise in temperature leaves this body practically unaffected. From this we may perhaps infer that polyprene monohydrochloride is a relatively very stable body.

All attempts to prepare polyprene hydrobromides and hydro-iodides ended in failure, and if such compounds should exist they can evidently not be obtained by the above-described method yielding the hydrochloride.

Interesting as the above discussed compounds are, as much from the purely chemical as from the technical point of view, their properties are quite unsuited for the purpose of the determination of the molecular weight of polyprene. The knowledge of this constant appears to me at the present moment of greater importance than any other point in relation to india-rubber. Long and wide experience with india-rubber and the colloids of the cellulose group, has given me the conviction that until we are in possession of this fundamental constant no material progress is possible, neither regarding the chemistry of india-rubber, nor concerning the question of the nature of the colloidal state in general.

It is further obvious that if the molecular weight of the colloids is indeed as high as is assumed to-day on reasonably plausible, if somewhat scanty, evidence, we can hardly expect to advance this important question by enormously increasing the size of the polyprene molecule by preparing addition products of it. In proportion with the increase of an already very large molecule falls the reliability of our methods for the determination of the molecular weight.

There appeared to me to be only two ways by which the desired end might be reached. The first resolves itself in the problem of detecting a chemical line of cleavage along which the polyprene molecule might be broken down into smaller, but clearly defined complexes, and the second would require the introduction into the polyprene molecule of groups which, as in the case of the hydroxyl or carboxyl group, would result in the formation of more chemically active bodies, possessing, at the same time, a greater range of solubilities in a greater variety of solvents.

The first of these two ways is obviously difficult to realise, as is already clearly illustrated by the very nature and reasons of the industrial applications of india-rubber, which lie in the pronounced resistance of india-rubber to any attack upon its molecule. The liability of india-rubber to oxidation is a well recognised fact, but, as I have shown above, it is highly probable that this oxidation process only leads to oxygenated addition products. There remains, therefore, only the remarkable breakdown process exhibited in the dry distillation of india-rubber, but, owing to the impossibility of controlling it, this process is, at least at present, totally unavailable for our purpose.

No controllable reaction is known, so far, to convert india-rubber into an oxygenated derivative containing a definite number of hydroxyl groups, but I believe that I have discovered such a reaction as the result of many attempts to find a solvent for polyprene tetrabromide possessing a moderately low solidifying or freezing point. In experimenting with phenol in this direction I observed that in working at a temperature only slightly exceeding the melting point of phenol (43° C.), one frequently succeeds in dissolving the tetrabromide more or less completely without any change taking place. Generally, however, already at this low temperature a very striking series of changes is noticeable; the white tetrabromide assumes a pale green, then successively a dull grey, dark blue, and finally a deep purple, almost crimson colouration. Simultaneously with this change of colour, the tetrabromide passes into solution and large volumes of hydrobromic acid are evolved.

The operation is best conducted on the water-bath, and as quite recent observations tend to show that it is advisable to prevent all access of moisture to the reacting mixture, it is desirable to connect the flask in which the reaction is carried out to a reflux condenser. 10 grms. of the tetrabromide are added to 100 grms. of absolute phenol, and this mass is heated for about 40 minutes on the water-bath. The operation is completed by heating for 10 minutes to 150° C. in an oil-bath. In order to remove from the dark purple liquid thus obtained, any particles of unaltered bromide which might be present, this liquid while still hot is filtered through some very close cotton fabric, or it is diluted with twice its volume of ether, filtered through a fluted filter, and the ether distilled off the filtrate. This solution is then run into a solution of 50 grms. of caustic soda in 1,000 c.c. of water, the resulting brownish purple solution heated until it is beginning to boil, and filtered. If the operation has been properly conducted, only inappreciable traces of an insoluble residue are retained on the filter. To the filtrate from 40 to 50 c.c. of acetic acid (50 per cent.) are now slowly added, when a brownish grey very fine precipitate is formed. The whole mass is now heated on a water-bath until this precipitate appears distinctly flocculent. It is filtered off, the still very strongly alkaline filter residue again suspended in about 600 c.c. of water, and distinctly acidulated with acetic acid. The colour of the precipitate is thereby changed into a light brown. The precipitate is again heated until distinctly flocculent, then filtered and washed neutral. After drying, the product thus formed is obtained as an amorphous, buff-coloured substance, which can easily be crushed into an impalpable powder. This substance is readily soluble in aqueous and alcoholic caustic soda, alcohol, acetone, ethyl ether, ethyl acetate, insoluble in benzene, chloroform, carbon bisulphide and petroleum ether. Its analysis gave the following figures :—

I.

0·1622 grm. substance	0·4785 grm. CO_2, 0·1184 grm. H_2O.
Calculated for $C_{34}H_{36}O_4$ ...	C, 80·16 ; H, 7·14.
Found	C, 80·15 ; H, 7·42.

The analysis agrees very well with the figures calculated for a body $C_{10}H_{16}(O.C_6H_5)_4$, tetroxyphenyl-polyprene, and its formation therefore would appear to be due to the very remarkable reaction.

$$C_{10}H_{16}Br_4 + 4C_6H_5OH = C_{10}H_{16}(O.C_6H_5)_4 + 4HBr.$$

The yield of this body was found, in a great number of preparations made, to amount very uniformly to from 60 to 60·5 per cent. of the weight of tetrabromide acted upon. This accounts almost exactly for one half of the tetrabromide taken. What happens to the other half, I have not yet been able to ascertain. Considering, however, the fact demonstrated by Gladstone and Hibbert (J. Chem. Soc. 1888, 686), that a solution of india-rubber in toluene can be heated to 200° C. without being thereby in the least affected, it seems highly improbable that the above described reaction which can be effected at water-bath temperature should lead to a breakdown of the polyprene molecule. Nevertheless, the inference to be drawn from the yield in the above process must be that the reaction in question is not so simple as it looks. This is fully borne out by the observation that, by confining the heating in the above process to from 90° to 95° C. in the water-bath, and treating the resulting solution in the same manner as before described, a body is obtained which in appearance scarcely differs from the first, and which behaves much in the same way towards solvents. On analysis this body gave the following figures :—

II.

0·1395 grm. substance	0·3860 grm. CO_2, 0846·0 grm. H_2O.
Calculated for $C_{34}H_{36}O_6$	C, 75·55 ; H, 6·66.
Found	C, 75·41 ; H, 6·73.

According to this analysis this body would appear to be tetroxyphenyl-dioxypolyprene.

The formation of this body is very difficult to explain, and this difficulty increases if we precipitate the phenol melts as above described by running them into a large volume (4,000 c.c.) of benzene. In this case precipitates are obtained, the colour of which, according to the conditions under which the reaction is carried out, varies from a somewhat dull crimson to a rich deep shade of indigo.

In carrying out the above-described reaction by heating on the water-bath only, and precipitating the melt by running it into 4,000 c.c. of benzene, a dark purple precipitate is obtained, separating out in very fine flakes. This is allowed to settle, the bulk of the supernatant liquid decanted off, the rest filtered, the filter-residue washed with benzene, and dried at from 40° to 50° C. The product formed is of a very fine deep indigo colour, and is easily crushed into an impalpable powder. It is soluble in alcohol, acetone, glacial acetic acid, and acetic anhydride. On standing, the purple colour of these solutions soon changes into a pale brown, but the colour of the solution in glacial acetic acid persists for a long time. The product appears to be very slightly soluble in chloroform. It is insoluble in ether, benzene, and carbon bisulphide. Its analysis gave the following figures :—

III.

0·1742 grm. substance......	0·4472 grm. CO_2, 0·1054 grm. H_2O.
Calculated for $C_{40}H_{41}O_{10}$ ...	C, 70·48 ; H, 6·02.
Found	C, 70·09 ; H, 6·78.

It will be seen that this analysis agrees tolerably well with the composition calculated for a body $C_{10}H_{16}O_5\ (OC_6H_5)_5$. I have, however, strong reasons to believe that this substance contains some slight impurity, to which, probably, its colour is due, but which, so far, I have not succeeded in removing. It will also be observed that this body would appear to be a still more complicated compound of the type of bodies represented by the above-described compound (II.) $C_{34}H_{36}O_6$.

I possess, at the present moment, no conclusive proof of the purity of this substance, or rather respecting the amount and nature of its impurity. But it is perhaps worth mentioning that the temperature at which it is prepared may be considerably varied from 100° C. downwards without any sensible alteration in the composition of the product. Thus 5 grms. of polyprene tetrabromide were heated with a solution of 50 grms. of phenol in 50 grms. of benzene, this solution boiling at 82° C. The reaction proceeded exactly as with phenol alone, but rather slower. The product, which was isolated by running the melt into a large volume of benzene, as before described, gave on analysis the following figures :—

0·2031 grm. substance......	0·5240 grm. CO_2, 0·1201 grm. H_2O.
Calculated for $C_{40}H_{41}O_{10}$ ...	C, 70·48 ; H, 6·02.
Found.....................	C, 70·40 ; H, 6·54.

On prolonged boiling with aqueous caustic soda, very rapidly on heating with alcoholic caustic soda, all these bodies split off phenol. On acidulating these solutions with acetic acid, the alcoholic solution being first diluted with much water, a flocculent brown precipitate is obtained, which is collected on a filter and washed neutral with water. After drying, it forms a light brown readily friable mass, which is easily soluble in dilute aqueous caustic soda, alcohol, ether, acetone, acetic acid, acetic anhydride; insoluble in benzene, chloroform, and carbon bisulphide. The analysis of this substance gave the following figures :—

IV.

0·2000 grm. substance......	0·4481 grm. CO_2, 0·1218 grm. H_2O.
Calculated for $C_{30}H_{36}O_{12}$ ...	C, 61·22 ; H, 6·08.
Found.....................	C, 61·11 ; H, 6·76.

This body may again be subjected to the above-named treatment with caustic soda, with the result of bodies with a still greater number of hydroxyl groups being formed. These bodies, like those before described, are precipitated from their solutions by means of acetic acid. Such a preparation, which, after drying, formed a dark brown somewhat gritty powder, the solubility of which in the solvents of this class of bodies is greatly diminished, gave the following results on analysis :—

V.

0·1661 grm. substance......	0·2501 grm. CO_2, 0·1030 grm. H_2O.
Calculated for $C_{30}H_{60}O_{30}$ ...	C, 40·00 ; H, 6·66.
Found.....................	C, 41·06 ; H, 6·88.

On repeating the above hydrating treatment successively darker products are obtained in the form of more and more colloidal precipitates, in form and colour greatly resembling colloidal precipitates of ferric hydroxide. The thorough washing of these precipitates is extremely tedious. After drying, they form very hard, almost black, resinous masses. I refrain from quoting analysis of these bodies, but may mention incidentally that, on drying, these colloidal products suffer very extensive dehydration.

I am continuing the investigation of these curious bodies, which, I believe, will furnish excellent material for the prosecution of the enquiry into the nature and constitution of india-rubber. The reaction to which we owe their formation is by no means confined to phenol. The cresols, carvacrol, thymol, as well as the naphthols, react in much the same way. Resorcinol, under the conditions above stated, reacts most energetically, forming an eosin-coloured mass. Pyrocatechol and hydroquinone react much more slowly. Of substituted phenols the action of the three chlorophenols and of the three nitrophenols was examined. The first-named yield chlorinated reaction products; the last-named appear to complicate the reaction by oxidation. Thiophenol acts in an entirely different manner, by simply eliminating the bromine of the tetrabromide as hydrobromic acid. Phenol-ethers, such as anisol or phenetol, do not react at all. This is almost conclusive evidence that it is the hydroxyl hydrogen of the phenol which forms the point of attack in the reaction in question, and this fact is at the same time the reason why, in the above formulæ, the oxygen appears as ether oxygen, and not as hydroxyl oxygen.

In my next communication I hope to be able to throw further light upon the nature of these bodies and their obviously close relation to india-rubber itself.

In conclusion I desire to express my best thanks to my assistant, Dr. Betteridge, for his assiduous support, especially in the latter part of the above work.

Discussion.

Mr. H. L. **Terry** wished to compliment Dr. Weber on the determined efforts he was making to unravel the mystery which surrounded our knowledge of the chemistry of india-rubber, though, looking at the present paper from a practical point of view, it could not be said that there was very much in it that appealed directly to the rubber manufacturer. The fact that the author had found the valuable insoluble part of Para rubber to be an oxygenated product was interesting in view of the fact that the amount of Spiller's resin and other intermediate oxidation products which a sample of manufactured rubber might contain was a direct index of the degree to which the rubber had deteriorated. He would like to know if Dr. Weber thought the presence of this insoluble body in the rubber of the euphorbiaceæ alone had anything to do with the well-known superiority of the rubber yielded by this order of trees over that of other orders in which this body was absent. The author had made what must be considered as an important pronouncement with regard to the drying of washed rubber by the vacuum process. A considerable difference of opinion existed with respect to drying by heat or vacuum, but from the statements Dr. Weber had made, backed up by figures showing the degree of oxidation taking place in the steam-heated chamber, it seemed that the case for the vacuum process was made out; that is, as far as theory was concerned. The matter of the greatly increased outlay on plant would, however, he thought, continue to act as a deterrent against the more general adoption of the process.

Dr. **Bailey** wished to ask Dr. Weber whether these various carbohydrate products were prepared under varying conditions, so that in the absence of recrystallisation they might have some criterion as to whether they had got a definite compound and not a body which was an amalgamation of the various constituents which had been put into the solution. He confessed ignorance of the history of the subject, and should like to know whether experiments had been made with regard to the viscosity and solubility of such matters in the fresh juice, because in colloidal substances all these properties would alter as time went on, and a good deal of light might be thrown on the subject and also on the nature of colloidal matter.

Dr. **Weber**, in reply, thought that Mr. Terry was under a misapprehension, caused, no doubt, by his not having the analytical figures for Spiller's resin before him. Spiller's resin possessed the same carbon-hydrogen ratio as india-rubber; consequently it could not be an oxidation product of india-rubber in the same sense as formic acid was an oxidation product of methyl alcohol, but must be an oxygenated addition product, comparable perhaps to oxidised or "blown" oils. On the other hand, the insoluble constituent of Para rubber no longer gave the carbon-hydrogen ratio of india-rubber, but it gave the same carbon-oxygen ratio as Spiller's resin, so that it would appear that by hydrogenating Spiller's resin it should be possible to revert to a compound similar to the insoluble constituent of india-rubber. He would, however, not go so far as to assume, with Mr. Terry, that the well-known superiority of Para rubber was due to this small quantity of its insoluble constituent. It was perfectly true that the physical properties of this constituent were very good, but at the same time he had some lingering doubt as to whether this constituent was not the cause of certain well-known difficulties and peculiarities noticeable in the vulcanisation of Para rubber. To settle an apparently simple question like this would require a great deal of time. On the face of it he felt rather apprehensive respecting the behaviour of this compound in the cold-cure process; the chloride of sulphur was almost certain to split off hydrochloric acid.

The oxidation which took place on drying the rubber in the ordinary air-stove was rather considerable, and vacuum-drying offered on this score great advantages to the manufacturer. His statement that the oxygen in india-rubber did not perceptibly affect its chemical behaviour did not contradict this; we had to distinguish the chemical from the physical effect of vulcanisation; and while even an undue amount of oxygen in the crude rubber might not perceptibly impede the chemical effect of vulcanisation; it certainly would detrimentally affect the physical result.

In reply to Dr. Bailey's question whether the compounds described had been prepared under varying conditions, he could give Dr. Bailey the assurance that in the course of the three or four years which this work had occupied an almost endless number of preparations of all these compounds had been made under the most varying conditions both by himself and his assistants, and only such compounds had been described which could always readily be obtained of uniform composition. Of course, as regarded the temperature at which these preparations had been made, no great variations were possible, most of the compounds being rather unstable at higher temperatures.

For a long time he had held the opinion that india-rubber was not a product of the metabolism of plant life, but was most likely a product of other changes in the cellulose tissues. From this point of view the insoluble part of india-rubber appeared to him particularly interesting. He certainly felt confirmed in his views by the readiness with which india rubber could be converted into such bodies as $C_{30}H_{32}O_{12}$ and $C_{30}H_{60}O_{30}$, bodies which exhibited in their properties, especially against hydrating and dehydrating agents, a striking resemblance to the carbohydrates.

THE ESTIMATION OF SULPHITES AND THIOSULPHATES IN PRESENCE OF EACH OTHER.

BY PROF. G. LUNGE, PH.D., AND D. SEGALLER, A.R.C.S.

A method for the estimation of sulphites and thiosulphates when in the same solution was published by Richardson and Akroyd (this Journal, 1896, 171). In their paper they say that they have tried Lunge and Smith's combined permanganate and iodine process for this estimation, but found the method tedious and, in their hands, incapable of giving good results. In proof of this they quote some results they obtained with the method, some of them showing more than double, others less than half, of the actual quantities present.

The method in question has been thoroughly repeated by us, and the following experiments will show that not only is it perfectly reliable, but that it has every one of the advantages which Richardson and Akroyd claim for their method. These operators obtained unreliable results through not following exactly the regulations laid down in the original paper (this Journal, 1883, 463) of Lunge and Smith, in which the method is described as follows:—

"The experimental work consists merely in two titrations, one with standard iodine, the other with standard permanganate, taking the precautions to add the latter in *large* excess to the alkaline or neutral solution, then acidifying and titrating back with excess ferrous sulphate and again with permanganate, as has been already explained."

We have in the course of our experiments found it sufficient to observe the precautions prescribed above. We have, of course, also observed the well-known general prescription for titrating sulphites by iodine, viz., to run the mixture to be examined into excess iodine till decolorised, and *not vice versâ*.

We proceed with the permanganate titration in the following manner:—Let the solution to be tested run in slowly into a large excess of the permanganate solution (almost twice as much permanganate as is used up in the reaction must be employed), then acidify after shaking well, add a known excess of ferrous sulphate solution, and titrate back with permanganate.

Provided the above precautions be taken, the method gives excellent results, as will be seen from the experiments quoted.

The following equations show the action of iodine and permanganate respectively:—

(1) $Na_2S_2O_3 + 2O_2 + H_2O = Na_2SO_4 + H_2SO_4$.
(2) $4Na_2SO_3 + 2O_2 = 4Na_2SO_4$.
(3) $2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$.
(4) $Na_2SO_3 + I_2 + H_2O = Na_2SO_4 + 2HI$.

If W = {weight of sulphur as thiosulphate oxidised to sulphate by …… } 1 c.c. $KMnO_4$ solution;
W′ = „ „ 1 c.c. iodine solution;
then 2W = {weight of sulphur as sulphite oxidised to sulphate by ……… } 1 c.c. $KMnO_4$ solution;
and $\frac{W'}{4}$ = „ „ 1 c.c. iodine solution.

In the mixture to be examined let—

S = weight of sulphur as thiosulphate.
s = „ „ sulphite.
N = number of c.c.'s permanganate required.
N′ = „ iodine required.

Then it is plain that—

$$\frac{S}{W} + \frac{s}{2W} = N \quad (1) \text{ or } 2S + s = 2WN.$$

$$\frac{S}{W'} + \frac{4s}{W'} = N' \quad (2) \text{ or } S + 4s = W'N'.$$

$$\therefore S = \tfrac{1}{7}(8WN - W'N').$$

$$s = \tfrac{2}{7}(W'N' + WN).$$

In the following experiments decinormal iodine and half normal permanganate were used. We employed for all of them the same solution of sodium sulphate, and another of sodium thiosulphate, which on being separately titrated gave the following results:—

(1) *Iodine Titrations.*

	C.c. Iodine.
(*a*) 25 c.c. thiosulphate solution titrated with iodine required …………	51·02
25 (β) „ „ „ „	51·00
25 (γ) „ „ „ „	50·98
Mean………………	51·00
(*b*) 25 c.c. sulphite solution titrated with iodine required………………	65·50
25 (β) „ „ „ „	65·50
25 (γ) „ „ „ „	65·51
Mean………………	65·50

(2) *Permanganate Titrations.*

(*a*) 25 c.c. thiosulphate solution were run into 150 c.c. permanganate solution.
Add 100 c.c. ferrous sulphate.
Titrated back, required 12·16 c.c. permanganate.
(25 c.c. $FeSO_4$ solution = 20·50 c.c. $KMnO_4$.)
∴ c.c.'s permanganate used up = 150 + 12·16 − 4 × 20·5 = 80·16 c.c. $KMnO_4$;
∴ 25 c.c. thiosulphate solution require 80·16 c.c. permanganate.

(*b*) 25 c.c. sulphite solution were run into 30 c.c. permanganate solution.
Added 25 c.c. ferrous sulphate solution.
Titrated back, required 3·63 c.c. permanganate.
∴ c.c.'s permanganate required = 30 + 3·63 − 20·5 = 13·13 c.c. permanganate.
(Mean of three experiments gave 13·11 c.c. permanganate.)

In the following experiments the two solutions were mixed in different proportions and then titrated, both iodine and permanganate, as prescribed by Lunge and Smith. If that method is correct, the results obtained with the mixture must agree with the sum of the results obtained when titrating the results separately.

I. A mixture of	Iodine.	Permanganate.
	C.c.	C.c.
25 c.c. $Na_2S_2O_3$	51·00	80·16
25 c.c. Na_2SO_3	65·50	13·11
50 c.c. mixture	116·50	93·27

20 c.c. mixture— C.c.
Required iodine ………………… 46·65
Calculated……………………… 46·60
Error = +0·05 = 0·11 per cent.
Required permanganate ………… 37·44
Calculated……………………… 37·31
Error = +0·13 = 0·28 per cent.

II. Mixture of	Iodine.	Permanganate.
	C.c.	C.c.
25 c.c. $Na_2S_2O_3$	51·00	80·16
50 c.c. Na_2SO_3	131·00	26·22
75 c.c.	182·00	106·31

25 c.c. mixture— C.c.
Required iodine ………………… 60·52
Calculated……………………… 60·68
Error = −0·16 = 0·26 per cent.

20 c.c. mixture— C.c.
Required permanganate ………… 28·38
Calculated……………………… 28·37
Error = +0·01 = 0·03 per cent.

III. Mixture of	Iodine.	Permanganate.
	C.c.	C.c.
25 c.c. $Na_2S_2O_3$	51·00	80·16
75 c.c. Na_2SO_3	196·50	39·33
100 c.c.	247·50	119·49

20 c.c. mixture— C.c.
Required iodine ………………… 49·32
Calculated……………………… 49·50
Error = −0·18 = 0·38 per cent.

20 c.c. mixture— C.c.
Required permanganate………… 24·05
Calculated ……………………… 23·99
Error = +0·06 = 0·22 per cent.

IV. Mixture of	Iodine.	Permanganate.
	C.c.	C.c.
25 c.c. $Na_2S_2O_3$	51·00	80·16
250 c.c. Na_2SO_3	655·00	131·10
275 c.c.	706·00	211·26

25. c.c mixture— C.c.
Required iodine ………………… 64·00
Calculated……………………… 64·18
Errors = − 0·188 = 0·28 per cent.

25 c.c. mixture— C.c.
Required permanganate………… 19·15
Calculated …………………… .. 19·20
Errors = − 0·05 = 0·26 per cent.

V. Mixture of	Iodine.	Permanganate.
	C.c.	C.c.
50 c.c. $Na_2S_2O_3$	102·00	160·32
25 c.c. Na_2SO_3	65·50	13·11
75 c.c.	167·50	173·43

20 c.c. mixture—	C.c.
Required iodine	44·81
Calculated	46·67

Errors = + 0·14 = 0·31 per cent.

20 c.c. mixture—	C.c.
Required permanganate	46·00
Calculated	46·24

Errors = – 0·24 = 0·50 per cent.

VI. Mixture of	Iodine.	Permanganate.
	C.c.	C.c.
125 c.c. $Na_2S_2O_3$	255·00	400·80
25 c.c. Na_2SO_3	65·50	13·11
150 c.c.	329·50	413·91

30 c.c. mixture—	C.c.
Required iodine	63·28
Calculated	64·10

Errors = – 0·82 = 1·26 per cent.

Required permanganate	82·52
Calculated	72·78

Errors = – 0·26 = 0·31 per cent.

Each test was performed over in this estimation. In most cases the error committee amounted to a small fraction of a per cent., only in one case it ran to a somewhat larger amount.

The solutions were then analysed by Richardson and Akroyd's method and practically identical results were obtained. But it is indeed difficult to see wherein the very great advantage over all other methods claimed by the above authors for their methods exists. Thus, to compare it with Lunge and Smith's method, in each case sulphides have to be precipitated and filtered off; then whereas in Richardson and Akroyd's method there are three titrations to be performed, in Lunge and Smith's there is only an iodine and a permanganate titration. As far as time is concerned the advantage, therefore, rests with the latter method. But we find this method also more accurate, as proved by the following figures, showing the results of both methods applied to known quantities of thiosulphate and sulphite solutions:—

$Na_2S_2O_3\,5H_2O$ present.	Found (Lunge and Smith).	Found (Richardson and Akroyd).
0·1086	0·1068	0·1050
0·1320	0·1301	0·1293
0·1648	0·1630	0·1618
0·1886	0·1868	0·1839

$Na_2S_2O_3\,7H_2O$ present.	Found (Lunge and Smith).	Found (Richardson and Akroyd).
0·0316	0·0321	0·0301
0·0825	0·0831	0·0802
0·0916	0·0939	0·0898
0·1131	0·1142	0·1110

New York Section.

Meeting held on Friday, February 23rd, 1900.

MR. THOS. J. PARKER IN THE CHAIR.

FREE FATTY ACID IN OLIVE OIL.

BY RUSSELL W. MOORE, A.M., M.SC.

For importation into the United States olive oil is divided into two classes: first, salad or edible oil, and, second, oil for mechanical and manufacturing purposes. This division has given rise to several differences of opinion between importers and the Customs authorities, which was hardly avoidable, especially in view of the fact that many Italians of the poorer class consume as food an oil which is much inferior in taste, appearance, and odour to those grades of oil which are used by large wool and silk mills. As a result, numerous samples have been submitted for test for the amount of fatty acid present. The following method was adopted:—

A pipette is used that will deliver exactly 7·05 grms. of oil, and the free acid titrated with quarter normal alkali in an ordinary oil sample bottle of four-ounce capacity with 50 c.c. of a 10 per cent. salt solution, using phenolphthalein as an indicator. After each addition of alkali the bottle and its contents are well shaken until a fairly permanent pink colour is obtained.

Since 7·05 is exactly one-fourth of the molecular weight of oleic acid, and quarter normal alkali is used, the number of cubic centimetres consumed of the latter is equal to the per cent. of free fatty acid present in the sample of oil under examination calculated to oleic acid. This method can also be used for solid fats by employing a previously warmed salt solution.

The following results were observed by Mr. N. J. Lane in the Customs Laboratory at New York:—

Olive Oils.

Percentage of Free Fatty Acid.	Number of Samples.		
	French Oil.	Italian Oil.	Total.
Less than 1 per cent.	11	..	11
1 to 1½ per cent.	9	10	19
1½ to 2 „	3	13	16
2 to 3 „	5	12	17
3 to 5 „	7	8	15
5 to 10 „	10	5	15
10 to 20 „	3	8	11
29·2 per cent.	1	0	1
			105

Two samples of Greek origin had an average percentage of 7·47.

Yorkshire Section.

Meeting held on Monday, January 29th, 1900.

MR. C. RAWSON IN THE CHAIR.

THE ABSORPTION OF BASIC CHROME SALTS BY SKIN.

BY H. R. PROCTER AND R. W. GRIFFITH.

The fact that leather could be produced by the action of basic chrome solutions on skin was pointed out by Knapp as early as 1858, but it was not till public attention was drawn to the advantages of chrome leathers by the success of the Schultz process that it received any practical recognition. The heavy royalties charged by the owners of the Schultz patent, and the litigation to which it gave rise, as well as the somewhat complicated nature of the process, which involved treatment of the skin with a bath containing acidified bichromate, and the subsequent reduction of the chromic acid absorbed by the skin by means of sodium thiosulphate, led Martin Dennis in 1893 to obtain an American patent for a process identical with that described by Knapp, which was granted on the ground that only basic chlorides would produce leather—a view which has since been shown to be entirely without foundation. It has been found that chrome alum, which is one of the cheapest commercial chromium salts, is quite as satisfactory in use as the more expensive chromium chloride.

Although the absorption of chrome salts by skin, and their tanning effect, has so long been known, considerable uncertainty exists as to the constitution of the basic salt absorbed; and the object of the experiments we are about to describe was to determine whether the absorbed salt possessed the same degree of basicity as that contained in the bath, or whether it was absorbed in a more or less acid condition. This point is not only interesting from a theoretical point of view, but is of considerable practicable importance, since, in order to strengthen a partially exhausted chrome bath for further use, it is necessary to restore it to the same degree of basicity as it had at first, and no easy means of determining this are at present known. It was further desired to ascertain how the absorption of the chrome salt was affected by the addition of common salt to the bath, which in practice is found to produce a softer, if perhaps a thinner leather.

These problems, most of which also arise with regard to alum tannage, have occupied the attention of several chemists. Berzelius (Lehrbuch, **9**, 372) expressed the opinion that in alum tanning a basic salt was absorbed, but Knapp ("Natur und Wesen der Gerberei," 1858) showed that in solutions of alum and aluminium chloride the residual solutions had the same composition as those before use—a conclusion which does not wholly correspond to the work of later investigators. The problem is by no means an easy one, since no practical method exists of measuring the acidity of the basic salts except that of determining the whole of the acids and bases present, and in all technical cases it is complicated by the presence of sodium and potassium salts of the same acids. Where salt is used the additional difficulty of the presence of chlorine in an unknown state of combination occurs, for, though no complete reaction takes place, theory shows that an equilibrium must be formed in which all the acids and bases present take part; and, as aluminium and chromium chlorides are freely absorbed by the skin, the salts fixed probably consist of a mixture of chlorides and sulphates. Owing to neglect of this point, an elaborate research of Reimer (Ding. Polyt. Jour. 205) failed to give any decisive result; and the following experiments have been made principally to clear up the points yet doubtful.

Our experiments were carried out with pieces of "sheep grain" (*i.e.*, the outer side of a split sheepskin) which had been delimed by a solution of boric acid and phenol, and afterwards well washed in several changes of water. The following was the method adopted in the first series of experiments. In each of two flasks was placed 10 grms. of chrome alum dissolved in water up to 80 c.c., and to this 3 grms. of crystallised sodium carbonate dissolved to 20 c.c. was gradually added. To one of these was further added 20 c.c. of a solution containing 7 grms. of common salt. Pieces of wet squeezed pelt, weighing 50 grms. each, were introduced into large stoppered bottles with 200 c.c. of water, which were placed in the rotating frame of a Kent's "Speedwell churn," which is found very convenient for such experiments; and to each of them, one of the chrome solutions was added in quantities of 30 c.c. at a time, at intervals of half an hour. When about half the chrome solutions had been added, the pelt was allowed to remain at rest in the solutions over night, and the operation continued in the morning till the pelt showed a uniform colour throughout. 20 hours was allowed for this tannage, in order as far as possible to reach a condition of equilibrium. After wringing the leather till nearly dry, a portion was taken from each piece and reserved for analysis. The remaining portions of the leather were then washed, and placed in separate bottles, each containing 1·5 grm. of borax in sufficient water to cover the leather, and were again placed in the churn frame, and rotated for half an hour, when the leathers were again wrung out, and dried for analysis. The volumes of the residual chrome liquors, which we may distinguish as "no salt chrome liquor" and "salt chrome liquors" were at this stage 356 c.c. and 352 c.c. respectively. We now placed in these liquors fresh pieces of squeezed wet pelt as before, that in the "no salt liquor" weighing 35 grms. and that in the salt liquor 33 grms. These were now churned in exactly the same manner as the previous pieces, the length of the tannage being 22 hours in each case. The leathers were wrung out of the solutions as before, and set aside for analysis without further treatment, being labelled "2nd tannage no salt" and "2nd tannage salt" respectively. This was similarly repeated to a third and fourth tannage. In each case the fourth tannage was very imperfectly tanned, and the liquor was nearly exhausted.

We now proceeded to the analysis of the leathers and residual liquors. As the ignited ash of chrome leathers is insoluble in acids, it was neccesary to bring the chromium into solution by fusion with soda and oxidation to chromate, which was subsequently acidified with hydrochloric acid, and the chromic acid estimated volumetrically with potassium iodide and thiosulphate—a method which previous work had shown to be very exact. The sulphuric acid was also estimated in a portion of the same solution. Where salt had been used, the chlorides were estimated in a separate portion of the leather. Unfortunately the necessity of fusion with soda rendered it impossible to estimate sodium and potassium in the ash, and as portions of the SO_4 (and Cl) were present in combination with these bases, fresh experiments were undertaken to determine their amounts.

It became evident from the results of these new experiments that the precautions taken in the first series were not sufficient to ensure the absolute uniformity of the different portions of the same tannage of leather taken for the different determinations, and that other sources of error existed which were afterwards eliminated, and it would be idle therefore to give numerical results or to employ them as the base of elaborate calculations; but it was quite clear, as was to be expected, that with progressive exhaustion of the liquors, the percentage of chromium taken up by the leather was correspondingly lessened, falling from 2·9 to 1·4 per cent. in the salt tannage, and from 3·7 to 1·0 per cent in the non-salt, reckoned on the weight of the leather dried at 100°, the later tannages being distinctly under-tanned. In fact, it may be taken that about 3 per cent. of Cr is necessary to produce a fully tanned leather. Comparing the two sets of tannages, it appears that salt does not facilitate the absorption of Cr, the amount taken up by the pelt being less, and the exhaustion of the bath less rapid and complete in its presence than without it. This is not necessarily a disadvantage in practice, and may partly account for the fact that leathers tanned with addition of salt are softer and thinner than in its absence. A somewhat similar action appears to be produced by salt in vegetable tannages.

The SO_4 found in the leather remained approximately constant throughout the experiments, amounting to about 2·6 of the dry leather weight on the average, and rather rising than diminishing as the bath became exhausted. About 1 per cent. of this, however, consisted in soluble alkaline sulphates easily removable by washing, and only mechanically retained by the leather; and we found later that a portion had also been driven off in ignition.

A second set of experiments was therefore undertaken in which these salts were removed by washing, and returned to the residual liquors before analysis, so as to determine the salts actually fixed by the leather, and the changes in constitution of the tanning liquors. The tanning solution for each experiment was made with 100 c.c. of 10 per cent. chrome alum solution, to which was added 25 c.c. of a normal sodium carbonate solution containing 1·32 grm. Na_2CO_3.

For each tannage 100 grms. of wet sheep grain was used in 200 c.c. of water, and to one 25 c.c. of a 20 per cent. solution of common salt was added, and the chrome solutions were introduced in successive portions as the tannage proceeded, as in the first experiments. The length of the tannage was 19 hours, at the conclusion of which the leathers were well squeezed and washed in successive portions of distilled water, which were added to the residual liquors till each was made to one litre. The leathers were air-dried and weighed, and portions were then weighed out for analysis and treatment with borax, while the residual liquors were also submitted to analysis. Portions of the leather samples were ignited at a low temperature to avoid loss of sulphuric acid and chlorine, but it was found that with the utmost care considerable amounts were expelled, and fresh analyses were therefore made in which the leather was moistened with Na_2CO_3 solution before ignition. This was effective in preventing loss of SO_4 and Cl, but introduced another difficulty. It is known that purified pelt contains a portion of organic sulphur, the amount being given by Stohmann and Langbein as 0·3 per cent.; and this, on fusion with Na_2CO_3 and the resulting chromate, was also oxidised into H_2SO_4. Nothing remained therefore but to determine and allow for the SO_4 so formed. The ash in each case was fused, as before, with sodium carbonate in excess, till the whole was oxidised to sodium chromate and soluble in water. The chromic and sulphuric acids and the chlorine were determined by the same methods as before, in aliquot parts of the resulting solutions.

The residual tanning solutions were similarly analysed, except that for determination of chromium a portion of the solution was oxidised with permanganate and described by one of us (this Journal, 1897, 414), so that in all cases the chromium might be determined by the same standard thiosulphate. In most cases several determinations were made and the average taken.

A test analysis made with skin and a known quantity of chrome alum gave almost exactly the theoretical value.

Table I. gives the result of these analyses. It was attempted to determine the SO_4 formed by oxidation of the pelt by ignition and fusion of a weighed portion with sodium carbonate and potassium bichromate, and a result of 0·124 grm. per 100 grms. of wet pelt was obtained; but as want of material prevented the analysis being made on the same skin as that used for the tanning experiments, and the results of repeated analysis of both skin and liquors showed an equal excess of SO_4 in both cases over that present in the original liquors, which could not be accounted for by the reagents used, it was thought best to assume that the excess was due to sulphur in the original pelt, the amount required (0·186 grm.) agreeing very closely with Stohmann and Langbein's value, which would yield about 0·180 grm. SO_4 on the weight of pelt employed. The differences of the chromium and chlorine from that originally present may be taken as experimental errors, as in both cases the actual quantities determined were only small fractions of the whole quantities employed, and in a material like leather it is impossible to secure absolute uniformity of the samples analysed.

Table II. gives the percentage composition of leathers obtained. It will be noted that in this, as in the previous experiments, a somewhat larger amount of chromium is fixed in the "no salt" leather than in the salt leather, while no great difference is to be observed in the total acid fixed.

In the salt tannage it is clear that a small part of the SO_4 has been replaced by Cl.

In both cases a portion of this acid is removed by the borax treatment, but it may be noted that if a more alkaline substance than borax is used, or even if it is used in such excess as to remove the whole of the acid, the quality of the leather is destroyed, and although no Cr is removed, it returns to an undertanned and horny condition. One of the most important points which it is desirable to settle is the question of the degree of the basicity of the salts fixed by the leather and remaining in the residual liquor.

This is calculated in Table III., in which the proportionate number of molecules of SO_4 and Cl_2 combined with Cr_2 is given in each case.

TABLE I.

Total Quantities Used and Found.

—	No Salt Tannage.		Salt Tannage.		
	Cr.	SO_4.	Cr.	SO_4.	Cl.
Residual liquor	0·369	3·032	0·420	3·162	2·910
Fixed in leather	0·725	0·994	0·625	0·864	0·150
	1·094	4·026	1·045	4·026	3·060
Difference	0·054	..	0·005	..	0·010
Less amount of SO_4 original in raw pelt	..	0·186	..	0·186	..
Theoretical constitution of unused liquor	1·040	3·840	1·040	3·840	3·050

TABLE II.

Percentage Composition of Leathers.

—	No Salt Leather. 20·07 grms. dried at 100° C.			Salt Leather. 20·74 grms. dried at 100° C.		
	Washed.	Extracted by Borax.	Left in Leather.	Washed.	Extracted by Borax.	Left in Leather.
Cr.	3·6	..	3·6	3·0	..	3·0
SO_4	5·0	3·0	2·0	4·2	2·5	1·7
Leather fibre by deduction	91·4	..	..	92·1	..	..
Cl.	..	..	..	0·7	0·5	0·2
	100·0			100·0		

TABLE III.

Relation of Cr_2 to Acids fixed in Leather and in Residual Liquors.

Unused Liquor	$Cr_2(SO_4)_{1 \cdot 8}$
No salt leather	$Cr_2(SO_4)_{1 \cdot 21}$
Residual, no salt liquor	$Cr_2(SO_4)_{2 \cdot 6}$
Salt leather	$Cr_2(SO_4)_{1 \cdot 2}. (Cl_2)_{0 \cdot 5}$
Residual salt liquor	$Cr_2(SO_4Cl_2)_{2 \cdot 3}$

Of course the formulæ given in Table III. are not intended to express the actual constitution of the salt, but only the relative number of molecules of Cr_2, of SO_4, and of Cl_2 present. Naturally these proportions are usually fractional, as the basic salts may be regarded as mixtures containing various proportions of normal salts and oxides and hydrates.

The numbers are obtained from the proportion:—

$$\frac{\text{Weight of } Cr_2}{104} : \frac{\text{Weight of } SO_4}{96} :: 1 : x.$$

and similarly with regard to Cl.

Taking first the simpler case of the "No Salt" leather. The compositon of the Cr salt in the original liquor is obtained by deducting the SO_4 present not only in the chrome alum as K_2SO_4, but also as Na_2SO_4 formed with the Na_2CO_3. In this case we find that the chrome salt is as Cr_2 is to $(SO_4)_{1 \cdot 8}$ in place of $Cr_2(SO_4)_3$, as it exists in the chrome alum. Turning to the unboraxed leather, we find that the proportion is $Cr_2(SO_4)_{1 \cdot 2}$ making it clear that a somewhat more basic salt has been fixed than that present in the original bath. Correspondingly we find that after deducting the alkaline sulphates from the residual liquor, the chrome salt remaining has become distinctly more acid, being $Cr_2(SO_4)_{2 \cdot 6}$. Turning to the salt tannage, very similar results may be observed: the basicity of the original liquor is the same of course as in the former case, though it is quite probable that in the acid constituent of the salt, Cl_2 has been partly substituted for SO_4.

In the residual liquor the proportion of Cr to SO_4 molecules is as 2 to 2·6, and we have no means of determining directly what proportion there is between the Cl_2 and SO_4, since alkaline salts of both radicles are present in the solution.

Turning, however, to the leather, we find that it contains in combination with Cr_2, 1·2 molecules of SO_4 and 0·5 molecules of Cl_2, showing, therefore, a greater total acidity than the "No Salt" leather, though the SO_4 alone is really somewhat lower. This, of course, should involve a lower acidity in the liquors, and the fact is that a part of the SO_4 which has been estimated as representing the acidity in combination with chromium in the liquor, has really been employed in liberating Cl from the salt and exists as Na_2SO_4. Deducting the amount of this as calculated from the Cl in the leather, and amounting to 0·3 molecules per Cr_2 in the liquor, we have left an actual acidity of 2·3 acid molecules for Cr_2, which is thus materially less than in the case of the "No Salt" liquor in which the acidity is as $Cr_2 : (SO_4)_{2 \cdot 6}$. It is therefore evident that used liquors require not merely strengthening for use, but also some addition of either chromic hydrate or an alkali to increase the basicity of the liquor.

If Na_2CO_3 were used for this purpose Na_2SO_4 would, of course, accumulate in the liquors and our experience is not enough to say whether this would prove injurious.

Should this be the case the use of barium or calcium carbonates or hydrates may be suggested as a way out of the difficulty.

Our results differ with regard to the fixation of a more basic salt than that in the solution from those of Messrs. Krutwig and Dalimier (Rev. Univ. des Mines, **48**, [3], 23, abstracted in this Journal, Vol. 19, page 58). They, however, operated with normal salts, $Cr_2(SO_4)_3$, and it is very possible that the basic salt in the liquor tends to revert to that form, allowing the dissolved oxide to be more freely taken up by the skin. Normal salts are, however, incapable (at least without the addition of salt) of making a satisfactory commercial leather.

It is probable that the basicity of the salts absorbed is influenced both by the constitution of that present in the original bath and by its concentration, since untanned skin has a very marked absorptive power for acids, and it is quite possible that with different conditions in these respects the relation between the Cr and the acid absorbed might be altogether reversed. We think, however, that it may be taken that our conclusions hold good under the conditions prevailing in practical tannage. After the skin has been washed from the chrome bath it is completely converted into leather, but it has been found that its durability and permanency are much increased by removing a still further portion of the acid contained in it by alkaline treatment. Borax is the agent most generally used for this purpose in about $\frac{1}{2}$ per cent. solution, though other alkaline compounds, such as suspended chalk or sodium bicarbonate solutions may be substituted.

Portions of the leathers produced, equal to about 12 grms. if dried at 100° C., were therefore treated each in 500 c.c. water with 2 grms. borax. No chrome was dissolved out, but considerable quantities of SO_4, and in the case of "Salt" leather of Cl, were withdrawn. Columns 2, 3, 5, and 6 of Table II. show the amount of acid extracted by the borax bath, and the consequent change of composition of the leathers.

It may be noted that the proportion of acids to Cr in the boraxed leathers are much reduced, the salts remaining in the leather being very basic, the relation being $Cr_2(SO_4)_{0 \cdot 6}$ in the "No Salt" tannage, and $Cr_2(SO_4)_{0 \cdot 6}(Cl)_{0 \cdot 1}$ in the case of the salt leather.

Since doing most of our analytical work, our attention has been drawn to an article by Paul von Schroeder on the estimation of Cr in chrome-tanned leather, published in the Deutsche Gerber Zeit. **42**, 153—156, and abstracted in this Journal, page 177. He incinerates about 3 grms. of the leather, and then oxidises the ash in the wet way with nitric acid and potassium chlorate. He subsequently evaporates frequently with concentrated hydrochloric acid to drive off the nitrous and chlorine fumes, the chromic acid becoming again reduced. In order to estimate volumetrically with KI and sodium thiosulphate, he subsequently oxidises with sodium peroxide, removing traces of hydrogen peroxide left by evaporation with a few drops of alcohol.

The results of this method, when very carefully carried out, are no doubt accurate, but the process is altogether unnecessarily complex, since mere fusion of the ash with dry Na_2CO_3 in an open crucible for a few hours will produce complete oxidation, and leave the chromic acid ready for immediate titration.

This can be easily carried out with one of the excellent Teclu burners, and is the method by which most of our analyses have been performed, and which by working on known quantities we have proved to give accurate results. But we have since found that by employing a mixture of two parts of dry Na_2CO_3 to one part calcined magnesia (light), with which the ash is thoroughly pulverized before fusion, we can obtain a thoroughly oxidized fuse from the ash of 3 grms. of leather within 20 minutes. It is necessary to dissolve the fuse in a little acid, as a small quantity of the chromic acid remains in combination with the magnesia.

ABSORPTION OF CHROMIC ACID BY SKIN FROM SOLUTION OF POTASSIUM DICHROMATE.

BY H. R. PROCTER AND E. F. HAMER.

(Contribution from the Leather Industries Laboratory of the Yorkshire College.)

In experiments by Heal and Procter, some account of which may be found in the present Journal, p. 248, 1895, it was shown that chromic acid liberated by addition of hydrochloric acid from a solution of dichromate was rapidly and almost completely taken up by skin, but that undecomposed dichromate was practically not absorbed. As it is frequently necessary to determine the amount of free chromic acid in a tanning liquor, further experiments were made, in conjunction with Mr. G. H. Russell, to ascertain whether a separation could be affected by hide powder, in course of which it became evident that the amount of absorption was affected by the presence of common salt in the solution. The following investigation was undertaken to ascertain to what extent, under different conditions, absorption from neutral dichromate could take place.

Experiments were first made with sheep grain (the outer side of a split sheepskin) which had been freed from lime by treatment with a solution of boric acid and phenol, and then thoroughly washed, and wrung out as evenly as possible. A moisture determination made on a portion showed that it lost 80 per cent. when dried at 100°. From the remainder two pieces of 5 grms. each were cut and marked I. and II.

Piece I. was placed in a stoppered bottle with 25 c.c. of solution of commercial potassium dichromate of 49 grms. per litre (approximately N/1 with regard to iodine) and 25 c.c. of water.

Piece II. was similarly treated, but 25 c.c. of a solution containing 2 grms. sodium chloride was substituted for the 25 c.c. of water.

The bottles were placed in the frame of a "Speedwell" churn, and shaken by rotation for 1 hour, allowed to rest for 30 minutes and again rotated for 1 hour, after which the skin was allowed to remain in the solutions overnight, so that equilibrium beetween the solutions within and without the pelt might be reached.

The original solution was titrated with N/1 NaOH and phenolphthalein, to which indicator normal alkaline chromates react neutral, and dichromates behave as dibasic acids, 1 mol. NaOH neutralising $\frac{1 \text{ mol.}}{2}$ of dichromates, or 147 grms. per litre of $K_2Cr_2O_7$. Thus the solution of 49 grms. per litre which is normal as an oxidising agent, is only N/3 as regards its alkali saturating power, 25 c.c. of the solution required 8·25 c.c. of N/1 NaOH, corresponding to 1·213 grms. dichromate.

The solution was also tested for free chromic acid by a method invented by Mr. C. B. Heal (this Journal, 1895, p. 250), which depends on the precipitation of a basic copper chromate from cupric sulphate by the least trace of neutral chromate in a boiling solution of dichromate. Titrated by this method, 25 c.c. of the dichromate solution required 6·25 c.c. of N/10 NaOH to produce a precipitate corresponding to 0·00625 grm. of CrO_3. This is apparently present as an impurity in the dichromate employed.

Liquor I.—This originally contained 25 c.c. of the dichromate solution, 29 c.c. of water including that derived from the pelt, and 1 grm of dry pelt. 10 c.c. of the residual liquor, titrated with N/10 NaOH consumed, on the average of several titrations, 15 c.c., therefore the whole liquor would consume $\frac{54 \times 1 \cdot 5}{10}$ N/1 NaOH = 8·1 c.c. corresponding to 1·191 grm. of dichromate, and showing a total absorption by the 1 grm. of skin of 0·022 grm. or 2·2 per cent. on the dry skin.

As, however, the original solution was shown by the copper test to contain free chromic acid equal to 0·018 grm. of dichromate, which would certainly be absorbed by the skin, this would leave an absorption of actual dichromate of only 0·004 grm. or 0·4 per cent.

Liquor II.—This contained the same volumes of solution as I. and the same solid constituents, with the addition of 2 grms. NaCl. As the result of repeated titrations, 10 c.c. required 14·95 c.c. of N/10 NaOH, equivalent to 8·07 c.c. of N/1 NaOH or 1·187 grm. dichromate, showing a total absorption of 0·026 grm. or 2·6 per cent. on the dry skin. Deducting for free chromic acid as before, the absorption of dichromate is 0·008 grm. or 0.8 per cent. on the dry skin.

In order to confirm and extend these results, a further set of experiments was carried out with hide powder instead of skin. That employed was from the Vienna Research Institute, and intended for tannin estimation. It contained little soluble matter, and about 14 per cent. of moisture.

5 grms. was employed for each experiment, and the solutions were the same as those used before, except that a new 5 per cent. dichromate solution was made with pure dichromate. This showed no evidence of any free chromic acid when tested by adding potassium iodide and starch, no iodine being liberated. The bottles were shaken in the churn for 2½ hours, and allowed to stand overnight. That without salt may be distinguished as IA, and that with salt as IIA. The hide powder was squeezed over and well washed, and the washings made up to 250 c.c. in each case.

Two methods were used in estimation of the dichromate, viz., that with soda and phenolphthalein already described; and the method with potassium iodide described by one of us in this Journal, 1897, p. 413. A measured quantity of the chrome solution, which must not contain more than about 0·05 grm. of potassium bichromate is placed in a stoppered bottle of 200 to 300 c.c. capacity, and 5 c.c. of concentrated HCl and 10 c.c. of a 10 per cent. solution of potassium iodide is added. The bottle is closed and well shaken, and allowed to stand at least a few minutes, and the liberated iodine is titrated with N/10 thiosulphate with addition of a little starch paste towards the end of the reaction. Each c.c. of the thiosulphate corresponds to 0·0049 of potassium dichromate or an equivalent quantity of chromic acid. The results of analysis of the original and residual liquors were as follows:—

STATEMENT OF RESULTS.

Amounts of Bichromate in 25 c.c. of Original Liquor.

—	Original Liquor.	No. IA Liquor.	No. IIA Liquor.
	Grms.	Grms.	Grms.
NaOH method	1·25	1·23	1·21
$Na_2S_2O_3$ method	..	1·225	1·21

Absorption.

—	In No. I. Liquor.	In No. II. Liquor.
	Grms.	Grms.
NaOH method	0·02	0·04
$Na_2S_2O_3$ method	0·025	0·04

Percentage on Weight of Hide Powder.

—	No. I. Liquor.	No. II. Liquor.
	Per Cent.	Per Cent.
NaOH method	0·4	0·8
$Na_2S_2O_3$	0·5	0·8

Since the two methods of analysis practically agree in their results, showing that the whole of the dichromate existed as such in the residual liquors, and since no free chromic acid existed in the original solution, the chromic acid must have been absorbed by the skin in the form of dichromate, and no appreciable reduction of the chromic acid can have taken place. It is evident that the presence of common salt in some way increases this absorption both in the case of wet skin and of hide powder, but the whole quantity absorbed is so small in proportion to the free chromic acid absorbed under similar conditions, which may amount to at least 8 or 10 per cent., that for technical purposes it may be neglected. The result strengthens Procter and Heal's conclusion (*loc. cit.*) that in tanning by the bichromate process the whole of the *free* chromic acid, and that alone, is taken up by the skin, and that in strengthening a bichromate bath, all that is necessary is to restore the total bichromate to its original strength, and to add the same quantity of acid as in the original operation. If this is done, any difficulty which may arise can only result from the accumulation of dissolved animal matter, and on the sulphates or chlorides formed in liberating the chromic acid, and the latter could, of course, be avoided by the use of CrO_3 instead of dichromate to strengthen the bath. The presence of excess of undecomposed dichromate was, however, essential, or at least advantageous, in practical work.

DISCUSSION.

Mr. T. **FAIRLEY** asked whether any reduction took place when bichrome was used; whether the addition of chromic hydrate to the liquor in the bath had been practically employed; and whether animal fibre which had taken up the full quantity of chromic hydrate was likewise saturated towards aluminium and ferric hydrates.

Mr. C. **RAWSON** enquired whether the strength of the bichromate bath described in the second paper could be kept up by the addition of chromic acid; and if the presence of salt (sodium chloride) had any beneficial effect on the tanning; and further, whether the 3 per cent. chromium referred to in the paper was Cr or Cr_2O_3.

Mr. F. W. **RICHARDSON** asked if the absorption of the chromic acid might be explained by dialysis.

Mr. W. M. **GARDNER** asked whether comparative experiments had been made with chromate acidified with sulphuric acid and with hydrochloric acid. In mordanting wool with bichromate the acid used played an important part, hydrochloric being much more effective than sulphuric acid, possibly on account of the formation of chlorochromic acid. He also asked whether ammonia was liberated during the tanning.

Mr. **HERBERT INGLE** asked whether the experiment had been carried out in a bright light or in the dark. If in bright light, then reduction of the chromate might take place as in the carbon printing process, and thus account for the small quantity of chromium fixed upon the leather.

Mr. **GARDNER** pointed out that in alizarine colour printing on chrome-mordanted wool, light had a very great influence.

Prof. **PROCTER**, in reply, stated that in a neutral bichromate bath no reduction seemed to take place, or at any rate only to a minimal extent.

The addition of chromic hydrate to the bath was very early introduced, and to a certain extent worked well—freshly precipitated chromic hydrate being pretty soluble in solutions of chromium salts. The method, however, was more costly than that of neutralising a part of the acid of a salt of chromium with sodium carbonate, and would not yield so basic a solution. He could not say whether fibre saturated to chromic hydrate could take up aluminium hydrate, but the fact that leather tanned with alum was apparently easily converted by substitution into chrome leather rather indicated that fibre saturated towards one of the hydrates was also saturated towards the others. Chrome seemed to fix itself better than alum in tanning, for chrome leather might be boiled with water without undergoing any change, but alum leather by the same treatment was easily brought back to an almost raw-hide condition.

The addition of bichromate to the bichrome bath brought about certain complications—notably the concentration of alkali salts, and it was customary for manufacturers to discard half-spent liquors rather than risk getting a poor quality leather from a re-made bath. Free chromic acid had not been commercially used for strengthening the bath, but would probably succeed if the exact quantity required could be determined. The common salt played no chemical part in the action, yet its presence was advantageous, for a softer leather was produced. One of the effects of sodium chloride in slightly acid solution on gelatinous fibre was a dehydrating action, and an acidified salt solution alone was capable of producing a sort of leather. The 3 per cent. of chromium referred to 3 per cent. of metallic Cr on the weight of the leather. The absorption was, no doubt, very closely connected with osmotic phenomena. Hydrochloric and sulphuric acids did not appear to behave differently in chrome tanning, except that in the former case some sodium chloride was formed. As the process was carried out at a low temperature it was not to be expected that ammonia would be formed, and no evidence of its presence was noticed.

Practically it was not advisable to expose skins in the yellow chromed condition to strong light, but during the tannage itself the skin was protected by the yellow colour of the chrome liquors to a considerable extent. Reduction by light did not proceed so far as to the production of green chromic salts, but stopped at the formation of brown chromo-chromates.

Obituary.

WILLIAM THORP, B.Sc. (LONDON).

A VICE-PRESIDENT OF THE SOCIETY OF CHEMICAL INDUSTRY (1897—1900); FORMERLY CHAIRMAN OF THE LONDON SECTION (1892—1894).

WILLIAM THORP was the son of the late Wm. Thorp, a London Brewer, and was born in the year 1846. He was educated at the City of London School, then in Milk Street, Cheapside, where he took the class prize in Chemistry. His first scientific training was received from the late Thomas Hall, B.A., for whom, as well as for the old school and its memories, he always evinced a deep affection. He was to the last a member of the "Old Boys' Re-union" as well as of the "John Carpenter Club" established 1851, and named after the founder of the City of London School.

From 1864 to 1867 he studied at the Royal College of Chemistry under Professors Hofmann and Frankland, and was eventually appointed Principal assistant and afterwards Chief Chemist under the latter, in the Laboratory of the Royal Commission on the Pollution of Rivers. In this capacity he was responsible for the analytical work in connection with the cognate subject of Water Supply, extending over ten years, when the Rivers Pollution Commission presented its celebrated report. In July, 1868, in consequence of the sudden and accidental death of Mr. C. H. Berger, of the firm of Lewis Berger and Sons, some promising experiments were left in an unfinished state, and the firm arranged with William Thorp to complete the research. This arrangement resulted eventually in his accepting the post of consulting chemist to the firm, whilst continuing the work of the Rivers Pollution Commission.

In 1875, the Commission having presented its report, Wm. Thorp was appointed chief chemist to Messrs. Lewis Berger and Co., and for twenty years his wide knowledge, sound judgment, and resourceful mind were devoted to the business. He was particularly successful in the preparation and treatment of vermilion, and carried out many experiments on the manufacture of white lead. In 1879, when the firm was converted into a Limited Liability Company, he acted as a Director and Assistant General Manager. At this period the firm established a white lead works at Sheffield, and the chemical departments there and in London were under his direct supervision. In 1895, he retired from active participation in the business, retaining, however, the position of consulting chemist until 1897.

In the same year (1895) he started practice at Victoria Mansions, Westminster, as a consulting chemist, taking over the laboratories of his friend, Mr. G. J. Snelus, F.R.S. His work here included an investigation on several new technical processes and particularly a research on the employment of cerium in the manufacture of steel.

Mr. Francis Sutton of Norwich testifies to the great assistance rendered him by his friend William Thorp, not only in revising his work on Volumetric Analysis, but for valuable additions to the subject of Water Analysis.

Of the Technological series, known as "Groves and Thorp's Chemical Technology," three volumes have been issued. In vol. I., devoted to "Fuel," W. Thorp took no special part, but in vol. II., on "Lighting," he participated, and wrote a large portion of vol. III., almost completing the subject of "Gas Lighting."

In 1866 William Thorp was elected a Fellow of the Chemical Society, and in that and the following year he contributed papers to the Society in conjunction with the late E. T. Chapman, from the Laboratory of the London Institution, "On the Relation between the Products of Oxidation and the Molecular Constitution of the Bodies oxidised," Parts I. and II., which appeared in the Journal. In 1880 he was elected a member of the Council of the Chemical Society. He was also a Fellow of the Institute of Chemistry, and at the time of his death, his name was standing for election as a member of the Council. He was a member of the Physical Society and also of the German Chemical Society.

William Thorp had been a member of the Society of Chemical Industry from its commencement in 1881. From 1892 to 1894, he filled the office of Chairman of the London Section. In 1891, he became a member of the Publication Committee; and though not enjoying good health, he rendered invaluable service on the editorial and Collective Index committees, in which latter he took a leading part.

In July 1894, he was elected an ordinary member of Council and became a vice-president in 1897.

Of a retiring disposition, comparatively few of those with whom he came in contact really learned to know him. Those who did however, bear unanimous testimony to his courtesy, liberality, breadth of view, consistency, and unimpeachable integrity. He was one of the most loyal of friends. An old and intimate friend and colleague writes of him :—"William Thorp was the soul of honour, and I know no one whom I would more implicitly trust"; and another friend, "He was one of the most lovable men I ever knew."

For many years he must have suffered much and constantly, but he was never heard to complain and was always cheerful and animated.

He died suddenly of spasm of the heart, on February 13, aged fifty-three years.

Journal and Patent* Literature.

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

I.—PLANT, APPARATUS, AND MACHINERY.

Stamp Mills [Gold]; Development of Gravitation. D. B. Morison and D. A. Bremner. Trans. Inst. of Min. and Metall., Jan. 7, 1900.

See under X., *page* 249.

PATENTS.

Furnaces for Melting Glass or Metal, and for Melting or Heating other Substances. R. W. James, London. From The American Stoker Co., New York, U.S.A. Eng. Pat. 2504, Feb. 3, 1899.

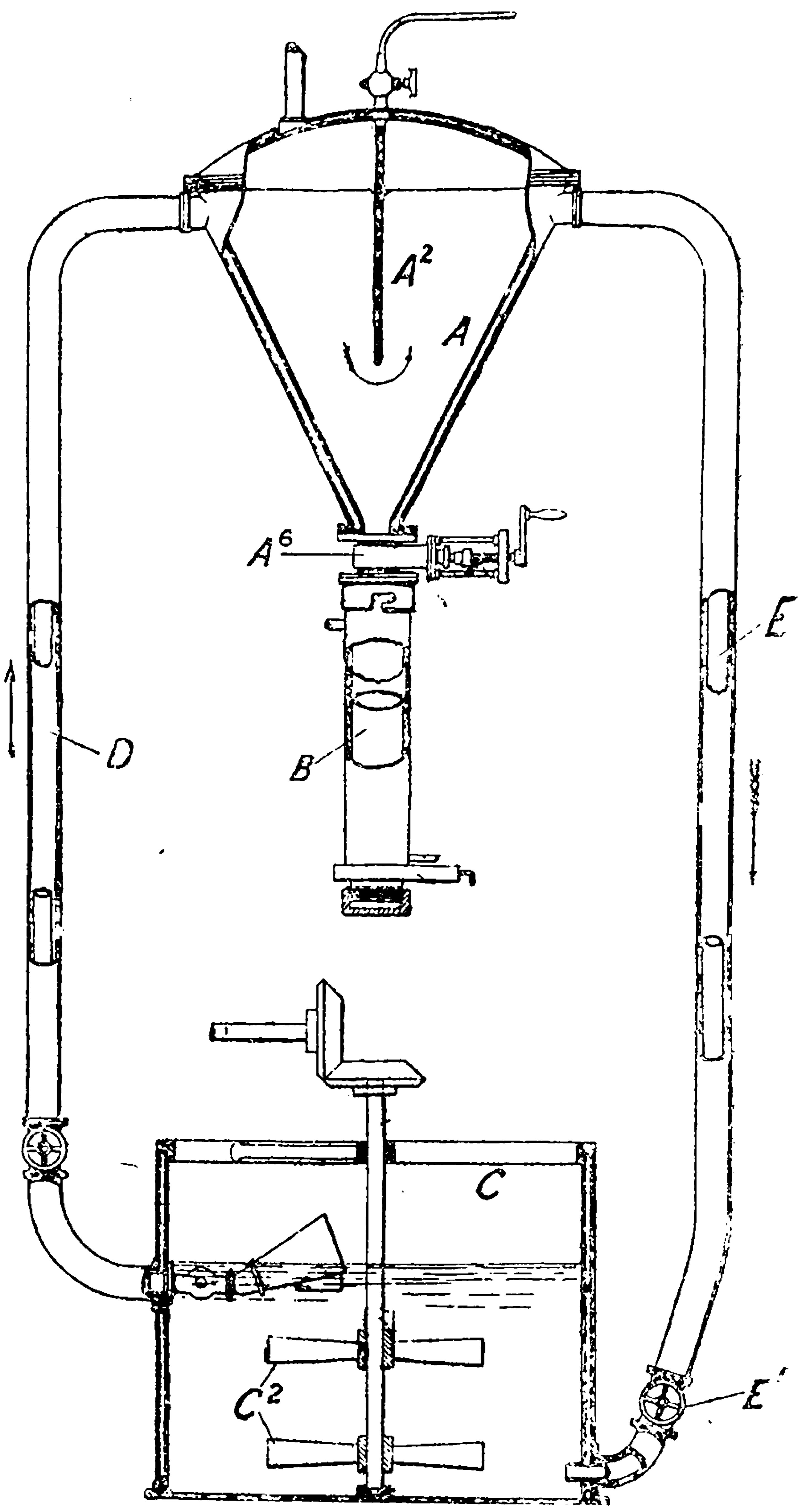
These furnaces are of the same kind as those described in this Journal, under VIII., page 247 (Eng. Pat. 2503), but there is a fuel feeding device at each end of the hearth; also, the system of flues therein described is not used, their place being taken by a regenerator at each end outside the heating fires. The parts claimed are the working hearth, the gas generators, each comprising an underfeed stoker and a chamber above the stoker for incandescent coke arranged within the shell of the furnace, and the regenerators also within the furnace shell, and alternately heated by the waste combustion products and heating the fresh air.—R. S.

Furnaces, Regenerative; for Heating Gas Retorts or for Analogous Purposes, and Special Construction of Bricks to be employed therein. J. H. Brearley, Longwood, Yorks. Eng. Pat. 7210, April 6, 1899.

Claim 1 is for the construction of bricks or slabs for use in building the furnaces or forming the dividing walls, so as to prevent leakage of gases, &c. Claim 2 describes the particular construction of brick, &c., intended, namely (*a*) with a longitudinal V- or other suitably shaped rib or ribs on one face engaging with a corresponding recess or recesses, (*b*) with half-circular or other suitably shaped grooves all around the brick, which grooves are afterwards filled with fire-clay, &c.; and (*c*) with opposing recesses at each end of the brick to form overlapping or rebated joints. Claim 3 is for U-shaped channels for conveying secondary air supplies to the combustion chambers. The last claim, No. 4, is for the combination of such bricks, passages, U-shaped channels, a longitudinal baffle wall or bridge on the crown of the producer arch, and method of controlling the distribution of secondary air supplies to the U-shaped channels.—R. S.

Separation of Solids [Pigments, Dyes, Fibres, &c.], from Liquids containing them. A. Ross, Sutton, Surrey. Eng. Pat. 3493, Feb. 16, 1899.

The separation is effected within the chamber A, which is connected by the valved pipes D, E, to the vat, boiler, or other container C. The liquid is agitated within the vat by paddles C^2, or by steam jets, &c., and is caused to circulate through the parts D, A, E, in the direction indicated by the arrows. The chamber A is provided with baffle-plates A^2, and the solid matter deposited passes through the valve A^6 into the receiver B, which is made detachable, so that, when filled, it can be replaced by an empty one, and is also provided with means for ejecting the contents. Two other forms of receiver are also described in the specification. The parts A, C, &c., may be jacketted, as shown, for the application of a heating or cooling medium to the system. In a modification, the pipe E delivers into a separate vessel, from which the liquid is drawn by a siphon, the circulation of the liquid from the vat C through the parts D, A, E, being also effected by siphon action.—R. A.



Filtering Apparatus. H. H. Lake, London. From Allgemeine Städtereinigungs-Gesellschaft mit Beschränkter Haftung, Wiesbaden, Germany. Eng. Pat. 4996, March 7, 1899.

A perforated cylindrical chamber, and a similar smaller chamber arranged eccentrically within the larger one, the filtering material partially filling the interspace between the chambers. This apparatus is revolved in a reservoir containing the liquid to be filtered, in which it is immersed, the liquid passing through the outer chamber and the filtering material into the inner chamber, from which the filtered liquid is discharged through a passage in one of the trunnions. The rotation of the apparatus is provided for to facilitate cleansing, by agitating the filtering material so as to free it from loose particles of impurities whilst a current of liquid is passed through it. The apparatus is applicable to the purification of drain-water in towns.

—R. A.

Concentrated or Viscid Solutions for Obtaining Solid Particles therefrom in the Form of Powder, Treatment of. J. Künstner, Obersedlitz, Austria. Eng. Pat. 6127, March 21, 1899.

The concentrated or viscid solution is heated to a high temperature in a closed vessel under pressure, and is then caused "to enter a large empty space," wherein it is brought, by the sudden reduction of pressure, and without the use of an atomizer, into a finely divided state, which facilitates the evaporation of the water, and the formation of the powder. The process is stated to be specially applicable for the manufacture of soap powders.—R. A.

Desiccating Granular and Slimy or Pasty Materials [Phosphates, Earth, Marl, Ore, Distillers' Grains, &c.] or the like; Process of and Apparatus for. G. Devaux, Bretueil, and H. Diedrich, Paris. Eng. Pat. 19,277, Sept. 25, 1899.

Air, which is first dried around a desiccating furnace, and mixed or not with the combustion gases from the furnace, is caused to circulate through a column or chamber in counter-current to a stream of the material under treatment, the material being constantly turned and stirred, so that it is all acted on by the desiccating gases. In the apparatus employed, the chamber is fitted with superposed plates, alternately perforated at the centre and the periphery, and the movement of the material is effected by ploughs, which are carried on a rotating vertical shaft, and are arranged to move the material from the centre to the periphery, and from the periphery to the centre, of successive plates.—R. A.

Cooking, Concentrating, and Evaporating Liquids [Sugar Solutions, &c.], Apparatus for. E. Shaw, London. Eng. Pat. 21,300, Oct. 10, 1898.

See Eng. Pats. 5803 and 19,262, of 1898. The apparatus comprises an externally heated coil or tube, a feed pump connected to one end of the tube, a collecting chamber, in which the concentrated liquid is separated from the vapour generated in the tube, in communication with the exit end of the tube, and means for varying the pressure in the said chamber. To prevent the variations of pressure within the chamber from affecting the feed, a valve, which will not open at less than atmospheric pressure, is placed in the communication between the pump and the tube.—R. A.

Emulsions: Apparatus for Use in the Making of. G. J. Blore, Manchester. Eng. Pat. 23,527, Nov. 25, 1899.

The apparatus comprises a cylindrical receptacle fitted with a cover, and a perforated dasher within the receptacle. The spindle of the dasher preferably passes through a packing gland in the cover, and is fitted with an operating handle. The cylinder may be made of cast iron, &c., and fitted with a tinned or enamelled lining, or may itself be tinned or enamelled, or it may be made of porcelain, &c. The dasher is of dome shape, to increase its action, and the bottom of the receptacle is of corresponding form. Suitable provision is also made for filling and emptying the receptacle.—R. A.

II.—FUEL, GAS, AND LIGHT.

Acetylene Gasholder: Explosion of an. T. Gigli. Chem. Zeit. 1900, **24**, [5], 37—38.

A copper gasholder of nearly 30 litres capacity was filled with acetylene in May, and about half the gas was used within a few days. The holder, half-filled with acetylene, was then allowed to stand until Nov. 8, when the acetylene was completely expelled by water. The holder was afterwards filled with oxygen, of which about a third was used within a few days, and the holder was again let stand. On Nov. 15, the slight vibration caused by mere handling caused a violent explosion, and the holder was shattered. The fragments were found, when dry, to be coated on the inside with a nearly black incrustation, which ignited and burnt with vivid scintillations when touched. The author concludes that this incrustation must have consisted of copper acetylide, which is known to be formed from acetylene and cuprous oxide in the presence of ammonia. Bullier's researches indicated that acetylene did not attack metallic copper, but as the incrustation on the fragments of the wrecked holder was considerably thicker than the layer of oxide which is commonly found on old copper, the author considers that in this case the acetylene had attacked the copper underlying the thin layer of oxide on the interior walls of the holder. The accident, he thinks, should serve as a warning against the use of copper gasholders for acetylene.—J. A. B.

Acetylene; Action of, on Copper, Formation of a Hydrocarbon, Cuprene. P. Sabatier and J. B. Senderens. Comptes Rend. **130**, [5], 250–252.

A current of pure acetylene is directed upon copper at a temperature of about 180° C. in the case of reduced copper, of 250° C. in that of copper foil or wire. For some time the gas is absorbed without apparent change in the structure of the copper, whilst colourless liquids, which are hydrocarbons of the ethylene series, condense in the cooler parts of the tube. After absorption is nearly complete, however, the copper begins to swell very considerably in bulk, till it ultimately chokes the tube. At the same time its colour becomes lighter, the condensed liquids become greenish, and now consist of a mixture of ethylenic and aromatic hydrocarbons, and gas escapes from the far end of the tube, and is found to consist chiefly of ethylene hydrocarbons, with some ethane and hydrogen, and a little acetylene. On removing some of the solid substance from the tube, and again exposing it to acetylene, it swells further in bulk, ultimately becoming a soft light or dark yellow substance, composed of an assemblage of matted filaments, and after gentle compression, strongly resembles touchwood. It burns in air with a smoky flame and an aromatic odour, and leaves a slight residue of copper oxide. It is a hydrocarbon of high molecular weight, having the empirical formula C_7H_6, and the authors propose to call it *cuprene*.—J. T. D.

Standards of Light; An Experimental Research on some. J. E. Petavel. Proc. Royal Soc. **65**, [422], 469—503.

The Harcourt pentane gas standard, and the Hefner-Alteneck amyl acetate lamp, have proved the most satisfactory of the numerous flame standards of light which have been proposed. They are, however, seriously affected by variations in the amounts of moisture and carbonic acid in the atmosphere. With reference to incandescent standards, the author found that the crater of a "silent" electric arc had an intrinsic brilliancy of about 147 candle-power per sq. mm., but that under the most favourable conditions, variations exceeding 5 per cent. were common. The Lummer and Kurlbaum standard, consisting of a strip of platinum foil, rendered incandescent by an electric current, of which only one-tenth of the total radiation is transmitted through a water-trough and utilized, he found to give a constant light, but the difficulties of reproducing the standard were almost insuperable, and the light displayed an undue preponderance of red rays. Violle's molten platinum standard he found could be reproduced only with great difficulty, but after many trials he arrived at the conclusions that under certain conditions the probable variation in the light emitted by molten platinum is not above 1 per cent., and that under improved conditions it might be still further reduced.—J. A. B.

Incandescence Mantles; Luminosity of Auer. H. Thiele. Ber. **33**, [1], 183—187.

For investigating the cause of the luminosity of incandescence mantles, it is difficult to find a source of heat which is quite free from objection. If the materials are enclosed in an apparatus, the walls of which become incandescent, the photometric results are vitiated by reflection; no apparatus constructed of graphite, nor even the electric arc between carbon pencils, is permissible, in view of the "catalytic" theory and the possible presence of combustible gases. Wehnelt's interrupter (Wied. Annalen, **68**, 260) produces in a simple way high-pressure currents, which can maintain arc-like phenomena for a considerable time, between platinum terminals, although the cathode is apt to melt.

An Auer mantle (thoria, 99; ceria, 1 per cent.) examined in the Wehnelt "arc" gave the same characteristic light as in the Bunsen flame. A mantle of magnesia, however, the luminosity of which in the Bunsen flame was not great, showed no appreciable difference from the Auer mantle when placed in the Wehnelt arc. In these tests when the mantles were moved, it was noticed that the stream of sparks followed the heated parts to some extent, so that apparently the insulation-resistance had been reduced by the rise of temperature, and the heat evolved by the conduction of the current would be a disturbing factor. Experiments with the following thoria-ceria mixtures were next made, as it seemed improbable that the maximum Joule effect would occur with just that composition which has the maximum illuminating power :—

Thoria.	Ceria.	Thoria.	Ceria.
Per Cent.	Per Cent.	Per Cent.	Per Cent.
100	0	90	10
99	1	75	25
98	2	50	50
97	3	0	100
95	5		

In the Bunsen flame the maximum illumination was given with 1—2 per cent. of ceria. In the Wehnelt arc the first members of the series showed no surprising differences, but with an increase of the ceria, the luminosity fell off quickly. But it was clear that the conductivity influenced the results, ceria having a much lower resistance than thoria.

With a sufficient tension of primary current there appears, over the Wehnelt arc proper, a flame-like phenomenon, which is apparently due to heated air. On immersing the thoria-ceria mixtures in this flame, successful results were obtained with those containing a moderate amount of ceria. With those rich in ceria, it was difficult to put the mantle far enough into the flame, as the electric arc was readily deflected on to the mantle. Under these conditions the maximum luminosity was obtained with the same percentage of ceria as is used in Auer mantles, and the author concludes that the high luminosity is a specific function of this mixture of thoria and ceria.—H. B.

Carbon Monoxide; Volumetric Determination of Small Quantities of. L. P. Kinnicutt and G. R. Sandford. J. Amer. Chem. Soc., 1900, **22**, [1], 14.

See under XXIII., *page* 275.

PATENTS.

Fuel; Apparatus for the Manufacture of Artificial or Patent. F. E. Finlayson, Swansea, and C. Cory, Swansea. Eng. Pat. 2231, Feb. 1, 1899.

The apparatus consists of a combined drying and mixing cylinder. The materials are fed into the upper or mixing portion, and pass through an adjustable opening to the lower or drying portion, through which hot air, furnace gases, &c. are passed, and from which the material passes to the moulds. A central vertical shaft passes completely through the cylinder, being provided in the upper part with mixing arms, and in the lower with a series of flat discs having sector-shaped openings, through which the material is forced, so as to drop to the disc below, by means of blades or scrapers secured to the side of the cylinder.
—R. S.

Fuel; Process and Means for the Conversion of Earth or Soil into. F. Kresser and G. Montag, Mannheim, Germany. Eng. Pat. 8067, April 18, 1899.

The fuel consists of from 92 to 94 per cent. of ordinary soil or earth, to which is added 6 to 8 per cent. of a material prepared by intimately mixing boiling pitch or resin with sawdust and earth, or sawdust or earth, the mixture being intimately stirred, simultaneously adding a half to one per cent. of 5 per cent. sulphuric acid and water. To the boiling pitch or resin, for the purpose of rendering it odourless, there may be added one quarter per cent. of any desired oil.
—R. S.

Furnaces; Impts. in and relating to Prevention of Smoke and Economisation of Fuel. A. G. Brookes, London. From E. Ott, Berlin. Eng. Pat. 12,489, June 15, 1899.

In these furnaces steam is sent through the fire bars, and claim 1 is for "the method of utilising a mixture of fire gases and fresh air as a vehicle for the introduction of steam into the fire grates of furnaces, in order to effect the prevention of smoke and the economisation of fuel therein." The five subsequent claims are for the construction of a tubular boiler furnace, and of a flame tube boiler furnace, arranged according to this method. There is a chamber connecting the ashpit and the channel, which leads the fire gases to the chimney, air pipes opening into such chamber, and a fan in the chamber to draw the fire gases and fresh air into the chamber, and to force the mixture into the ashpit. The ashpit has a water-distributing pipe, so as to mix finely divided water with the gaseous mixture.—R. S.

Blast-Furnace and other Gases; Apparatus for Separating Dust and the like from. A. Hebelka, Coblenz, Germany. Eng. Pat. 20,150, Oct. 7, 1899.

The blast furnace or other dust-laden gases pass into the top of a cylindrical chamber, and thence down to an outlet near the bottom. Across the chamber are a number of inclined deflector plates, upon which the dust falls. The dust slides down to the bottom of the plates, and thence out through a small opening to one or two narrow vertical channels, in which the dust accumulates.—R. S.

Furnaces; Apparatus for Injecting Liquid and Pulverised Fuel into. E. A. Vetillard and E. Scherding, both of Paris. Eng. Pat. 23,238, Nov. 21, 1899.

Injectors of the kind described in Eng. Pat. 8589, 1899 (this Journal, 1899, 819), have "an apparatus with mixing jets, of which apparatus a first injector takes in the air, which mixes with steam ; this mixture, having reached the opening of a second and conical injector, drawing up through a central pipe, the pulverised fuel and air, and the whole of the fuel mixed and well diffused being projected into the furnace, passing through a gasified mixture of liquid fuel, steam, and air; this gasified mixture being produced by a third injector, drawing up the liquid fuel, mixing it with the steam, and injecting it in the form of a cylindrical jet, whilst two co-axial injectors convert this cylindrical jet into an annular jet, caught between a layer of external air and the mixture of pulverised fuel and air entering through the internal pipes, the result being that the pulverised and liquid fuel, the steam and the air, are thoroughly mixed and diffused." There are also claimed for use with these injectors, a funnel, a distributing and mixing box for the pulverised fuel, and the arrangement of pipes and cocks described.—R. S.

Coke, Charcoal, and the like; Manufacture of, and Apparatus therefor. A. Schenk, Bergedorf, Germany. Eng. Pat. 23,085, Nov. 20, 1899.

The applicant claims "the process for increasing the solidity and density of coke, charcoal, and the like, which consists in charging the ovens or furnaces, in which the coking material is brought to the necessary temperature, with gases or vapours containing carbon, without admission of air, or with substances which are capable of generating gases or vapours containing carbon." He also claims the various forms of apparatus illustrated. In three cases these consist of arrangements whereby the gases, arising when fresh fuel is heated, are caused to pass through the material, already charred, and subjected to an intense heat. In the other case, such intensely heated material exhausted of gaseous products is subjected to a stream of gases, or vapours, or tar, oil, or the like, containing carbon.—R. S.

Liquid Fuel Burner. G. W. White, Chattanooga, U.S.A. C. Bradshaw, Birmingham, U.S.A.; O. T. Gregory, Paducah Co., U.S.A. Eng. Pat. 24,684, Dec. 12, 1899.

These burners are called "fire logs" and are intended to be placed in the fire chambers of stoves, furnaces, boilers, or the like, or in open fireplaces. Each burner or "log" consists of a horizontal tube or cylinder, closed at each end, and provided at its centre with an inlet or oil supply pipe on its lower side, and with an escape pipe on its upper side. The escape pipe leads the gas to a horizontal pipe parallel with the log, and which is provided with a number of branch tubes projecting backwards, and having several small jets. One of these jets is directed upon the "log." The burner is first heated by oil placed in a pan beneath it. When heated, the gas is vaporised and is then burned at the orifices, the special jet playing upon the burner, heating the latter sufficiently to vaporise the oil used, so long as the burner is in use.—R. S.

Gas Burners for Heating Purposes. B. J. B. Mills, London. (From La Société Anonyme des Fontaines à Gaz, Fontaines-sur-Saône, France.) Eng. Pat. 3552, Feb. 17, 1899

THE claims are for "A gas heating apparatus, consuming low-pressure gases characterised by the supply of air being obtained by means of the combustion of the gas burning on the surface of one or several wire-gauze tubes of suitable form, arranged horizontally or vertically, the said supply of air being obtained partly through the wire gauze and partly through exterior openings arranged around the jets supplying the gas to the burners." Also for a movable sleeve to regulate the size of the air-inlet openings round the gas jet, and an arrangement for fastening the wire gauze, so that it can be replaced when desired.—C. S.

Water-Gas; Manufacture of. A. J. Boult, London. (From E. Fleischer, Dresden-Strehlen, Germany.) Eng. Pat. 4495, March 1, 1899.

TWIN generators are employed and are heated to different temperatures. Coal is then added to the cooler of the two and steam blown through it in the same direction as the air blast, the resulting gases being afterwards conducted through the second generator, in a direction opposite to the course taken by the air blast in heating up. Or the process may be modified by alternating the procedure so that one generator, after being heated by a blast, and before being re-heated, is employed first for the second phase of the process, and then for the first phase of the following operation, only the less highly heated generator through which steam had first been passed being re-heated by a hot blast after every completed gas period, the other generator from which the water gas was finally evolved at the last gas period and which is not then re-heated by a hot blast, then receiving the steam current for the next generation of gas. In another modification the air-blast phase is carried out as in Eng. Pat. 29,863 of 1896.—C. S.

Gas, Manufacture of Coal and other Illuminating. H. G. Colman, Birmingham. Eng. Pat. 4742, March 4, 1899.

IN order to increase the illuminating power of the gas and more completely remove the naphthalene, the inventor eliminates the tarry matters (preferably by centrifugal force) whilst the gas is still warm (100°—140° F.) and on the way to the condensers. The next stage is to divert a portion of the gas before the condenser is reached, this portion being afterwards added to the cooled gas in the condenser, so that the hydrocarbons in the former are returned to the bulk at a time when they can be retained and increase the illuminating power. Finally, before entering the condenser, the gas is passed through a washer where it bubbles through a seal of heated liquid hydrocarbons, whereby the naphthalene is removed and the gas enriched with the vapour of volatile hydrocarbons.—C. S.

Acetylene Gas Burners. E. J. Dolan, Philadelphia (Pa.), U.S A. Eng. Pat. 6116, March 21, 1899.

To produce a brilliant, flat, smokeless flame, and provide sufficient air for perfect combustion and cooling the gas tip, the inventor employs a tip pierced by an arched slot not exceeding $\frac{14}{1000}$ths of an inch in width, $\frac{30}{1000}$ths in. in depth, and $\frac{3}{16}$ths in. in radius. This is covered by a cap with a larger slot directly above that of the tip, and arranged so as to leave an air chamber, into which air is admitted at a point intermediate between the two slots. The same procedure may be applied to multiple-flame burners.

—C. S.

Gas, Apparatus for Washing. N. A. Guillaume, Paris. Eng. Pat. 11,868, June 7, 1899.

THE apparatus, which is worked on the counter-current principle, consists of a number of superimposed rectangular troughs fitting tightly together by ribs and grooves, vertical screws and bevel-wheel gear being provided in the columns of the frame in order to keep the joints tight. The troughs are each divided into a large compartment and a small one, the latter serving as vertical conduit for the outlet of the washed gases. Water communication between the washing troughs is effected by a series of splayed tubes dipping into cups in the next lower trough in each case. The gas enters through long narrow openings at the bottom of the apparatus, and is kept in contact with the water by hoods with serrated lower edges; it then escapes at the ends by bubbling up through the water and ascends through the other troughs, being finally discharged from the top one by way of the said conduit. In the lowermost trough the gas is delivered as a horizontal sheet to meet the overflow water from the upper troughs, and is then passed through the series described.—C. S.

Gas Generators. N. A. Guillaume, Paris. Eng. Pat. 12,139, June 10, 1899.

IN gas generators operating by suction, the inventor claims "a fuel charging device comprising a hopper closed at its upper end by a hinged lid or cover secured in its closed position by hinged bolts and nuts, a sliding plate for closing the bottom of the hopper and a bolt or retaining device for securing the said plate in its closed position, a system of levers connecting the securing bolts for the top cover to the bolt or retaining device for the sliding plate or "button cover" so that both covers cannot be open at the same time, and springs arranged beneath the sliding plate to press it against the bottom of the hopper to prevent vibrations of the plate by the action of the suction pump or motor."—C. S.

Petroleum and other Hydrocarbons, Process and Apparatus for Gasifying. A. J. Boult, London. From C. de Haupt, Brussels. Eng. Pat. 23,724, Nov. 28, 1899.

THE process consists in circulating a mixture of superheated steam and finely divided or vaporised hydrocarbons (together with air so as to produce partial combustion and generate greater heat) in a conduit heated sufficiently to produce decomposition of the steam, "the oxygen of which combines with the carbon of the hydrocarbon to form an oxide of carbon which becomes mixed with the hydrocarbon evolved by the process."

The apparatus comprises two serpentine or other tubes, one serving as a steam generator, the other as a gasifier, and both provided with feed-tubes, injectors and regulating screws, the hydrocarbon feed being also fitted with an air-inlet. The generator tube also communicates with the tube through which the hydrocarbon passes to the gasifier, "the end of the latter tube forming a conduit, for the gas produced, to the cooling, purifying or like apparatus."—C. S.

Gas Scrubbers. G. Zschocke, Kaiserslautern, Germany. Eng. Pat. 24,056, Dec. 2, 1899.

THE inventor claims an improved scrubber with perforated loose scrubbing material placed in a number of compartments in which they are caused to roll, one over another, by the action of perforated wings or vanes mounted on a rotating central shaft.—C. S.

Gas from Gasoline and other Inflammable Liquids; Device applicable to Lighting and Heating Apparatus for Generating. M. Posno, Paris. Eng. Pat. 24,203, Dec. 5, 1899.

THE device comprises a lamp body, a main supply wick, a gas-generating chamber, and an oblique auxiliary burner, which latter is provided with an air tube opening into the burner, and also with a second tube surrounding the lower end of the burner tube and the wick and dipping into the liquid fuel. The auxiliary burner is employed to heat one or more tubes to which the liquid to be vaporised is fed by the main wick, and which unite in the generating chamber leading to the main burner.—C. S.

Acetylene Gas; Apparatus for Generating. The Read-Holliday Acetylene Company, Ltd., W. Cardno and R. Holliday, all of Huddersfield. Eng. Pat. 7325, April 7, 1899.

A WATER container is attached by screw fastenings and an elastic packing ring to a subjacent chamber, containing receptacles filled with carbide, to which water is fed, drop by drop, through one or several valves, from the water container.—J. A. B.

Acetylene Gas, Apparatus for Generating. A. Rouma, Liége, Belgium. Eng. Pat. 24,364, Dec. 7, 1899.

A DRUM, with chambers packed with carbide, is rotated on a vertical axis by means of a weight and cord, so that each chamber in turn discharges its contents through a shoot into water. The rotation of the drum is interrupted, through the action of a special controlling device, when the bell of the gas-holder, into which the gas evolved passes, rises above a certain point.—J. A. B.

Acetylene Lamps. F. W. Barratt, Wimborne, Dorset. Eng. Pat. 2699, Feb. 7, 1899.

WATER percolates on to carbide from the lower chamber of a water container. The upper chamber replenishes the lower through a pipe leading from the bottom of the former to a short distance above the bottom of the latter.—J. A. B.

Acetylene and other similarly obtained Gases; Apparatus for Generating. H. R. Bean and H. R Ringwood, both of Plaistow, Essex. Eng. Pat. 6029, March 20, 1899.

A CARBIDE holder comprises several compartments having inlet tubes for water at different levels. The compartments are bolted together, and when the whole is immersed in water, they operate in turn.—J. A. B.

Acetylene Gas, Apparatus for Generating. J. W. Robinson and H. Hadley, both of Enfield, Middlesex. Eng. Pat. 6328, March 23, 1899.

WATER is supplied to carbide in generating vessels from a tank placed above a bell gasholder through a pipe with a valve operated by a lever actuated by the rise and fall of the bell of the gasholder, which receives the generated gas. When the bell is raised to the full extent, and the valve is closed, gas subsequently generated displaces water from the gasholder tank by forcing it into the superposed tank. —J. A. B.

Incandescent Vapour or Gas Burners. P. R. J. Willis, Kingston-on-Thames. From A. A. Arnott, Topeka, and W. A. Granville, New Haven, U.S.A. Eng. Pat. 1607, Jan. 24, 1899.

THERE is described in great detail an incandescence vapour burner in which liquid hydrocarbon is vaporised, the vapours mingle with air, and burn beneath an incandescence mantle as usual.—H. B.

Incandescence Mantles; Apparatus for Incinerating. C. Clamond, Paris. Eng. Pat. 2178, Jan. 31, 1899.

THERE is described and claimed apparatus by which the burning-off of impregnated mantles is effected automatically. The mantle is suspended from a hook which is attached to a vertically sliding rack, geared to clockwork, and carrying a downwardly inclined Bunsen burner. The fabric is first burned off from the top downwards, the clockwork extinguishing the Bunsen burner at the proper time. The mantle is then lowered over a Bunsen burner, the strong flame of which, burning within the mantle, plays upon it as it is gradually raised by the clockwork. The mantle is now again lowered and subjected to a strong radiating Bunsen flame inside, and a strong Bunsen flame on its top during its gradual ascent, the incineration being thus completed.—H. B.

Electrical Incandescence Lamps of the Nernst Type. V. I. Feeny, London. From the Allgemeine Elektricitäts Gesellschaft, Berlin. Eng. Pat. 45, Jan. 1. 1900.

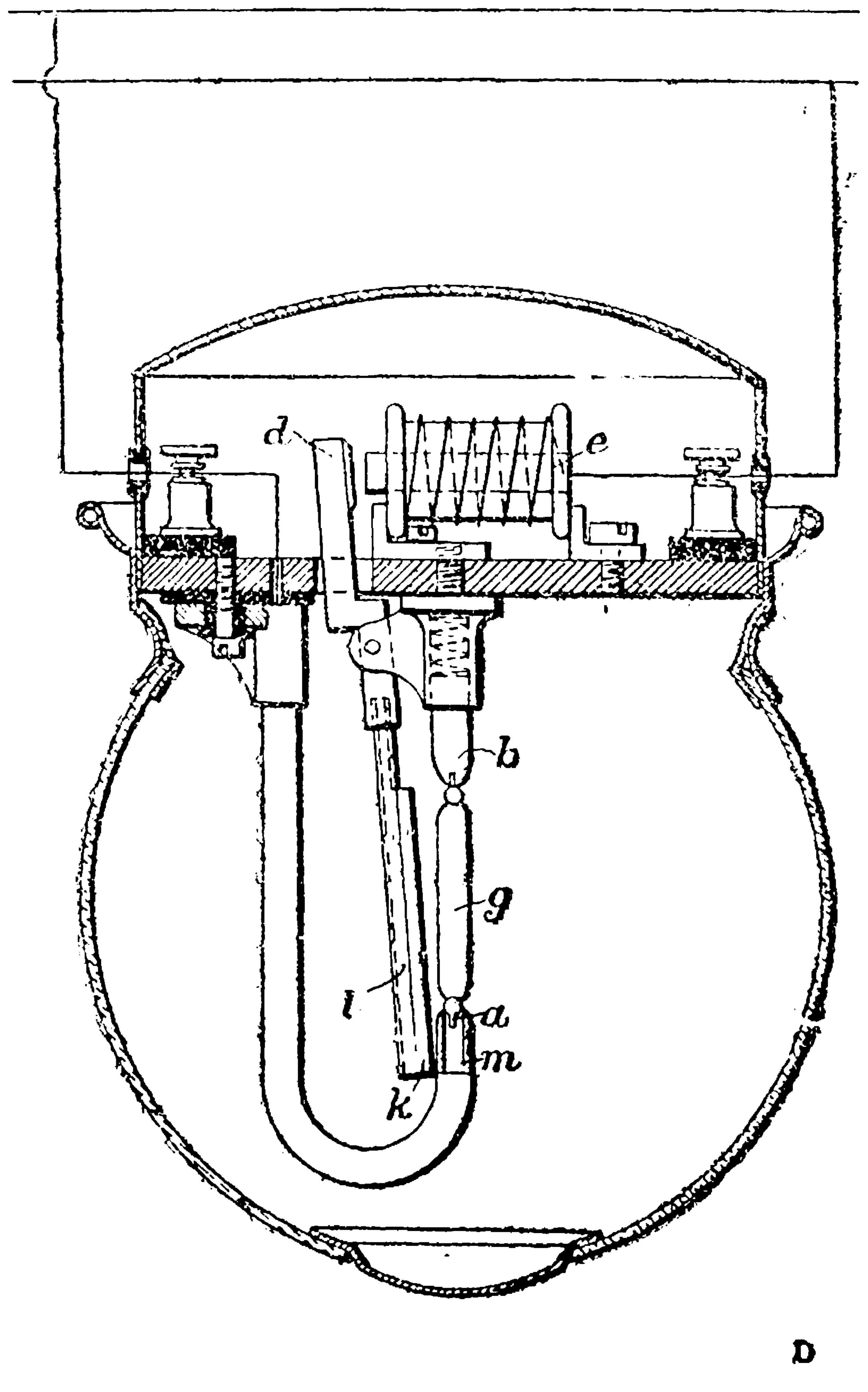
FOR heating the filaments of lamps of the Nernst type (Eng. Pat. 23,470 of 1897; this Journal, 1898, 1031) to the temperature at which they become conductors, heating bodies are used, which are themselves capable of emitting light during the heating period, and which are cut out of the circuit by means of an automatic current-interrupting apparatus when the Nernst filament has become incandescent. In one form of lamp the Nernst filament is suspended inside a small tube, which is closed at the lower end; round the outside of the tube there is coiled an ordinary carbon filament which is surrounded by an ordinary vacuum bulb, and when the current is passed through the carbon filament, it quickly heats the Nernst filament. In other forms both the Nernst filament and the heating body are enclosed within the vacuum bulb. The carbon filaments may be replaced by silicium or silicium-carbon filaments, when both filaments may be used in free air, "since the use of the silicium filament for exciting the incandescence rod only lasts for some fractions of a minute, and thus a total durability of a few hours is amply sufficient for practical purposes."—H. B.

Electric Incandescent Lamps of Glowing Refractory Substances. A. Howard, London. Eng. Pat. 4725, March 3, 1899.

THIS relates to improvements upon Jablochkoff's lamp (Eng. Specification, 1996 of 1877). Mica, either alone or coated with other refractory substances, is to be used as the filament or "candle." The filament is to be attached to the terminals by means of screw clamps, instead of soldering. To serve for the preliminary heating of the filament, the latter is coated electrically with magnesium or aluminium, or it may be coated with celluloid; in each case the coating is ignited either by an "arcing-fuse" or by an externally applied flame. In the former case the residue of magnesia and alumina remains as part of the incandescent filament. The filament is enclosed within a removable protecting sleeve or cylinder of glass. The patentee's object is to manufacture cheap "candles" that may be renewed as often as the lamp is to be re-lighted. —H. B.

Electrical Incandescence Lamps. Siemens Bros. and Co., Ltd., London. From Siemens and Halske, Berlin. Eng. Pat. 6220, March 22, 1899.

THIS relates to lamps containing refractory bodies that require to be heated in order to become conductors, and



the figure shows a complete lamp constructed according to the invention. Between the two electrodes *a* and *b* is inserted the refractory incandescence body *g*; *l* is a conducting rod, pivotted, in conducting connection, to *b* and faced with a carbon rod *k*; the upper end *d* is the armature of an electro-magnet *e*, the winding of which is included in the main circuit. On passing a current of moderate intensity through the lamp, the armature *d* is drawn towards *e*, and the conducting rod *l* is drawn back from the electrode *a*, whereupon the arc is produced between *k* and *a*. The contiguous lower end of *g* thus becomes heated and conducting, so that the arc now passes between *k* and *g*. The rod *g* gradually becomes heated throughout its entire length, the arc passing upwards simultaneously, until *g* becomes completely incandescent, when the arc disappears altogether and the current passes directly between *b* and *a* viâ *g*. The lower electrode *a* may be provided with a renewable copper cap *m*. There are claimed the above method of effecting the preliminary heating, and a lamp of the kind described above.—H. B.

Graphite, Purifying. E. Teisler, Borsdorf, Germany. Eng. Pat. 23,233, Nov. 21, 1899.

See under VII., *page* 246.

Regenerative Furnaces for Heating Gas Retorts or for Analogous Purposes, and Special Construction of Bricks to be employed therein. J. H. Brearley, Longwood. Eng. Pat. 7210, April 6, 1899.

See under I., *page* 229.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, PETROLEUM.

Wood; Modern Distillation of. F. A. Bühler. Zeits. f. angew. Chem. 1900, [7], 155—165.

The author states that there are but few works in Europe on a large scale, and that the processes and products are nearly identical at all of them. The works must be so situated that beech wood is obtainable at about 5 to 6 marks per cubic metre. Pine and birch are occasionally distilled instead of beech. The distillation is usually carried out in horizontal or vertical retorts 3 metres long and 1 metre in diameter. Laboratory researches show that the yield of products on the large scale of working is capable of great improvement. Each retort carbonises about 1,000 cubic metres of wood per annum. The wood is stored for nearly two years before it is carbonised, with the object of reducing the water it contains from 35—50 per cent. to 20—25 per cent. The retorts are drawn about 16 hours after charging, with all possible speed, and the charcoal is set aside to cool in closed iron receptacles for about 36 hours. The less rapid the cooling, the less liable is the charcoal to ignite spontaneously. The liquid distillate collects in wells in which the tar settles down from the crude pyroligneous acid, the latter being pumped up to stills, in which the crude wood spirit is distilled off, leaving the crude acid, which is saturated with lime. The wood spirit is frequently purified by rectification, and pure methyl alcohol and acetone recovered from it. The crude calcium pyrolignite contains tarry matters, which are driven off by heat, and the commercial gray salt, containing not less than 80 per cent. of calcium acetate, is thus obtained. In some cases large iron ovens, traversed by heating pipes, are used in place of the retorts for carbonising the wood. Many attempts have been made to dry the wood by artificial means prior to carbonisation, but they have not proved successful.

An installation capable of dealing with 30,000 cubic metres of wood per annum costs about 280,000 to 300,000 marks. A return of 15 per cent. on the outlay can be secured only under exceptionally favourable conditions. Beech ordinarily yields about 8 per cent. of grey calcium acetate, 1 per cent. of methyl alcohol, 24 per cent. of wood charcoal, and 8 per cent. of tar. Assuming that the market prices per 100 kilos. are, for the acetate, 14 marks, for the methyl alcohol, 100 marks, and for the charcoal, 4 marks, the receipts from the carbonisation of 30,000 cubic metres of wood will amount to 323,000 marks. The tar is in most cases valueless. The expenditure, including wages, general charges, cost of wood, coal, and other materials, varies very much from time to time. The raw material, however, is gradually becoming more costly, and the higher expenditure thereon must be met by efforts to improve the yields, to utilise the by-products, and to reduce the manufacturing expenses.—J. A. B.

Petroleum, Origin of. C. Engler. Ber. **33**, [1], 7—21.

With reference to the researches of Krämer and Spilker (this Journal, 1900, 36) on petroleum from diatomaceous remains, the author is of opinion that, although the fat in such remains may, like other fats, furnish petroleum when distilled, there is not sufficient ground for the assumption that they may have constituted a principal source of existing petroleum deposits, the balance of evidence collected by numerous observers tending to show that the bulk of the oil must have been formed from *marine* deposits of animal or vegetable matters, and not in fresh-water lakes. Furthermore, he points out that the bitumen in peat mosses should not be attributed to diatom wax without further investigation, since Guignet and others have found considerable quantities of vegetable wax (from leaves, &c.) in peat. —C. S.

Vinegar; New Method for the Acetimetric Valuation of. Durieu. J. Pharm. Chim. **11**, [1], 22.

See under XXIII., *page* 278.

Ammoniæ Manufacture; Soda Washer for Liquor. O. Pfeiffer. J. f. Gasbeleucht. **43**, [6], 80.

See under VII., *page* 244.

Cresols; Application of the Bromine Substitution of, for their Quantitative Determination. R. Clauser.

See under XXIII., *page* 278.

Petroleum; Solidified. A. Conrady. Apoth. Zeit. **14**, 767. Chem. Zeit. Rep. **24**, [1], 8.

See under XII., *page* 255.

PATENTS.

Continuous Distillation and Desiccation of Substances; Viscid, Pulverulent, and Granular. H. M. C. Knopp, Eidelstedt, and E. Westphal, Stellingen-Langenfelde, Germany. Eng. Pat. 3558, Feb. 17, 1899.

The material (sawdust, the waste of dyewood and tan, leather parings, &c.) is admitted through a charging hopper into a drying retort, wherein it is propelled forward by helical spirals, and caused gradually to drop through a shoot into a second retort, also provided with a screw spiral, and situated within a structure fitted with heating apparatus, so that the material, being moved in a direction opposite to that of the flow of the hot gases, is carbonised. The first retort is swept by the exhaust gases of the furnace. The products of distillation escape from the second retort into a cooler, whilst the waste gases may be admitted as fuel into the heater. The carbonised residues are passed through a shoot into a third retort (the "cooling retort"), in which they are cooled before being discharged. The contents of the first shoot prevent the gases in the second retort from entering into the first, whilst their passage into the cooling retort is avoided by disconnecting the second and third retorts from each other by means of two sliding dampers provided in the shoot connecting the distilling retort and the cooling retort.—D. B.

Wood; Treatment of, to obtain therefrom Charcoal and other Products. W. H. Philipson, Greenodd, Lancaster. Eng. Pat. 5923, March 18, 1899.

For the production of charcoal and other products from wood economically, and with little or no external heating, the wood to be treated is placed in a series of brick chambers, so arranged that each chamber becomes consecutively the last, intermediate, and first of the series. Heated air under a slight pressure is introduced into the first chamber, containing wood in a partially charred condition, the products of combustion are then passed through wood in several other chambers. The escaping gases are conveyed through appliances for the purpose of separating tar, acetic acid as acetate of lime, and other products, and are finally burnt in an oven in order to heat the air which is supplied to the first chamber of the series.—D. B.

IV.—COLOURING MATTERS AND DYESTUFFS.

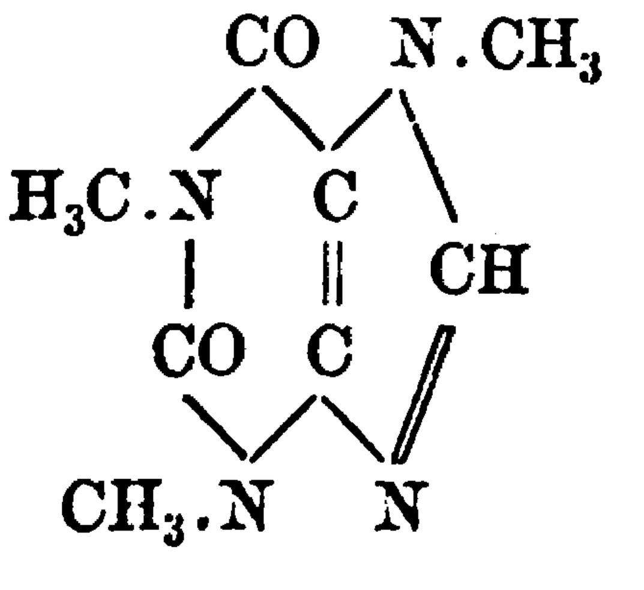
Aniline Still [*Nitrobenzene Reduction*]; *Probable Cause of Explosion of an.* Actien-Gesellschaft für Anilin-Fabrikation. Berlin. Chem. Ind. **23**, 1900, [2], 26–27.

The factory at Rummelsburg was on Oct. 7th, 1899, the scene of the explosion of a reducing still, as to which the following facts are now published. Since 1869, the reduction of nitrobenzene, nitrotoluene and nitroxylene, &c., to the corresponding amines, had with one exception been carried out down to the above date without mishap. With regard to the arrangement of the plant, the reducing stills are connected with condensers, which during the reduction condense the water, nitro- and amido-compounds distilling over. This distillate runs into a receptacle, from which it can be raised by means of a pump, to a vessel standing above the still, and from this it is run periodically into the still again. On the day of the accident, the still had been charged with nitroxylene, iron, acid, &c., and the reduction was proceeding normally, when at 9 a.m. the pump broke down. The foreman gave notice at once to a fitter, but the repair was not completed by 3 p.m. when the explosion occurred. Shortly before 3 p.m. the still became very hot. This rise in temperature would be accounted for if further quantities of ground iron had been added without returning the distillate or adding fresh water to the still. If no iron had been added the reaction would cease. That the materials were not at fault was shown by the fact that an adjacent still charged with exactly the same materials, worked normally throughout. Before the foreman had time to apprise his superior of the heating of the apparatus, the explosion took place. The still was lifted about 2 feet, and the bottom bursting, the contents, a black, pitchy mass, were blown about the factory. In determining the cause of this, it was evident that at the time of the explosion an excess of nitroxylene had acted on xylidine in presence of ferrous chloride at a high temperature, and with little water present. Laboratory experiments demonstrated that mixtures of nitroxylene, xylidine, and ferrous chloride, the first-named being in excess, reacted more and more rapidly, with a rise in temperature, and at about 200° C. became explosive. Mixtures containing an excess of xylidine or a deficiency of iron, did not react, or only feebly. The product of the reaction had the same properties as that obtained in the explosion described. It consisted of a pitchy, partly carbonised mass, from which water extracted small quantities of a magenta dyestuff. The same result was obtained after removing the water and heating a portion of an incomplete reduction from a still in the same stage as that at which the explosion took place. It would thus appear probable that the explosion was due to the action of an excess of a nitrocompound on an amine in presence of ferrous chloride at a high temperature and in the absence of water.

—T. A. L.

Diazocaffeïne. M. Gomberg. Amer. Chem. J. **23**, 1900, [1], 51–69.

Although caffeïne is usually regarded as an aliphatic substance, it behaves in many of its reactions more like an aromatic compound. According to recent researches (Ber. **30**, 553) its constitutional formula is—



and its amido-compound, in which the amido group is attached to the tertiary carbon atom, gives an unstable diazocaffeïne, on treatment with nitrous acid. The substance has hitherto only been obtained in solution, but combines readily with aromatic amines and phenols. It will also combine with certain aliphatic compounds, and gives with aceto-acetic ester a derivative which crystallises from chloroform in dark blue crystals with a greenish reflex. The substance is said to possess in a high degree the properties of a dyestuff, and dissolves in water to a reddish-violet solution, which becomes deep blue when warmed, returning to the original colour on cooling. The author regards it as a disazo compound.

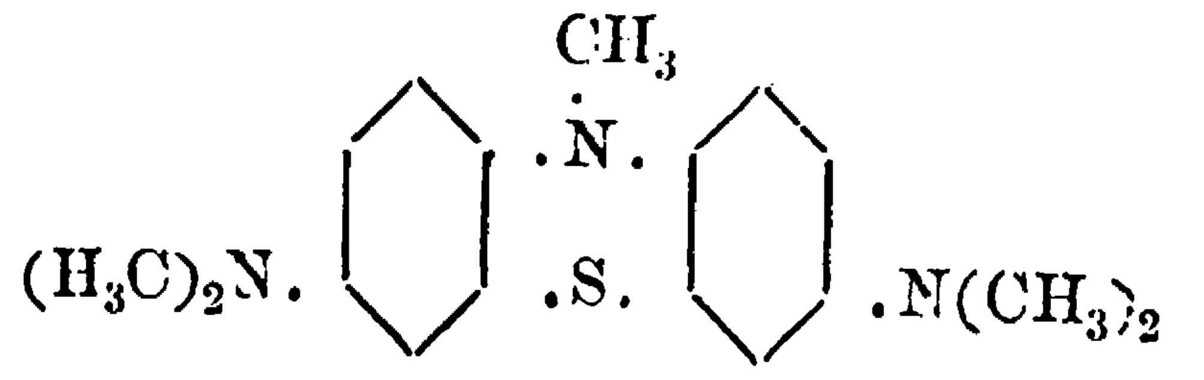
Amidocaffeïne (obtained by heating chlorocaffeïne and alcoholic ammonia in an autoclave to 150°—160° C.) is best diazotised by dissolving it in 5 times its weight of hydrochloric acid (sp. gr. 1·2), cooling to – 18° C., and running in sodium nitrite solution at – 10° C., with vigorous agitation. The strongly yellow solution of the diazo compound remains clear for an hour if kept in a freezing mixture, but will not yield an insoluble salt, nor could it be reduced to the hydrazine by stannous chloride. It appears to form normal azo compounds with phenol, dimethylaniline, and *m*-phenylene diamine, whilst it is noteworthy that the azo compound obtained by combining it with β-naphthol is said to be soluble to a deep-red solution in hot dilute alkali hydroxide.—T. A. L.

Methylation; New Method of, M. Prud'homme. Bull. Soc. Chim. **23**, 1900 [3], 69—71.

By the action of formaldehyde and nascent hydrogen in an acid solution, certain leuco bases and dyestuffs containing amido groups may be methylated. The reaction is expressed by the following equation:—

$$R.NH_2 + CH_2O + H_2 = RNHCH_3 + H_2O.$$

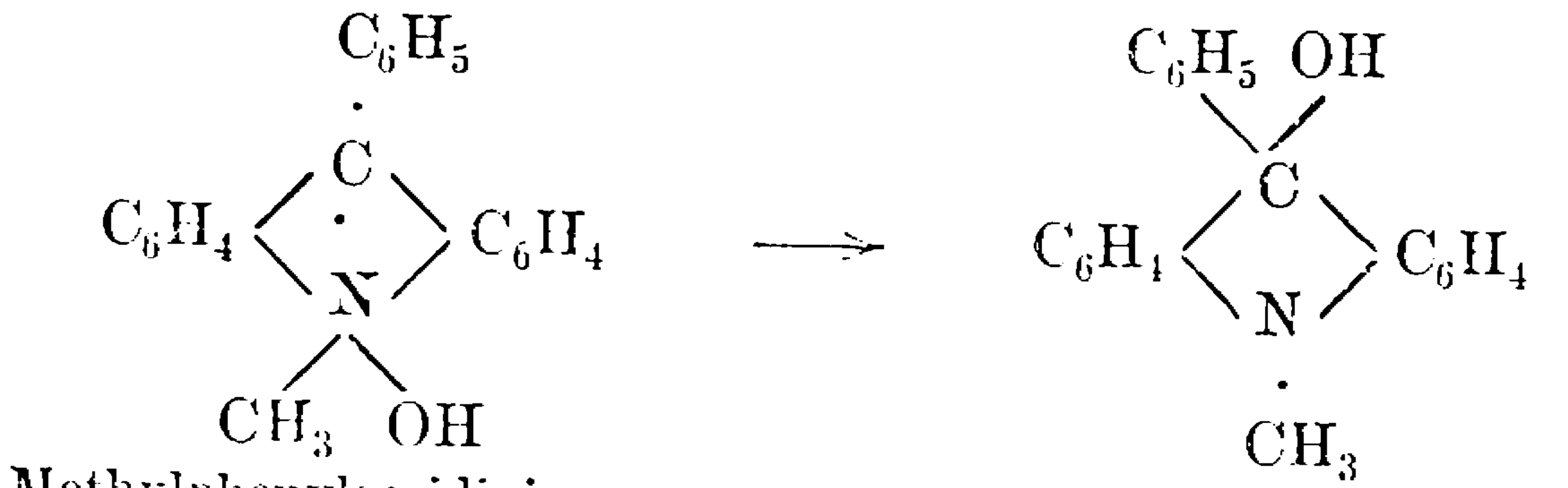
For example, a solution of rosaniline to which is added zinc dust, hydrochloric acid and formaldehyde is warmed for about a quarter of an hour to 75°—80° C. The colorless solution after filtering and cooling is oxidised in presence of acetic acid with 1 molecular proportion of lead peroxide for each molecule of rosaniline. The lead is removed with sodium sulphate and the dyestuff is salted out. It gives on cotton mordanted with tannin and tartar emetic, violet shades identical with Hexamethyl Violet. If anything, perhaps the shade of the new violet is somewhat more bluish, probably due to the fact that commercial rosaniline does not consist of pararosaniline, but is a mixture of C_{19} and C_{20} rosanilines. In a similar manner, rhodamine from *m*-amidophenol yields a methylated derivative giving brilliant red shades on silk, wool, and on cotton mordanted with tannin. By employing the same reaction with Lauth's Violet, it is converted into tetramethyl thionine or Methylene Blue. If, however, the reaction be pushed further, a new dyestuff is formed which gives greener shades than methylene blue on mordanted cotton. Probably the imido group is substituted so that the leuco compound has the formula:—



By treating phenosafranine solution (1 grm. per litre) with zinc and hydrochloric acid at 75°—80° C. for a short time, decolorisation takes place. The addition of formic aldehyde turns the solution purple-red. If the dyestuff be separated at this stage by filtration and precipitation with salt, it is found to give shades on mordanted cotton similar to those obtained from Witt's Neutral Red. On prolonging the reaction for some minutes, the purple-red becomes violet, and finally after about half an hour, the colorless liquid on oxidation turns blue, whilst the dyestuff precipitated with salt gives purple-blue shades on mordanted cotton. The new dyestuff appears to be an induline. It dissolves in sulphuric acid with an olive colour, becoming bluish-grey on dilution, whilst the sulphuric acid solution of the safranines is generally green, turning blue and then violet on dilution. Possibly here, methylation of the azonium nitrogen takes place after reduction to the leuco derivative.—T. A. L.

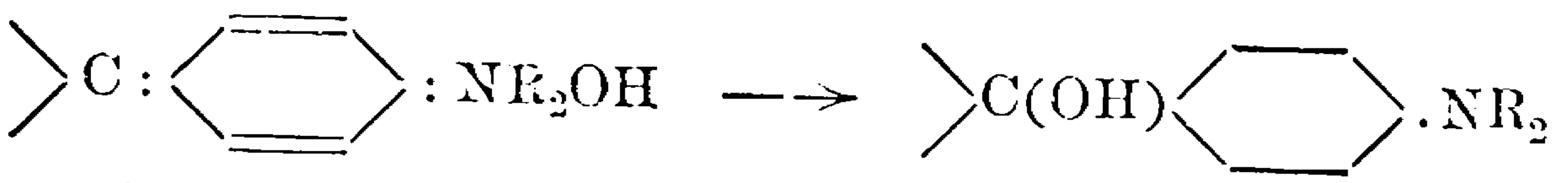
Colour bases into Pseudo-ammonium-hydrates, cyanides, and sulphonates; Conversion of. A. Hantzsch and G. Osswald. Ber. **33**, [2], 278—317.

The alkylhaloids of the acridine series, as Hantzsch and Kalb (Ber. **32**, 3109) have found, are converted into ammonium bases which, however, decompose very rapidly in aqueous solution into isomeric insoluble non-electrolytes. The latter are formed by the movement of a hydroxyl group from the ammonium nitrogen to a carbon atom, as follows:—

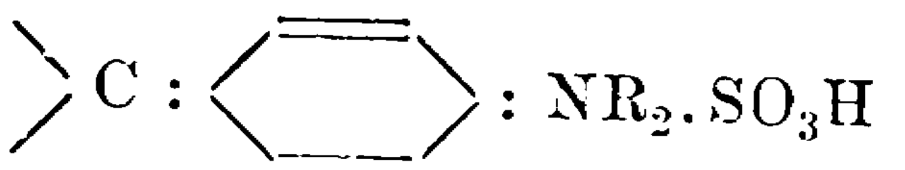

Methylphenylacridinium hydroxide.
Methylphenylacridol.

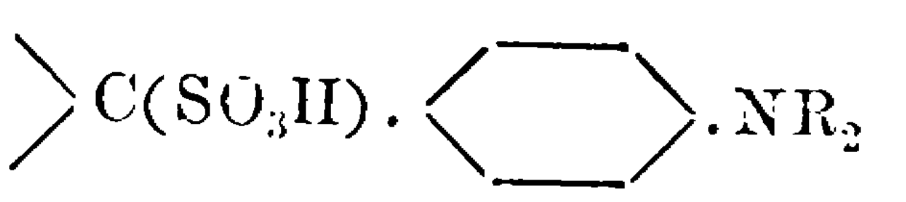
Hitherto these compounds have been considered as true ammonium derivatives, whereas they are now known as pseudo-ammonium hydrates, and are actually carbinols.

Similar transformations of several dyestuffs of the quinonoid ammonium salt type are described in the present paper. The first products like the acridinium bases change more or less readily to non-conducting insoluble colourless "Pseudo-dyestuff bases" which frequently (but not always) *e.g.*, in the triphenylmethane series, are identical with the already known carbinols. In the case of the hydrates this can be represented by the following scheme:—


True Ammonium Base.
Pseudoammonium Base (Carbinol).

The same change takes place in the case of the cyanides and also of the sulphur dioxide addition products, which latter are colourless, and hence are sulphonic acids of the carbinol type, (2) and not quinonoid ammonium sulphites (1) which would be coloured.

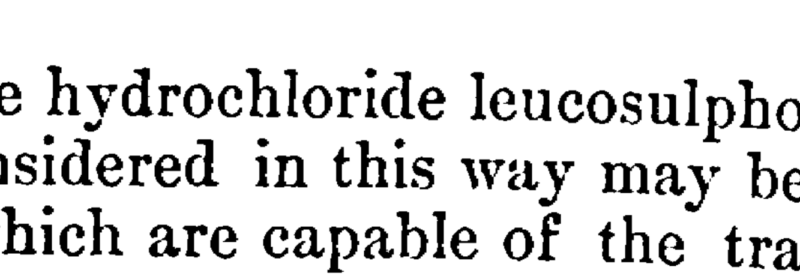
(1.)


(2.)


These transformations stand on a parallel with the conversion of the diazonium salts into the normal or syn-diazo compounds (pseudo-diazonium compounds). With regard to the nomenclature of the new compounds, the authors place the prefix leuco before the word hydrate (or carbinol), cyanide or sulphonic acid, so that, for instance, in the case of Brilliant Green, tetra-ethyldiamidotriphenylcarbinol would be termed Brilliant Green leucohydrate, the derivative $[(C_2H_5)_2N.C_6H_4]_2{:}C(C_6H_5)SO_3H$ is Brilliant Green leucosulphonic acid and the sulphate of this acid—

$$H_2SO_4[(C_6H_5)_2N.C_6H_4]_2 : C(C_6H_5)SO_3H$$

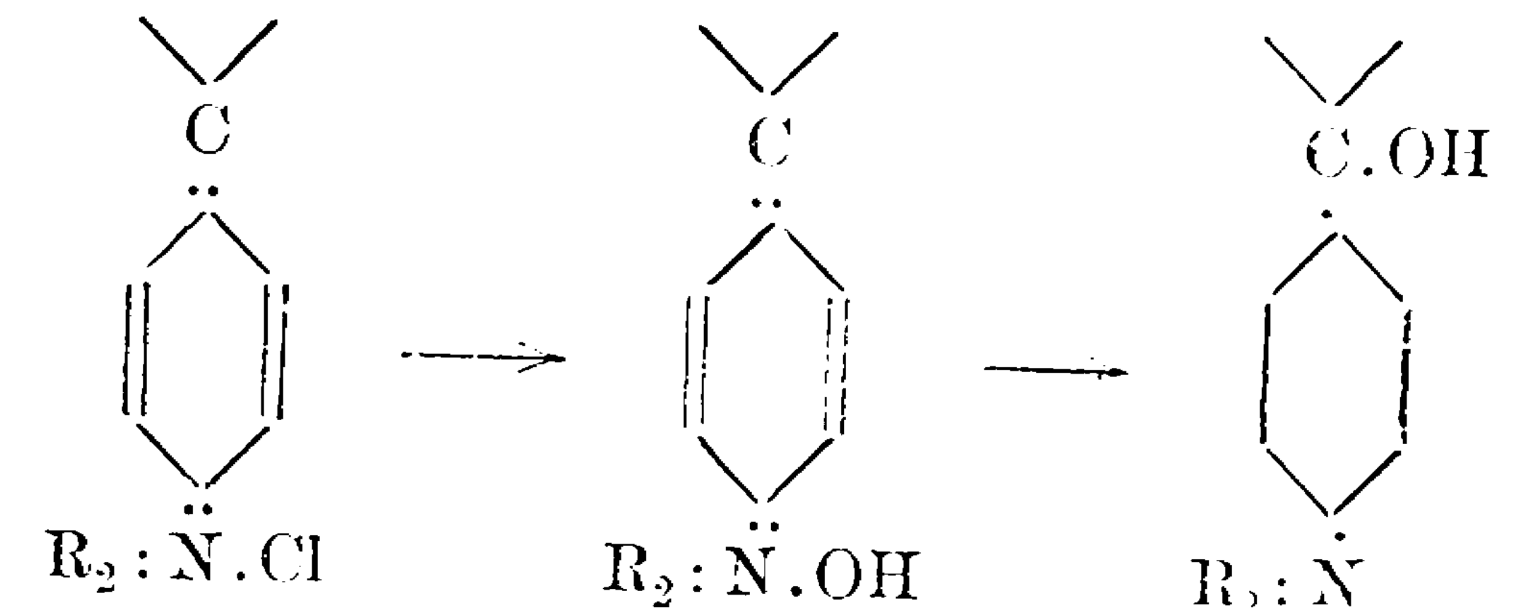
is Brilliant Green sulphate leucosulphonic acid. In the same way the so-called magenta sulphurous acid—


$$\left.\begin{array}{r}[H_2N.C_6H_4]_2\\ HClH_2N.C_6H_4\end{array}\right\rangle C.SO_3H$$

is pararosaniline hydrochloride leucosulphonic acid.

Dyestuffs considered in this way may be divided into two groups, those which are capable of the transformation, and those which are not. To the first group belong bases of the tri- and di-phenylmethane series, such as Crystal Violet, Pararosaniline, Brilliant Green and Auramine, further the rosindones, rosindulines and flavinduline, whilst the second group contains the safranines and thiazimes (Methylene Blue group).

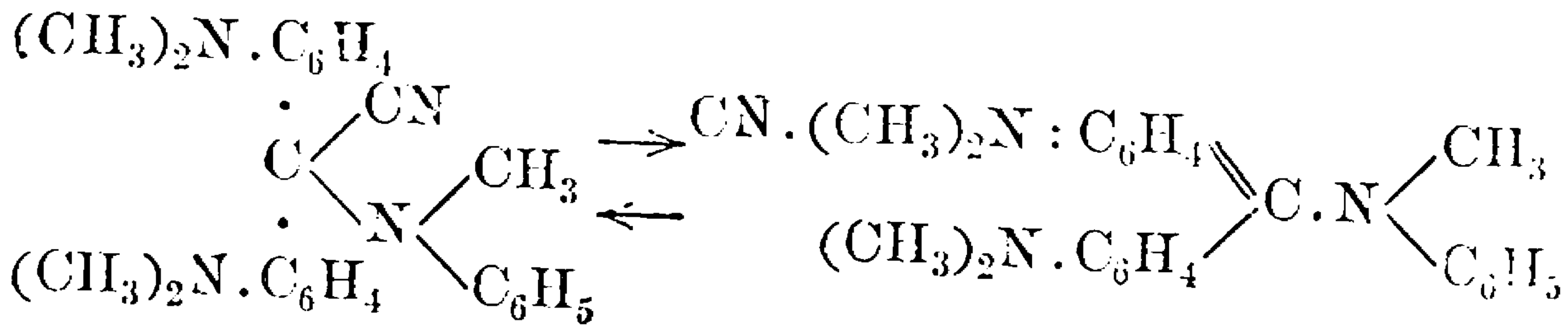
The decomposition is represented by the following scheme:—


Dyestuff Salt.
True Ammonium Colour Base (Labile).
(Colourless) Pseudo-ammonium Base (Carbinol) (Stable).

The transformation is traced most readily and also quantitatively by an electrical method. The electrical conductivity of the system (Dyestuff ammonium salt [chloride or sulphate] + sodium hydroxide = sodium chloride or sulphate + true ammonium base) falls continuously, finally reaching the salt or sodium sulphate value, showing that the isomerisation of the true ammonium to the pseudo-base is complete.—T. A. L.

Auramine; Constitution of. A. Stock. Ber. **33**, [2], 318—320.

The auramines, according to Graebe (Ber. **32**, 1678; this Journal, 1899, 752), in contradistinction to the author's views (J. prakt. Chem. **47**, 401), are salts of the imines of diamidobenzophenones and the existence of disubstituted auramines was called in question. The author has now obtained, in a crystalline form, the thiocyanates of methylphenyl- and of diphenyl-auramine. On treatment with alkali, methylphenylauramine gave tetramethyldiamidodiphenylketone and methylaniline, identified by its acetyl compound. Albrecht (Ber. **27**, 3294; this Journal, 1895, 475), by acting with potassium cyanide on auramine hydrochloride, obtained hydrocyanauramine, which, when decomposed by acids, gave off ammonia, yielding an unstable green dyestuff. An analogous compound can be obtained from methylphenylauramine thiocyanate, the resulting compound *methylphenylhydrocyanauramine* separating from ether in large yellowish crystals melting at 99° C. On decomposition with acids, methylaniline is given off and an unstable green dyestuff results. Methylphenylhydrocyanauramine is very readily reconverted into the original dyestuff. Even on warming with alcohol a yellowish-red solution is obtained in which evidently part of the compound is converted into the tautomeric cyanide of methylphenylauramine so that the two compounds are co-existent in the solution:—

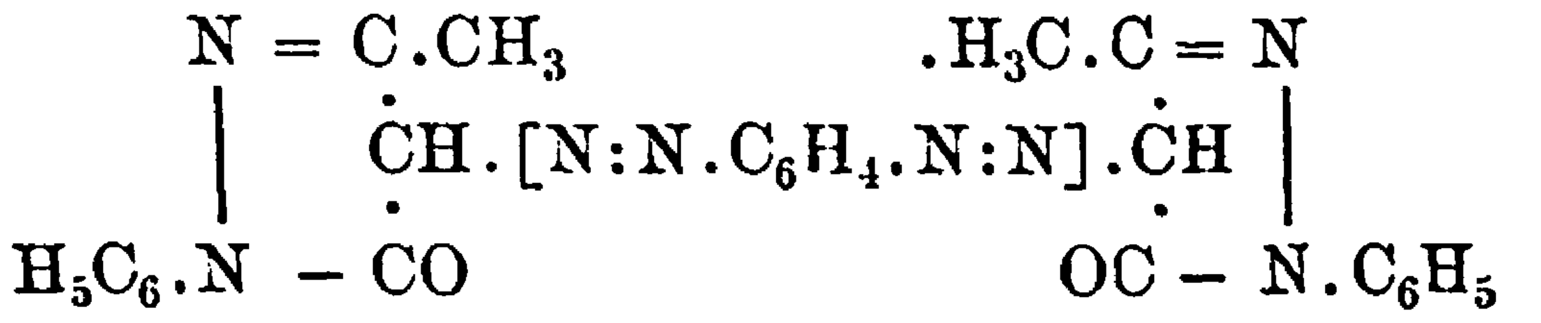

$$\begin{array}{l}(CH_3)_2N.C_6H_4\\ \quad\dot{C}\begin{array}{l}\diagup CN\\ \diagdown N\begin{array}{l}\diagup CH_3\\ \diagdown C_6H_5\end{array}\end{array}\\ (CH_3)_2N.C_6H_4\end{array} \begin{array}{c}\longrightarrow\\ \longleftarrow\end{array} \left.\begin{array}{r}CN.(CH_3)_2N : C_6H_4\\ (CH_3)_2N.C_6H_4\end{array}\right\rangle C.N\begin{array}{l}\diagup CH_3\\ \diagdown C_6H_5\end{array}$$

On warming an alcoholic solution of methylphenylhydrocyanauramine with sulphur, that portion present as cyanide takes up sulphur yielding the thiocyanate. A fresh portion of the original compound then isomerises to cyanide, which again is further converted into thiocyanate. Such a solution, after warming some time with sulphur, shows all the reactions of methylphenylauramine. Acids and alkalis decompose it into methylaniline and ketone, whilst ammonia converts it into auramine.

It is also noteworthy that *hydrocyanphenylauramine* melting at 160° C. (from phenylauramine hydrochloride and potassium cyanide) and Albrecht's hydrocyanauramine, on treatment with sulphur in alcoholic solution yield phenylauramine and auramine respectively.—T. A. L.

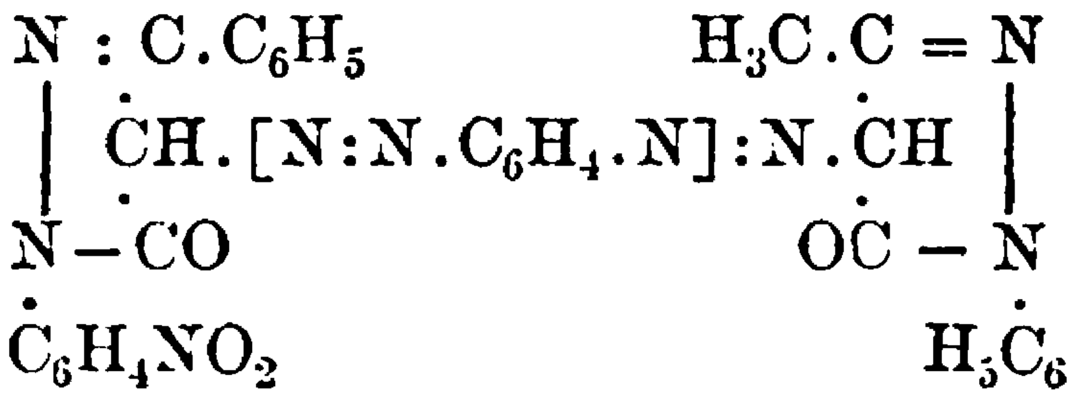
Paraphenylene Diamine; Fatty-aromatic Azo and Disazo, Combinations of. (Bülow's Reaction.) C. Bülow. Ber. **33**, [1], 187—199.

The starting point for these combinations is *p*-amidoacetanilide, the diazo compound of which on combination with aceto-acetic ester gives derivatives which readily yield with phenylhydrazine, semicarbazide, or hydroxylamine, azopyrazolones and isazoxolones. On hydrolysis with caustic alkalis or with acids, the acetyl group is removed and the resulting substances contain an amido group para to the existing azo group. When this is diazotised, the compound can again be coupled with acetoacetic ester, and the resulting derivative then condensed with phenylhydrazine, in this manner forming, for example, the disazo-dyestuff, having the constitution—



$$\begin{array}{lcr} N = C.CH_3 & & .H_3C.C = N \\ \vert \quad \dot{C}H.[N{:}N.C_6H_4.N{:}N].\dot{C}H \quad \vert & & \\ H_5C_6.N - CO & & OC - N.C_6H_5 \end{array}$$

The same product can also be obtained in other ways, one of which consists in combining in acetic acid solution, diazotised *p*-amidoacetanilide with phenylmethylpyrazolone (from phenylhydrazine and aceto-acetic ester) splitting off the acetyl group, diazotising, and again combining with the above pyrazolone. Other compounds described are those obtained by combining diazotised *p*-amido-acetanilide with a phenol or amine or a derivative thereof, splitting off the acetyl group, rediazotising and combining with a hetero-cyclic component, or else with a 1·3-diketone and subsequent condensation with a hydrazine or hydroxylamine, or else the operation may be performed in the reverse order. Again other disazo hetero-cyclic compounds may be obtained by employing different aliphatic compounds and also different hydrazines or hydroxylamines for combination with the diazo-derivatives and subsequent condensation, and these compounds may be obtained in a number of different ways. For example, eight methods can be employed for obtaining the compound represented by the following formula—



$$\begin{array}{lcr} N : C.C_6H_5 & & H_3C.C = N \\ \vert \quad \dot{C}H.[N{:}N.C_6H_4.N]{:}N.\dot{C}H \quad \vert & & \\ N - \dot{C}O & & \dot{O}C - N \\ \dot{C}_6H_4NO_2 & & H_5\dot{C}_6 \end{array}$$

In describing the method for diazotising *p*-phenylene-diamine-azo-phenyl-methyl-pyrazolone, the author states that the orange-red diazo solution exhibits *Bülow's reaction.* To obtain this reaction, the solution is poured into an ice-cold sodium acetate or carbonate solution when it turns blue, an indication that the amido group in the benzene ring para to the azo group has been completely diazotised.—T. A. L.

Red and Violet Dyestuffs; Manufacture of New. A. Abt.
Bull. Soc. Ind. de Mulhouse, 1899, 319—320.

These dyestuffs are obtained by the condensation of 2 mols. of nitrosodimethylaniline hydrochloride with 1 mol. of fluoresceïn, or of a halogen-, nitro-, or sulphonic acid derivative thereof. The principal member of the new group is prepared as follows:—100 grms. of fluoresceïn are dissolved in 1·5—2 kilos. of previously warmed commercial acetic acid, to which, after cooling, 1,000 grms. of nitrosodimethyl aniline are added, in small portions at a time, the mass being kept well stirred. When solution is complete, the mass is gradually heated, with stirring to 80° C., and maintained at that temperature for 3—4 hours, after which a sample of the newly-formed red dyestuff should be easily soluble in water. If not, the heating must be continued until the requisite solubility is attained, after which the mass is poured into 10 times its bulk of cold water, any precipitate filtered off, and the new dyestuff "salted-out" from the filtrate.

Careful manipulation should ensure a yield in dyestuff of 90 per cent. of the nitroso derivative taken.

By substitution, for fluoresceïn, of its chlorine, bromine, or iodine derivatives, shades merging from red into pure violet are obtained.

The new dyestuffs, the sodium and potassium salts of which are very soluble in water, are bodies analogous to the Eosines, but much redder and almost without fluorescence. Their distinguishing features are greater fastness to soap and to light, particularly the latter. By "alkylating" them, violets of great beauty and fastness are obtained.—J. A. P.

Yellow Colouring Principles of Various Tannin Matters.—VII. A. G. Perkin. Proc. Chem. Soc. **16**, [219], 45.

The colouring matter of the leaves of *Arctostaphylos uva-ursi* (bear-berry) and *Hæmatoxylon Campeachianum* (logwood) is *quercetin,* and this is accompanied by a second substance, probably *myricetin,* to which the green colour of its alkaline solutions are due. *Gallotonnic acid* occurs in some quantity in the latter leaves. The leaves of *Rhus metopium* contain *gallotannin, myricetin,* and a trace of *quercetin,* but the stem of this plant, unlike *R. cotinus,* and *R. rhodanthema* is devoid of colouring matter. The sparing solubilities of acetylmyricetin and dibromoquercetin have been employed for the separation of myricetin and quercetin. The leaves of *Robinia pseudacacia* contain a feeble colouring matter, *acacetin,* $C_{16}H_{12}O_5$, which yields an *acetyl* derivative, $C_{16}H_{10}O_5(C_2H_3O)_2$, colourless needles, m. pt. 195°—198°, and on fusion with alkali *phloroglucinol,* p-*hydroxybenzoic acid,* and a trace of *protocatechuic acid.* Acacetin contains one methoxyl group, on removal of which a colouring matter, $C_{15}H_{10}O_5$, results, having the reactions of apigenin. It is thus probably an *apigenin monomethyl ether.* The leaves of *Myrica gale* and *Coriaria myrtifolia* contain respectively *myricetin* and *quercetin.* Although a relationship frequently exists between the tanning and the colouring matters of the same plant (Trans. Chem. Soc. 1897, **71**, 1138), there is no rule on this point, for exceptions are somewhat numerous.

Morphine; The Nitrogen-Free Decomposition Products of. W. E. Vongerichten.

See under XXIV., *page* 283.

Hydrosulphurous Acid. A. Bernthsen and M. Bazlen.

See under VII., *page* 244.

PATENTS.

Acridine Derivatives; Production of. C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Germany. Eng. Pat. 5461, March 13, 1879.

This specification is supplementary to Eng. Pat. 16,475 of 1898 (this Journal, 1899, 826), and describes the use of other primary aromatic amines, such as *o*-toluidine, *m*-xylidine, acetyl-*p*-phenylene diamine, &c., in place of *p*-toluidine in the manufacture of acridine derivatives. For instance, 6 kilos. of anhydroformaldehyde-*o*-toluidine are slowly added to 10 kilos. of β-naphthol at 150° C. The condensation is complete in a short time, and, after distillation, the product can be purified by recrystallisation from a mixture of benzene and petroleum spirit, when it melts at 143° C. The corresponding compound from *m*-xylidine melts at 152° C. An aqueous solution of acetyl-*p*-phenylenediamine on treatment with the theoretical quantity of formaldehyde yields on cooling, white crystals of anhydroformaldehyde-acetyl-*p*-phenylene diamine, melting at 195°—200° C. 5 kilos. of this product and 12 kilos. of β-naphthol are fused at 150°—180° C. The melt, after cooling, is washed with dilute caustic soda lye, and the acetylamidonaphthacridine is recrystallised from alcohol, forming yellow needles melting at 255° C. On hydrolysis, it yields amidonaphthacridine, melting at 238° C. The following alternative method for obtaining naphthacridine is also described. A mixture of 10 kilos. of β-naphthol and 8 kilos. of aniline is heated to 160° C., and 1 kilo. of trioxymethylene is added. The excess of amine and β-naphthol is removed by distillation, and at about 430° C. naphthacridine distils over. After washing the product with dilute caustic soda lye, it is recrystallised from a mixture of benzene and petroleum spirit, when it forms yellowish crystals, melting at 129°·5 C.—T. A. L.

Dinitrosulphonic Acid of the Aromatic Series, A New. [*Dinitrochlorobenzene Sulphonic Acid.*] C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 6436. March 24, 1899.

The manufacture of dinitrochlorobenzene sulphonic acid [$NO_2 : Cl : NO_2 : HSO_3 = 1:2:3:5$] by the two following processes is claimed. (*a*) Nitrochlorobenzene potassium sulphonate is dissolved in fuming sulphuric acid (25 per cent. SO_3), and fuming nitric acid is added, the nitration is effected at 120°—130° C., the product thrown into water and recrystallised. (*b*) Chlorobenzene is heated on the water-bath with ordinary and fuming sulphuric acid. After cooling, fuming nitric acid is added, and the nitration completed at 40° C. After standing, fuming sulphuric acid (60 per cent. SO_3) and potassium nitrate are added, and the mixture heated to 120°—130° C. The product is then isolated as before.—A. C. W.

Black Disazo Colouring Matters. C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 6583, March 27, 1899.

According to Eng. Pat. 24,527 of 1897 (this Journal, 1898, 916) black disazo dyestuffs are obtained by combining diazotised *p*-amidodiphenylamine sulphonic acids with *α*-naphthylamine, again diazotising, and finally coupling with naphthol sulphonic acids. In the present specification the same process is applied to derivatives of *p*-amidodiphenylamine, containing a carboxylic instead of a sulphonic acid group. These amidodiphenylamine carboxylic acids are obtained by condensing 1·4·6-chloronitrobenzoic acid with aromatic amines or their carboxylic acids, and reducing the products so obtained. For example, *p-nitrodiphenylamine carboxylic acid* is obtained by condensing 1·4·6-chloronitrobenzoic acid with aniline. The product crystallises in yellowish-green needles melting at 232° C., and is converted, on reduction, into *p-amidodiphenylamine-carboxylic acid*, which crystallises in nearly colourless needles melting at 228° C. If the diazo compound of this product be combined with *α*-naphthylamine, the intermediate compound again diazotised and combined with R salt, a valuable disazo dyestuff is obtained, giving dark black shades on wool from an acid bath.—T. A. L.

Black-Brown Dyestuff directly Dyeing Cotton. C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 7022, April 1, 1899.

By condensing 1.4.6-chlorodinitrobenzene with 1.2.4.5-amidocresol sulphonic acid in alcoholic solution in presence of sodium acetate, the patentees obtain dinitro-oxyphenyltolylamine sulphonic acid, which, on heating with sulphur and alkaline sulphides, gives a direct-dyeing colouring matter. For example, 30 kilos. of sulphur and 75 kilos. of sodium sulphide are heated to 110° C., and 25 kilos. of the above condensation product are stirred in. The melt is then heated to 140°—150° C. until it begins to thicken, when the temperature is raised to 150°—160° C. until the mass becomes dry. The product is then powdered, and can be employed directly for dyeing. It is soluble in water containing a little sodium sulphide, giving a brownish-grey solution, and dyes cotton blackish-brown shades. The product dissolves in sulphuric acid with a brown colour.
—T. A. L.

Blue Dyestuffs, directly Dyeing Cotton. C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Germany. Eng. Pat. 7023, April 1, 1899.

According to Eng. Pat. 25,234 of 1897 (this Journal, 1898, 1139) direct dyeing colouring matters are obtained by fusing oxynitrodiphenylamine or its homologues, with sulphur and alkali sulphides, the shades varying from greenish- and violet- to blue-black. It is now found that if the melt be extracted with 10 times its weight of alcohol and the residue after filtering, dried, the resulting product dyes unmordanted cotton indigo blue shades. It gives a violet solution in water and a cornflower blue colour in concentrated sulphuric acid.—T. A. L.

Brown Dyestuffs directly Dyeing Cotton. C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 7348, April 7, 1899.

By fusing amidocresol sulphonic acid—

$$(CH_3:NH_2:SO_3H:OH = 1:2:4:5)$$

with sulphur and alkali sulphides, a brown dyestuff is obtained, which is said to be very strong and fast. For instance, 25 kilos. of the above-mentioned amidocresol sulphonic acid is added to a mixture of 75 kilos. of sodium sulphide and 30 kilos. of sulphur at 115°—120° C. until the mass is dry. After cooling, the product is powdered, and the brownish-black powder can be employed directly for dyeing. It gives a yellowish-brown solution in water, a brown colour in concentrated sulphuric acid, and dyes unmordanted cotton deep-brown shades.—T. A. L.

Diphenylamine Carboxylic Acid and Colouring Matters [Black] therefrom, Manufacture and Production of New Derivatives of. G. W. Johnson, London. From Kalle and Co., Biebrich-on-the-Rhine, Germany. Eng. Pat. 5581, March 14, 1899.

By fusing certain hydroxydinitro-diphenylamine carboxylic acids with sulphur and sodium sulphide, dyestuffs are obtained which give black or violet-black shades on unmordanted cotton from salt baths containing sodium carbonate and sulphide. Subsequent treatment with chromium fluoride or copper sulphate increases the intensity and purity of the shades. The hydroxydinitrodiphenylamine carboxylic acids are obtained as follows:—

Hydroxydinitrodiphenylamine Carboxylic Acid A.—Chlorodinitrobenzoic acid (m.p. 200° C.), obtained by nitrating *o*-chlorobenzoic acid in sulphuric acid solution, is condensed in presence of sodium carbonate, with an equivalent quantity of *p*-amidophenol. In this case, the carboxyl and hydroxyl groups are heterocyclic. By substituting *p*-amidosalicylic acid ($OH:CO_2H:NH_2 = 1:2:4$) for the *p*-amidophenol in the foregoing example, the resulting product is termed B, and possesses the hydroxyl and carboxyl groups in the same ring, whilst if, in this example, the dinitrochlorobenzene be replaced by dinitrochlorotoluene ($CH_3:Cl:(NO_2)_2 = 1:2:3:5$), the product obtained is referred to as hydroxydinitrodiphenylamine carboxylic acid C. For instance, the first of these compounds (A) is obtained by dissolving 24·6 kilos. of chlorodinitrobenzoic acid, 11 kilos. of *p*-amidophenol, and 30 kilos. of sodium acetate in 400 litres of water. The mixture is boiled and agitated for six hours. When cold, the condensation product is precipitated by adding 30 kilos. of hydrochloric acid and on recrystallisation from boiling water forms red needles or prisms melting at 105° C. The sodium salt is easily soluble in hot, less so in cold water. For the preparation of a dyestuff, 500 kilos. of crystallised sodium sulphide are dissolved in a small quantity of hot water in a vessel heated in an oil-bath. 75 kilos. of flowers of sulphur and 100 kilos. of the sodium salt of hydroxydinitrodiphenylamine carboxylic acid A are stirred in. The temperature of the melt is then raised in 3—4 hours to 140°—150° C., and maintained thereat until the mass becomes dry and friable. When cold it can be used directly for dyeing. The product forms a powder with a pale bronzy lustre, and is readily soluble in water to a greenish-black solution. It dissolves in sulphuric acid to a greenish-blue solution, from which water precipitates the dyestuff in black flocks. Cotton is dyed deep black from a salt bath, the shade being improved by a subsequent treatment with metallic salts.—T. A. L.

Diphenylamine Carboxylic Acid and Colouring Matters therefrom; Production of Derivatives of. G. W. Johnson, London. From Kalle and Co., Biebrich-on the-Rhine, Germany. Eng. Pat. 6245, March 22, 1899.

This specification is supplementary to Eng. Pat. 5581 of 1899 (see above), and describes the preparation of homologues of oxydinitrodiphenylamine carboxylic acids and colouring matters therefrom by fusing these products with sulphur and sodium sulphide. The new compounds which are dinitrophenyloxytolylamine carboxylic acids are obtained by condensing 1.2.4-chlorodinitrobenzene with the amidocresotic acids $CH_3:NH_2:CO_2H:OH = 1:3:5:6$ and $1:2:4:5$ in an alkaline medium. On treating these compounds with sulphur and sodium sulphide as already described in the preceding specification, they yield black substantive cotton dyes.—T. A. L.

Aromatic Dinitro-Compound [Dinitrochlorobenzene] and a New Diamido-Compound [Chloro-m-phenylene Diamine] Derived therefrom. C. D. Abel, London. From The Actiengesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 5766, March 16, 1899.

The patentees find that 1.3.5-dinitrochlorobenzene can be obtained directly from *m*-dinitrobenzene by treating it with chlorine at 100° C. in presence of ferric chloride until 1 atomic proportion of chlorine has been absorbed. After washing with water, the compound can be recrystallised from alcohol, when it forms white needles melting at 59° C. On treatment with a reducing agent (iron or zinc dust and a mineral acid or stannous chloride and hydrochloric acid), it is converted into chloro-*m*-phenylene diamine, which crystallises from water in colourless crystals melting at 103° C. When employed as a developer, it yields with suitable compounds valuable greenish-black shades.
—T. A. L.

Fast Black Shades on Wool. J. Y. Johnson, London. From The Badische Anilin and Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 6244, March 22, 1899.

Certain disazo dyestuffs, some of which are described in the lapsed Eng. Pat. 10,845 of 1888 (this Journal 1889, 538), obtained from *o*-amidophenol-*p*-sulphonic acid yield shades varying from violet to brownish black, which have no technical value. The patentees find that these shades may be rendered fast and valuable by treating the fibre simultaneously or subsequently with a chromate or a chrome salt. The dyestuffs employed, are obtained from *o*-amidophenol-*p*-sulphonic acid, α-naphthylamine as a middle component, and a naphthol, dioxynaphthalene or a sulphonic acid as an end component. For instance, 30 kilos. of merino are dyed in a bath containing 900 litres of water, 1·5 kilos. of calcined glauber salt and 1 kilo. of the dyestuff from *o*-amidophenol-*p*-sulphonic acid, α-naphthylamine and β-naphthol-3′-sulphonic acid. The goods are dyed neutral for 15 minutes at the boil when 2 kilos. of sodium bisulphate are added in 4 equal portions at 15 minute intervals. The bisulphate may be replaced wholly or in part by acetic, sulphuric, or other acid. When the bath is exhausted, 600 grms. of potassium bichromate are added, and the goods are boiled for another half hour, after which, they are well washed and worked up in the usual manner. The shades produced are fast to acid and alkali, and are said to be fast to washing, fulling and light.—T. A. L.

Para-diamidodiphenylamine-sulphocarboxylic Acid; Manufacture of. O. Imray, London. From The Farbwerke vormals Meister, Lucius und Brüning, Hoechst a/M., Germany. Eng. Pat. 6950, March 30, 1899.

By condensing 1 . 4 . 6 . chloronitrobenzoic acid with phenylenediamine-sulphonic acid and subsequent reduction, the patentees obtain a product capable of tetrazotisation, and this, on combination with the usual components, forms substantive cotton dyestuffs. A solution of 20·2 kilos. of 1.4.6.chloronitrobenzoic acid, 18·8 kilos. of *p*-phenylenediamine sulphonic acid, and 17 kilos. of soda ash in 200 litres of water, is heated for 5—6 hours under an inverted condenser. The solution becomes orange-brown and when the condensation is complete, an excess of hydrochloric acid is added and the precipitated nitroamidodiphenylamine sulphocarboxylic acid is filter-pressed. The press-cakes are then reduced directly by boiling in 250 litres of water with 50 kilos. of iron and 5 kilos. of glacial acetic acid for about half an hour. The solution is then made alkaline with sodium carbonate and filtered from the precipitated iron oxide. The free acid is precipitated in greyish-green needles by adding acetic acid. The yellow tetrazo compound is soluble in water and turns an intense red when treated with sodium carbonate.—T. A. L.

Basic Dyestuffs [Yellow]; Manufacture of New. J. Imray, London. From La Société L. Durand, Huguenin et Cie, Basle, Switzerland. Eng. Pat. 7025, April 1, 1899.

By the action of phthalic anhydride on mono-alkylated *m*-amidophenols in presence of zinc chloride, there are formed besides the symmetrical dialkylrhodamines, certain by-products which are yellowish-brown and are soluble in alkalis. After purification by dissolving in ammoniacal alcohol and precipitation with acids, the product is recrystallised, when it forms yellow needles, and is converted into a dyestuff by treating with alcohol in presence of sulphuric acid. For instance, 20 kilos. of the crystals obtained from ethyl-*m*-amidophenol as described above, are heated for 12 hours with 80 kilos. of alcohol, and 80 kilos. of sulphuric acid monohydrate. The mixture is poured into about 800 litres of water kept alkaline with caustic soda, and at a temperature not exceeding 35° C. The precipitate is dissolved in alcohol together with a little hydrochloric acid and is filtered. On adding water or a further quantity of aqueous hydrochloric acid until the solution becomes turbid, the product crystallises out on standing and is purified by several recrystallisations. It gives yellow shades on tannin-mordanted cotton.—T. A. L.

Black, Substantive, Sulphurised Dyestuffs; Manufacture of. J. Imray, London. From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, Paris, France. Eng. Pat. 7349, April 7, 1899.

The dyestuffs obtained by oxidising mixtures of *p*-phenylene diamine or *p*-amidophenol with phenols or naphthols, when fused with sulphur and alkali sulphides, yield black substantive dyestuffs for cotton. For instance, 10 kilos. of the dyestuff obtained from *p*-phenylene diamine and α-naphthol are stirred into 15 kilos. of sulphur at 130°—140° C., the temperature during four hours being gradually raised to 200° C. The friable brittle mass obtained, is gradually added to from 60—80 kilos. of fused crystallised sodium sulphide. The pasty melt is heated to about 200°—250° C., when a reaction ensues, and after the mass has become hard and brittle it is ready for use.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

PATENTS.

Cotton, Ramie, or other Fibrous Materials, with Mercerising or other Liquors in Doubling or like Machinery; Apparatus for Treating. H. E. Aykroyd, Thornfield, Yorks. Eng. Pat. 54, Jan. 2, 1899.

This invention relates to the mercerising of threads simultaneously with the process of "doubling." The threads from two bobbins are passed through various troughs (suitably placed along the machine) containing the mercerising and washing fluids. The rollers in the mercerising fluid are of glass; as the thread emerges from one trough, it is passed through squeeze rollers before entering the next, and so on.—C. M.

Decolorising Paper and Textile Fabrics, after Immersion in Cuprammonium Solution. J. Williams, London. Eng. Pat. 1358, Jan. 19, 1899.

The process for decolorising paper and textile fabrics which have become coloured by immersion in a waterproofing solution of cuprammonium, consists in eliminating the said colouring by immersing the fabric in a bath composed of a solution containing precipitated cyanide of zinc dissolved to saturation in a solution of ammonia (30—70 lb. of ammonia gas per 100 gallons), then removing the fabric from the bath, pressing it, and finally drying it by heat. Care must be taken to maintain the ammoniacal zinc cyanide solution at its proper strength. The green hydrated compound of copper and cellulose is thus decomposed with the formation of white copper cyanide and zinc oxide.
—J. F. B.

Cellulose; Solution of. M. Fremery and J. Urban, Aachen, Germany. Eng. Pat. 6557, March 27, 1899.

The inventors claim a process for increasing the solubility of cellulose or hydrates of cellulose or of hydrocellulose, in direct solvents of cellulose, such as in cuprammonium or zinc chloride solution, characterised by subjecting the said materials, previously to the dissolving of the same, to an energetic treatment with oxidising or reducing agents, *e.g.*, with bleaching agents or with parchmentising agents, such as sulphuric acid of 59° B. This preliminary treatment enables solutions of cellulose in cuprammonium to be prepared up to 10 per cent. concentration, whereas the concentration of the solution of untreated cellulose is only about 4·5 per cent. The treatment must not, however, be carried too far, otherwise the cellulose molecule will be affected, and only thin, watery solutions will be obtained. The economy of the processes for the manufacture of artificial silk is considerably increased by working with the more concentrated solutions so obtained.—J. F. B.

Animal or Vegetable Fibre; Apparatus for the Treatment of. International Fibre Syndicate, Ltd., Dublin, and A. G. Dear, London. Eng. Pat. 3193, Feb. 13, 1899.

The fibre placed on suitable bands, is first passed through tanks containing "suitable degreasing and degumming" solutions, and then, after thoroughly washing and squeezing, it is opened by being rotated on a rapidly rotating roller. The fibre is then bleached by passing through a heated chamber containing bleaching gas.—C. M.

Washing Cellulose Threads, Films, &c. M. Fremery and J. Urban, Aachen, Germany. Eng. Pat. 6641, March 28, 1899.

The claim relates to a process for freeing cellulose threads, strips, or the like from the chemicals used in their manufacture, consisting in allowing washing liquid to flow over a number of spools containing the threads, &c., arranged one over the other, and moving up the lower spools as the washing of the upper ones is completed. The spools are supported horizontally on frames, the water is delivered from perforated pipes immediately on the top spools, and falls thence on each succeeding row of the same. When the spools of the top row are washed, they are removed and the others receive a step upwards, a fresh row being inserted at the bottom.—J. F. B.

Mercerising Cotton Fabrics under Tension. E. Simon, Villefranche, France. Eng. Pat. 25,638, Dec. 28, 1899.

An apparatus in which the cotton, having been mercerised, passes between rollers, to prevent longitudinal shrinkage. Transverse shrinkage is prevented by passing the fabric over a pair of inclined rotatory discs, which are adjustable as to distance apart and obliquity. These are in combination, also, with a pair of endless chains. Just before the fabric leaves the stretching apparatus it is thoroughly rinsed with water, then with dilute acids, and finally with water.
—C. M.

Vegetable Fibres for Paper-Making, Textile and other Purposes; Preparing. B. S. Summers, Chicago, U.S.A. Eng. Pat. 6346, March 23, 1899.

See under XIX., *page* 267.

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

Wool Dyeing; Application of Logwood in. Leipziger Färber- und Zeugdr.-Zeit. 1900, **49**, [8], 76.

Logwood is usually dyed upon either a chromium or iron mordant. As is well known, chromium is fixed on the fibre in two states of oxidation. The author in his paper proceeds to study the behaviour of logwood towards the "green" or non-oxidising chrome mordanted wool (chromic oxide), and towards "yellow" or oxidising chrome mordanted wool (chromic acid).

Green or Non-oxidising Mordant.—If wool be mordanted with chrome alum, chromium fluoride or oxalate in presence of oxalic acid, and dyed with haematoxylin, only a very weak colorisation is developed. If the process be carried out in an atmosphere of hydrogen, no colour is produced. On the other hand, if an equal quantity of haematein be employed, fine though rather light blue shades are obtained, which never approach a black, however much haematein be used. This proves that the true colour of the haematein-chromium lake is blue and not black. "Unfermented" or fresh logwood gives very poor results when dyed on the green mordant, whilst "fermented" or aged logwood yields dark blue shades (since this contains the colour as haematein) much duller than those obtained from pure haematein, as some of the brown oxidation products of haematein are always present and are fixed by the mordant.

The oxidation of haematoxylin is favoured by the presence of alkalis, hence it is customary in practice where "unfermented" or fresh logwood chips are employed to add calcium acetate to the dye bath. The same effect is obtained quicker by the use of hydrogen peroxide in suitable amount.

The Yellow or Oxidising Mordant.—Sodium and potassium bichromate must necessarily act as oxidising agents before they are reduced to the chromic condition, and it is possible to regulate the mordanting so that the reduction takes place either during that process or in the dye-bath or partly in each. This is effected by varying the amount of cream of tartar, tartaric or lactic acid used in the mordanting bath, or by altering the time of boiling. The reduction in the dye-bath is brought about by the haematoxylin or haematein and can be regulated by varying the composition of the mordanting bath. If the amount of oxidation be insufficient, the haematoxylin is, of course, wasted, and if too great, then the same results are obtained as would be produced by dyeing with haematein on a mordant of similar oxidising power.—H. I

Browns and Greys, Production of. J. Censi. Bull. Soc. Ind. de Mulhouse, 1899, 310.

By the action of water on certain nitroso derivatives of tertiary bases, brown and grey dyestuffs have been obtained. Thus, nitrosodimethyl aniline and its hydrochloride yield on warming with water, a brown, which may be rendered brighter by the addition of ammonia to the water, in which case the warming is continued until a sample dissolved in water exhibits the desired tint. After subsequent cooling, the precipitate is filtered off, and well washed.

The new base has been employed in the forms of hydrochloride, acetate, and oxalate. It can be applied to the various fibres "direct."

On long warming, the brown dyestuff is converted into a grey, but passes back into brown under the influence of oxidising agents.

By extending the above reaction to other bases, the following colours have been obtained:—From nitrosodimethylaniline hydrochloride, yellowish-brown; from nitrosodimethyl-*o*-toluidine, orange; from nitrosomethyl diphenylamine, blue.—J. A. P.

Blue Dyestuff used for German Infantry Uniforms. Leipziger Färber- und Zeugdr.-Zeit. 1900, **49**, [5], 46—49.

According to recent regulations the blue cloth used for the uniforms of the German infantry regiments has to be of lighter shade than formerly, and the dye must consist exclusively of Indigo and not of Alizarin Blue. It is further stipulated that the goods must be yarn and not piece-dyed.

The reasons for the changes are probably the facts that in light shades, Alizarin Blue is not so fast to light as Indigo, although it rubs off less and consequently wears better. Piece-dyed Indigo is well known to be more easily rubbed off than when dyed in the yarn.

In order to obtain good even results, care must be taken to use only one kind of wool, or, if mixtures be employed, only such wools as show a similar affinity for the dyestuff should be used. Under any conditions, however, the dyeing of the new light shades is much more difficult than the older darker ones. Whether a slight topping of too light pieces in the indigo vat is allowable, is still in dispute.

The shade prescribed for the military "Moltons" ("Meltons"?) is lighter than previously employed, but they may be dyed with Alizarin Blue, as they are less subject to wear and more loosely woven, so that better penetration of the colour is obtained.

For dyeing them, Alizarin Cyanine, Anthracene Blue SWX, and Acid Alizarin Blue are almost exclusively employed. The method of application of the first is well known, but Alizarin Blue SWX and SWX extra are very suitable for certain shades. According to a recent method they are dyed with the addition of 6 per cent. of ammonium acetate and 4 per cent. of oxalic acid to the dye bath, and after boiling one hour the goods are chromed by the addition of 2·5 per cent. of chromium fluoride and boiled until the colour is developed.

Light shades of the cloth for military "Moltons" are dyed with Acid Alizarin Blue B.B. (M. L. & B.). The bath, to which 25 per cent. of Glauber's salt and 4 per cent. of sulphuric acid has been added, is boiled and then filled up with cold water. When the goods are entered and after running for some time, the colour (2·3 per cent.), dissolved in boiling water, is added. The temperature is then raised to the boiling point, and after boiling for 1½ hours it is allowed to fall and 2·5 per cent. of chromium fluoride dissolved in hot water gradually added. The goods are then "chromed" by heating for 1—1½ hours, until the colour is developed.—H. I.

Dyed Goods; Effects of Finishing and Storage on. Textile Colorist, 1900, **22**, [253], 8—9.

The writer calls attention to the well-known difficulty experienced by dyers, when matching-off a given sample of cloth, in allowing for the changes of shade brought about in dyed goods by the sizing and finishing operations. Although the former has the most effect, yet very marked changes result from the thorough drying and hot-pressing to which fabrics are subjected after dyeing. Blue colours, both substantive and adjective, are especially subject to alteration by heat, becoming redder in tone; and some of the yellows, under the combined influence of heat and moisture, change to orange. The storage of dyed goods, bobbins of yarn, &c., in direct sunlight, or near boilers, so that when the fires are raked the gases from the furnace come in contact with them, or near open windows close to a dyehouse or bleach works, often gives rise to defects for which the dyer is wrongly blamed.—H. I.

Chlorate-prussiate Discharge; Impts. in the. Koechlin Bros. Bull. Soc. Ind. de Mulhouse, 1899, 317—318.

An addition to the original "discharge colour," of salts easily dissociating at 100° C., such as the ammonium salts, and those of aluminium with non-volatile acids (*e.g.*, sulphates, oxalates, tartrates, citrates, &c.), renders it applicable to almost all dyestuffs, no matter in what strength they are dyed. Thus, it is effective on any shade of "Alizarin V," whether produced by dyeing or by padding and steaming, and whatever the mordant employed.

For Alizarin-Blue, -Green, -Orange, and -Black, the Gallocyanines, Cœruleïn, &c., it is equally effective.

It is also applicable to Logwood, "Bark," and Persian-berry shades, and to such as are dyed with St. Denis Red, Magenta, Resorcinol Green, Cachou de Laval, &c.

Methylene Dark-blue is not completely discharged—a tinge of violet remaining, whilst Aniline Black, Alizarin Yellow, the Naphthol Colours, and Kern and Sandoz' Orange remain unchanged.

The "discharge colour" is prepared by mixing together 10 litres of aluminium chlorate (or a mixture of aluminium and ammonium chlorates) and 1 kilo. of starch (proportion of 3 kilos. to every 600 grms. of crystals); then warming to 80° C. and when nearly cold again, adding 400—800 grms. "red prussiate," and 2—2½ kilos. of ammonium tartrate, citrate, or oxalate.

After printing with this mixture, the cloth is steamed in the Mather and Platt apparatus at 100° C., then soaped and washed. Yellow discharges may be obtained (1) by omitting the ammonium tartrate—or similar substance, and incorporating in the "colour" a lead salt, a subsequent passage through bichrome being of course necessary; or (2) by a direct addition of either Kern and Sandoz' Orange or of Alizarin Yellow, along with a suitable chromium salt. A *Blue* discharge is obtained by passing through an "iron solution" before washing.

For pinks, Safranine may be used.

If tartrates, &c. be not employed the discharged white may be coloured by adjective dyestuffs which will combine with the alumina fixed there, without, as a rule, greatly affecting the "ground."

Recipes for Aluminium Chlorates:—(1) 670 grms. of aluminium sulphate, 460 grms. of barium chlorate, 2 litres of water; the mixture to stand at 38° Tw. (2) 50 kilos. of aluminium sulphate, 50 litres hot water, then 13 kilos. of potassium chlorate.

Recipe for double Aluminium and Ammonium Chlorate:—680 grams of ammonium alum, 960 grams of barium chlorate, 2 litres of water.

In each case allow to stand, and decant clear solutions for use.—J. A. P.

Aniline Black and Diazo-Dyestuffs; Nitrite as Resist for. W. Popielsky. Färber-Zeit. **11**, [3], 39—40.

Aniline Black.—This method depends on the fact that the diazo-compound formed by aniline salt and nitrous acid does not undergo oxidation. By impregnating a fabric with aniline black paste and printing over a nitrite-resist there is produced in the printed parts the diazo-compound, which on subsequent ageing, prevents the formation of the Aniline Black. In this way various coloured effects can be obtained upon Aniline Black by using suitable dyestuffs with the nitrite-resist. For a white-resist, sodium nitrite is employed, properly thickened with gum tragacanth.

Auramine, Rhodamine, or Malachite Green are combined with the sodium nitrite for coloured resists, but in this case albumin and zinc acetate are used in addition.

If β- naphthol be used instead of Auramine, &c., cachou shades are obtained which are produced as follows:—impregnate the fabric with Aniline Black, print, steam, wash in hot water, and finally develop the black in potassium bichromate solution.

Diazo-Dyestuffs.—The process is based on the fact that β-naphthol and nitrous acid yield nitroso-β-naphthol, which does not combine with the diazo-compound.

The author has not succeeded in getting a pure white by this method, since the nitroso-naphthol formed on the fabric cannot be completely washed out.

The process is carried out in the following manner:—the fabric is impregnated with β-naphthol and sodium nitrite (20 grms. per litre), dried, printed with coloured-resist, passed through solution of diazotised dyestuff, washed in cold water, run through tannic acid solution at 40°—50° C. (10 grms. per litre), washed in hot water, fixed with antimony, soaped at 40° C., and again washed.

The coloured-resist is composed of Crystal Violet or Brilliant Green and tartaric acid with thickening.

It is evident that dyestuffs which contain a NH_2 group must not be used in either of the above processes.—J. E. H.

Silico-Tungstic Acid as a Mordant for Basic Aniline Colours. A. Scheurer. Leipziger Färber- und Zeugdr.-Zeit. 1900, **49**, [6], 60.

A study of the behaviour of silico-tungstic acid towards alkaloids by C. Bertrand (this Journal, 1899, 404) led the author to devise the following mode of application:—

If sodium tungstate be dissolved in sodium silicate a certain quantity of a white precipitate (soluble in soda) separates. The filtered solution, on boiling, also deposits a precipitate, and in the cold is precipitated by organic and mineral acids, by ammonia, carbolic acid, and by sodium phenate.

If goods be treated with the solution and dried, this precipitate is left on the fibres as a mordant, the amount fixed can be further increased by boiling in acetic acid. This mordant possesses strong affinity for basic dyestuffs. The colour produced with Methylene Blue is fast to soap up to 100° C., but, on boiling, the colour lake is attacked, and in 10 minutes loses three-quarters of its strength. Silico-tungstic acid, however, gives lakes which resist soap better than those derived from tungstic acid alone.—H. I.

Mordanting Wool with Chrome. R. Löwenthal. Färber Zeit. 1899, **10**, [19], 301—303; and [20], 317—321.

The compounds of chromium principally employed in mordanting wool, are the bichromates of sodium and potassium. The active constituent of both of these is chromic acid, which is liberated when their solutions are acidified with sulphuric acid. Chromic acid is not itself a mordant, but it becomes changed during the process of mordanting, or afterwards, into chromic hydrate which is the real chrome mordant. Instead of applying the acid and effecting its reduction to chromic hydrate, the latter may be directly introduced into the fibre by the use of certain chromium salts. The results, however, are in general inferior to those obtained when chromic acid or a bichromate is used.

Chromium fluoride is employed in the proportion of 4 per cent. of the weight of the wool to be mordanted, along with 2 per cent. of oxalic acid. The wool is introduced into a warm solution of these compounds, which is heated in half an hour to the boil, and is boiled 1—$1\frac{1}{2}$ hour. The drawbacks to the use of the salt are the harsh feel which it imparts to the wool, its relatively high cost, and the fact that in applying it copper vessels are required. On the other hand, owing to its not possessing any oxidising action, it is to be preferred to chromic acid when the dyestuff on the fibre, or subsequently to be applied, is sensitive to oxidation. It is also very suitable for use in the single-bath method of mordanting and dyeing.

Chromium oxalate, which may be readily prepared by dissolving chromic hydrate in a solution of oxalic acid, has been proposed by Hummel and Liechti (this Journal, 1894, 222 and 356) as an efficient and cheap mordanting salt. An amount, equivalent to 4 per cent. of chrome alum, gives without further addition to the mordanting bath, an excellent mordant.

Chromium acetate has been recommended by the Badische Anilin und Soda Fabrik for use in the dyeing of pale shades of Alizarin colours by the single bath method.

Chromic acid appears to be employed only in the United States of America, where its use has been patented by O. P. Amend (Eng. Pat. 30,039, of 1897; this Journal, 1898, 243). An advantage connected with its application is that the operation of mordanting can be performed at a temperature below the boil, the wool being, in consequence, left in a better condition for spinning and weaving. The following directions are given:—Heat the mordanting bath to 75°—80° C. Add to it 6 per cent. (of the weight of the wool) of a 30 per cent. solution of acetic acid. Introduce the wool and work it for half an hour. Add 1 per cent. of chromic acid and work for another half hour. Then add a 6 per cent. solution of sodium bisulphite, raise the temperature to 75°—80° C., and maintain it at this for half an hour. Drain the wool well and dye with the addition of 1 per cent. of a 20 per cent. solution of ammonia, in place of the usual addition of acetic acid. With regard to this process, the author finds that the chromic acid may be replaced by a mixture of potassium bichromate and sulphuric acid. Further, the proportion of chromic acid given is not sufficient for a full mordanting. Again, sulphuric acid may be employed instead of acetic acid. Excellent results have been obtained in cloth dyeing with a mordant produced by the use of 3 per cent. of potassium bichromate, 3 per cent. of sulphuric acid, and a 12 per cent. solution of sodium bisulphite. An improvement may be made in the process by reducing in a separate bath the chromic acid, which is absorbed by the wool, thus keeping the chroming bath for repeated use. The difference between this process and the ordinary process of mordanting wool with chrome is, therefore, essentially in the temperature at which the operation of mordanting is conducted. Under otherwise similar conditions, less chromium is deposited on the wool at temperatures below the boil than is deposited at the boil.

When the mordanting is accomplished by means of the bichromate of sodium or potassium, the wool is immersed in a lukewarm solution of 2—4 per cent. of its weight of the bichromate. The solution is then gradually heated to the boil and boiled for 1—$1\frac{1}{2}$ hours. Of 3 per cent. of the salt applied, only 1 per cent. enters into reaction with the wool, the remainder for the most part becoming transformed into the normal chromate which remains in the bath. To utilise the mordanting salt more effectually, an assistant must be employed, that most commonly preferred being sulphuric acid in the proportion of 1—3 per cent. Under the influence of this, about two-thirds of the chromium applied to the wool becomes absorbed by it. The mordant is not, however, so even as that which is obtained when an alkali bichromate is used alone. It is, therefore, to be recommended only in connection with the dyeing of dark shades. Hummel and Gardner (this Journal, 1895, 452) have shown that hydrochloric acid and nitric acid act much more powerfully than sulphuric acid in promoting the absorption by wool of chromium from solutions of alkali bichromates. With 2 per cent. of potassium bichromate and from 4 to $7\frac{1}{2}$ per cent. of hydrochloric acid (32° Tw.), the wool is strongly mordanted, the whole of the chromium being absorbed. The mordanting is probably not so even as when potassium bichromate and sulphuric acid are used. Practical experience must decide the value as a mordanting assistant of the product known as "Egalisol," which, it is stated, is a compound of boric and sulphuric acids.

The process of reducing by the action of sodium bisulphite the chromium which is present in the form of chromic acid on wool mordanted by means of chromic acid or a bichromate (this Journal, 1890, 60), is but little practised on account of the necessity for an additional bath. The reduction is more conveniently accomplished by the use of organic acids or acid salts in the mordanting bath. Of these that most used is tartar, which gives an even mordant suitable for the majority of dyestuffs. The chromium tartrate which is formed in the mordanting bath in the course of the operation dissociates partly in the bath, and partly on the fibre, into chromic hydrate and tartaric acid. A portion of the mordant thus precipitated in the bath is deposited upon the wool. The mordant consists chiefly of chromic hydrate, with a small amount of chromic acid. Suitable proportions for medium shades are 3 per cent. of potassium or sodium bichromate, and $2\frac{1}{2}$ per cent. of tartar; for pale shades, 1 per cent. of each; for very dark shades, 4 per cent. of the former, and 3 per cent. of the latter. The amount of chromium fixed on the fibre is not appreciably increased by the action of the tartar. Oxalic acid has more influence on the fixation of the chromium, resembling in this respect sulphuric acid, but acting less powerfully. 1—$2\frac{1}{2}$ per cent. of it is used, along with 3—4 per cent. of potassium bichromate. The mordant being less regularly fixed and less reduced than that from potassium bichromate and tartar, its use is restricted to the dyeing of medium and dark shades. Lactic acid, in the proportion of 3—5 per cent. of a 50 per cent. solution, and 2—3 per cent. of potassium bichromate, give a stronger, browner mordant than tartar and the same proportion of the bichromate. With an addition of sulphuric acid, the chromium is still more completely deposited on the fibre, a mordant for dark shades being obtained with $1\frac{1}{2}$ per cent. of potassium bichromate, 3 per cent. of lactic acid (50 per cent.), and 1 per cent. of sulphuric acid. Dreher (this Journal, 1898, 921) has suggested the employment of ammonium sulphate in place of sulphuric acid. This salt is gradually decomposed in the bath, the liberated sulphuric acid entering into action. The compound known as Lignorosin (Eng. Pat. 19,005 of 1897; this Journal, 1898, 45; see also this Journal, 1898, 923), when applied with sulphuric acid and potassium bichromate, imparts to the wool a pale brown colour.

A mixed chromium-copper mordant, frequently used in dyeing with Logwood and other natural dyestuffs, is obtained with 2 per cent. of potassium bichromate, and 3 per cent. of copper sulphate, with or without the addition of sulphuric or oxalic acid. The mordant is unsuitable for Alizarin dyestuffs generally.

In mordanting for dyeing with Alizarin Blue, Alizarin Cyanine, Anthracene Blue and certain other dyestuffs, chromic acid and its salts should not be used. Chromic acid is especially injurious in mordanting wool which has been dyed with Indigo. In such a case potassium bichromate may be used with a small quantity of tartar or lactic acid. When the dyeing with Indigo is performed after mordanting, chromic acid may be employed in this operation, but it is advisable to complete the reduction by a passage through a bath of sodium bisulphite (Ger. Pat. 84,974). Schnabel and Ernst have shown that the destructive action of alkali bichromates upon Indigo is decreased by the addition of tartar and lactic acid, and is increased by the addition of sulphuric acid, and to a greater degree by that of oxalic acid. The greatest action is, however, exhibited by a mixture of potassium bichromate, copper sulphate and sulphuric or oxalic acid.—E. B.

Bronze Printing. Textile Colorist, 1900, **22**, [253], 3—5.

All classes of textile fabrics may be printed with metallic powders for decorative purposes.

Two methods of application are in use: for thin goods, the ordinary cylinder machine is employed, whilst for thick, rough, or pile fabrics, the "Perrotine," or even hand-blocks are used.

The vehicle must be one that will so fix the powders to the material that even brushing will not remove them, also, their brilliancy must not be destroyed.

Two classes of "fixers," or vehicles, are at present employed in the trade: egg or blood albumin, and various varnishes having caoutchouc as their base. As an example of the former, the following mixture may be cited:—½ gall. of gum tragacanth paste (65:1,000) and 1¼ galls. of egg albumin solution (1:1) are, when thoroughly mixed, incorporated with 8 lb. of the metallic powder, giving a total volume of nearly 2 galls. The gum tragacanth paste and the egg albumin solution are prepared in the usual way by allowing the dry materials to soak in cold water, &c.

In using the above mixture on the cylinder machine, a lap apron is employed to furnish the colour from the box to the printing roller, whilst for block printing, the ordinary pad suffices. This method will not answer for pile fabrics, especially if they be of silk. Before printing, the goods are slightly damped so that the paste has a better ground to hold on, and after printing they are dried without the application of heat, and steamed for 15—20 minutes. If the fabric has been previously dyed, as is generally the case, it should be well washed before printing, which is the last operation. The paste has no effect on the dyestuffs.

The rubber paste is made from pure unvulcanised india-rubber, as the sulphur in the vulcanised rubber would injure the colour of the metallic powders. The following mixture may be used:—2 lb. of rubber and 4 lb. of camphor oil are heated together in a pan until a homogeneous mass has been obtained. After cooling to a certain extent, 1 quart of "naphtha" is added for thinning, followed by 1 pint of pure copal varnish, and the whole mixture is thoroughly stirred. After further cooling, the requisite quantity of the bronze powder is introduced and well mixed.

By the printing direct with a pale thinned copal varnish or a good quality of gold size, and dusting on the powders, various pleasing effects are producible.

With regard to the metallic powders, there is a wide range of shades in the market, and the colour mixer does not get his particular shade by mixing two powders (a method which does not give good results), but buys them direct. The colours obtainable vary from silver to reddish-brown, including yellows, greens, and blues of great brilliancy.

Other quick drying pastes have been tried, but the solvents proved too volatile for good work.

For hand block printing, the paste used by wallpaper printers answers fairly well; although it will not withstand prolonged wear, it is useful for velvets and plushes of short pile such as are used for albums and cheap plush covered boxes.

The main point in printing all such goods is that the impression shall be sharp, and applied with such force as shall effectually prevent the particular fibres from again rising.—H. I.

Hydrosulphurous Acid. A. Bernthsen and M. Bazlen. Ber. 1900, **33**, [1], 126.

See under VII., *page* 244.

PATENTS.

"Jigger" Machines for Dyeing. H. Newell, Walsden. Eng. Pat. 2707, Feb. 7, 1899.

A combination with a "jigger" dyeing machine of squeezing rollers and a plaiting apparatus. A trough is placed under the squeezing rollers, from which a pipe conducts the recovered dye liquor to the dyeing machine. One set of squeezing rollers may be made to serve several "jiggers," arranged side by side.—E. B.

Book-binders' Cloth. S. L. Wood and The Park House Dyeing Co., Salford. Eng. Pat. 3914, Feb. 22, 1899.

The warp and weft of the cloth are dyed with different colours, and the woven fabric then finished, calendered, and backed in the usual way. In order to render the sized fabric resistant to moisture, it is treated with a solution of gelatin, and afterwards with a solution of formaldehyde or potassium bichromate. Another method is to coat the woven cloth with a clear solution of celluloid in amyl acetate.—C. M.

Dyeing [Blues from Immedial and Vidal Blacks, &c.], Impts. in. R. B. Ransford, Upper Norwood. From L. Cassella and Co., Frankfort-on-Maine, Germany. Eng. Pat. 4069, Feb. 23, 1899.

In the presence of alkalis, a mixture of superheated steam and air, at a temperature exceeding 100° C., converts Vidal and Immedial Blacks and Kryogene Blue, on vegetable fibres, respectively into dark greenish-blue, dark reddish-blue, and very dark blue. As the process of dyeing with these dyestuffs is performed in a bath containing sodium sulphide, which is sufficiently alkaline for the purpose, the dyed materials are conveniently treated with the air and steam, before they are washed and dried. Ammonia may also be used as the alkali necessary for the reaction, being introduced in the state of gas into the oxidising apparatus (see also this Journal, 1899, 491).—E. B.

Indigo on the Fibre [Kalle's Indigo Salt], Production of. G. W. Johnson, London. From Kalle and Co., Biebrich-on-Rhine, Germany. Eng. Pat. 7779, April 13, 1899.

Fabrics composed of vegetable fibres are printed with a mixture of 0·7 kilo., of "Indigo salt" (Eng. Pat. 4287 of 1893; this Journal, 1894, 145) dissolved in 1·3 litres of water, 2·5 kilos. of British gum thickening, and 5·5 kilos. of caustic soda-lye (40° B.). The fabrics are dried, steamed without back cloths for 7 minutes, washed, soured and again washed. The blue produced is bright and has a reddish tinge. The advantages of this method, compared with the older method (this Journal, 1893, 998 and 988), are that the "Indigo salt" is "utilised to a much greater extent" and produces much deeper shades, and that it can be printed along with steam colours.—E. B.

Dyeing Fabrics or Yarns made of Cotton or other Vegetable Materials [Drabs from Oxides of Copper and Iron] and Producing Figures on such Fabrics. F. I. Horrocks, Sale. Eng. Pat. 7888, April 14, 1899.

The fabrics are impregnated with mixed solutions of iron and copper salts, precipitable by alkalis, and are passed into solutions of the latter. Suitable salts and concentrations of solutions are:—Copper sulphate, 8° Tw.; ferrous sulphate, 2°–6° Tw.; sodium carbonate, 2°—4° Tw. Fast drab colours are obtained.

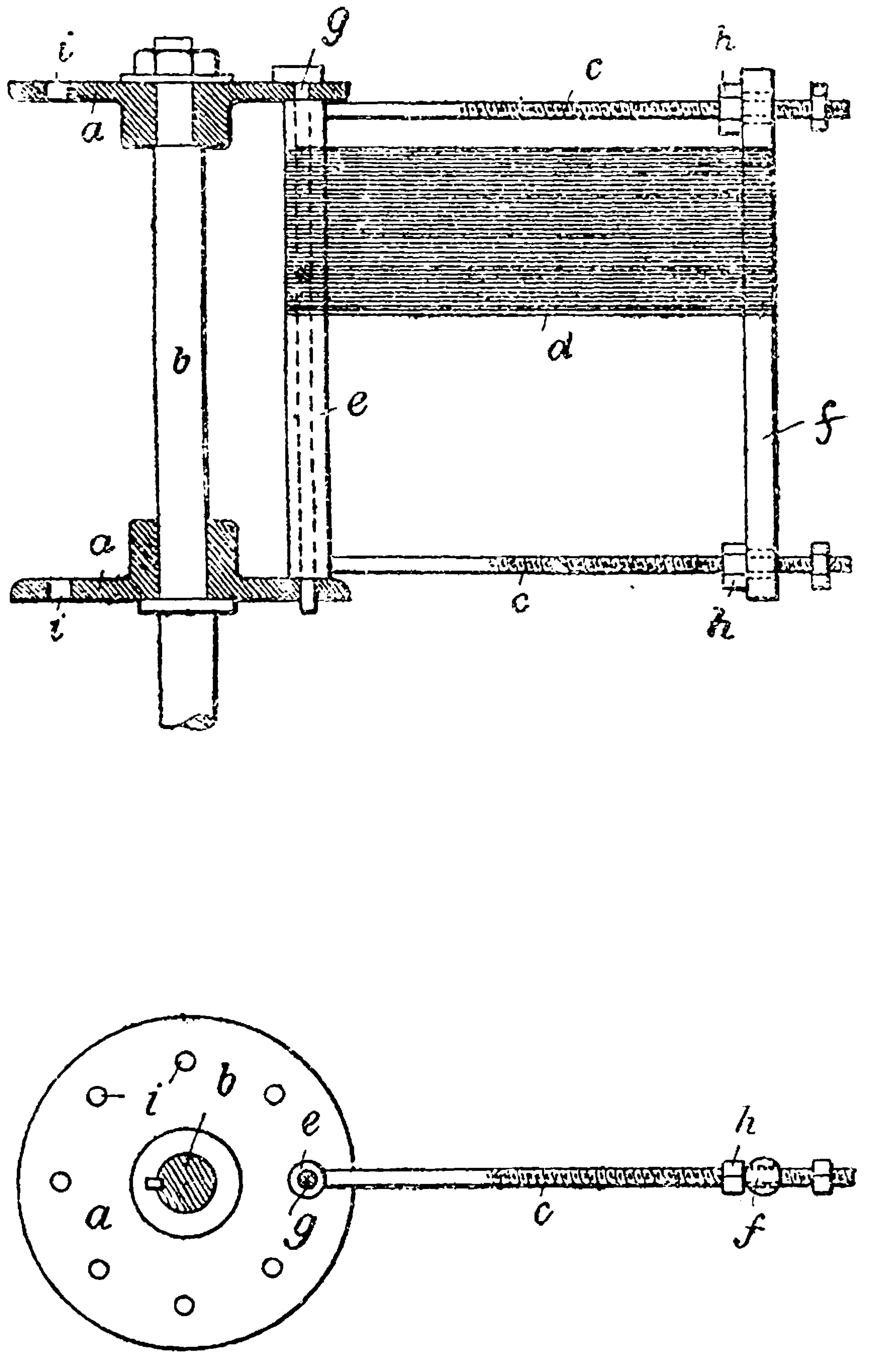
Patterns are produced by engraving a roller and employing it as one of a pair of squeezing rollers in removing the excess of the solution of iron and copper salts, the fabric being passed into the fixing bath immediately after it has been squeezed. The parts of the fabrics which are pressed by the raised portions of the roller, retain less of the solution, and become less coloured than the other parts. —E. B.

Dyeing, Washing, Bleaching; Impts. in the Method of, and in the Apparatus used in. P. Schirp, Barmen, Germany. Eng. Pat. 22,948, Nov. 17, 1899.

A number of dye-tanks are placed above a common collecting vessel. The requisite solutions are placed in the latter and are pumped into the dye-tanks from which they flow, through the materials to be dyed, back to the collecting vessel. The solutions in the tanks must be maintained at a level sufficiently high to submerge the materials contained therein. This is effected by regulating the outflow by means of valves attached to the pipes connecting the tanks and collecting vessel.—E. B.

Stretching Frames for Hanks or Skeins of Yarns or the like in Mercerising, Bleaching, Dyeing, and like Operations. W. Herschmann, Vienna, Austria. Eng. Pat. 23,098, Nov. 29, 1899.

The yarns to be treated are placed upon frames (see figure), composed of rods *c* and bars *e* and *f*. Tension is put upon them by means of nuts *h* working in screw-threads upon the rods. The frames are fixed upon two discs *i*, attached to a vertical shaft *b*. When this is revolved, the centrifugal action tends to move the outer away from the inner bars, and thus further to stretch the yarns.



Apparatus for Lustreing Cotton Yarns.

—E. B.

Solids [Pigments, Dyes, Fibres, &c.] from Liquids containing them, Separation of. A. Ross, Sutton, Surrey. Eng. Pat. 3493, Feb. 16, 1899.

See under I., *page* 229.

VII.—ACIDS, ALKALIS, AND SALTS.

Hydrosulphurous Acid. A. Bernthsen and M. Bazlen. Ber. **33**, 1900, [1], 126—132. See also this Journal, 1898, 1109; 1899, 452.

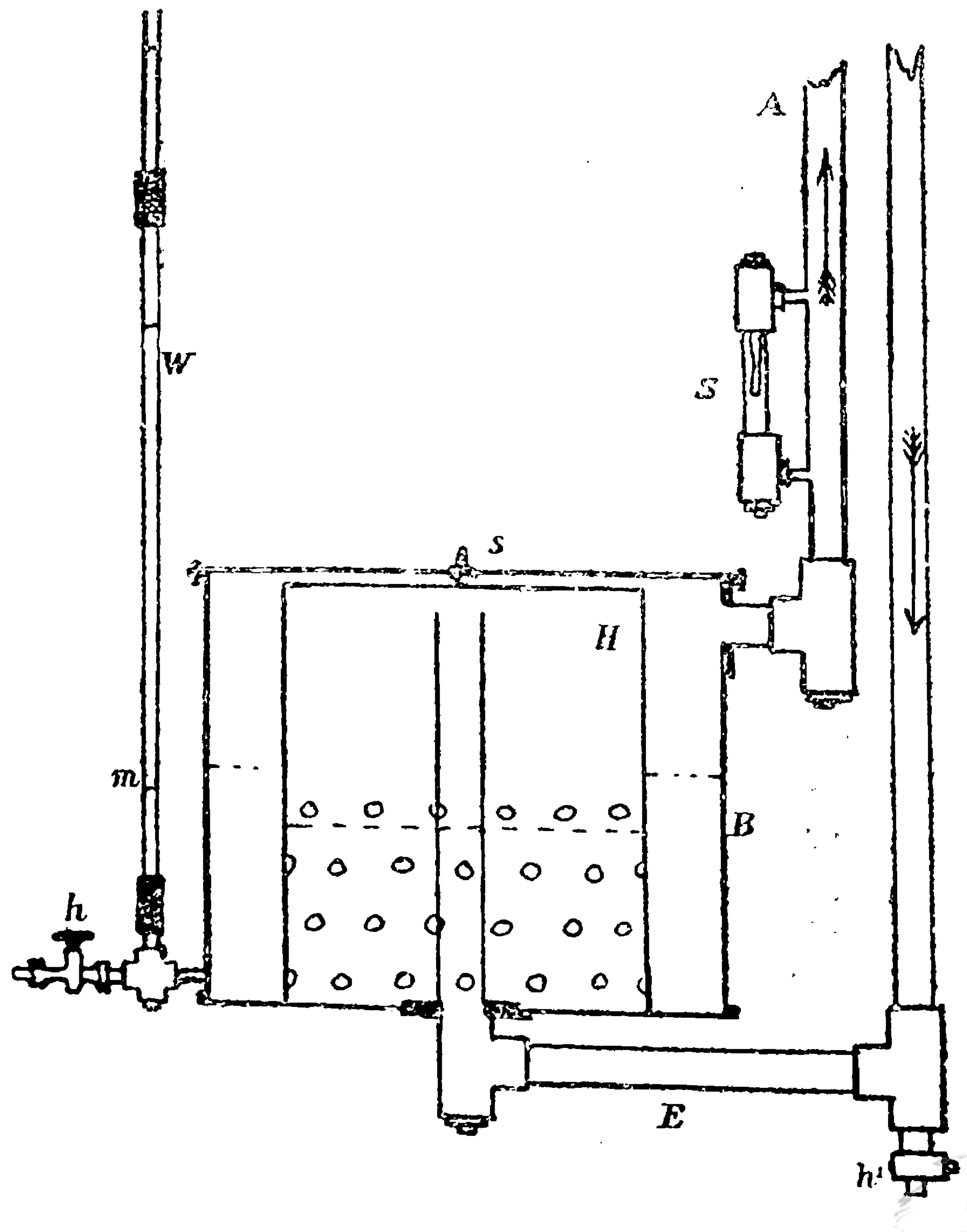
In the ordinary method for the preparation of sodium hydrosulphite by means of zinc dust and sodium bisulphite, a considerable proportion of the sulphurous acid is lost, owing to the formation of normal sodium and zinc sulphites. Attempts to obviate this loss by the addition of other acids have met with but partial success. If, however, sulphurous acid be added, the whole of the bisulphite is converted into hydrosulphite—

$$2NaHSO_3 + SO_2 + Zn = Na_2S_2O_4 + ZnSO_3 + H_2O.$$

On the addition of milk of lime to the resulting mixture, the zinc, calcium, and sulphurous acid are precipitated and the solution contains sodium hydrosulphite only. By the use of gaseous sulphur dioxide, solutions are technically obtained of 15°—16° B., 10 kilos. of which reduce nearly 2 kilos of indigo. From this solution, salt separates solid sodium hydrosulphite; if it be dissolved in the warm liquid, fine crystals are obtained on cooling. The hydrosulphite is obtained in stable form, if it be filtered in the absence of air, the mother liquor removed by aqueous followed by pure acetone and then dried in a vacuum. The crystallised salt possesses the formula $Na_2S_2O_4.2H_2O$, it reduces sulphonated indigo quantitatively, and the yield in the above process is almost double what would be the case if the formula were $NaHSO_2$. In the moist state, the salt rapidly oxidises in the air forming sodium pyrosulphite with evolution of heat. The dry salt loses little of its reducing power on keeping for months in a closed vessel. The conclusions of Grossmann (*loc. cit.*) are criticised, the authors have observed no indications of the existence of acid salts, the addition of a small quantity of acid to the neutral hydrosulphite causes at once a red coloration, and on standing sulphur separates.—A. C. W.

Ammoniæ Liquor, Manufacture; Soda Washer for. O. Pfeiffer. J. f. Gasbeleucht. **43**, [6], 89—91.

The author pointed out in an earlier communication (this Journal, 1898, 231) that when gas liquor was heated prior to its distillation for the production of liquor ammoniæ, about two-thirds of the ammonia present in it existed in the free state, and that consequently it was only necessary before distilling to add an amount of lime equivalent to the remaining one-third to the charge of previously heated liquor in the still. Practically, however, a slightly greater proportion of lime facilitated complete working off of a charge of gas liquor in a reasonable time; for instance, whereas with the theoretically equivalent amount of lime, 10 hours were required to reduce the ammonia in the treated liquor to 0·03 per cent., an excess of 20 kilos. of lime for working a charge of about 3 cb. metres of gas liquor, brought about the same result in eight hours. The theoretical amount of lime (CaO) was calculated as 20 kilos.



per cb. metre of gas liquor. The cost of the additional 20 kilos. of lime was far less than the expenditure on steam and wages for two additional hours.

Provided there is no hitch in the working, and the previous heating of the liquor has been adequate, no sulphuretted hydrogen will pass through the usual train of milk of lime washers, condenser and charcoal filters to the vessels in which the ammonia gas is absorbed in cooled water. But if the previous heating has not sufficed to drive off the acid gases, sulphuretted hydrogen may pass forward and contaminate the product and foul the whole train of apparatus. Such ill effects may be avoided by the insertion, between the charcoal filters and the absorption vessel, of a washer charged with a solution of caustic soda in ten times its weight of water. Such a washer, as used at the Magdeburg gas works, is shown in the annexed figure, in which B is a covered cylinder 0·67 m. high, and 0·80 m. in diameter; and H a cylinder, 0·65 m. high and 0·56 m. in diameter, perforated, to a height of 0·30 m. from its open bottom, in holes 0·015 m. in diameter. The tops of the cylinders are connected at *s*. The solution of soda is run in through the cock *h*, until it rises to the mark *m* on the glass tube W, which is continued upward to a height of 2 m. by $\frac{1}{4}$-inch gas piping to serve as a safety pipe in case of excessive back pressure in the apparatus. The ammonia gas from the charcoal filters enters the inner cylinder by the pipe E, and having escaped through the perforations to the outer cylinder passes by the pipe A to the absorption vessels. S is a glass tube communicating at both ends with A, and containing a slip of paper saturated with solution of lead acetate. The solution of soda in the washer must be changed as soon as discolouration of this lead paper indicates that sulphuretted hydrogen is present in the ammonia gas issuing from the washer.—J. A. B.

Magnesium; Action of, on Saline Solutions.

H. Mouraour. Comptes Rend. **130**, [3], 140—141.

Magnesium reacts, with more or less rapid evolution of hydrogen, not only on solutions of magnesium salts (Lemoine, Comptes Rend. **129**, 291), but also on solutions of many alkali salts, particularly ammonium salts. Probably the activity of ammonium salts is due to their dissolving the magnesia formed, and thus keeping clean surfaces of metal exposed; but the evolution of hydrogen along with the deposition of metal which occurs when magnesium acts on salts of copper, lead, tin, &c., can hardly be thus explained.—J. T. D.

Ammonium Amidosulphite. E. Divers and M. Ogawa.

Proc. Chem. Soc. **16**, [219], 38.

Dry ammonia does not combine with sulphur dioxide at a low temperature. When slightly moist, it does so, with such rise of temperature that the product of the union is at once partly decomposed. But by dissolving the ammonia in dry ether, free from alcohol, and passing the sulphur dioxide into the solution, kept very cold, the product is precipitated and undecomposed. It is colourless, very deliquescent, and unstable in the air. It dissolves in water with a hissing sound, and, when the water has been well cooled, yields a solution which gives the reactions of ammonium sulphite only. It is also very soluble in absolute alcohol, forming the salt lately described by the authors, ethyl ammonium sulphite. It is formed from two mols. of ammonia and one mol. of sulphur dioxide. In a dry atmosphere, at temperatures such as 35° C. it loses nearly half of its ammonia, as well as a little water. After the decomposition, no sulphite-forming substance remains, and the mass is somewhat orange-coloured from the presence of a very small quantity of a red substance. A very little sulphate is also present.

The undecomposed salt appears to be correctly named *ammonium amidosulphite*, and to have the formula $NH_2.SO_2.NH_4$. The preparation of this salt proves that Rose's assertion, that the gases unite only in equal volumes forming an orange substance which at once decomposes with water into sulphate and trithionate, is altogether wrong, he no doubt having failed to prevent the decomposition of the product first formed.

Alkali Nitrites; Colour of. E. Divers. Proc. Chem. **16**, [219], 40.

As Boguski (J. Russ. Chem. Soc. 1899, **31**, 543) has published his observation that a solution of sodium nitrite had a slight yellow colour, which he could not remove, and which he attributes to the presence of an impurity, the author wishes to recall attention to his own statement (this Journal, 1898, 1146) that alkali nitrites have a slight yellow colour, especially in solution. The author desires now to reiterate his claim for accuracy as to this statement. The colour of the potassium salt is much more manifest than that of the sodium salt, principally because of its ability to yield exceedingly concentrated solutions. Solutions of potassium nitrite, three parts of salt in one of water, are strongly yellow, particularly when thoroughly free from turbidity. Solutions of equal strength are equally coloured, whatever has been the source of the nitrite. Alkali nitrites are yellow in the fused state. The author is confident that Boguski's promised endeavour to remove the colour of sodium nitrite will be as futile as were those of chemists, long years ago, to remove the pink colour from manganous salts. Boguski's observation that the solid nitrite is sometimes apparently without colour, depends upon the well known optical effect of fine division in lessening the colour of transparent solids.

Mixed Potassium Nitrite and Nitrate; Solubility of.

E. Divers. Proc. Chem. Soc. **16**, [219], 40.

By crystallising out, as far as possible, the nitrate which is mixed with potassium nitrite in its usual method of preparation, there remains a mixture of about three parts of nitrite to one part of nitrate, and this constitutes the ordinary nitrite of commerce. This mixture is soluble in one-fourth its weight of water, although the pure nitrite requires one-third of its weight, and the pure nitrate about four times its weight of water to dissolve it. Accordingly, when a solution of three parts of pure potassium nitrite in one part water is digested with powdered potassium nitrate at the common temperature, it readily dissolves up one part of this salt.

Sulphur Dioxide with Oxygen; Combination of. E. J. J. Russell and N. Smith. Proc. Chem. Soc. **16**, [219], 41.

The authors have found that when a mixture of sulphur dioxide and oxygen acts on certain oxides, in addition to the absorption of the sulphur dioxide, part of the sulphur dioxide and oxygen combine forming sulphur trioxide, this being apparently due to the "surface action" of the oxide. The extent of this "surface action" varies with the nature and physical conditions of each oxide. No sulphur trioxide was ever found unless a simultaneous absorption of sulphur dioxide occurred; when manganese peroxide and sulphur dioxide, dried by phosphorus pentoxide, were brought together, no absorption took place, nor was any sulphur trioxide produced on the addition of dried oxygen.

If a mixture of dried sulphur dioxide and oxygen be passed over well-dried platinised pumice heated to 400—450°, very little sulphur trioxide is formed, and the drier the materials the less is the combination.

Sulphur, Estimation of Gaseous Compounds of. E. J. Russell. Proc. Chem. Soc. **16**, [219], 41.

See under XXIII., *page* 278.

Potassium Chloride; Electrolysis of. A. Brochet.

Comptes Rend. **130**, [3], 134.

See under XI. A., *page* 252.

Alkali Persulphates and Hydrogen Peroxide; Valuation of. B. Grützner. Chem. Centr. 1900, **1**, [7], 435.

See under XXIII., *page* 276.

Alkali Chlorides and Nitrates; Conductivity of Aqueous Solutions of. F. Kohlrausch and M. E. Maltby. Preuss. Akad. Wiss. Berlin, S. Ber. 1899, **36**, 665.

See under XI. A., *page* 252.

Nitrates; New Method for the Determination of. C. M. van Deventer. Chem. Centr. 1900, **1,** [4], 265.

See under XXIII., *page* 277.

Iodic Acid in Nitrate of Soda; Estimation of. R. Auzenat. Monit. Scient., Feb. 1900, **14,** 72.

See under XXIII., *page* 277.

PATENTS.

Sulphuric Acid; Apparatus for Concentrating. J. L. Kessler, Clermont Ferrand, France. Eng. Pat. 26,169, Dec. 10, 1898.

This apparatus is easily cleansed from solid impurities separating when such acids as those obtained from the Glover's tower, or the residues of the acids of nitration of organic acids, are concentrated. The lower part of the apparatus, the "saturex," which receives the acid to be concentrated from a series of superposed plates above, is subdivided into a number of channels in which the hot gases admitted to the apparatus, circulate. The partitions are moulded of pumice moistened with an alkali silicate solution of 20° or 30° B., strongly dried or calcined. The channels have closed openings in the surrounding lead casing, whereby they may be cleaned; there are also transverse gutters terminating in lateral openings. A stated depth of acid is maintained in the "saturex," in order that the deposits may become granular, and leave the acid clear. The hot gases pass upwards from the "saturex" through suitable perforations into a tower in which a series of superposed plates or slabs are ranged, internally recessed in the form of troughs the bottoms of which are perforated. Porcelain parts, with caps and other details, are so fitted that a hydraulic seal is formed, so that the ascending hot gases necessarily traverse the acid which is charged primarily into this part of the apparatus, from which the "saturex" is fed, and, as they pass upwards through tier after tier, give up much of their heat, and escape, with water vapour, into a flue at the top. The level of the acid in each plate is adjusted by a cap and tube arranged to form the liquid seal referred to, so that the rate of flow may be regulated. Apertures are formed on the vertical sides of the plates, closed by stove plugs, and covered by small plates of lead soldered to the external casing, to facilitate the cleansing of the plates and the replacement of their internal parts. The entire apparatus is cased in lead. It is a claimed feature of the invention that the acid systematically supplied should be of higher concentration than chamber acid.—E. S.

Titanium; Production of Compounds of. F. M. Spence, D. D. Spence, and H. Spence, Manchester. Eng. Pat. 4183, Feb. 25, 1899.

Material containing titanic acid, such as the residue left from the manufacture of aluminium sulphate from bauxite, is fluxed with a stated proportion of sodium hydrogen sulphate ("pyro-" or "bisulphate") and the product is leached with cold or hot water or dilute sulphuric acid, the degree of acidity being so adjusted that insoluble titanic acid may not separate on boiling. The solution, cleared from insoluble matter, is then concentrated to effect the separation of the soluble crystalline compound of titanic acid, having the formula $TiO_2 2(SO_3)Na_2O$, in which sodium may be replaced by potassium or ammonium, by using in the preparation the "bisulphate" of either instead of the sodium salt. In case the titanic acid material be readily soluble in sulphuric acid, either before or after a preliminary fritting with sodium carbonate, it may be dissolved in an excess of the hot acid, and to the cleared solution sodium, potassium, or ammonium sulphate is added; on concentration, the soluble titanic salt crystallises out, from which, by known methods, any desired compound of titanium may be obtained.—E. S.

Cyanogen Compounds, Obtainment of. J. Mactear, London. Eng. Pat. 5037, March 7, 1899.

A mixture of about two volumes of gaseous ammonia with one volume of carbonic oxide is made in a closed chamber, and is led into a decomposing chamber, in which the mixture is heated to from 1,800° to 2,000° Fahr. Ammonium cyanide and water vapour, the products of the reaction, are led through coolers into suitable absorption vessels containing an alkali hydrate. The ammonia set free is recovered for re-use. Instead of pure CO, a mixture of carbonic oxide with nitrogen and hydrogen (such as "producer gas") may be used with the ammonia. The solution of the alkali cyanide obtained is evaporated, preferably *in vacuo*, to obtain a crystalline mass.

The decomposing apparatus is of "an oblong shape with open ends and perforated flanged extensions fitted with perforated end covers, cemented, and metal bound thereto, and serving to lead the mixed gases from a branched main to the chamber interior and the resulting products from the chamber to a branched outlet main." There is a perforated diaphragm above the bottom, supporting a catalytic substance nearly filling the vessel, from the cover of which depend tubes of refractory material "equi-spaced as regards each other and also as regards the chamber sides, and containing electric coils or resistances adapted to heat the chamber equably." The internal heating of the chamber by electric resistances may be supplemented by electric heaters without, or by other means of heating.—E. S.

Liquids and Gases, Contact between; Bodies used in Reaction and the like Apparatus [*" Glover Towers," &c.*] *for Facilitating.* O. Guttmann, London, E.C. Eng. Pat. 24,847, Dec. 14, 1899.

Reaction bodies of the kind, and for the purposes, described in Eng. Pat. 14,774 of 1896 (this Journal, 1896, 700), are, according to this present invention, made solid, of spherical or other regularly curved form, and with curved depressions, or cup-like cavities, geometrically disposed on their surfaces.—R. A.

Sodium Persulphate; Manufacture of. B. J. B. Mills, London. From A. and L. Lumière, Lyon-Monplaisir, France. Eng. Pat. 25,081, Dec. 18, 1899.

The claim is for "the preparation of persulphate of sodium by means of persulphates of barium, by treating this body in very concentrated solution with sulphate of sodium or else with 'soda, the carbonate, or the bicarbonate of soda,' after a preliminary treatment by sulphuric acid; the solutions of persulphate of sodium thus formed being filtered and evaporated *in vacuo*, or in the open air, at only a slightly elevated temperature." The sodium persulphate is thus obtained as small anhydrous crystals, very soluble in water.—E. S.

Graphite, Purifying. E. Teisler, Borsdorf, Germany. Eng. Pat. 23,233, Nov. 21, 1899.

Graphite is first treated with hydrofluoric acid, and then with sulphuric acid or with an acid sulphate, to remove impurities.—E. S.

VIII.—GLASS, POTTERY, ENAMELS.

Water in Hot Glass; Absorption of. C. Barus. Phil. Mag. 1899, **47,** 461—479. (See also this Journal, 1899, 832.)

Continuing his work on the solution of glass in water at high temperatures (210° C.) and pressures, the author finds that in the absorption of water in hot glass, the action shows a definite limit when the solution has reached a certain concentration. This would appear to be in favour of the view that an ordinary solution, or a definite compound is formed, but the author gives reasons for regarding the solution as colloidal. He regards the first or opaque stage, as the swelling of a coagulated colloid in contact with its solvent, the subsequent clear stage being due to the fusion of this saturated and coagulated colloid.—A. S.

Enamelling upon Metals. H. H. Cunynghame. Journ. Soc. Arts, **48,** 137—145, 150—156, 162—168, 173—184.

The different styles of enamelling are cloisonné, champlevé, bassetaille, Limoges painted, and miniatures. The enamels used consist of flint glass with various proportions of oxide of lead. That used for watches and jewellery contains 20 or 30 per cent.; the use of 45 per cent. produces a dense flint suitable for enamels in the Limoges style. Large additions of soda or potash increase the fusibility of enamels, but render them more easily affected by moisture. Borax glasses are also affected by moisture, besides being very liable to crack. The colouring is imparted by the addition of certain metallic oxides. Gold gives a red; uranium and antimony, a yellow; cobalt, a blue; manganese, a violet; various mixtures of iron, chromium, copper, zinc, and cobalt, give greens; a mixture of cobalt, manganese, and iron, produces black; iron and manganese, and also nickel, give brown; oxide of tin, white arsenic, or phosphate of lime, produces an opaque white; iridium, a black or grey; platinum, a grey; cobalt blue, combined with uranium, an indigo; yellowish-green iron; mixed with oxide of iron, manganese forms a red. The various shades of colour are largely dependent upon the states of oxidation. About 2 per cent. of potassium nitrate may be added to prevent the lead from becoming reduced. The longer the heating, the better the result. It is desirable to try each enamel before it is used, by running cold water over it whilst just so hot that it can be touched. If this does not crack it, it may be considered sound.

Limoges painted enamels.—These are generally executed upon pure copper, of a thickness, for small plaques, of about 0·35 mm. The copper is shaped on a block of boxwood or pewter, hollowed out concave, and is then trimmed with a pair of shears. The plate is cleaned by plunging whilst hot into weak nitric acid, after which it is scrubbed with some fine powder and water, and burnished. The enamel is prepared for use by pulverising it in a mortar and levigating with water until even grains are left that will pass through a sieve of 75 meshes to the linear inch. On applying the powdered enamel to the plates, either water, or weak solutions of certain gums are used to make it adhere. Enamels of the same character should be used on back and front, and the thinner the coats, the less the liability to cracking. The plates after drying are fired in a muffle furnace. Paillons consist of thin sheets of pure gold, silver, or platinum, embedded in transparent enamel, and are used to impart brilliancy. They should be perforated with about 200 fine holes to the square inch, to allow of the passage of the air and steam from beneath the metal. The paillons may be either smooth or creased; they are applied with gum and well pressed down. Such shading or lines as are required are painted upon them in black sesquioxide of iridium. The paillons having been fixed, the plates are then fired to fuse all firmly down. The coats of colour are next laid on in a state of moist powder, being well dried with blotting paper and smoothed down to an even surface. Thorough drying and firing complete the work.

Cloisonné enamel.—This is usually employed for jewellery and is executed with fine gold. For the cloisons the gold wire is annealed and drawn through a very fine rectangular hole in a draw plate. The wire is then bent into the shape required, placed in position and soldered with fine 18-carat wire solder by the aid of a blow-pipe. After pickling in nitric acid, the enamel is laid on. The firing is carried out in a furnace burning alcohol. If desired, the enamel may be filled above the cloisons and the surface finally ground down and polished. Silver work is pickled in dilute hydrochloric acid.

Plique-à-jour is filigree work executed in gold or silver filled up with transparent enamels. The enamel must be soft and the initial heat very great.—H. H. B. S.

PATENTS.

Compound Glass or Metal Plates or Articles; Improved Manufacture of. P. T. Sievert, Dresden, Germany. Eng. Pat. 522, Jan. 9, 1899.

The object of this invention is the production of metal-coated glass plates, to be used in the manufacture of large acid-proof vessels. One method consists in applying a cold sheet of lead to a heated glass plate, and then rolling the two together. The glass being suddenly chilled by the lead, a number of surface cracks are produced, forming a "key," to which the lead adheres. Better adhesion may, however, be obtained by casting the glass plates with grooved or other uneven surfaces. By another method, molten glass is poured into cold press-moulds, by which it is cracked and granulated, and then molten metal is poured upon it, and the whole united by pressure. A further claim consists in the combining of small pieces of heated glass with heated metal by the application of pressure.

—H. H. B. S.

Furnaces for Making Glass or Melting Metal. R. W. James, London. From The American Stoker Co., New York, U.S.A. Eng. Pat. 2503, Feb. 3, 1899.

The glass, &c., is treated on an open hearth, beneath and at the ends of which are air spaces to keep the hearth cool. At one end is an underfeed stoker, consisting of an Archimedean screw, a hopper, an air or steam inlet, and air or steam tuyère openings. The products of combustion pass over the hearth, out through openings in the end wall, down flues to other horizontal flues below the hearth, and thence to the chimney. Air is delivered by an upper cross tube to a number of vertical passages leading to horizontal passages, both sets of passages being arranged between the flues for the products of combustion. The highly heated air thus obtained is admitted to the furnace through openings above the fire. The items claimed are a receptacle for coal, means for introducing coal and air below an incandescent bed of coke in the furnace, means for introducing highly heated atmospheric air above the incandescent bed of coke, an open hearth to contain the elements to be heated, and a series of longitudinal passages for hot waste gases, intervening with passages for fresh air.—R. S.

Furnaces for Melting Glass or Metal, and for Melting or Heating other Substances. R. W. James. Eng. Pat. 2504, Feb. 3, 1889.

See under I., *page* 229.

Waste Sand from Glass Works; Utilisation of. D. Herman, Prescot, Lancashire. Eng. Pat. 5353, March 11, 1899.

This invention refers to the purification of waste sand from the grinding of plate glass, &c., so as to render it suitable for use in the manufacture of glass. The sand is first graded by levigation, the coarser portion being returned to the grinding benches. The finer portion, which contains the impurities, *viz.*, particles of iron and iron oxide, is dried, passed over a magnetic separator, and then extracted with a solvent for iron oxide, such as hydrochloric acid.

—H. H. B. S.

Pottery; Drying Kilns for Earthenware and the like. C. Dansard, Poppelsdorf, Bonn, Germany. Eng. Pat. 23,948, Dec. 1, 1899.

The construction of the kiln is as follows:—There is a drying chamber for the reception of the goods, beneath which are two parallel passages, one for the supply of heated air and the other for the withdrawal of the air used. The air is heated in a furnace, communicating by way of a conduit with the hot-air passage. The passage for the withdrawal of the air used communicates with a chimney. By means of orifices connecting the hot-air passage with the outside, cool air can be let in as desired. The passages, inlet and outlet orifices, &c., are all provided with dampers.

—H. H. B. S.

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

Sulpho-Aluminate of Lime [in Hydraulic Cements]; Composition of. M. Deval. Bull. de la Soc. d'Encourag. l'Ind. Nat. 1900, **5**, [1], 49—54.

Le Chatelier and Michaëlis have shown that the swelling of hydraulic cements in sea water may be attributed, in part, to the formation of sulpho-aluminate of lime. This compound was discovered by Candlot, but its composition has not, up to the present, been defined.

The author prepared samples of the compound in two ways, *viz.*, from sulphate of alumina and calcium hydrate and from tricalcium aluminate and sulphate of lime. The samples prepared by the two methods had the same composition, represented by the formula—

$$Al_2O_3.3CaO,3(CaO.SO_3) + 28{\cdot}5\ H_2O$$

which does not differ sensibly from that found by Michaëlis.

In both methods of preparation, the course of the reaction was followed by testing, at different stages, the amount of sulphuric acid not fixed but remaining dissolved in the liquid. The reaction was found to be complete when the sulphuric acid fixed and the alumina were in the ratio of three equivalents to one equivalent.—A. S.

PATENTS.

Cement; Manufacture of. J. Hastie, Edinburgh, and J. Gill, Leith. Eng. Pat. 27,431, Dec. 29, 1898.

The dried raw materials are mixed and ground in a mill. They are then passed into a second mill, in which they are further ground to a fineness sufficient to pass through a sieve of 2,500 holes to the square inch. The finely ground material is carried from this mill by air-exhaust, and is deposited in a collecting chamber, from which it is elevated and fed into the moulds of a pressing machine. The pressed bricks are then mechanically conveyed to the kiln.
—H. H. B. S.

Cork, Treatment of Refuse, to form a Tenacious Composition for Structural and other Purposes. F. Bartleet, Bury, Lancaster. Eng. Pat. 2809, Feb. 8, 1899.

Cork powder is mixed with a solution made by dissolving shellac, resin, copal, or other adhesive substance or combination of substances in a suitable solvent. The resulting mass is allowed to dry, and is then rammed into moulds and baked at a temperature of 200°—300° F. The following mixture is said to be suitable for most purposes: $2\frac{3}{4}$ lb. of shellac dissolved with $\frac{3}{4}$ lb. of borax and $3\frac{1}{2}$ galls. of water for each cwt. of cork. The finished material can be turned, cut, sawn, polished, or otherwise treated.
—H. H. B. S.

Portland Cement; Manufacture of. L. White, London. Eng. Pat. 4813, March 1, 1899.

The slurry, either before or after drying, is mixed with powdered fuel, and the mixture is then burnt in a rotary furnace. The improved draught obtainable by the use of the rotary furnace is said to render this possible.
—H. H. B. S.

Portland Cement; Manufacture of, and Apparatus therefor. L. White, London. Eng. Pat. 4814, March 4, 1899.

The operations of drying, calcining, and clinkering the slurry are carried out in three separate rotating furnaces, placed one above the other. The carbonic acid gas disengaged in the middle or calcining furnace is drawn off and passed through heated fuel, and thus converted into carbonic oxide, which is then used to heat the lower furnace.
—H. H. B. S.

Cement; Manufacture of. W. Seidel, Biebrich on the Rhine, Germany. Eng. Pat. 17,608, Aug. 31, 1899.

The raw materials are fed into "spherical mills" and ground with water, just sufficient being added to form a paste containing about 32 per cent. This paste is then further ground under millstones, after which it is kneaded with sufficient of the finely-ground and dry raw materials to reduce the proportion of water in the mixture to 22 per cent. The mixture so produced is finally made up into bricks and burnt in the usual way.—H. H. B. S.

Wood; Apparatus for Impregnating Long Pieces of. G. F. Lebioda, Boulogne-sur-Seine, France. Eng. Pat. 19,362, Sept. 26, 1899.

The apparatus consists of a horizontal cylinder, in the interior of which is a steam worm for heating the fluid. The wood is introduced into the cylinder upon a waggon frame, moving upon rollers working upon slide-bars affixed to the interior of the cylinder. At each end of the cylinder is a cover, hung upon hinges, and capable of being tightly closed. The covers have each four cylindrical excrescences, in which there are adjusted and arranged hollow cylinders having inlet and outlet openings for the passage of the fluid. These hollow cylinders have "inserted plates provided with a number of perforations, surrounded by annular sharp-edged bosses or cutters, with the object of enabling tree trunks or long pieces of wood to be held fast between the perforated plates, and of facilitating the penetration of the impregnating fluid in the direction of the fibres." Passing through each cylinder, and made tight by a stuffing box, is a spindle, having a thread at one end, by means of which such cylinder can be tightly screwed down to the wood. The wood having been introduced and the covers closed, the liquid is let in at one end under pressure. After penetrating the wood in one direction, the operation can be reversed and the liquid caused to pass in the other direction.—H. H. B. S.

X.—METALLURGY.

High Temperatures; Colour Definitions of. H. M. Howe. Eng. and Mining. J., Jan. 20, 1900, 75.

The author refers to a recent communication by M. White and F. W. Taylor (this Journal, 1900, 140) as to the temperature which corresponds to each of the colour names, "a dull red," "a light yellow," &c. He then compares in a table, his own results, obtained in 1891, those of White and Taylor (*loc. cit.*) and those of Pouillet (Comptes. Rend. 1836, **3**, 784); and points out that whilst his results agree in all cases within 98° F. with those of White and Taylor, Pouillet's results differ from the latter by much larger amounts, ranging from 172° to 467° F. On an algebraic average, the author's results differ by about 51° F., and Pouillet's by about 300° F. from those of White and Taylor.—A. S.

Magnetic Properties of Ingot-Iron Plates; Influence of, on Annealing. H. Kamps. Chem. Zeit. Rep. 1900, **24**, [2], 9. From Stahl u. Eisen, 1899, **19**, 1120, 1154.

The relation between the chemical composition and the magnetic properties of iron, is but little understood as yet, nor is it known why annealing sometimes improves, and at other times is detrimental to the quality of the iron. Experiments on the effect of the duration of the time of annealing gave negative results, inasmuch as, within the temperature limits tried, no such influence could be observed. The experiments only proved that the maximum induction was but little affected, whilst the residual magnetism showed marked differences, and that with a given sample of iron only a certain (characteristic) degree of excellence can be imparted with regard to its quality. The author finds that the alteration of the magnetic properties of iron by annealing, bears a distinct relationship to the internal changes of the plates, especially to changes in the form of the carbon and iron, and in the size of the grain. Since mechanical softness and good magnetic qualities are known to be related, it is clear that hardening carbon, if present even in minute quantities, must exert an influence on dynamo iron. The increase in hysteresis losses due to mechanical work in the cold, can only be explained by a change in the form of the iron. An explanation of the "ageing" of dynamo plates may be found in the allotropic theory of iron. The time taken in cooling alone causes the differences in the magnetic qualities of plates annealed at the same temperature, the time occupied in raising them to the maximum temperature, being quite without effect. Of the elements present, iron and carbon in their various forms alone influence the magnetic properties directly, the others act indirectly by their effect on the structure and on the two principal elements. Silicon and aluminium increase, whilst phosphorus and manganese diminish the permeability.—W. G. M.

Steel, Influence of Copper on the Quality of. A. L. Colby. Stahl u. Eisen, 1900, **20**, 55.

The results of the author's experiments on the influence of copper on the mechanical properties of steel, confirm those obtained previously by Wasum. Neither a propeller shaft with 0·565 per cent. of Cu, nor a gun tube with 0·553 per cent. of Cu, showed any defects after forging and hardening; armour plates with 0·575 per cent. of Cu, welded and flanged well, and answered all the requirements of the American navy. Bessemer steel with 0·11—0·65 per cent. of C. and 0·292—0·486 per cent. of Cu, showed no red-shortness on rolling, nor did Bessemer steel for rails, Martin steel with 0·075 per cent. of Cu for fire boxes, or nickel steel with 0·08 per cent. of Cu for bicycles. It was also found that copper exhibited but little tendency to liquate.—W. G. M.

Silver Smelting in Mexico; Development of. O. H. Hahn. Paper read before the Inst. Mining and Metall., Feb. 21, 1900, 1—69.

The author gives a lengthy detailed account of the modern methods of silver smelting in Mexico, with descriptions of the different forms of plant, and also of figures as to cost of working, &c.

Analyses are given of the different varieties of ore, of the fluxes and fuels used, and of the slags and mattes produced.

It is stated, in conclusion, that with the development of many new deposits in all parts of Mexico, copper will come more and more into use for the extraction of the silver and gold, especially as the deposits of lead ore are pressed to a rate of production which cannot be maintained indefinitely.
—A. S.

Silver and Lead, Losses of, in Lead Smelting. L. D. Godshall. Eng. and Mining J., Jan. 13, 1900, 43.

This article is a criticism of a communication by M. W. Iles (this Journal, 1899, 1023) on the subject of losses of silver in smelting.

Losses in lead smelting occur mainly in slags, and the flue dust from blast furnaces, and also from volatilisation and dust in roasting. If the silver and lead are present in the slags in an oxidised state, separation is impossible by any form of settling arrangement. The slag may also hold in suspension particles of matte, and in rare cases, extremely small shots of bullion. The losses in the first case are readily determined by taking daily samples of the slag. In the second case, however, a sample taken after the slag has been allowed to settle for a few minutes may not represent the true content of silver and lead, since a portion of the matte in suspension would have settled to, or towards the bottom. The value of the matte thus lost would be similar to that produced and saved during the same period, except when the charge contains much zinc, in which case, "light-float zinc matte" is produced, which invariably contains more zinc and less silver than the normal matte produced at the same time.

With regard to the question of roasting, Iles states:—"It cost per ton to roast this ore, 3·417 dols. on the fuse boxes and sinter boxes; on the Brown roasters it cost 2·504 dols. per ton. The question may well be asked, did it not cost too little? That is to say, if there was much zinc present it should be roasted better in order to convert the sulphide of zinc into an oxide." The author considers that the cost of roasting was quite high enough, and he gives the following results obtained at Tacoma with the Godshall roasting furnace during a test-run of 15 days:—60 tons of ore, containing 40·5 per cent. of sulphur, were roasted per day; the roasted material contained 1·85 per cent. of sulphur. Cost of fuel, 15 cents per ton; fireman, 8 cents; feeder, 7 cents; total, 30 cents per ton. The fuel used was partly fine bituminous coal, and partly slab wood. As to the amount of sulphur which should be left in roasted ore, the author is of opinion that it is unadvisable to endeavour to convert the whole of the zinc sulphide into oxide, on account of the expense involved in roasting zinc-lead ores until they contain less than 3·5 or 4 per cent. of sulphur; also the heavy loss of silver experienced in such an operation owing to the presence of zinc, forms a serious item, for such zinc when oxidised, invariably carries with it, if excess of oxygen be present, a very considerable quantity of silver.

Iles refers to the difficulty of saving light, floating zinc matte. The author states that he has smelted, for months at a time, material producing slags containing 8 to 10 per cent. of zinc, frequently with a high percentage and a low percentage of iron, and a high percentage of silica, and with fully three-quarters of the charge made up of fine ore from mechanical roasters, the so-called dry ore containing from 7—9 per cent. of sulphur, and from 8—10 per cent. of zinc. Even under such conditions, however, it was possible to save the whole of the matte, whilst the loss of silver was not serious, provided the percentage of lead on the charge was kept high. The most serious and apparently insurmountable difficulties were the irregular, and at times, slow, running of the furnace, and the exceeding great difficulty in maintaining a proper reduction, whilst forcing the furnace.—A. S.

Gravitation Stamp Mills [Gold]; Development of. D. B. Morison and D. A. Bremner. Trans. Inst. of Min. and Met. Jan. 17, 1900.

The authors have made a series of tests on the crushing efficiency of stamps under conditions varying as to weight of stamp, rapidity of drop, screen area, screen mesh, &c., and they give tables of the results of these tests and of formulæ for the determination of the many factors concerned in the action of a stamp battery cam, with their application to three typical cases. The deductions from the tests are as follows:—(1.) That the crushing capacity increases directly as the number of drops per minute and the weight of the stamp, other conditions being constant. (2.) That the screen area of an ordinary standard single-discharge 5-stamp mortar-box is adequate for a crushing rate of 10 tons per stamp per 24 hours at 132 drops per minute. This means a screen duty of 6·55 lb. per square inch per hour, which is below the limit at which the screen begins to control the crushing capacity of the mill. Only a depth of 12 in. of the entire 17 of the front screen was found to be effective even with the splash of a high speed stamp. (3.) That increased coarseness of screen mesh accelerates the crushing rate in an increasing ratio. (4.) That considerable variations in the screen mesh make only a slight difference in the percentage of the extreme fines and of the extra coarse sands. And (5) that advantage might accrue from the use of coarser screens, with the consequent acceleration of the rate of crushing and a reduction of slimes. A high speed and violent agitation would also assist in preventing slime production, although the proportion due to soft clayey material cannot be affected by such conditions.

It is pointed out that a 5-stamp mill has a higher crushing rate per stamp than a 1-stamp mill under similar conditions of weight of stamp, height, and rate of drop, although the screen duty is much higher in the former case. This is explained by the fact that at 132 drops per minute, the stone, after being washed off the die by one blow, cannot get back for the succeeding one, whereas with a 5-stamp mill the blow of one stamp tends to throw the material on to the dies of the others.

To gain a greater percentage of the blow energy by increased rigidity of mortar-box and foundations, it is advisable to use concrete in place of timber piles for foundations, and to insert heavy cast iron blocks between the box and the concrete to provide inertia. The authors recommend the high speed stamp (132 drops per minute) as possessing many advantages over the cam stamp.—A. W.

Gold and Platinum Mining in the Ural Mountains. D. A. Louis. Trans. Inst. of Min. and Met., Jan. 17, 1900.

The Ural mining district comprises an extensive area, about 600 miles in length and 70 miles in width, and contains many gold-bearing reefs and alluvial deposits. The author has visited parts of this district and now describes the geological and mineral nature, together with the methods of working, of a few mines not hitherto recorded. The gold extraction appliances are very primitive, although sometimes quite new and working well, and they are generally made of wood, except in localities recently approached by railways. In these, modern appliances are being gradually introduced, with the result that breakers, stamps, vanners, chlorination, and cyaniding plants are working side by side with the old appliances.

At the Myass mines, the clayey sands and gravels, which may contain from 9 to 10 grains of gold per ton, are roughly screened by washing the fine particles by means of water sprays, through the perforated bottom of a circular iron tank on to an inclined table 30 to 40 ft. long and 6 ft. wide, to which are fixed amalgamated plates, matting, and battens. The amalgam, gold, and heavy gravel from this table are then washed in a finishing cradle, and the amalgam subjected to distillation in an iron tray placed on a tripod standing in water, with an iron bell over the whole, the heat being supplied by charcoal burning on an annular shelf on the outside of the bell, whilst the mercury is partially caught in the water.

In the Tcheliabinsk locality the gold varies from $1\frac{2}{3}$ to to 7 dwt. per ton, and the output of one mill was 25 tons of ore per day, the gold yield being 40 gr., with 16 gr. remaining in the tailings to be further extracted by cyanide or other means.

Platinum is found in swampy alluvial deposits in some localities on both the European and Asiatic slopes of the mountains. The alluvials are similar to those bearing gold, being from 2 to 7 ft. in thickness, and are covered with unproductive gravel, clay, and sand, with a vegetable soil above all. The earth is stripped and the alluvial laid bare or got at by shallow shafts, the working and washing then being similar to that of the gold extraction. The alluvials are sometimes obtained by undercurrent hydraulic treatment. Native platinum is found in the metamorphosed rocks. There are many other rare metals and minerals in this district, as well as gems and ornamental stones, the commoner metals, such as silver, copper, lead, iron and manganese, and chrome iron ore.—A. W.

Gold-Aluminium Alloys. C. T. Heycock and F. H. Neville. Proc. Roy. Soc., Feb. 9, 1900, **66**, 20—21.

The authors give an abstract of their research on the freezing-points of a series of gold-aluminium alloys ranging from pure gold to pure aluminium. The original paper contains an equilibrium curve for the liquid alloys and the various solid bodies that can form in them, in which the ordinate is the freezing point on the air-centigrade scale, the abscissa being the composition of the alloy expressed in atomic percentages of aluminium. The curve consists of seven branches, each corresponding to a state in which one particular solid crystallises first, and the seven solid substances are found to contain the following bodies:—Gold; Au_4Al, with a melting or freezing point at about 550° C.; Au_5Al_2, or perhaps Au_8Al_3, at 575°; Au_2Al, at 625°; a hypothetical body, probably AuAl, the freezing point of which is not given by the curve; $AuAl_2$, Roberts-Austen's purple alloy, at 1,062°, apparently identical with the melting point of gold; and aluminium.

There are three well-marked angles of eutectic mixtures, one at 527° C. with 3·6 per cent. by weight of aluminium, probably a mixture of Au_4Al and Au_5Al_2, another at 569° with 8·36 per cent. of aluminium, corresponding to Au_3Al_2 but a mixture of Au_2Al and AuAl, and a third at 648° with 1·87 per cent. of gold, a mixture of $AuAl_2$ and Al. These results show that the lowest melting point mixture is the one containing 3·6 per cent. by weight of aluminium, and this small percentage lowers the melting or solidifying point of gold from 1,062° to 527°. This effect of a small quantity of aluminium on gold, and also the fact that the freezing point rises to that of gold as the compound $AuAl_2$ is reached, have already been pointed out by Roberts-Austen. All the above compounds are white in colour, with the exception of the purple $AuAl_2$.

The microscopical examination of polished and etched sections shows that the structure of the solid alloy harmonises with the freezing point indications, and that the structure varies and repeats its variation with the approach to the summit of each branch in the curve.

As an example, the pure alloy Au_2Al, indicating the summit of one branch, shows hexagonal polygons filling nearly the whole field and separated only by very fine lines of impurity, whereas the alloy slightly below the summit shows a ribbon-like network surrounding the polygons, and still further below, the polygons of Au_2Al are scanty, with indications of their free crystallisation in the surrounding liquid, whilst finally, at the lowest or eutectic point of the branch, the large crystals of Au_2Al are absent and the field is filled with mother substance, which is sometimes, but not necessarily always, the eutectic mixture.

Some of the alloys very rich in aluminium were photographed by Röntgen rays, and enlargements made from the negatives. By this means, a much better picture of the structure was obtained than by the photo-micrograph of the surface.—A. W.

Gold; Novel Association of. H. F. Collins. Trans. Inst. of Mining and Met., Feb. 27, 1900.

The value of the ore at the Santa Fé Mine, Chiapas, Mexico, is due chiefly to a bornite containing about 1 oz. of gold per ton, which, in the deeper ores, is fairly constant, and is not completely extracted by mercury, thus indicating its chemical combination probably with sulphur, since selenium and tellurium are absent. Grains of a steel-grey brittle mineral were noticed in some of the bornite. A few grains were picked out, finely ground, passed through a 120-mesh sieve, and the fine powder submitted to analysis. The resulting figures led to the empirical formula—

$$(NiCuFeCo)S,CoS_2,$$

with silver, 42 oz. per ton, and gold, 173 oz. Several particles of free gold were left on the sieve, equivalent in amount to over 200 oz. per ton. The amalgamation of about a gram of the carefully picked pure mineral with mercury, by grinding in a mortar in the presence of a little cyanide solution, showed that "294 oz. per ton" of the gold were extracted and amalgamated, whilst "59 oz." were left in the residue. The author thinks that these results indicate the presence of combined, as distinguished from finely disseminated metallic gold in the mineral.—A. W.

Molybdenum; A New Crystallised Sulphide of. M. Guichard. Comptes Rend. **130**, [3], 137—140.

Molybdenum disulphide, MoS_2, at a high temperature dissociates, yielding a sesquisulphide, Mo_2S_3, which crystallises in needles. This at a higher temperature dissociates, giving sulphur and metallic molybdenum, whilst when heated to redness in sulphur vapour it regenerates the disulphide. The sesquisulphide is readily attacked by fluorine, chlorine, or oxygen at a red heat, and at the same temperature in the air, it is slowly converted into the trioxide, which volatilises.—J. T. D.

Bronzes, Japanese. W. Elkan. Chem. Zeit. Rep. 1900, **24**, [3], 23. From the Protokoll der Sitz. des Vereins zur Beförd. des Gewerbefleisses, Oct. 2, 1899, 187.

Most cast wares of Japanese origin are made by the *cire-perdue* process. Most vases (with which class of work this article is chiefly concerned) are cast in loam moulds from which the wax is melted out. The casting of such a vase is described step by step in the original paper. The Japanese alloys differ from those of this country chiefly in containing a large proportion of lead, which improves the patina at the expense of the strength. The following table illustrates the difference in composition between the Japanese and European bronzes:—

——	Cu.	Sn.	Zn.	Pb.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Modern Japanese bronze	81·62	4·61	..	10·21
	76·60	4·38	6·53	11·88
German and French bronzes	88·55	2·42	3·29	4·72
	86·60	6·60	3·30	3·30
	91·49	1·70	5·35	1·37

Sometimes a little antimony is added just before casting, as shown by the following analysis:—Cu = 68·25, Sn = 5·47, Zn = 8·88, Pb = 17·06, and Sb = 0·34 per cent. The proportion of lead in this alloy was abnormally high; hence the metal took a beautiful patina, but cast very badly with many defective places and a large number of pin-holes, which, however, were very cleverly concealed.—W. G. M.

Graphite in Pig Iron; Determination of, by Loss. G. Auchy. J. Amer. Chem. Soc. **22**, [1], 47.

See under XXIII., *page* 276.

PATENTS.

Ore Treatment; Separating Metallic from Rocky Constituents, and Apparatus therefor. F. E. Elmore, Leeds. Eng. Pat. 21,948, Oct. 18, 1898.

The pulverised ore suspended in water is mixed with a thick oil, such as the tarry residue of mineral oils. This adheres to the metallic but not to the rocky particles. The mixture is made in a helically ribbed drum, from the end of which it flows into a settler. From this settler, the lowest layer of tailings passes away to a tank, where it is settled for re-treatment or run off as waste. The upper or oily layer with its contained metallic particles, is led into a rapidly revolving drum or centrifugal separator containing a little water. As the drum revolves, the metallic particles pass from the oil into the water and form a layer on the side of the drum, whilst the oil flows over an inwardly-projecting lip into the drum-casing, whence it is removed by a pipe to be used over again.

As alternatives, the oil may be removed by heating, or by means of a solvent such as benzoline.—J. H. C.

Furnaces for Making Glass or Melting Metal. R. W. James, London. Eng. Pat. 2503, Feb. 3, 1899.

See under VIII., *page* 247.

Furnaces for Melting Glass or Metal, &c. R. W. James, London. Eng. Pat. 2504, Feb. 3, 1899.

See under I., *page* 229.

Ore-Concentration and Separation; Impts. in. W. A. Macfadyen, Pretoria, South African Republic. Eng. Pat. 2747, Feb. 7, 1899.

The finely crushed ore is passed first through a magnetic separating apparatus to remove the paramagnetic particles. The residue is then passed through a strong magnetic field, the air within which has been rendered a good conductor of electricity by means of a radiation from a Geissler or other similar vacuum tube containing salts of uranium, thorium, "polonium, or radium," and excited by a high-tension current of electricity. In this way the diamagnetic particles may be diverted and separated as a concentrate, whilst the waste passes on to the dump.—J. H. C.

Blast Furnaces or Cupolas; Construction and Working of. A. J. Henderson, London. Eng. Pat. 5114, March 8, 1899.

Combustible gases or vapours are blown into a well below a perforated arch or floor which supports the charge, and through which the molten material can flow as it is produced.—J. H. C.

Furnaces; Continuous Steel Billet Heating. R. W. James, Queen Victoria Street. From The American Stoker Co., New York, U.S.A. Eng. Pat. 2505, Feb. 3, 1899.

In these furnaces the steel billets are forced continuously through the furnace by means of steam operated pistons, which force them from a truck on to tracks in the furnace, and thence through the furnace. The billets are finally delivered from the other, or fire end, down inclined tracks. The furnace is heated by solid fuel, supplied with oxygen, and the flame passes both above and below the billets. Two forms of furnace are illustrated, the first containing one under-feed stoker or group of stokers, the other, two such stokers or group of stokers. The following parts are claimed in combination with a continuous steel billet heating furnace, namely, means for maintaining a practically continuous feed of billets through the furnace, one or more under-feed stokers, passages for applying the heat both above and below the billets, and one under-feed stoker situated beneath the billets and another at the rear end of the furnace.—R. S.

Furnaces; Metallurgical and other like, for Calcining, Reducing, Carbonising, and other Purposes. J. A. Drake, Halifax. Eng. Pat. 5710, March 16, 1899.

A metallurgical regenerative furnace, for the above purposes, is constructed with a gas-producer in a central position or between and separating the ovens or kilns to be heated, whereby sinking for sub-structure is avoided. The producer occupies the central front, whilst a series of flues and air channels for heating the air supply (this Journal, 1899, 281) is built in the central back portion, the various horizontal ovens or muffles, one above the other, on either side, running nearly the full depth from front to back in an arched chamber enclosing the whole. The hot air meets the gas from the producer in the top of this chamber, and the flames pass down over the series of ovens to the flues below, the products of combustion being conveyed thereby to the air heating structure at the back, and onwards to the stack.—A. W.

Blast Furnace and other Gases; Apparatus for Separating Dust and the like from. A. Hebelka, Coblenz. Eng. Pat. 20,150, Oct. 7, 1899.

See under II., *page* 231.

Copper or Copper Alloys, Method of Uniting, with another Metal. W. G. Clark, South Australia. Eng. Pat. 10,763, May 23, 1899.

Copper is united to another metal by heating and applying, whilst still hot, a finely divided flux, composed of the following materials, substantially in the given proportions: Silica, 31·25 per cent.; carbonate of lime, 25 per cent.; carbonate of soda, 25 per cent.; iron filings, rich ironstone, or sulphate of iron, 12·5 per cent.; and sulphate of copper, 6·25 per cent. The operation is utilised more especially for coating iron or steel with copper, but may also be used for welding purposes.—A. W.

Gold and other Metals, Mixing or Aërating Apparatus for Use in the Extraction or Recovery of. W. Duncan, Queensland. Eng. Pat. 12,749, June 19, 1899.

The apparatus consists of a vat, transversely constructed as a semicircle, with a horizontal revolving shaft running lengthwise near the top centre, having arms arranged helically, and with revolving gear at one end and taps at various levels at the other. The revolving shaft carries the arms successively into and through the sludge, out again and back through the air, thus agitating and aërating the sands or tailings during leaching or other treatment. With repeated leachings or washings, the agitation is stopped to allow settling and decanting, the mud being finally washed out during agitation.—A. W.

Steel and other Materials; Manufacture of Cast Blocks and Masses of. L. Perin, France. Eng. Pat. 20,939, Oct. 19, 1899.

Steel or iron scrap, galvanised iron, or other suitable materials, are melted in a cupola with a mixture in specified proportions of ferro-manganese, ferro-silicon, and aluminium; lime and magnesia are added, and coke is used as fuel. For very hard steel, small proportions of tungsten, ferro-chromium, and nickel may also be added.

The metal thus produced is cast in sand moulds of the shape desired; the castings are placed in vessels and surmounted by iron ore, lime, chips, and scales of rolled iron. The vessels with their contents are re-heated in an annealing furnace for from 1 to 5 days in a temperature sufficiently high for the ore to take up all excess of carbon from the pieces.—J. H. C.

Fertiliser rich in Phosphoric Acid, and a Phosphorus Pig Iron; Method of Producing. A. J. Leopold, of Forselles, St. Petersburg. Eng. Pat. 21,484, Oct. 27, 1899.

See under XV., *page* 258.

Aluminium or Aluminium Alloy; Joining of Pieces or Articles of. M. Schmidt, Berlin. Eng. Pat. 24,123, Dec. 4, 1899.

The surfaces to be joined are cleansed by friction with a rod of carbon or platinum. Sometimes an electric current of low potential is employed; in such cases the rod forms the anode, whilst the piece to be cleaned is the cathode of the circuit. The welding temperature may either be produced by the electric current only or by extraneous sources, such as the oxyhydrogen flame.—J. H. C.

Compound Ingots; Manufacture of. S. A. Cosgrave, Pennsylvania, U.S.A. Eng. Pat. 24,719, Dec. 12, 1899.

An ingot mould is prepared with a removable core or partition coated with a fluxing material such as silica, or a deoxidising material, such as carbon. Molten metal is poured or forced into the mould so as to fill the space surrounding the core or on one side of the partition. As soon as this metal has sufficiently solidified, the core or partition is withdrawn, and the remaining space is filled with the other metal which it is desired to "compound" in the ingot. When produced in this way, the two metals are found to be "inter-diffused" or "decrementally" merged into each other, and the compound ingot can be forged or worked without danger of separation or fracture.—J. H. C.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

(*A.*)—ELECTRO-CHEMISTRY.

Electrified Bodies; The Discharge of, and the Formation of Ozone. P. Villard. Comptes Rend. **130,** [3], 125—127.

The author looks on the discharge of electrified bodies by flame or incandescent objects as due to the emission of cathode rays. On this hypothesis the discharge by flames is placed in the same category with that by oxidising phosphorus and by ultra-violet light, whilst the production of ozone in these cases is referred to one and the same cause, for it is known that the Lenard rays have the property of ozonising air.—J. T. D.

Solutions other than Aqueous Solutions; Electrical Conductivity of. A. T. Lincoln. J. Phys. Chem. 1899, **3,** 457—494.

The author has investigated the electrical conductivity of certain substances dissolved in a large number of solvents other than water. The general laws which are applicable to aqueous solutions do not as a rule apply to solutions in other solvents. The degree of dissociation calculated from conductivity measurements is quite different from that calculated from the elevation of the boiling point. No relationship could be discovered between the dissociating power of a solvent and its dielectric constant or its degree of association.—J. S.

Alkali Chlorides and Nitrates; Conductivity of Aqueous Solutions of. F. Kohlrausch and M. E. Maltby. Preuss Akad. Wiss. Berlin, S. Ber. 1899, **36,** 665.

Re-determinations have been made of the conductivities of solutions of alkali chlorides and nitrates, reliable electrical standards and thermometers, and salts and water which have been carefully purified being used, and special care being taken in the manipulation of the electrical measurements and in the determination of the concentration of the solutions. The results obtained are somewhat smaller than those previously accepted as the most accurate, especially in the most dilute solutions, where differences of $\frac{1}{2}$ to $2\frac{1}{2}$ per cent. are found.

The results show that up to and including the concentration of 0·002 grm. mol. per litre, the law of independently mobile ions is exactly true; in more concentrated solutions this is not the case. The diminution of the equivalent conductivity with increasing concentration is roughly proportional to the square root of the equivalent conductivity. —A. S.

Potassium Chloride; Electrolysis of. A. Brochet. Comptes Rend. **130,** [3], 134—137.

On electrolysing, at 20° C., a 20 per cent. solution of potassium chloride, to 100 c.c. of which 0·1 grm. of potassium bichromate and 0·2 grm. of potassium hydroxide has been added, hypochlorite and chlorate are both formed from the beginning, and increase in amount as the electrolysis proceeds; but the chlorate continues to increase in amount for some time after the hypochlorite has reached a maximum at which it remains constant. Ultimately, the hypochlorite in solution corresponds with about 23 grms. of chlorine per litre, and the yield of chlorate corresponds to about 70 per cent. of the total chlorine. High temperature and strong alkalinity are therefore necessary to the formation of chlorate. Working, as Foerster has done, without the presence of chromate, the yield of chlorate is much less, a large part of the energy of the current being then employed in reducing the hypochlorite.—J. T. D.

Hydrates in Solution. W. D. Bancroft. J. Phys. Chem. 1899, **3,** 551.

The author draws the following conclusions as regards hydrates in solution:—

(1.) Hydrated electrolytes are decomposed by dilution.

(2.) The presence in solution of a hydrated electrolyte will not account for variations from Ostwald's dilution law, except in so far as it is not permissible to treat the concentration of the water as constant.

(3.) Treating the concentration of the water as a constant, the ratio of the concentrations of a hydrated and a dehydrated ion will remain the same, provided there be no secondary electrolytic dissociation.—A. S.

Hypochlorites; Electrolytic Production of. A. Sieverts. Zeits. für Elektrochem. 1900, **6,** [27], 364—370, and [28], 374—378.

The author begins by recapitulating the results of the recent investigations on the electrolytic production of hypochlorites, as follows:—

(1.) The fraction of the current utilised for the formation of hypochlorite gradually diminishes during the progress of the electrolysis, depending on the amount of hypochlorite present, and finally becomes zero when the concentration of the hypochlorite has reached a maximum value. According to the latest researches of Haber and Grinberg (this Journal, 1899, 690—691 and 1028), and Müller (Zeits. anorg. Chem. 22, 48), this maximum value, for low temperatures, is 0·5—0·6 grm. of hypochlorite oxygen per 100 c.cm. The moment this value is exceeded, production of chlorate only takes place.

(2.) Oettel's experiments (this Journal, 1895, 492) have shown that a high current density is favourable to the production of hypochlorite. With regard to detail, he has also proved that a high cathodic current density tends to diminish the reduction of the hypochlorite at the cathode, and that a high anodic current density decreases the evolution of oxygen at the anode. From his figures representing the "chlorine ratio," it may also be concluded that a high anodic current density lessens, or even prevents, the formation of chlorate. By increasing the current density, therefore, the factors which prevent the production of hypochlorite are minimised, *viz.*, reduction, evolution of oxygen, and formation of chlorate, and hence the yield of hypochlorite is increased.

(3.) Increase of temperature occasions increased reduction and formation of chlorate, and therefore diminishes the yield of hypochlorite.

(4.) As long as the concentration of the chloride solution does not fall below 1·2-normal, then, according to Müller, any further change in the concentration of the solution is without effect on the production of hypochlorite. It is only when the electromotive force necessary to drive a certain current through the solution becomes so high as to produce a rise of temperature at the electrodes, that its influence is felt.

These results are in harmony with the theoretical views of Foerster (this Journal, 1900, 154), especially with regard to the influence of current density. The only investigations at variance with the above statements are those of Schoop (this Journal, 1895, 864), who found that, in the case of a 5 per cent. calcium chloride solution and of a 3 per cent. sodium chloride solution, the yield of hypochlorite diminished with an increase of current density. Haber has already attempted to reconcile the contradictory observations of Schoop and Oettel, and the author has further taken the matter up and now confirms, in the first part of his paper, the statement that the yield of hypochlorite increases with increased current density.

In the second part of his paper the author deals with a statement by Foerster and Bischoff (this Journal, 1898, 666—667), to the effect that an electrolytically prepared hypochlorite solution possesses higher bleaching properties than an ordinary bleaching powder solution of the same chlorimetric value. This statement is also confirmed along with the suggested explanation that the electrolytically prepared solution is comparable with an acidified bleaching powder solution, inasmuch as the electrolytically prepared solution already contains free hypochlorous acid, at any rate in the case of an electrolysed calcium chloride solution. Such a solution was found to contain about 50 per cent. of the hypochlorite oxygen in the form of free hypochlorous acid, which bleached in a few minutes, whereas a corresponding solution of a neutral or slightly alkaline solution of sodium or potassium hypochlorite only exerted its full bleaching effect after the lapse of several hours. Full experimental details are given.—J. S.

Wood Pulp; Electrolytic Bleaching of. A. Navarre. Ind. Electrochim. 1890, **3**, 49.

See under XIX., *page* 267.

PATENTS.

Primary Batteries. [*Reservoir.*] T. Heffernan, London. Eng. Pat. 2144, Jan. 31, 1899.

A carbon electrode, contained within a porous pot, is placed in an open vessel containing an iron electrode, and the arrangement is such that when the current is taken off, and the potential of the generator falls, gas generated in the porous pot collects in its upper portion, and in a reservoir in connection with it. As the pressure of the gas increases, it forces the electrolyte out of the pot into a receptacle, whereupon fresh electrolyte is admitted to the pot in an automatic manner, and the potential of the generator is restored to its normal condition. By means of water or other liquid, the capacity of the gas reservoir may be increased or diminished, and the regulation of the level of the electrolyte in the porous pot, and the immersion of the carbon electrodes in it, secured, so as to keep an even potential difference.—G. H. R.

Ozone; Apparatus for the Production of, by Electricity. J. H. Lamprey, Brockley. Eng. Pat. 5322, March 10, 1899.

The ozoniser, which is of the type described in Eng. Pat. 11,810, 1894 (this Journal, 1895, 585), is placed in a casing which is provided with means for the exit and entry of air at different parts of it, together with a rotatory fan actuated by an electric motor.

The claim is for the "arrangement of an ozoniser of the type referred to within a casing, having provision for entry and exit of air at different parts thereof, and the combination therewith of a rotatory fan, and an electric motor for actuating the said fan."—G. H. R.

(*B.*)—ELECTRO-METALLURGY.

Chromium; Electro-Deposition of. S. Cowper-Coles. Paper read before the Inst. of Mining and Metallurgy, Dec. 20, 1899.

The author states that bright deposits of chromium on a silvered copper surface can be obtained by electrolysing a solution of chromic chloride (1 : 3) at a temperature of 190° F., with a current density of 40—50 ampères per sq. ft. In the cold a black non-adherent deposit was obtained, the potential at the terminals of the cell being about 4 volts. Gas was evolved at both anode and cathode during the deposition of the metal. In order to obtain good deposits, it was found necessary to add an excess of hydrochloric acid to dissolve the precipitated oxide, and form a clear solution.

The author determined the comparative reflecting power of various electro-deposited metals; and the relative diminution of reflecting power due to the tarnishing caused by exposure for 4½ hours at a distance of about 1½ ins., to a small arc light.

Metals.	Comparative Reflecting Power.	Relative Diminution of Reflecting Power due to Tarnishing (Unit Law).
Silver	100	10
Chromium	100	44
Platinum	74	22
Palladium	64	35

—A. S.

PATENTS.

Ore; Process of Smelting, and Electric Incandescent Furnaces therefor. F. E. Hatch, Michigan, U.S.A. Eng. Pat. 24,723, Dec. 12, 1899.

The electricfurnace consists of a horizontal cylinder composed of electric, insulating, refractory material, on the inner surface of which are placed refractory conductors adapted to be heated to a high degree of temperature, and divided into groups. These conductors are heated in sequence to a state of incandescence by an electric current, which is then cut off, and the conductors are placed successively in contact with the material to be smelted, which is mixed with a suitable reducing agent, so that whilst the material is being smelted, other conductors are being heated by the current. Means are provided for rotating the cylinder, and there is a commutating device for directing the current through the different groups of conductors in succession.

—G. H. R.

Electro-Plating; Apparatus for Use in. H. H. Lake, London. From H. R. Boissier, New York, U.S.A. Eng. Pat. 4415, Feb. 28, 1899.

In the centre of the tank is an upright tube, in which fits a second tube or rod, which may be reciprocated vertically. Near its upper end is a metal holder, provided on opposite sides with sockets, which support depending work holders, on which large work may be suspended directly, and which is electrically connected with one pole of the source of electric current. For small work a cage is employed, journalled on the hanger, and held in place by insulating nuts. The periphery of the cage is provided with a series of hard rubber teeth, and a pawl is arranged to catch between these teeth on the rising movement of the cage, and thus impart a partial revolution to it. The anodes are placed on each side of the cathode.—G. H. R.

Electro-Deposition of Metals. [*Triple Cyanide Bath.*] A. Zimmermann, London. From Dr. Courant, Berlin, Germany. Eng. Pat. 5468, March 13, 1899.

The electrolyte for potassium cyanide baths is formed by treating the potassium cyanide double salt of the metal to be deposited with sulphurous acid. The method for the preparation of the triple salt is described, and the triple salts of gold, silver, nickel, and zinc may be similarly prepared.—G. H. R.

Platinum, Gold, and Silver; Method and Apparatus for the Manufacture by Electro-Deposition of Articles Plated with. L. Bourdillon, Marseilles, France. Eng. Pat. 20,237, Oct. 9, 1899.

The process consists in electrolytically coating with copper one of the faces of an extremely thin sheet of platinum or gold, without affecting the other face, and may be also applied to copper, or any other metal, or to two or more metals successively, some of them serving as intermediaries for the others. Articles already made of extremely thin platinum or gold may be plated in the same way. The apparatus required, consists of a vat containing an ordinary copper plating bath, in which is placed a drum of some material, such as aluminium, which is not acted on by the electrolyte, and round which is wound a sheet of platinum or gold, of a thickness corresponding to that of the plating which it is desired to obtain. Copper anodes are arranged on either side, whilst the cathode is formed by the cylinder and a brush. The axle of the drum is provided with paddles which agitate the bath on its rotation.—G. H. R.

Inlaying Metal on Materials; Process of Galvanically. F. Eppler, Berlin, Germany. Eng. Pat. 25,594, Feb. 3, 1900.

For this process for producing inlaid work with any desired metal on any desired material, grooves are made on the ground material by chemical or mechanical action, such as a sand blast, corresponding to the pattern to be inlaid, and by a further process these grooves are partially filled with any desired kind of material, coloured or otherwise, so that the galvanic deposition to follow will adhere to them closely, and take place quickly and evenly. To secure the deposit, the grooves are undercut so as to give their section the form of a trapeze, and holes are bored in the bottom starting in different directions, in which pins of less valuable material may or may not be inserted. The heads of these pins may be covered by the deposit, or reach above it, so as to add to the ornamentation. Wires may be placed in the narrow grooves, and secured by passing their ends through holes in the ground material, or they may be sewn into it, whilst wire netting may be pressed into the wider grooves. In the case of non-conducting materials a style of soft metal is rubbed over the pattern in order to render the surface of the grooves conducting.—G. H. R.

Electrolysis (of Nickel) and Electrolytical Apparatus. U. L. P. Le Verrier, Paris, France. Eng. Pat. 5781, March 16, 1900.

This process for the electrolytic refining of pig nickel is also applicable in all cases where it is required to separate two metals, one of which can be precipitated in the state of peroxide. The electrolytic trough is divided into two compartments, in one of which the electrodes are placed. The other compartment is much narrower, and communicates with the former at the lower part, and forms a siphon. The bottom of the trough is in the form of a hopper to facilitate the accumulation of the precipitate. The bath is composed of a neutral and oxidising solution, and after it has undergone electrolysis, it is discharged through cocks provided in the outer wall of the smaller compartment, and is returned to the apparatus after having been clarified and renewed. Means are also provided for automatically brushing off the deposits formed on the cathodes.—G. H. R.

XII.—FATS, OILS, AND SOAP.

Macassar Oil. J. J. A. Wijs. Zeits. physik. Chem. **31**, 255—257. Chem. Centr. 1900, **1**, [6], 374.

The oil was extracted by means of benzine (petroleum spirit) from the seeds of *Schleicheria trijuga* obtained from the Celebes. The kernels weighed 60 per cent. of the seeds and contained 70·5 per cent. of fat, 3·5 per cent. of water, and 12 per cent. of proteïd matter. The fat melted at 22° C., determined by Crossley and Le Sueur's method, a portion however remaining solid at this temperature. It contained 3·12 per cent. of unsaponifiable matter, and had the saponification number, 215·3; Hehner number, 91·55; iodine number, 55·0; Reichert-Meissl number, 9; and acid number, 19·2. The fatty acids melted at 52°—54° C., and had the iodine number, 58·9; and acid number, 191·2—192·0. The volatile acids consisted of acetic acid together with a little butyric acid. 45 per cent. of the non-volatile fatty acids was solid; the liquid portion had the iodine number, 103·2.—A. S.

Sulphonic Acids containing the Stearic Radicle.

E. Twitchell. J. Amer. Chem. Soc. 1900, **22**, [1], 22—26.

Benzene-stearo-sulphonic Acid.—When a mixture of benzene and oleic acid is treated with an excess of sulphuric acid, any excessive rise of temperature being avoided, the following reaction takes place—

$$C_6H_6 + C_{18}H_{24}O_2 + H_2SO_4 = C_6H_4(SO_3H)C_{18}H_{35}O_2 + H_2O.$$

On treating the product with water and boiling, two layers are formed. The lower is a clear aqueous liquid which contains the sulphuric acid and whatever benzene sulphonic acid has been produced. The upper layer, a viscous oil, contains the benzenestearosulphonic acid, and the impurities which do not dissolve in dilute sulphuric acid. This layer is washed with hydrochloric acid and then repeatedly with petroleum spirit, which removes unaltered oleic acid and benzene, and fatty derivatives such as oxystearic acid and anhydrides. The compound is now pure with the exception of small quantities of water and hydrochloric acid, which are expelled by drying at 100° C.

Benzenestearosulphonic acid is dibasic, and the acid hydrogen of the sulphonic group can be titrated with methyl orange as indicator, whilst the acid hydrogen of the fatty acid group can be titrated by continuing the operation with phenolphthalein as indicator.

Theoretically, 100 parts of oleic acid produce 156·03 parts of benzenestearosulphonic acid. In two experiments, after making a deduction for oleic acid which had combined to form other substances, the author obtained yields of 151·0 and 156·7 parts from 100 parts of oleic acid which had combined with the benzene and sulphuric acid.

In its physical and chemical characteristics, benzenestearosulphonic acid shows some resemblance to stearosulphonic acid. At ordinary temperatures it is a sticky semi-solid mass which becomes a viscous oil at 100° C. Like its mono- and di-alkali salts it is soluble in water in all proportions, yielding colloidal solutions which form a lather on shaking, and is precipitated (salted out) on adding any strong acid, base, or salt. It differs from stearosulphonic acid in not being decomposed on boiling with water, and in yielding potassium sulphite instead of potassium sulphate when fused with potassium hydroxide. On being heated with hydrochloric acid it is only very slowly decomposed. A small quantity heated for 3½ hours in a sealed tube at 170° C. was only decomposed to the extent of 15 per cent.

Naphthalene-stearosulphonic Acid.—This is prepared and purified in the same manner as the benzene compound, which it resembles in its general character, though it is rather less fluid. Its composition corresponds with the formula $C_{10}H_6(SO_3H)C_{18}H_{35}O_2$.

Phenol-stearosulphonic Acid ($C_6H_3OH(SO_3H)C_{18}H_{35}O_2$) is obtained in the same way as the other compounds, and closely resembles them in character. (This Journal, 1898, 588.)—C. A. M.

Saponification; Theory of the Process of. J. Lewkowitsch. Ber. 1900, **33**, [1], 89—99.

The author gives in a number of tables the experimental results from which he concludes that during the saponification of fats and oils mono- and di-glycerides are formed as intermediate products (this Journal, 1899, 1631).

Fractional Saponification.—In the experiments, several pounds of tallow or cotton-seed oil were saponified with soda or lime as in the ordinary manufacturing process. From time to time, samples were withdrawn, treated with hydrochloric acid, washed and dried. The acetyl value and the Hehner and saponification values of the acetylated products were determined, whilst the amount of free fatty acids calculated from the acid value, showed to what extent the saponification had progressed.

It is pointed out that in drawing conclusions from the acetyl value, the only complication which might arise would be if the original fat had a definite acetyl value, but even in such cases, if the old theory of saponification were correct, the fat at all stages of the saponification would show practically the same acetyl value, and not yield higher values as would be the case in the presence of mono- or di-glycerides. The Hehner and saponification values of the acetylated products also yield evidence of the presence or absence of these lower glycerides, as is seen in the accompanying table.

Thus, if the older view of the course of saponification were correct, the Hehner value of the acetylated products could only fluctuate between 95·73 and 100, and the saponification value between 189·1 and 197·5, but in the presence

—	—	Hehner Value.	Saponification Value.
Tristearin.....	$C_3H_5(O.C_{18}H_{35}O)_3$	95·73	189·1
Stearic acid ...	$C_{18}H_{36}O_2$..................	100·00	197·5
Monoacetyldistearin	$C_3H_5(O.C_2H_3O)(OC_{18}H_{35}O)_2$	85·3	252·7
Diacetylmonostearin	$C_3H_5(O.C_2H_3O)_2(OC_{18}H_{35}O)$	64·2	380·8

of mono- or di-stearin there would be a far greater variation.

As a characteristic example, the following results of one of the experiments may be quoted.

Saponification of Cotton-seed Oil with Calcium Hydroxide.

—		Acid Value.	Acetylated Product.		
			Acetyl Value by Filtration Process.	Hehner Value.	Saponification Value.
Original oil acetylated ..		0·0	11·7	93·5	..
Partially saponified oil	1	0·5	14·9	94·5	206·3
	2........	0·6	20·0	92·84	209·2
	3........	16·0	43·15	92·0	230·1
	4........	17·6	59·2	89·1	240·0
	5........	19·9	28·3	92·35	215·3
	6........	53·4	24·9	93·8	214·8
	7........	73·2	32·4	93·6	223·4
Fatty acids obtained by means of alcoholic KOH, acetylated.		199·45	13·8	99·4	216·4

Of this experiment the author remarks that the fairly rapid rise of the acetyl value accompanied by a slower rise of the acid value points to the predominance of the first or first two steps in the saponification, whilst the subsequent rapid decrease of the acetyl value points to the decomposition of the monoglyceride predominating.

In fact, only the mean values of the concurrent phases of the operation are determined in these experiments, since simultaneously a molecule of diglyceride may be decomposed into monoglyceride and fatty acid, or a molecule of monoglyceride into glycerin and fatty acid, whilst a triglyceride molecule is only passing through the first stage.

In one of the experiments the saponification proceeded with such rapidity that but for the fact that a high acetyl value was obtained at the commencement, the results might have appeared to support the older view of saponification.

—C. A. M.

Petroleum ; Solidified. A. Conrady. Apoth. Zeit. **14**, 767 ; Chem. Zeit. Rep. **24**, [1], 8.

Aluminium oleate, prepared by precipitating dissolved aluminium sulphate with common soap solution, is recommended, the addition of 10—30 per cent. of warm petroleum furnishing a product of the consistency of vaseline suitable for impregnating boot soles.—C. S.

PATENTS.

Fatty Acids and the like ; Apparatus for the Distillation of. M. Blumski, Odessa, Russia. Eng. Pat. 2858, Feb. 8, 1899.

An apparatus is claimed for distilling fatty acids, tar, and the like, by means of superheated steam. The steam, before passing into the substance to be distilled, is caused to pass through a worm placed in the substance itself, for the purpose of utilizing the heat of the latter to superheat the steam. When the steam is to be greatly superheated, it is first allowed to pass through tubes placed in the furnace flue. The still is composed of a copper or cast-iron vessel, provided with a dome-shaped cover, and is surrounded with a masonry furnace. The steam, after passing through the worm, enters the substance by means of a perforated pipe at the bottom of the still.—W. P. S.

Soap ; Manufacture of. B. J. B. Mills, London. From A. Lumière and A. Nicolle, Lyons, France. Eng. Pat. 25,357, Dec. 21, 1899.

The patentees add a quantity of sodium or potassium "bibasic phosphate" to soap during its manufacture, for the purpose of neutralising any free soda in the same.

—W. P. S.

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

(*A.*)—PIGMENTS, PAINTS.

PATENT.

Lead Oxide and White Lead ; Manufacture of [by Oxidation], and Apparatus therefor. W. E. S. Bunn and E. J. Case, Peoria, Illinois, U.S.A. Eng. Pat. 25,130, Dec. 19, 1899.

Coarse particles of metallic lead are agitated and ground under water in an open vessel, till the mass is reduced to powder ; and the mixture is brought into a closed cylinder, where it is agitated with air under pressure. The unoxidised lead is then separated from the litharge by elutriation ; and, when it is desired to manufacture white lead, the latter is treated as before in another cylinder, with compressed carbon dioxide. Suitable apparatus is figured in the specification.—F. H. L.

(*B.*)—RESINS, VARNISHES.

Linoleum, Testing of. H. Burchartz. Mitt. königl. tech. Versuchsanst. 1899, **17**, [6], 285—291.

Experience has shown the advantages which linoleum possesses over other materials used for the same purposes, and what requirements a sample should fulfil. It has also been found that linoleums of different origins vary much in their properties.

Linoleum is prepared according to two methods, namely, that of F. Walton, in which ground cork, oxidised linseed oil, kauri copal, and rosin are incorporated together, with the addition of pigments, and calendered on to a jute canvas (this Journal, 1896, 75—79) ; and that of Taylor, in which the linseed oil is not thickened by natural oxidation, but by boiling (corticine). The author states that no kauri copal, which makes the linoleum pliable and the surface glossy and non-porous, is used in corticine.

The methods used for testing linoleum at the Royal Berlin Technical Experimental Station include determinations of specific gravity, weight per square metre, thickness, resistance to wear, the action of weak acids, alkalis, and petroleum, bending tests over mandrels of 10 to 45 mm. diameter, tensile strength, extensibility, and of permeability to water (see this Journal, 1895, 587).

Three samples of German made linoleum gave the following results :—

(1.) Specific gravity: 0·834, 1·222, 1·180 ; (Linoleum powder),—1·342, 1·325.

(2.) Thickness: 3·8, 3·6, 4·0 mm.

(3.) Weight per square metre: 3·580, 4·085, 4·280 kilos.

(4.) Wear: 2·2, 1·6, 1·1 c.c. per 50 sq. cm. surface for 440 revolutions of the disc (see this Journal, 1895, 587).

(5.) Bending tests : Samples began to break when bent over mandrels of 23, 25, and 30 mm. diameter respectively. On warming, the linoleum was less pliable.

(6.) Tensile strength :—In the direction of rolling : 42·8, 34·3, 28·0 kilos. per sq. cm. ; breaking length : 445, 298, 353 metres. At right angles to above: 57·5, 53·8, 46.6 kilos. per square cm. ; breaking length: 603, 468, 570 metres.

(7.) Water and petroleum exerted little or no influence on the colour of linoleum. The three samples were not affected very differently by acids and alkalis.

(8.) All the three samples were equally waterproof. The requirements for linoleum of one State department are as follows : "The linoleum must be brown and unprinted, must have a smooth and glossy surface, and contain so much oil that it possesses india-rubber-like elasticity. It must not be hard or brittle, and must not change its colour on washing with water and drying."—H. I.

PATENTS.

Composition or Treatment for Rendering Materials Suitable for being Written upon in an Erasable Manner. H. Briggs, Idle, Yorkshire. Eng. Pat. 5606, March 15, 1899.

The object of this invention is to render fabrics, pasteboard, and the like, capable of being written upon with pen or pencil, capable of having the writing erased and of being used over again, and also to give them (especially ordinary school "slates") a white surface, on which blue or black marks are more visible than usual.

The material is first brushed over with a solution of isinglass, and then wetted with methylated spirit, 1 pint; "French white gum shellac," 10 oz.; isinglass, $\frac{1}{4}$ oz., which is rubbed over the surface with a pad of cotton wool till the solvent evaporates, and the whole is well polished. Extra smoothness may be obtained by calendering before or (and) after the application of the second composition; and coloured grounds can be produced by the addition thereto of suitable dyes.—F. H. L.

Sealing Material, for Rendering Sheet Metal Joints tight, particularly applicable for Sheet Metal Cans and the like, for the Storage and Transport of Solid or Liquid Substances containing Oil, &c.; Manufacture of. W. Ruttenberg and M. van Zanten, both of Dordrecht, Netherlands. Eng. Pat. 17,423, Aug. 28, 1899.

The manufacture of a non-metallic sealing substance is claimed, consisting of a mixture of vegetable and animal glues or adhesive materials and carbohydrates, together with a precipitating agent, such as calcium chloride or other metallic salt. As an example, 40 parts of glucose are mixed with 30 parts of joiners glue, previously digested with 30 parts of water, and to the mixture is then added 0·1 to 1·0 per cent. of calcium chloride. In addition, bitter substances, such as assafœtida, having a neutralising effect on the carbohydrate may be incorporated.—A. W.

(C.)—INDIA-RUBBER, &c.

India-Rubber in West Africa. (Report by R. Schlechter to the German "Colonial Committee.") Gummi-Zeit. 1900, **14**, [17], 267.

Kickxia appears to be very promising for plantation. Most of the seeds imported into Cameroon from Lagos germinated, and are rapidly developing into robust looking trees. On the plains near Stanley Pool large quantities of "root rubber" are found, but the product is of very poor quality. At Leopoldville the milk of several species of Ficus was examined, all of which yielded only the well-known sticky masses. At Coquilhatville the famous Bossanga was found to occur, and was identified as a species of Costus. Manihot Glaziovii has been planted in this locality, but, as in Cameroon, was found to yield an insufficient amount of milk. The Kickxia which is indigenous in this district is Kickxia latifolia Stapf from which no india-rubber is obtainable. All the india-rubber of the Congo is obtained from the interior, two or three weeks' journey from the coast. Only one of the Landolphias of the Congo yields no rubber. The rubber is obtained by detaching a Landolphia of sufficient dimensions from the trees, supporting it along the ground by means of wooden forks, and making incisions at distances from 1 to 1·5 feet. The milk which is thus obtained is coagulated without boiling by means of the juice of the Bossanga plant, the coagulated mass well kneaded and drawn out into sausage shaped masses. These are the next day divided into small sections, which are subsequently air dried during a period of two months. The high quality of Congo rubber is therefore evidently due to the entire removal from it of all the water. The effect of the acid contained in the juice of the Bossanga, in this direction, should be determined by experiment.—C. O. W.

Caoutchouc; A new Mode of Extracting, from the Barks of various Plants, especially of Landolphia. A. Arnaud and A. Verneuil. Comptes Rend. **130**, [5], 259—261.

The dried barks are pulverised and sifted to remove fine powder. The residue is saturated with hot water, and ground to a paste, which is again passed through a sieve under water. The magma which has not passed the sieve is again submitted to milling till the caoutchouc has agglomerated into spongy masses, when the whole is thrown into boiling water. The caoutchouc floats to the surface, and is worked into a single mass or cake, which is then purified in the ordinary way by passage through differential rollers. The yield from *Landolphia* is about 8 or 9 per cent. from the stem-bark, 14 or 15 per cent. from the root-bark. The process is extremely simple, and yields a rubber free from resins or fatty substances.—J. T. D.

PATENT.

Caoutchouc; New Manufacture of Compound for Mixing with. O. Imray, London. From W. V. McManus, New York. Eng. Pat. 17,655, Aug. 31, 1899.

Five lb. of rape oil and 5 lb. of powdered graphite are heated to about 44° C., sprinkled with about 8 fluid oz. of sulphuric acid of "66 per cent." and 6 oz. of sulphur chloride, and the whole well agitated. The material is then ground in water, washed with a weak solution of caustic soda, and finally incorporated with pure rubber in the proportion of about 4 : 6 parts. The inventor prefers to heat the vessel in which the composition is made electrically by means of "insulated electric resisting conductors," as he finds that when "electrical influence" is employed, the compounding of the materials is greatly promoted, and the substance requires much less washing in alkali to remove the by-products. Zinc white or white lead may be used instead of the graphite.—F. H. L.

XIV.—TANNING, LEATHER, GLUE, SIZE.

Kips; East India. Leather Trades' Rev. **33**, [726], 115. (See this Journal, 1900, 60.)

It was formerly the custom in the tanning of East India kips to tan out in "shifts," which required from 7 to 12 weeks, according to the character of the goods; three or four new liquors being used, also a good valonia, mimosa and gambier liquor as a finish, in which they remained from four to seven days. By laying such goods away, the time is considerably reduced, the leather remaining in the handlers about four weeks for the light, and six weeks for the heavier hides; and then from one to two weeks in the layers. By this method the goods are better "filled," especially in the flanks, they weigh more, are brighter in colour, and more suitable for colour work, whilst in the layers they do not require to be handled, which means economy of labour.

After the goods have been drawn from the layers, and allowed to drain overnight, they are drummed in sumach, prepared as follows:—A bag of sumach is scalded in a tub or vat and allowed to remain overnight, and warm water is then added until a stiff paste is produced. Fifty kips are then placed in the drum with six buckets of the sumach paste, and "run" for about 20 minutes; the goods are afterwards washed in hot water, and allowed to drain over a horse. After draining for three days, they are oiled with a brush on the grain side with cod oil, which has been previously warmed. The tables on which the oiling is done should be of such dimensions that a full-sized kip can be opened out. When the 200 kips of a whole pack have been oiled, they are allowed to lie for at least 24 hours for the oil to soak in.—J. G. P.

Tannage; New System of Electrical. Carl Lucknow and Franz Toerisser. Leather Trades' Rev. **33**, [726], 118.

A new system of elecrical tannage forms the subject matter for a patent recently taken out in Germany by the above named. The patentees claim a process for making hides conductors of electricity by stretching them on a perforated metal plate, of effecting the same result by rubbing them with plumbago, and by precipitating in the inner tissue of the hide, by electrolysis, a metallic substance. In practice, the hides are stretched tightly across a metallic conductor in the form of a plate, so that a perfect contact is assured between the two. The conductors, or plates, in order to facilitate the circulation of the electrolytes, should be provided with perforations at intervals, or a wire netting with small meshes may be used, or of charcoal of similar external shape. The plates, which serve as cathodes, are of the same material and form. In order to protect the conductors upon which the hide is stretched from being attacked by the anions, (*viz.* hydrogen and the acid radicals), they may be polished with plumbago, or plated with platinum, or made of certain alloys which are little affected by these substances. A similar result may be attained when the hides are coated or impregnated with a metallic substance so that their conductivity is correspondingly increased, and thus they are fitted to act as anodes. They may, therefore, be brushed with plumbago, or the hides may first of all be soaked in a solution of acetate of lead, and then transferred to one of chloride of potassium, by which means, basic chloride of lead is deposited in the fibrous tissue.

After being thoroughly washed, the hide thus prepared may be suspended as a cathode in a suitable bath. The hide having thus had a metallic conductivity imparted to it, must now be placed in a suitable liquor to serve as an anode. The solutions to be electrolysed principally consist of tannins or of salts containing tannins—the so-called tannates. In practice, where feasible, in order to accelerate the process without injury to the leather, the conductivity of the electrolyte is increased by the addition of certain salts, acids and bases of easy solubility, the anions of which, by acting upon the pores of the hide, also exercise a plumping effect which facilitates the penetration of the tannin and improves the quality of the leather.

The solutions to be electrolysed may further consist of such substances as are used in the manufacture of tawed leather or leather which has been treated with alum. Only a very moderate current is employed. The tannage may be commenced with one of from 50 to 100 ampères per square metre of anode surface, and can subsequently be gradually increased. The tension is about 3 volts.

During the process of the tannage, the liquors must be frequently strengthened, and fresh tanning materials added as required. When the tannage is complete, the leather is taken out and finished by the usual methods.—J. G. P.

Seal Skins for Black Levant; Manufacture of. Leather Trades' Review, **33**, [726], 128.

In the preparatory process, the skins, after being well cleansed in the usual way, are freed from adhering flesh and blubber and then "limed," the skins being first put into an old lime, and worked on into stronger pits, until the liming effect is complete, three or four weeks being generally required for the operation. The skins are then unhaired in the ordinary way, and split, followed by puering with dog manure, drenching, and a thorough working out on the beam to remove all traces of lime salts, and the complex collection of organic compounds, which the tanner terms "filth."

From the beam house, the skins are usually put into a weak gambier liquor of 10° Bk. [Barkometer. Sp. gr. of 1·025 = 25° Bk.], being handled often in the early stages until well struck through. They are then passed to a stronger liquor of 15° Bk., made up largely of best oakwood extract, and finished in a liquor of 20° Bk. "Block" gambier is often used for the sake of cheapness, but "cube" gambier is perhaps the cheaper in the end, by reason of its being less adulterated.

When the skins are tanned, they are finished off in a good sumach bath, lightly oiled, and dried out in the ordinary way. To "finish" the skins, they are damped back, struck out, slightly stiffened and set. If a bold grain be required, they are embossed and again dried out; if a natural grain be wanted, the goods may be blacked at once, giving as a "finish" a good bottom coat of logwood, prepared by extracting 1 cwt. of best Campeachy logwood, 5 lb. of carbonate of soda, with 45 gallons of water, until the logwood is extracted, the liquor being at once drained off. Iron vessels are often used, but as the wood contains a small percentage of tannin which combines with the iron, this tends to destroy the brilliant colouring properties of the logwood. The material should, therefore, be extracted in a steam-jacketed copper extractor.

A simple "black" for seasoning seal skins is made with 1 quart of water to 3 quarts of good iron liquor, to which about 1 lb. of good Scotch glue is added. This is brushed on warm, and well rubbed in. If a "powder" grain is required, the solution must be used cold, and only half the above amount of glue added.

After "airing" the skins off slightly in a stove, they are ready for "wet graining," commenced by crossing from belly to belly, followed up by crossing from shank to shank, and finishing off by "breaking up" from neck to butt.

The embossed or printed skins are dried out after embossing, wet-grained, and blacked as above, using a larger quantity of glue, which gives to the goods a crisp feel. The skins are then dried out in a hot stove, laid away to cool before "fluffing" and seasoned with three-quarters of a pint of milk, and one and a half pints of blood, added to a gallon of water. This is rubbed well in with a stiff brush, and the goods again dried out in the stove. The skins are then damped back with a mixture of milk and water (1 in 6), and rolled up in half dozens, grain to grain, for a few hours. They are then ready for "rolling on." A No. 2 roller is suitable for fine grains, and a No. 4 for a bolder grain, commencing across butt to fin. The skins are then grained off, after which they are oiled with best warm Newfoundland cod oil.—J. G. P.

Tanning Materials and Basic Dyestuffs; Ullmann's Method for the Valuation of. A. Heinemann. Chem. Zeit. **24**, [7], 58.

See under XXIII., *page* 279.

Glue; Examination of, and Determination of the Gelatin. A. Ruffin. Chem. Centr. 1900, **1**, [3], 233.

See under XXIII., *page* 279.

PATENT.

Hides; Improved Process for Treating. L. Friedländer, Rixdorf, near Berlin. Eng. Pat. 1317, Jan. 19, 1899.

The hides, before or (preferably) after tanning, are treated with a solution of resin in carbon bisulphide (1 : 2), or with a solution thereof in some hydrocarbon solvent. It is claimed that these liquids produce a specific effect upon the albuminous substances of the hide, so that the leather is rendered more durable, whilst low quality hides are made better and stronger.—F. H. L.

XV.—MANURES, Etc.

Plant Soil Tests; Danger of Erroneous Conclusions from. H. J. Wheeler. 15th Annual Convention of Official Agricultural Chemists, Washington, 1898. U.S. Dept. Agric. Bull. 1899, [56], 60–62.

In testing the effects of various manures on the crops of maize yielded by a certain soil selected for the purpose, the author found a great increase in the available phosphoric acid of the soil after the application of a lime dressing. The soil which was deficient in phosphoric acid available for plant food before liming and which required the use of phosphatic manures in order to obtain a good yield, was found afterwards to contain an abundance of that constituent, which was only made available after the neutralisation of the soil acidity by lime. The precaution of testing the effect of lime should, therefore, always be taken before reporting that any particular soil is in need of phosphatic manure.

—J. F. B.

Cocoanut Ash; Analysis of. F. Bachofen. Chem. Zeit. 1900, **24**, [3], 16.

The proportions of the several parts of a nut taken from a palm growing in a suitable white sandy soil, were found to be:—1. Husk, 57·28 per cent. 2. Inner shell, 11·59 per cent. 3. Copra (the white mass containing the cocoanut oil), 18·54 per cent. 4. Milk, 12·58 per cent.

The husk contains 1·63 per cent. of its weight of ash, the percentages for the inner shell, copra, and milk being 0·29, 0·79, and 0·38 respectively. Analyses of the ashes from the separate parts gave the following results :—

——	Husk.	Inner Shell.	Copra.	Milk.
SiO_2	8·22	4·64	1·31	2·95
$Fe_2O_3+Al_2O_3$	0·54	1·39	0·59	Trace
CaO	4·14	6·26	3·10	7·43
MgO	2·19	1·32	1·98	3·97
K_2O	30·71	45·01	45·84	8·62
Na_2O	3·19	15·42	..	..
$NaCl$	45·95	15·56	5·01	26·32
KCl	..	..	13·04	41·09
P_2O_5	1·92	4·64	20·33	5·68
SO_3	3·13	5·75	8·79	3·94

Estimations of water and nitrogen gave the following percentage numbers :—

——	Husk.	Inner Shell.	Copra.
Moisture	65·56	15·20	52·80
Dry substance	34·44	84·80	47·20
Nitrogen as NH_3 in moist substance	0·167	0·122	0·612

The value to the soil of ash constituents such as the foregoing is clear, and, as a matter of fact, in Ceylon it has for long been largely the custom to return systematically to the soil those portions of the nut not useful as a foodstuff, so as to replace the mineral matter abstracted.—T. H. P.

Fertilizers on Calcareous Soils; Use of Ammoniacal. E. Giustiani. Ann. Agron. 1899, **25**, 325—335.

A SERIES of experiments was made, in which air, both moist and dry, was passed through sand mixed with various amounts of calcium carbonate, and known amounts of ammonium sulphate. For comparison, pure sand and calcium carbonate were also employed separately; and, in some experiments, basic slag was mixed with the sand instead of calcium carbonate.

With moist air, about a quarter of the ammoniacal nitrogen was lost in 48 hours in presence of calcium carbonate, the amount of the carbonate being practically without effect. When dry air was employed, nearly all the nitrogen was lost.

Basic slag acted still more quickly than calcium carbonate, and, with 3 per cent. of the slag, nearly all the nitrogen was removed in 20 hours.—N. H. J. M.

Fertilizer Experiments on Crops. A. C. True. U.S. Dept. Agric. Farmers' Bul. 107, 1899, 5—7.

A LARGE number of fertilizer experiments with various crops have been made in Massachusetts, and results obtained which are of great value to farmers who employ the same system on similar soils. Where other conditions prevail, this system must be adopted with caution. In the districts where the experiments were made, the grain is not sold, and, consequently, the loss of phosphates is limited, especially as foods rich in phosphates are purchased. The farmers, however, sell hay, straw, and vegetables, and in this way part with a good deal of potash. Hence the crops are liable to be richer in phosphoric acid than in potash; and whilst the soil undergoes exhaustion in potash it accumulates phosphoric acid.

Under similar soil conditions, maize, clover, rye, and beans seem to especially require potash, whilst grasses and oats require nitrogen; and mustard, cabbages and swedes, phosphates. Single manures, to be mixed as required, are preferable to "complete special fertilizers."

Continuous use of potassium chloride may so far deplete the soil of lime that an occasional application of lime to the soil may be required; potassium sulphate is sometimes preferable.

When more than 150 lb. of nitrate of soda is applied per acre, half of it may be kept back until the plants are 3 or 4 ins. high.—N. H. J. M.

Oil Cakes; Manurial Value of. L. Malpeaux. Ann. Agron. 1899, **25**, 111—126.

As regards the after-effects of cake applied as manure, it was found that whilst rape cake and earth-nut cake, which had but little effect the first year, were very effective the next year, sesame, cameline, and castor-oil cake had less effect.

It is of importance, but not equally in the case of the different cakes, to apply them in the autumn. Rape and earth-nut cakes give very unsatisfactory results if applied at the same time as the seed, especially if the sowing be followed by a dry summer.

Sesame cake proved to be the best as manure, whilst the values of the other cakes decrease in the following order: poppy, earth-nut, cameline, colza, castor-oil, cotton, and palm. If applied in the spring, the cakes recommended are poppy, sesame, castor-oil, and cameline.

The effect of oil cakes depends on several conditions, and the order of manurial value as given above is not to be considered absolute.—N. H. J. M.

Mechanical Soil Analysis; Rapid Method of [Centrifugal]. C. G. Hopkins. 15th Ann. Conv. of Official Agricult. Chem. Washington, 1898.

See under XXIII., *page* 279.

PATENT.

Fertilizer rich in Phosphoric Acid, and a Phosphorous Pig Iron; Method of Producing. A. J. Leopold of Forselles, St. Petersburg. Eng. Pat. 21,484, Oct. 27, 1899.

A FERTILIZER slag rich in phosphoric acid and a phosphoric pig iron suitable as a very fluid molten metal for castings, are produced in one process by melting in a cupola or blast-furnace a mixture of coal, iron scrap, fluxes, if required, and a phosphoric rock, such as apatite, in proportions convenient for the said purposes. The phosphoric pig iron may be further treated by the basic Bessemer or Siemens-Martin process, the resulting slag being also suitable as a fertilizer.—A. W.

XVI.—SUGAR, STARCH, GUM, Etc.

Cane Sugar Factory, Technical and Practical Notes on the. E. Marchand Bey. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1899, **17**, [6], 396—406.

IN double pressing, the bagasse should be soaked with hot water immediately on leaving the first mill, in order to allow as much time as possible for the softening of the cells, and the conveyor to the second mill should be enclosed to prevent cooling. By introducing water below the moving bagasse as well as above, the maximum useful effect is realised owing to uniform exhaustion.

Mills considered of excellent make cannot usefully support a greater pressure than 200 or 220 kilos. per sq. cm. on the generatrix of the delivery roller, while a pressure of 300 kilos. with other modifications would give a yield of 76 to 82 per cent. of juice according to the quality of the canes. The ordinary type of mill gives 75 per cent. The juice left in the bagasse amounts to 13 per cent., but its quality is lower than that of the cane so that the loss of sugar is 1·173 per cent., where, with the extra crushing power mentioned above, it would be reduced to 0·9 per cent. There is no possible means of extracting the sugar entirely.

Diffusion, by increasing the quantity of juice extracted, would likewise reduce the purity and entail expense in the chemical operations needed for purification; also, the exhausted pulp is inferior to bagasse as a combustible.

It might be suitable for canes of high and uniform quality, but is quite unsuitable for bagasse which contains much foreign matter. Next to mills, however, diffusion in closed vessels is the best process; washing, maceration, or levigation in the open air is condemned on every hand.

Dried bagasse from the fields still contains about 15 to 20 per cent. of moisture and represents 14 per cent. of the cane; its calorific power is, however, inferior to Newcastle coal, one kilo. of this coal being equal to 2·077 of dry bagasse. The wet bagasse direct from the mills is burnt in some places; it contains 50 per cent. of moisture, and has about 55 per cent. of the calorific power of dry bagasse. The ordinary grates are not suitable for burning it in this condition, as the constant stirring needed would allow too much air to enter; gasogenes are therefore necessary. It may also be dried by utilising the heat of the chimney gases, but the ventilation needs careful attention to avoid risk of ignition, and in any case, other fuel must be used to reduce the moisture to 20 per cent.

The juice extracted at the various mills is mixed, and is consequently received in a diluted condition. To control the extraction, the volume V_1 found must be corrected to the normal volume V at 20° C. If d be density of the mixed juice and D that of the real juice at the first mill, then $V = V_1 \times \frac{d-1}{D-1}$.

With juice of high purity that is to be boiled to grainy sugar for refining, simple defecation to neutrality with lime is sufficient. It is preferable to add lime in the cold and heat afterwards, and then defecate. The colour obtained is improved. Inferior or spoiled juice requires carbonating, sulphiting, &c., just as beetroot juice does.

Animal charcoal is the best filtering agent. Inversion caused by char filters need not be feared, provided the filter be not used too long.

Exhaust steam is to be preferred in the first vessel of the triple or multiple effect, since high pressure steam darkens the juice and causes inversion. The juice may conveniently be concentrated to 29° B. at 55° C., its volume when dilute being reduced in this way by 70 per cent. The viscosity of the syrups after boiling may be enormously reduced by sulphiting, which also improves the colour.

Centrifugalising is relatively difficult owing to the viscosity of the mother syrup of the massecuite; a charge of 50 kilos. of massecuite requires about 10 minutes. Cane generally needs about three times the number of machines that beetroot does. Chemical purification would reduce this. The returned syrup should be separated according to its quality, the best being that which runs after centrifugalising with steam and later still. But a simpler method is to allow the whole syrup to rest in a tank, when a natural separation takes place in about 12 hours. The purity at the surface of the tank is about 0·79, at the bottom, 0·90; the former is used for the second jet sugar, the latter is returned to the direct liquor.—L. J. de W.

Raw Beetroot and Diffusion Juice; Influence of the Lead Precipitate on the Polarisation of. J. Weisberg. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1899, **17**, [5]. 361—363.

Opinions are still divided as to the influence of the lead precipitate on the polarisation of juice to which one-tenth volume of basic acetate of lead has been added. According to Sachs the polarisation is increased by about 1 per cent.. and 0·10 should be deducted from the result. Pellet, who made simultaneous tests with other reagents, alcohol, acetic acid, and sulphurous acid, concludes that the influence is nil.

The author followed the method of Scheibler, operating on double volume, but diluting six or seven times, and instead of using pipettes, weighing the juice.

From diffusion juice at 14·3° Brix, quantities equal to twice, four times, and six times the normal weight (German) were weighed out, and 5, 10, and 15 c.c. respectively of basic lead acetate of 27° Baumé were added and the solutions made up to 220 c.c.; the readings were 22·5, 45·0, and 67·5 in a tube of 400 m.m.

Twice, six times, and seven times the normal weight of juice pressed from fresh beetroot chips (17·1° Brix) were treated in like manner with 5, 15, and 17·5 c.c. of lead acetate; the readings were 26·35, 79·10, and 92·20 respectively.

Juice pressed from very rich beetroots was taken (20·8° Brix), and once, and seven times the normal weights were used and 2·5 and 17·5 c.c. of basic lead acetate added; the readings were 8·35 and 58·45 respectively.

From these results it is concluded that the precipitate does not increase the polarisation.—L. J. de W.

Raw Sugar; Invertive Power of. L. Jesser. Oesterr-ung. Zeits. Zucker-Ind. u. Landw. **28**, 626—628; Chem. Centr. 1900, **1**, [2], 106.

The author made determinations of the invertive power of some raw sugars. The alkalinity of the sugar was determined before and after two hours boiling. The difference gives the amount of lime or alkali which has been neutralised by the acids that are formed from the sugar on boiling and from the invert sugar on boiling with alkali. Thus the amount of invert sugar can be calculated, and also that of the saccharose destroyed. It was found that the amounts of sugar destroyed on boiling solutions of raw sugar were very different with the individual sugars, and that the extent of decomposition bore no ratio to the alkalinity. The alkalinity of a sugar is therefore no sure indication of its goodness. It is also stated that the alkalinity of, and the amount of alkali contained in a sugar are by no means synonymous terms.—A. S.

Sorbose, Production of. G. Bertrand. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1899, **17**, [6], 385—396.

Sorbose does not exist ready formed in the juice of mountain ash berries, but is produced by the oxidation of the sorbite contained in the latter under the influence of a bacterium analogous to or identical with the *Bacterium xylinum* of Brown, which is usually introduced by the small red vinegar fly (*Drasophila cellaris*). The most suitable concentration of the juice for the production of sorbose is 1·05 or 1·06 at a temperature of 29°—30° C. The yield of sorbose is 80 per cent. of the sorbite present.—L. J. de W.

Syrups and Similar Products; New Method of Determining the Water in. O. Molenda. Chem. Centr. 1900, **1**, [1], 71.

See under XXIII., *page* 280.

Sugar Analysis [Beet Molasses]; Report on. R. S. Hiltner. 15th Ann. Conv. of Offic. Agricult. Chem., Washington, 1898.

See under XXIII., *page* 279.

Effluent Water from Sugar Factories; Proskowetz's Method for the Purification of. von Rosnowski and Proskauer. Chem. Centr. 1900, **1**, [2], 135.

See under XVIII. B., *page* 265.

Effluent Water from Sugar Factories; Proskowetz's Method for the Purification of. M. Hönig. Chem. Centr. 1900, **1**, [2], 149.

See under XVIII. B., *page* 266.

l-Arabinose; Down-grade Products of. [Pentose to Tetrose.] A. Wohl. Ber. 1899, **32**, [18], 3666.

See under XXIV., *page* 282.

d-Erythrose. O. Ruff. Ber. 1899, **32**, [18], 3672.

See under XXIV., *page* 282.

PATENT.

Sugar Powder; Direct Manufacture of. A. Vallez and F. Giraud, Montpelier, France. Eng. Pat. 24,213, Dec. 5, 1899.

A sufficiently pure solution of sugar or green syrup is boiled in an open, steam-heated pan until it reaches a certain point of high concentration. It is then poured boiling into a special apparatus, which by pugging and kneading eliminates the water in the form of vapour without further heating, and transforms the sugar into a pulverulent mass. The apparatus in question consists of a vessel, the bottom of which is made up of two cylindrical segments in which powerful pugging shafts with arms rotate, the arms of one shaft being furnished with flat, rake-like ends;

the vessel is arranged to tip over without interfering with the motion of the shafts. The principle on which the process depends lies in the fact that the latent heat of the concentrated boiling solution is greater than that necessary for the vaporisation of all the water, and its utilisation is facilitated by the constant renewal of the surfaces of evaporation by the mechanical motion.—J. F. B.

XVII.—BREWING, WINES, SPIRITS, Etc.

Badly Germinating Barley; Use of Bleaching Powder for the Impt. of. Cerny, Oesterr. Brauer and Hopfen-Zeit. 1900 [1], through Woch. für Brauerei, **17**, [1], 5.

Barley badly damaged by rain, also partly heated and mouldy, with dark coloured husks, was sampled into two separate heaps. One heap was worked up in the ordinary way and the other was steeped in calcium hypochlorite solution made by mixing 10 kilos. of bleaching powder with a hectolitre of water and diluting with 50 hectolitres of water. The chlorinated water was used as the last steep and left on the barley for 24 hours. The two lots of barley were then malted under parallel conditions. The hypochlorite produced a lighter colour both of the steeped barley and the kilned malt. The germinative energy and germinative power was considerably greater and the development of the rootlets and acrospires had the character of a healthy growth in the case of the malt prepared from the hypochlorite steep; the formation of mould was either entirely suppressed or considerably restricted. The malt from the plain-steeped barley, on the other hand, contained so many ungerminated and mouldy corns as to be unfit for brewing purposes. The beer from the hypochlorite barley was perfectly good, and it appeared to have more body and a thicker and more permanent foam than ordinary beer.

—J. F. B.

Barley; Treatment of, in the Steep and Influence of Aëration in the Steeping Vat. W. Windisch. Woch. für Brau. 1900, **17**, [3], 33—34.

The author has frequently expressed his doubts as to whether it is really necessary to steep the barley at all, or at least, whether a very short steeping would not suffice, and thus bring the operations on the large scale more into accord with those in the laboratory. A friend of his has put through some experimental maltings with barley simply washed and then supplied on the floor with the necessary moisture from cans. Without being able to definitely recommend the substitution of this method for the usual one, the results were in every way satisfactory, and warrant systematic investigation of the comparative efficiency of the two methods. Another brewer varied the time and extent of steeping, with the result that barley steeped for 24 hours germinated well, whilst that steeped for 3 days became mouldy.

Aëration in the Steeping Vessel.—A brewer named Grohn writes to the author, informing him of the splendid results obtained by a full aëration of the barley during steeping. It is well known that barley respires during this process and requires air, but most maltings have their steeping-vats in the germination room, where the air is heavily charged with carbon dioxide. He prefers a colder temperature and plenty of fresh air. He finds that, apart from artificial aëration, the best way is to follow the English custom and to supply the steeping water by means of a rotating sprinkler. Water may be economised by closing the outflow cock and letting the vat fill up intermittently, but allowing the water to escape continuously during the last 24 hours. Barley so steeped can be malted much cooler and begins to germinate in 24 hours as against 3 days without aëration. Thus, there is a clear gain of 2 days' germination, which may be utilised by working the heaps at a cooler temperature on the floor. The germination of barley so steeped is quite uniform.—J. F. B.

Malting-floors and Malting. F. Bousquet. La Bière, **7**, [11], 161—162.

In a brewery, the malting floor is preferably situated underneath the steeping vat, and should be partly underground, 7—10 ft. below the outside level, in order to facilitate the maintenance of a uniform temperature. The floor may be formed of 8—10 ins. of well-rammed clay covered with a 4-inch layer of concrete, over which is laid the actual paving material. Bricks are too porous, absorbing the moisture of the malt too readily, and also affording at the joints a harbouring place for mould fungi. Asphalt is liable to crack and soon becomes hot, whilst cement also exhibits the first-named defect unless properly laid; provided however a cement of good quality be taken, mixed with a predetermined quantity of sand, carefully spread, and kept moist until properly set, it will form an excellent and very durable floor. Solenhofen tiles are said not to absorb moisture when once properly damp, but are expensive. In any case a slight fall should be provided, but not so steep as to drain off too much water.

The principal point to consider with regard to the walls is that they should offer as little harbourage as possible to mould, and be easily cleaned and disinfected, cement or faïence tiles being the best material, though these may be cheaply replaced by a coating of enamel, pitch, or lime-wash.

For ventilation, windows should be arranged on the north side, so that direct sunlight is excluded; they should be on a higher level than the couch of malt, and preferably swung on pivots. Small channels should be arranged in the lower part of the walls to facilitate the escape of the heavy carbon dioxide liberated during germination.—C. S.

Malt; Kilning. R. Gifhorn. Allgem. Zeits. für Bierbrau. **27**, [49], 1172—1174.

In order to prevent gelatinisation of the starch, and the resulting production of steely malt, the first stage of kilning, *viz*., drying the malt, should be performed at a temperature not exceeding 44° C., preferably between 31° and 38° C., this temperature also preserving the diastase. To develop colour, the malt, containing 8—12 per cent. of moisture, must then be heated at about 63°—75° C. (in the malt) and afterwards at 88°—100° C. (measured in the air), the resulting colour depending on the proportion of moisture, the rapidity of heating, and the temperature employed. The duration of exposure at 63°—75° C. also influences the development of aroma.

For malt of the "Pilsen" type, it is necessary to dry well at a low temperature before proceeding to heat (gradually) to 50°—63° C. This gives a pale malt of faint aroma, high in diastase, and therefore yielding a pale, highly attenuated beer of vinous flavour. For "Vienna" malt a higher finishing temperature (75°—100° C.) is required to develop the colour and aroma necessary to the production of a golden yellow beer; whilst for malt of the "Munich" type, the initial stage is abbreviated, the malt with 10—15 per cent. of moisture is kept for several hours at 63°—75° C., and finished at 88°—100° C. (air temperature), the product being brown and aromatic, and yielding a dark beer of decided aroma. Though no hard and fast rule can be laid down as regards manipulation on the kiln, the object of drying the malt on the upper floor is most readily effected by subjecting the material to as few turnings as possible; whilst on the lower floor, more frequent turning is desirable in order to secure uniformity of curing and colour, especially towards the end of the process.—C. S.

Starch; Saccharification of, by Malt Diastase. A. Fernbach. Ann. de la Brass. et de la Distill. 1899. Through Woch. für Brau. 1900, **17**, [3], 34—37.

Former investigators have recorded the observation that diastatic action is stimulated by the presence of small quantities of acid (Compare Ling, this Journal, 1898, 478). The author comes to a different conclusion, and lays stress on the distinction between the acidity of acid salts and that of free acids. The chief impurity of precipitated diastase consists of ash, and it is noted that the diastase prepared from *Aspergillus niger* contains only about one-tenth of the quantity of ash present in that from malt extract. Strictly comparative experiments with increasing additions of small quantities of acid, were made, and the maltose produced in a given time, was determined. It was found that, with the exception of small quantities of phosphoric acid, the addition of acid, even in the smallest proportions, caused a decrease in the velocity of the transformation, which retardation increased with the quantity of acid used, until the saccharification was entirely inhibited. By previously heating the diastase with acid for an hour, it was proved that the retarding effect of the acid on the saccharification was not due to any destructive action of the acid on the diastase. The acidity of malt is due not to free acids but to acid salts, principally acid phosphates. Phosphates have a specific action on the saccharification process; neutral [di-basic] phosphates are alkaline to Methyl Orange; they also have a harmful effect on the saccharification. Diastase acts best in a medium in which the phosphates have a neutral reaction to Methyl Orange; acid [mono-basic] phosphates are therefore favourable; free phosphoric acid is most harmful. Small quantities of acid oxalates, tartrates, and sulphates have very little influence on the velocity of diastatic action; no retardation is produced by larger quantities of the normal salts, but the free acids act powerfully. The first effect of adding small quantities of acid to the malt extract is the transformation of the phosphates; therefore the influence of the first additions of acid will depend on the proportion of normal phosphate present. If this be high, the addition of acid, and especially of phosphoric acid, will stimulate the hydrolytic process by the conversion of the harmful normal phosphate into the favourable acid phosphate; the retarding effect will only appear when the phosphates have been saturated and free acid occurs in the mixture. The purer the diastase and the starch, the smaller the quantity of acid required to produce a retardation. The above results will naturally render the methods for the determination of diastatic power, as practised at the present time, of doubtful value.—J. F. B.

Pressed-Yeast; Process of Purification of Molasses for the Preparation of. M. Fuhrmann. Ger. Pat. 108,334, Aug. 6, 1898; through Zeits. Spiritusind. 1900, **23**, [2], 11.

The albuminoids and other organic impurities present in molasses render them unsuitable for use in pressed-yeast manufacture. The present process for their purification consists in the treatment of molasses of any alkalinity or acidity at any dilute concentration with alum at a temperature of 70° C. The alum precipitates the albuminoids and other matters in a manner and to an extent not effected by any other reagent. The result is a clear and odourless molasses, and the fact that the cane sugar is inverted by the treatment does not in any way interfere with its use for the above purpose. Excess of alum is removed by precipitation with lime.—J. F. B.

Molasses for Pressed-Yeast and Spirit Manufacture, Process of Purification of. A. Marbach and M. Fuhrmann. Austrian Pat. 516; through Zeits. Spiritusind. 1900, **23**, [5], 35.

The process consists of the treatment of the molasses with zinc dust and sulphurous acid. The molasses are diluted to 30 per cent. concentration, warmed by steam to 70° C., and treated with sulphurous acid to strong acid reaction. At this temperature the cane sugar is inverted, and the acidity brings about the precipitation of a large part of the albuminoids. A quantity of sulphurous acid is then added to the hot liquor sufficient to bring the acidity up to 0·2 per cent.; at the same time the calculated quantity of zinc dust is added in the form of a sludge, and the temperature is raised to near the boiling point. Immediate and perfect clarification then ensues, and the liquor is neutralised with lime, heated, and filtered in the press.—J. F. B.

Yeast; Multiplication of, without Fermentation, in Presence of Limited quantity of Air. A. Rosenstiehl. Comptes Rend. 1900, **130** [4], 195—198.

In the examination of cider the author has frequently observed the multiplication of yeast without the evolution of carbon dioxide. The yeasts in question (*apiculatus* and *ellipsoideus*), when taken from cider and grown on solidified must plates, can ferment artificial must directly in a normal manner, but if they are sown from the plates into natural apple juice they show this property of reproduction without fermentation. The cells are $\frac{1}{2}$ or $\frac{1}{3}$ smaller than usual and seem to be in an enfeebled state; they only regain their normal fermentative properties after two or three cultures in natural must. The enfeebling of the yeast was proved to be due not to the action of an oxydase, but to the presence of tannin. If sufficient gelatin be added to precipitate the tannin of the must the fermentation is normal, but if such an excess of gelatin be used as to redissolve the tannin precipitate, no fermentation takes place, and the cells only give off a very small quantity of gas. In total absence of air or in hydrogen gas there is neither multiplication nor fermentation; the phenomenon described above takes place at the expense of the air dissolved in the must. The author proved that the favourable effect of the gelatin is not due to the nitrogen introduced; and, although the yeasts ferment artificial must in the ordinary way, they lose this property if tannin be added. With a yeast of enfeebled vitality the fermentative property is lost sooner than the reproductive property.—J. F. B.

Yeast; Action of Precipitants on Expressed Extract of. R. Albert and E. Buchner. Woch. für Brau. 1900, **17**, [4], 49—51.

Experimenting on the precipitation of an active enzyme from the expressed yeast extract, the authors poured 50 c.c. of extract into 600 c.c. of absolute alcohol, and rapidly filtered off the precipitate produced. This, when washed and dried *in vacuo*, was of a horny nature and only partially soluble in water. When ground up with sand and suspended in a solution of sugar the fermentation produced was equal in extent to that brought about by the original extract, but its commencement was somewhat delayed owing to the time required for re-hydration. Attempts were made to extract the zymase from this substance by digesting with water and with 10 per cent. sugar solution; but the fermentation tests with the filtered extracts indicated a considerable deficiency of zymase owing either to imperfect extraction or destruction. Other experiments were made with mixtures of alcohol and ether instead of alcohol alone for the precipitation. The precipitates so obtained were more easily filtered, and were friable instead of horny, but the fermentative powers of their filtered extracts were very little better than those with alcohol alone. The results obtained by fractionally precipitating the yeast extract with alcohol were not satisfactory as regards fermentative power, but they suggested that the presence of zymase in all these precipitates is not due to the insolubility of the enzyme in the precipitant, but rather to the mechanical inclusion of zymase in the precipitate formed. R. Rapp has used acetone as a precipitant in the proportion of 400 c.c. of acetone to 100 c.c. of extract, but he found that, when the dried precipitate was suspended in a solution of sugar, a considerable decrease of fermentative power had taken place. Methyl alcohol, apparently, brings about the complete destruction of the zymase when used as a precipitant.—J. F. B.

Beer, Turbidity in Trade Casks. Allgem. Zeits. für Bierbrau. **27**, [38], 909—910.

When beer is found to be turbid in the trade casks, the cause is usually to be sought in the presence of young and active yeast cells, newly developed in the beer, old cells due to careless racking from the storage cask, or bacteria—the latter resulting from lack of cleanliness in the brewery. In the first case the beer may have been insufficiently matured, or the pitching yeast has probably been deficient in attenuative power and left too much unfermented extract, which the unavoidable residual living cells, stimulated by aëration in racking, then attack so as to produce haze.

Dead yeast cells giving rise to turbidity, may be drawn off from the storage cask in racking, the more easily if the taps are suddenly opened and shut, the shock causing the sediment to rise; and the same result may ensue if the beer be racked from a bunged cask without the employment of counter-pressure. Even filtration is not always efficient, and therefore samples should be examined for turbidity from time to time during racking.

For bacterial turbidity the sole remedy is a thorough purification of all parts of the brewery, utensils, &c.

—C. S.

Secondary Fermentation [Distillery]; Temperature of. Delbrück. Zeits. Spiritusind. 1900, **23**, [2], 9.

It is quite probable that the usual temperature of secondary fermentation, especially in distilleries provided with cooling worms and in the winter time, is too low. Formerly the mash used to warm up to 35°—36° C. during primary fermentation, which was undoubtedly far too high, but the secondary fermentation generally took place at about 27·5° C., and was quite satisfactory. From some small experiments the author is of opinion that both primary and secondary fermentation should be conducted at 27·5° C., certainly never below 25° C., but the temperatures of secondary fermentation prevailing in practice are more often 22·5°—20° C. It is true that primary fermentation can be carried out at 17·5°, 15°, and even 12·5° C., although it is slow. But then the conditions of nutrition are very different; in the primary fermentation the sugar is concentrated, and there are no inhibitive compounds present, but in the secondary fermentation, although the quantity of yeast is something like seven times as great, the sugar is only at about 2 per cent. concentration; and the presence of alcohol, acids and carbon dioxide, which, owing to sluggishness, is not removed, has a strong retarding effect. Further, this 2 per cent. in the case of potato mashes is mostly dextrin, and dextrin is not fermentable unless diastase be present. There is no secondary fermentation unless there is secondary diastatic action, and all the above conditions retard such action, its maximum temperature being high. The author invites communications from practice as to whether the temperature of 27·5° C. is not the best, and as to how far the temperature naturally sinks in winter if primary fermentation be kept down to 27·5° C., also whether artificial warming during secondary fermentation be permissible.—J. F. B.

Secondary Fermentation, Temperature of. F. Neumann. Zeits. Spiritusind. 1900, **23**, [4], 27.

In reply to the question raised by Delbrück (see preceding abstract), the author remarks that in practice he works vats of 4,433 litres capacity, and uses a mechanical cooling stirrer by Hornung and Schreibner. In warm weather the mash is set up at 17·5° C. and in cold weather at 18·7° C. The first mash is mixed with both yeasts in the preliminary vat and then passed into the fermenting vats, the second mash is added direct without yeast. This operation is completed at 1 p.m. By 4 a.m. next morning the temperature has risen to 25° C., and the froth is at its full height. The stirring apparatus is then started, and the temperature at 7 a.m. has risen to 27·5° C., water is passed through the cooling pipe, so that when the primary fermentation begins to die down at 3 or 4 p.m., the temperature has only risen to 29°—30° C. The cooler is then taken out and an hour later 80 litres of cold water are added per vat, another 80 litres being given at 6 a.m. on the third day. Before this latter addition the temperature is generally about 31° C. and the secondary fermentation proceeds until, at the time of distillation, the temperature has sunk to 28° C. The density of the wash before distillation is from 0·5° to 1° Balling from a sweet mash of 23°—24° Balling. It has sometimes happened that the cooling water has been left running too long, and in such cases the temperature before distillation has been about 25° C., and the density 2° Balling. The maintenance of the temperature during secondary fermentation depends largely on working vats of a sufficient size. The author once had occasion to work large vats and small vats side by side, but the attenuation in the small vats was not satisfactory, owing to a rapid fall of temperature; he thinks that in the case of small vats, heat should be supplied by warming the fermentation room to a suitable temperature.

—J. F. B.

Fermentation; Influence of Oxygen on, and on the Fermentative Energy and Reproductive Power of Various Races of Yeast, under Different Conditions of Nutrition. G. Korff. Allgem. Zeits. für Bierbrau. **27**, [46], 1097—1099; [47], 1123—1124; [48], 1145—1147; [49], 1169—1170.

The following general conclusions are drawn by the author from the researches detailed in the paper:—

Moderate aëration favours the reproductive energy and power of Saaz and Frohberg yeast, but has a deterrent influence on Logos yeast; it also increases the fermentative energy of Saaz and Logos yeast, but reduces that of Frohberg yeast; and favours the fermentative power of Frohberg and Logos yeast whilst being inert towards Saaz yeast. Oxygen increases the reproductive energy in all cases, as well as stimulating the reproductive faculty, though in this latter respect, moderate aëration has more effect on Frohberg yeast. In every instance, fermentative energy and power are diminished by oxygen.

Hydrogen, or total exclusion of oxygen, restricts the reproductive energy of Saaz and Logos yeast, but has no effect on Frohberg yeast; in all cases, however, it reduces the reproductive power. Furthermore, hydrogen reduces the fermentative energy of Saaz and Frohberg yeast, but is without influence on Logos yeast; on the other hand it increases the fermentative power of Frohberg and Logos yeast, but is inert towards Saaz yeast.

These divergent effects are due to the varying requirements of, or susceptibility to, oxygen on the part of the several yeasts mentioned. Neither the admission nor exclusion of oxygen can increase the vital activity of yeast in any one or other direction, the same law holding good for all, that the reproductive energy and power vary inversely as the fermentative energy and power; hence the total working capacity of the cell is a definite quantity, which, however, varies with the kind of yeast. Fermentation can, therefore, no longer be regarded as a purely pathological process.—C. S.

Wort; Phenomena occurring in the Aeration of. C. Bleisch and R. Schweitzer. Zeits. für d. ges. Brauw. **22**, [39], 515—519; [40], 531—537; [41], 547—551.

I.—*Oxygen absorbed Mechanically.*—Working with Paterson's modification of the Schützenberger method, the authors found the total absorption of oxygen, to the point of saturation, by wort to be as follows:—

Temperature.	Oxygen Absorbed per Litre by	
	7·4 °/₀ Ball. Wort.	14·4 °/₀ Ball. Wort.
	C.c.	C.c.
62·5° C.	2·4	2·4
50° C.	3·3	2·8
37·5° C.	3·6	2·9
25° C.	4·0	3·5
12·5° C.	5·0	4·0
5° C.	5·6	4·4

saturation being effected in two minutes.

The comparative influence of air passed respectively through and over the wort was also examined, the amount of oxygen absorbed in the first case being 2·56 c.c., and in the other 2·28 c.c. in two hours, the saturation quantity (2 minutes agitation) being 3·5 c.c. Hence brisk movement is more efficacious than prolonged exposure.

With regard to the effect of the depth of wort in the cooler, it was found that in cooling down from boiling point to 25° C., 2·4 c.c. of oxygen were absorbed per litre when the liquid was 2 c.m. in depth, decreasing to 1·8 c.c. when the depth was increased to 6 c.m., and to 0·8 c.c. with a depth of 23 c.m. In each case the diffusion of the oxygen was uniform throughout the liquid.

Further experiments showed that the oxygen absorbed mechanically, does not enter into combination when the wort is kept at a low temperature, the amounts being practically identical (5·3 and 5·1 c.c) before and after 24 hours' storage at 6°—7° C. It is therefore concluded that the yeast added to the cooled wort works up the mechanically absorbed oxygen direct, without any previous chemical combination taking place. Compared with solutions of saccharose, the oxygen of wort is apparently uninfluenced by the composition of the liquid, and is solely a function of temperature and mechanical movement.

In practice, the use of the cooler does not particularly favour oxygen absorption, agitation of the wort being only admissible whilst the temperature is still high and before sediment has begun to form. The refrigerator is more suitable, provided a good current of (preferably sterilised) air is passed over the apparatus, to prevent the formation of a superficial layer of water vapour on the walls. An equal increase in oxygen absorption is effected during the time the wort is running into the fermenting tuns, and by "rousing" after pitching.

II. *Combined Oxygen.*—Finding that the sealed flask method pursued by Pasteur and Jörgensen in estimating the amount of oxygen entering into combination with wort gives unreliable results, owing to the high pressure in the apparatus, the authors devised an apparatus (illustrated and described in the paper) for carrying out the examination at ordinary pressure; and from the results obtained, they concluded that more oxygen enters into combination with the wort during the time it is being boiled with the hops than during aeration on the cooler; that such combination is greatly facilitated by agitating the wort whilst still hot on the cooler; but that under the usual conditions of working that vessel, only a very small quantity of oxygen enters into combination with the wort. Furthermore, that the shallower the wort on the cooler, the more oxygen is absorbed and combined, immediate stirring and fanning being advisable for increasing the effect when the layer of wort is deep.

Concerning the part played by the various constituents of wort in combining with oxygen, it is concluded, from investigations made with pure solutions and referred to the average composition of wort, that maltose combines with 71·3 per cent. of the total oxygen fixed, dextrose and levulose with 14·5 per cent., and the other substances with the remaining 14·2 per cent., assuming that the constituents behave in the same manner in wort as they do when alone. To confirm this action of maltose, a 14 per cent. solution of this body was kept at 85° C. for 5—6 days, with occasional shaking. The colour changed to dark brownish-red and the liquid gave a strongly acid reaction. On neutralisation with ammonia and treatment with acetic acid and lead acetate, a small brown precipitate, soluble in sodium hydroxide, was formed, though none could be obtained from the original solution. On distilling the oxidised liquid with steam, traces of furfural were detected, but no maltol.

All wort constituents capable of oxidation are darkened in colour by the change, hop extract in particular; and an unwelcome red tinge is liable to be produced when the boiling of the wort is unduly prolonged, or the goods are kept too long at high temperatures.

III. *Influence of Oxygen on the Formation of Cooler Sludge.*—Experiments having shown that the separation of sedimental matter from the wort proceeds equally well in an atmosphere of hydrogen as in air, it is concluded that neither the absorbed nor the combined oxygen in the wort has any effect on this deposition, which must therefore be regarded as solely a physical phenomenon.

IV. *Influence of Absorbed and Combined Oxygen on Fermentation.*—The amount of oxygen consumed by yeast during the first 24 hours of fermentation, varies according to the nature of the yeast, one variety examined having reduced the oxygen content of the wort from 4·3 c.c. per litre to 0·58 c.c., whilst another only consumed 1·1 c.c. in the same period. In point of attenuation, the experiments detailed go to show that there is little difference between hot and cold rousing previous to fermentation, but that aeration during that process increases the attenuation. The head was found to be darker in colour, less compact, and lower in the case of hot-aerated worts. The (low fermentation) yeast, however, breaks better, and forms a more compact and whiter sediment when the wort has been aerated cold. No special influence on attenuation could be traced to the combined oxygen. Though in primary fermentation, abnormal phenomena, due to improper aeration, may be more or less compensated in secondary fermentation, it is probable that prolonged aeration of the wort, whilst still hot, may result in damage to the flavour and power of retaining head, owing to the susceptibility of the albuminoid matters to oxidation at high temperatures. In open coolers there is less risk of over-aeration of the wort at the hot stage (when oxidation occurs) than in closed coolers, and an efficient draught in the latter is essential at this stage to remove volatile products. Prolonged aeration of the wort for use in pure-culture yeast apparatus should be avoided, the best course being to draw off the wort when cooled to 50°—60° C. and re-boil in the steriliser without further aeration. The wort should be well roused in the cold before pitching, but not during fermentation in the propagating cylinder, except in so far as the operation of removing the supernatant carbon dioxide by a current of air affords a certain amount of surface aeration.—C. S.

Attenuation; Influence of the Boiling and the Hopping of the Wort upon the. Dworsky. Allgem. Zeits. f. Bierbrauerei und Malzfabrikation, 1899, 235.

The author tested (1) wort from the clearing bottom, unboiled and unhopped, (2) the same, boiled but not hopped, (3) super-hopped wort. Wort No. 1 attenuated 53·6—55·0 per cent.; worts No. 2 and No. 3 attenuated equally 64·5—70·0 per cent.; differences occurred in the primary attenuation of the two worts, but these were equalised after four weeks' cellarage, so that the hops had no effect on the final attenuation. The samples differed widely in appearance and taste; the unboiled, unhopped wort cleared slowly and incompletely, whilst that boiled and hopped cleared best. The beer from No. 1 had an insipid taste, not at all like beer; that from No. 2 tasted of malt or bread, not unpleasant, something like malt wine; the beer from No. 3, the super-hopped wort, was unpleasant, and tasted strongly of hops. The retention of foam by the first two samples was good, but the foam of the strongly hopped beer was deficient, and consisted of large bubbles. No. 1 contained vigorous large yeast cells, greatly contaminated by foreign organisms; No. 2 contained good yeast, not so impure as No. 1; whilst the yeast cells from No. 3 had thick walls, and many small cells were present.

—J. F. B.

Pitch [Brewer's], Clear and Cloudy, for Pitching Casks. Gambrinus. Allgem. Anzeig. für Brau. **15**, [38], 1468—1469.

Although clear colophony toughened with rosin oil is the best material for pitching casks, being free from volatile and ill-flavoured impurities, an erroneous opinion—founded on the appearance of the natural resin formerly used—is prevalent that cloudiness is an indication of quality, and this has led to the addition, by manufacturers, of such bodies as iron oxide, heavy spar, lead chromate, &c., all of which are injurious to the beer. Even such chemically inert adjuncts as ground bark are not free from objection, since the rough surface of such pitch affords shelter to yeast cells, and increases the difficulty of cleaning the casks, to say nothing of the irregularity of the covering layer in consequence of the diminished fluidity of such adulterated pitch. The alleged advantage of cloudy pitch that it enables dirty patches to be more readily detected is illusory unless the pitch be very nearly black, so that in reality there is no good reason for the preference, and it is better to employ the pure clear preparation.—C. S.

Amylomyces Rouxii and Amylo-Fermentation Processes. A. Marbach. Oesterr. Chem. Zeit. 1899, **2**, 178—181; through Zeits. Untersuch. Nahr. Genussm. 1900, **1**, 50—51.

This mould fungus was first isolated by Calmette in 1892 from Chinese yeast. An ancient method of preparing spirit from rice is in use in the East Indies and China, in which this fungus plays the several parts of saccharifying ferment and fermentation exciter simultaneously. The alcohol obtained from 100 kilos. of rice amounts to 18 litres, barely one-third of the possible yield.

Amylomyces was first used industrially at Seclin, near Lille. From 300 hectolitres of brewery "settlings" (schlempe) Boidin and Collette obtained 270 litres of alcohol. It was found better to add the *amylomyces* first and allow it to act, and then afterwards to add the yeast. The actual method of working was as follows:—The fermentation vats used were similar to those employed for the preparation of pure yeast cultures, and of large size. The mash was sterilised by heating to a pressure of two atmospheres. It was then cooled to 38° C. (the most suitable temperature for the development of the fungus) and the *amylomyces* added. The stirring apparatus was now started and the ventilation carefully regulated. After about 20 hours, when a microscopical examination shewed that the mycelium of the fungus had spread throughout the whole mass, the mash was cooled to 33° C., the air supply cut off, and a few cubic centimetres of a pure yeast culture added.

After a further 96 hours the fermentation was completed.

The tables given in the original paper show that after the action of the *amylomyces,* some starch is still in suspension and in solution; that the saccharification and fermentation can proceed simultaneously; and that finally all the starch is converted into sugar and fermented. 97·5 per cent. of the theoretical yield of alcohol was obtained.
—W. P. S.

Rum Factory in Martinique; Chemical Control of a. Saussine. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1899, **17**, [5], 367—370.

As in Guadeloupe, several kinds of rum are made, according as raw juice, boiled juice or sugar works molasses are used. The rich syrups from the centrifugals of country sugar works using the old-fashioned Wetzel evaporation apparatus are not to be compared to the molasses of central factories, and give a rum of excellent quality which is almost entirely exported.

Rum from raw juice is intended for immediate consumption and is known locally as *tafia grappe blanche.* It is considered difficult to keep. Rum intended for exportation is made from juice sterilised by concentration in a Père Labat apparatus to 10°, 20° or 40° B. which is then known as battery syrup.

In order to keep down the temperature and preserve the natural bouquet, it is of advantage to use a number of small fermentation vats so as to increase the surface exposed, the usual size being 2 to 3,000 litres capacity.

According to the richness of the syrup, from 10 to 12 per cent. is taken and diluted with wash and water. Some distilleries use no spent-wash, but the larger ones add from 55 to 65 per cent. The glucose is usually from one-third to half the crystallisable sugar in the molasses.

Starting with a density of 7° B., a complete attenuation may be reached. The larger distilleries start at 9° to 10° B. and the fermentations seem to stop at 4° or 5° (1·030), the resulting wash being 1·040 at 28° C. The density in a good fermentation begins to fall the second day, and is complete in five to six days. The average temperature on the island is 27° C. on the west coast. Near the sea, fermentation is sometimes carried on at 40° C., but on the higher levels, below 30° C. With an initial temperature of 33° or 34° C. there is first an increase of 4° to 5° the first few days, then a slower fall, but with an initial temperature of 38° C. there is at first a fall and then a rise of temperature. In bad fermentations the temperature always falls. The acidity is attributable to a fixed or only slightly volatile acid in the wash, which is transferred from one operation to another; during the fermentation there is very slight increase of acidity. Almost all rum distilleries use small quantities of sulphuric acid, 0·02 grm. per litre. In good fermentations the acidity (as H_2SO_4) varies from 4 to 4·5 at the start, to 6 or 6·5 at the finish, for the wort; 7·5 for the wash, and 0·5 for the tafia.

The wash, set to cool, forms a deposit useful as manure. The excess, not required for diluting fresh molasses, is run into the sea. It has been found to contain 15 grms. of nitrogen per litre. It reduces Fehling solution strongly, for it contains aldehydes. Furfuraldehyde is easily recognised. On distilling for some time at 100° C., a liquid slightly heavier than water is obtained, possessing the odour of ordinary tafia. It appears therefore to contain a reserve of aromatic principles, only slightly volatile, and of which the alcoholic vapours carry off a small quantity on traversing the trays of the column.

Statistics dating from 1818 give the following definitions: —The first spirit coming over in the distillation is rum, and stands at 25° (Cartier). Tafia follows; it is sold at 28° (Cartier). The rest is weak spirit, and is rectified for spirit. The rum distiller has done good work when he has obtained a gallon of tafia from a gallon of syrup.—L. J. de W.

Fermentation Carbon Dioxide. E. Luhmann. Zeits. für d. ges. Kohlensäure-Ind.; Allgem. Anzeig. für Brau. **15**, [47], 1828—1829.

In the author's opinion, the recovery of fermentation carbon dioxide in breweries will not pay wherever there are other cheap sources of this gas, owing to the large capital expenditure entailed in fitting up plant, and to the difficulty in purifying the gas. In the case of distilleries, the intermittent character of the process would entail too great an outlay for storage cylinders, to be profitable.—C. S.

Wine; Detection and Estimation of Aldehydes in. M. Ripper. Chem. Centr. 1900, **1**, [7], 436.

See under XXIII., *page* 280.

PATENTS.

Yeast; Treatment of. R. Rückforth, Stettin, Germany. Eng. Pat. 4709, March 3, 1899.

The process consists in first congealing the yeast, breaking up the mass so formed, and bursting the cells by rapidly heating to a temperature of about 15° C., finally extracting the proteïns by pressure or lixiviation. The burst cells may be subjected to the action of proteolytic ferments before extraction, if desired.

The congelation of the yeast is preferably brought about by exposing the washed yeast to a temperature of −12° to −15° C. The bursting of the cells is then effected by heating the crushed mass to 15° C. by means of warm water or else by passing it between heated cylinders.—J. F. B.

Racking Beer; Apparatus for. H. W. Colby, Chicago, U.S.A. Eng. Pat. 9221, May 2, 1899.

The pressure in the barrel is maintained at an equilibrium with that in the tank by an open pipe from the upper part of the barrel to the upper part of the tank; the liquid is caused to flow by gravity from the tank at a higher level, and the air displaced by the liquid rises through the air-pipe into the tank. To allow of the complete filling of the barrel without waste or overflow, the air-pipe is provided with a closed receptacle which will contain a quantity of liquid approximately equal to that displaced by the filling tube inserted in the barrel, this liquid enters the barrel when the tube is withdrawn.

The valve in the air pipe is automatically opened before the valve at the bottom of the filling tube is opened.

The claims, 36 in number, enumerate the various combinations of devices which may be used in the apparatus to secure the fulfilment of the above conditions.—J. F. B.

Producing Wine by previously Sterilising the Must of Grapes. E. P. y Mendivil, Saragossa, Spain. Eng. Pat. 11,876, June 7, 1899.

The process consists of previously sterilising the must of grapes by purely physical means without in any way altering its chemical composition, as, for example, by evaporating the must *in vacuo* at a temperature not exceeding 65° C., restoring the moisture lost in this operation, and treating the must with yeast specially selected, and possessing qualities suited to the nature of the must and the taste of the consumer.—J. F. B.

XVIII.—FOODS; SANITATION; WATER PURIFICATION, & DISINFECTANTS.

(*A.*)—FOODS.

Chinese Tea, and Certain of its Substitutes. E. Collin. Pharm. Chim. **11**, [1], 15—21; and [2], 52—59.

The leaves of many plants of diverse geographical origin have been used as adulterants of tea. Those which resemble the Chinese leaf in shape, odour, and astringency, and possess dentate margins, such as *Fraxinus excelsior, Sambucus nigra, Spiræa salicifolia,* and *Trigonella cœrulea* were formerly employed. When these leaves, cut in small pieces, are treated with a solution of caffeine, they so far resemble the genuine article that sophistication has often been undetected. Since, however, the histology of Chinese tea has been better known and microscopic examination more frequently employed, leaves which possess similar anatomical elements, such as those of *Camellia japonica, Phyllirea angustifolia,* and *Olea europæa* have been more generally made use of. The Chinese have also adapted themselves to meet the advance of expert knowledge; a spurious tea was exported some years since in considerable quantity under the name of "Canton made Tea" or "Imperial Tea," which Riche found to contain no caffeine, and the author showed to be composed of a leaf, other than that of *Camellia thea,* the botanical source of which is undetermined. Under the name of "Kaporie tea," the leaves of *Epilobium angustifolium* are largely employed in Russia as a substitute for tea, a considerable trade being done in the product. The leaves of *Epilobium hirsutum* are also employed for the same purpose. In the same country, a fictitious tea is also made from the leaves of *Vaccinium arctostaphylos,* and occasionally from those of *V. myrtillus,* which, under the name of "Caucasus tea" or "Thé du Dutais," has a considerable consumption. Figures of microscopical sections of the leaves, and a minute account of the distinctive histological elements of Chinese tea, and of the two last named substitutes accompany the communication.—J. O. B.

Margarine; Detection and Estimation of Cane Sugar in. Mecke. Chem. Centr. 1900, **1**, [7], 435.

See under XXIII., *page* 280.

PATENTS.

Bread; Making of, with Unground Corn. P. Rakhmanoff, Moscow, Russia. Eng. Pat. 7142, April 5, 1899.

The bread is made with corn, which has been peeled or had its exterior coat removed by treating it in a rotating drum with a solution containing 7 grms. of soda per litre. The corn is stirred in and discharged from the drum by a worm. After leaving the drum, the grains pass through brushes and a washing tank. An acid fermented liquid called "kvass" is added to the prepared corn, and allowed to act for 12 hours at a temperature of 100° to 110° F. The liquid is then run off, and the soaked grains ground up and made into loaves, &c.

The "kvass" is made by mashing fresh soaked corn with green malt, and then heating to a temperature of 154° to 163° F. The resulting sweet mass is left to cool and to enter into acid fermentation. Water is now stirred in and pressed yeast added. The final liquid thus obtained is used as above.—W. P. S.

Sterilising Liquids; Apparatus for. J. Y. Johnson, London. From Compagnie Générale pour la Conservation des Liquides, Paris. Eng. Pat. 23,828, Nov. 29, 1899.

The liquid (beer, wine, milk, &c.) to be sterilised is heated in a rotating cylinder, which is in valvular communication with another circular rotating vessel. Both vessels are provided with steam heating pipes and steam jackets. The second cylinder receives the liquid which is forced out of the main vessel on a pre-determined pressure being attained. This second cylinder is also previously filled with sterilised air, or carbonic acid gas under pressure.—W. P. S.

Heating and Pasteurising Liquids; Apparatus for. E. Edwards, London. From L. C. Nielsen and P. V. F. Petersen, Copenhagen, Denmark. Eng. Pat. 24,304, Dec. 6, 1899.

The apparatus claimed is one for sterilising liquids, especially milk and cream. It consists of a cylindrical metal vessel surrounded by a steam jacket. On the outside of the vessel, but in the steam chamber, are fixed "drip rings," or flanges, to carry off condensed water from the surface of the vessel. A stirrer is provided, having an arrangement of plates across its axis, and extending almost to the side of the vessel, serving to separate the milk or other fluid into layers. Any air liberated, escapes through holes at the centre of these plates. The liquid to be sterilised continuously enters at the bottom and is removed at the top of the cylinder. The waste pipe at the bottom of the steam chamber is bent so as to form a drain-trap, in order to separate the outside air from the chamber. A vent-cock on this waste pipe or in the bottom of the steam chamber allows any air which may be disengaged by the steam to escape.—W. P. S.

(*B.*)—SANITATION; WATER PURIFICATION.

Effluent Water from Sugar Factories; Proskowetz's Method for the Purification of. von Rosnowski and Proskauer. Vrtljschr. f. ger. Med. u. öffentl. Sanitätswesen, **16**, Suppl. Heft. 54—98. Chem. Centr. 1900, **1**, [2], 135—137.

According to this process, the effluent waters are treated with milk of lime, and, after settling in clarification reservoirs, where they gradually lose their alkaline condition, they are conducted on to a drainage area, the draining tubes of which are about half a metre below the surface, and lead into the open, so that there is free access of air. The water is then led on to a second drainage area, which is doubly drained at depths of about 1 metre and 1·5—2 metres. The water coming from the first drainage area is already neutral, or even faintly acid, and should, in the second area, undergo a similar alteration, such that the constituents previously not precipitated by lime, have now become precipitable. A further quantity of lime (lime water) is therefore added at this stage, and, after allowing the precipitate produced to settle, the purified water can be led back again to the works.

At a sugar factory in Sadowa, Bohemia, the first addition of milk of lime is dispensed with, and the effluent water, after settling in a clarification reservoir and in a cooling pond, passes on to the two drainage areas successively, and then, after a strong addition of lime, finally on to a small drainage field. It was found that 96·7 per cent. of the nitrogenous organic matter was removed from the water, and that its oxygen absorption power was decreased by about 92·3 per cent.

The purification began in the clarification reservoir and in the cooling pond, being effected partly by settling and partly by processes of putrefaction and fermentation, for which the composition of the effluent water and the temperature (36·5°—26° C.) are very favourable. In the first drainage field, which, according to Proskowetz, should act as an "oxidation field," as also in the second drainage field, the destruction of the organic matter only proceeded by putrefaction. The water from the second drainage area contains the organic matter in a condition such that it can be removed by addition of lime. At Sadowa, much too large a quantity of lime is used, notwithstanding which, the purified water is clear and colourless, possessing, however, on account of the strong addition of lime, and the defective condition of the final small drainage field, a faintly alkaline reaction, and still smelling somewhat of ammonia. On standing in undiluted condition, and after dilution with varying amounts of river water, no occurrence of fetid putrefaction could be observed. The authors do not hesitate to introduce into public water-courses, water of the quality exhibited by the samples of the purified effluent waters referred to.

The process, as carried out at Sokolnitz, Moravia, approximates more closely to Proskowetz's directions, and the results obtained are equally satisfactory. The organic matter remaining in the purified water is chiefly in the form of soluble fatty acids.—A. S.

Effluent Water from Sugar Factories; Proskowetz's Method for the Purification of. M. Hönig. Oesterr.-ung. Zeits. Zucker-Ind.-u. Landw. **28**, 630—642; Chem. Centr. 1900, **1**, [2], 149.

The author has observed Proskowetz's method for the purification of effluent waters from sugar factories (see preceding abstract), during a whole campaign at Sokolnitz, and has examined samples in different phases of the process. His results agree in the main with those of von Rosnowski and Proskauer. The normal course of the purification process is not influenced to the smallest extent by the alkalinity of the effluent water before it passes to the drainage beds, but, in order to obtain good and quick settling in the clarification reservoirs, the water flowing from the latter should possess a degree of alkalinity corresponding to 60 mgrms. of CaO per litre. Only a portion, and indeed the smaller portion of the effluent waters from a sugar factory (cutting-presses, diffusion, bone-black washing, &c.), need so thorough a purification, the remainder can be purified sufficiently by allowing to settle. At Sokolnitz, where the total water amounts to 6,000 cb. m. daily, 600 cb. m. are purified by Proskowetz's process, and the total drainage surface, which is unusually large, is 3·75 hectares.—A. S.

Water; Detection of Nitrites in Potable, by Means of 1.1′.4.3′-Amidonaphthol Disulphonic Acid. H. Erdmann. Zeits. f. angew. Chem. 1900, [2], 33.

See under XXIII., *page* 277.

PATENTS.

Sewage and other Liquids to and from Filter Beds; Apparatus for the Automatic Delivery and Discharge of. E. Brown, Sussex. Eng. Pat. 941, Jan. 16, 1899.

The apparatus comprises large float vessels in chambers communicating with the filter-beds, mechanically connected with valves for admitting the sewage to the filter-beds, and for discharging the same therefrom, and self-acting means for allowing the float devices to actuate the valves, when the liquid has arrived at a predetermined level in the several filter-beds.—L. A.

Sewage and other Polluted Waters; Apparatus for Use in the Purification of. H. Tulloch, London. Eng. Pat. 1641, Jan. 24, 1899.

Apparatus for the anaerobic treatment of sewage consists of a tank, the lower part of which is a chamber, of which the arched perforated roof supports a bacteria bed divided into sections by vertical partitions. The tank is closed by a moveable cover rendered air-tight by a water seal. The sewage flows down a central channel into the lower chamber, and rises through the bacteria bed. A central pipe with horizontal swivel arm which travels round the floor of the chamber, enables the deposit to be removed while the treatment is going on. The same pipe serves for the admission of air, steam, gases, or cultivations of bacteria. Means are provided for ensuring the equal flow of the sewage through all the different sections of the bed.

A similarly constructed tank is used for aerobic treatment, the effluent in this case being sprinkled on the top of the bacteria bed and filtering downwards into the chamber below, to which air is supplied by special means.

—L. A.

Purification of Sewage and other Waters; Supplying Air to Filters for the. W. M. Ducat, London. Eng. Pat. 2373, Feb. 2, 1899.

It is necessary that the large volumes of air supplied to aerobic bacteria beds in winter be warmed sufficiently to maintain the temperature above 37° F. This may be done by external heat; but it is now proposed to utilise the heat generated by bacterial action in the filter bed itself, and carried away in the effluent. Thus the fresh air supplied to the filter may be drawn through thin metal pipes laid in the effluent channel.—L. A.

Sewage; Purifying. D. Cameron, F. J. Commin, and A. J. Martin, Exeter. Eng. Pat. 2461, Feb. 3, 1899.

Air is introduced into sewage or sewage effluent by causing the latter to pass through a chamber down which water is showered, the minute air-bubbles which are carried down into the sewage by the falling water being trapped by coke or similar rough material which is spread on the floor of the chamber or carried on trays at a higher level. Or, the sewage may be caused to flow through perforated walls, in the holes of which the air becomes engaged, the water being showered into the spaces between the walls. The water thus showered into the sewage may consist of sewage effluent already wholly or partially purified, which will carry with it the bacteria necessary for the process of purification. Thus, part of the sewage of a town may be intercepted and purified at a higher level, and the purified effluent showered into the main body of sewage at a lower level. Improvements are also described in the design of channels for distributing sewage over the surface of a filter bed; also for the collection of the effluent from the bottom of the bed. The invention also describes an arrangement of valves for bringing a second set of filters into use automatically, when those previously in use become unable to discharge their contents through the rise of tidal water or otherwise.—L. A.

Sewage and Trade Effluents; Method of and Means for Treating. A. Pickles, Wakefield. Eng. Pat. 5293, Mar. 10, 1899.

The apparatus consists of seven series of seven tanks placed vertically one over the other. Each tank is divided into four chambers, and each chamber consists of an upper compartment containing filtering material and a lower empty compartment. The different tanks and chambers of each series are connected by pipes in such a way that the sewage which is delivered into the lower compartment of the end chamber of the highest tank passes up through the filter bed and overflows into the lower compartment of the next chamber, and so on from chamber to chamber and tank to tank until it reaches the bottom and flows away in a purified condition. The seven series of tanks are for use in rotation on the seven days of the week. The sewage before treatment is screened, and "the solids are dried and burnt to provide the steam and power required in the process."

—L. A.

Sewage; Treatment of. T. J. Barnard, Truro. Eng. Pat. 20,759, Oct. 17, 1899.

Sewage is filtered through coal-slack, coke, and charcoal, then upwardly through slaked lime, and again filtered. The filtering material is ultimately mixed with tar and made into fuel.—L. A.

Water; Purification and Softening of. A. J. and P. A. G. Bell, Manchester. Eng. Pat. 5423, March 13, 1899.

The apparatus described consists of a combination of suitable tanks for containing chemical solutions, connected with a measuring cylinder arranged so as to work in ratio with a force pump, by which the chemical solution is mixed with water in proper proportions; a closed settling tank, into which the said mixture is forced; one or more mechanical filters in connection therewith; and an outlet pipe, with a bye-pass regulated by suitable valves, for containing a neutralising chemical and mixing it with the effluent water.—L. A.

Water or other Liquids; Apparatus for Purifying. L. Gathmann, Washington, Columbia. Eng. Pat. 21,451, Oct. 27, 1899.

Suspended matter is first removed by centrifugal action. The water is then sterilised by an electric current, and is finally subjected again to centrifugal treatment to remove suspended matter which has been formed by the electrical treatment or has escaped the preliminary process.—L. A.

Water; Purification and Filtration of, and Apparatus therefor. A. Schautz, Dayton, Montgomery, Ohio. Eng. Pat. 25,085, Dec. 18, 1899.

In order to soften water for brewing, boilers, medicinal, and domestic uses, it is allowed to fall over heated pipes on to a bed of heated boulders, over which it is made to flow, and is then agitated by jets of steam, and filtered through gravel.—L. A.

(*C.*)—DISINFECTANTS.

PATENTS.

Antiseptic Compounds. B. J. B. Mills, London. From Stafford Chemical Company, New York. Eng. Pat. 2277, Feb. 1, 1899.

80 lb. of glucose are dissolved in 4 galls. of water, and to the solution, 44½ lb. of borax are added, and heated until dissolved. To the hot solution, 40 fluid oz. of hydrofluoric acid of about 56° B. are gradually added, and then 96 oz. of benzoic acid, and the whole is stirred until solution is effected. The solution is allowed to cool, and made up to 18 galls. by addition of water; 0·5 per cent. of formaldehyde is finally added after the solution has been clarified by settling. The solution is stated to be a germicide.—L. A.

Dipping Sheep [SheepDip] and other Purposes; Compound for. Odam's Manure and Chemical Co., Ltd., and H. E. Macadam, London. Eng. Pat. 9400, May 4, 1899.

This is a solution of calcium polysulphides, made by boiling sulphur and lime with water, and decanting the clear solution from the residue.—L. A.

XIX.—PAPER, PASTEBOARD, Etc.

Paper; Influence of Folding and Printing on the Strength of. W. Herzberg. Mitt. königl. tech. Versuchsanst, zu Berlin. 1899, 269—284.

A series of experiments was made in the Royal Berlin Technical Experimental Station with the view of studying the effect of various forms of mechanical treatment on different papers.

Folding.—The apparatus used was that of Kirchner, which consists of a heavy (7·5 kilo.) iron roller (150 mm. diameter), provided with a central flange fitting exactly into a grooved rail, along which the roller runs. A strip of the sample paper, 15 mm. broad, likewise exactly fitting the groove, is folded back upon itself, laid in the groove, and the roller run over it first in one direction and then in the other. After removal, the paper is tested for tensile strength in the usual way, the expansion being at the same time noted.

The difference between these numbers and those obtained with the original unfolded sample expressed in per cent. of the latter gives the "loss in breaking strength" and "loss in stretching power" of the paper on folding, respectively. Some 87 normal papers (this Journal, 1898, 263) were examined, and it was found that (1) the loss in stretching power was greater, both in the machine direction and at right angles to it, than the loss in breaking strength; (2) the loss in breaking strength in the machine direction is greater than at right angles to it; (3) and the loss in stretching power in most cases obeys the same law. The results are tabulated at length in the original. The lowest mean loss in breaking strength observed was 4·7 per cent. and the greatest was 48·5 per cent., and the lowest loss in stretching power was 14·3 and the greatest was 78·7 per cent. (see also this Journal, 1895, 881—882).

Printing.—It was found that printing exerts no influence on the breaking strength or stretching power of paper except with newspaper, which shows, after printing, a slight decrease in the former, although the latter property remains unaltered.—H. I.

Wood Pulp; Electrolytic Bleaching of. A. Navarre. Ind. Électrochim. 1890, **3**, 49—51.

In order to avoid the dynamos of special design, and also to prevent corrosion of electrodes, Corbin has adopted, at Lancey, the following modification of the Hermite process. The trough contains two electrodes at the ends, and 11 intermediate insulated electrodes, all fixed in frames, fitting in tightly. The plates are all of platinum, and the intermediate plates serve as double electrodes. The connecting cables are fixed outside the trough, and by varying the number of intermediate plates, any tension may be applied. At Lancey, the 13 plates receive currents of 150 ampères at 120 volts. The electrolyte is sea-salt solution, and the process is continuous. Each cell can bleach 750 kilos. of bisulphite pulp to extra whiteness.—A. S.

PATENTS.

Decolorising Paper and Textile Fabrics, after immersion in Cuprammonium Solution. J. Williams, London. Eng. Pat. 1358, Jan. 19, 1899.

See under V., *page* 239.

Vegetable Fibres for Papermaking, Textile, and other Purposes; Preparing. B. S. Summers, Chicago, U.S.A. Eng. Pat. 6346, March 23, 1899.

The claims, 12 in number, relate to the combination of a pair of digesters or vats, connected together, with means for producing and maintaining a difference of gaseous pressure inside them so as to force the liquid from one digester to the other. The digesters are provided with movable covers, and the difference of pressure may be produced either by excessive pressure or vacuum in one or other of the digesters. A heater is also provided in each of the digesters for heating the contents of the same or for producing a steam pressure for forcing the liquid from one to the other. The necessary combination of pipes and valves for fulfilling the above conditions is claimed. Since the fittings, &c., of the digesters are such that they can be used either separately or together, the fibres may be repeatedly treated with the successive solutions necessary, with the greatest economy of liquid and without handling.—J. F. B.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

Pure Yttria; New Process for the Separation of the Gadolinite Earths, and the Preparation of. W. Muthmann and R. Böhm. Ber. **33**, [1], 42—49.

When potassium chromate solution is added to a neutral solution of salts of the gadolinite or cerite earths, precipitates are obtained of the general formula—

$$R_2(CrO_4)_3 + n\ H_2O,$$

n being in most cases = 8. By fractional precipitation with K_2CrO_4 solution, using the readily soluble bichromates of the earths, the authors have devised an expeditious method of separating the latter. The conditions necessary for success are (1) both solutions must be dilute; (2) the liquid must be kept boiling violently during the precipitation; and (3) the precipitate must be as fine as possible and brought into most intimate contact with the liquid. 40 grms. of "Yttrium oxydat. pur." of commerce (which was found to contain 12 to 15 per cent. of Er_2O_3 and small quantities of Nd_2O_3, Pr_2O_3, Sm_2O_3, Ce_2O_3, &c.) were triturated with about 90 grms. of chromium trioxide, and added to 1 litre of water, whereupon the earths dissolved as bichromates. K_2CrO_4 solution was added till turbidity set in, when the liquid was brought almost to the boiling point and a strong current of steam was led through. K_2CrO_4 solution was now added slowly drop by drop, so that each drop was at once distributed throughout the violently agitated liquid. Fraction 1 was filtered off after 250 c.c. of 10 per cent. K_2CrO_4 solution had been added (time taken, 2 hours): it contained about 25 per cent. of erbia. The filtrate was evaporated to the original volume, 1 litre, as was done in each case, to ensure uniform conditions, and a second fractionation was made in the same way as above, the earths from fraction 2 containing about 20 per cent. of erbia. A further quantity of this fraction was precipitated during the evaporation of the filtrate. Fraction 3, obtained with 300 c.c. of 5 per cent. K_2CrO_4 solution, contained about 10 per cent. of erbia. It consisted of a mixture of orange prisms and yellow needles. Fraction 4, obtained in the same way as fraction 3, also consisted of a mixture of prisms and needles: on mechanically separating them, the orange salt was found to contain 32·5 per cent., the yellow needles, 40 per cent. of oxides. Fraction 5, obtained in the same way, as well as the further precipitate obtained during the evaporation, consisted of beautiful orange-coloured prisms of almost pure yttrium chromate. Fraction 6, obtained by adding 250 c.c. of 10 per cent. K_2CrO_4 solution, was uniform, and yielded pure yttria. The solution remaining contained the impurities other than rare earths. Fractions 5 and 6 were purified by repeated precipitation as oxalates, and the atomic weights were determined by weighing as sulphates and then as oxides, the results being 90·12 and 88·97 (O = 16) respectively, the latter corresponding with Cleve's figure, 89·02, for pure yttria.

For ascertaining the progress of the fractionation, the authors use the specific gravity determination, from which useful data can be had, as the specific gravity of yttria is about 5, that of erbia 8·8, and that of ytterbia 9·2. The following shows the observations during the above fractionations :—

Fraction.	Weight.	Density.	Volume.
	Grms.		C.c.
1	1·1	6·06	3·41
2	19·6		
3	7·4	5·62	1·31
4	7·3	5·43	1·34
5	5 7	4·87	1·28
6	1·5	4·83	
	42·6		7·34
Raw material ...	41·29	5·62	7·35

From these results the authors conclude that there was present some quantity of a colourless earth with a specific gravity between those of yttria and erbia.—H. B.

Medicinal Plants of the Dutch East Indies.—G. Planchon. J. Pharm. Chim., **11**, [2], 49—51.

The following notes are abstracted by the author from a series of communications by Boorsma to the "Mededeelingen uit's Lands plantentuin," published in Batavia, forming a record of the researches conducted in connection with the State Botanical Garden at Buitenzorg.

Plants containing alkaloids. Popovia piscocarpa, Endl., N. O. Anonaceæ contains a base of slight activity. *Cyclea peltata,* H. F. and Th., N. O. Menispermaceæ yields cycleine, analogous to beberine or buxine, found in the roots of *Chondrodendron tomentosum* R. and P. *Ancistrocladus vahlia* Arn., N. O. Dipterocarpeæ contains a feeble base, both in the leaves and the bark. *Lunasia costulata* Miq., closely resembling, if not identical with, *L. amara.* Clum. (*Rabelaisia philippinensis* Planch.), from which the Negritos of Luzon prepare an arrow poison, has yielded Boorsma an alkaloid acting as a heart poison; Plugge has isolated a glucoside, rabelaisin, from the same source. *Chloroxylon swietina* D. C., N. O. Meliaceæ, contains the alkaloid chloroxyline. *Enchresta horsfieldii,* Bensl., N. O. Leguminosæ, contains a toxic alkaloid, which, according to Plugge, is cytisine; the seeds are reputed to possess remedial properties for respiratory diseases. Among the Loganiaceæ, *Spigelia anthelmia* L. gives an active poison spigeline, while *Strychnos tieuté* Lesch. contains strychnine unaccompanied by brucine. *Haasia Squarrosa,* Z. and M., N. O. Lauraceæ contains a heart poison in the bark and leaves. *Justicia adathoda* L., N. O. Acanthaceæ contains vasiarine, and *J. gendarussa* a bitter alkaloid. *Phalænopsis amabilis* Lindl., N. O. Orchidaceæ, yields an alkaloid fatal to frogs.

Plants containing Glucosides.—Curanga amara Juss., N. O. Scrophulariaceæ, contains curangin. *Rhododendron javanicum,* Reims, and *Pernettya repens,* Zoll., both give andromedotoxin. Saponin is found in *Polygala venenosa;* in several Araliaceæ; in *Duranta plumieri,* Jacq., N. O. Vebenaceæ; in *Ficus hypogaea,* N. O. Moraceæ, and in *Sloanea javanica* Miq., N. O. Miliaceæ.

Plants containing undetermined Poisonous Principles.— Gynartocarpus venenosa, Boerl., N. O. Moraceæ, yields a milky juice which is a violent poison. The lily, *Gloriosa superba* L., gives an amorphous bitter powder, which kills guinea pigs; and *Kickxia arborea,* Blum., an albuminoid which kills earthworms, and is also used medicinally as a vermifuge.

Plants containing Toxic Acids.—In the family Meliaceæ some species contain poisonous acids, which are similar in character; for example, the fruits and bark of *Dysoxylon acutangulum,* Miq., *D. alliaceum* Bl., *D. amooroïdes* Miq., *var. otophora* K. and V., and *M. caulostachylum* Miq.

The following also contain poisonous acids :—*Chisocheson divergens* Bl., *Lausium domesticum,* Jack., and *Heynea sumatrana* Miq.—J. O. B.

Ricinine; Preliminary Communication upon. T. Evans. J. Amer. Chem. Soc. 1900, **22,** [1], 39—46.

Extraction of Ricinine from Castor Seed Cake.—Tuson isolated from castor beans by extraction with boiling water and subsequent extraction of the residue from the aqueous extract with alcohol, a substance to which he gave the name of *Ricinine* (J. Chem. Soc. **17,** 195). Starting from castor press-cake as his raw material, the author has extracted by both Tuson's and Schulze's method (Ber. **30,** 2197) a product identical in each case, and thus identifying Schulze's *Ricidine* with Tuson's *Ricinine.* It was found advantageous to substitute boiling toluene for alcohol in Tuson's method. On suddenly cooling the toluene extract, the ricinine was deposited in small prismatic crystals. When pure it melted at 193° C., and had a formula approximating $C_{12}H_{13}N_3O_3$, $C_{16}H_{16}N_4O_4$, or $C_{16}H_{18}N_4O_4$.

Bromine Derivative of Ricinine.—On adding bromine or bromine water to a moderately concentrated aqueous solution of ricinine, and heating the liquid on the water bath, long colourless radiated needles were obtained. The pure substance melted at 232° C., and had the formula $C_{16}H_{14}Br_2N_4O_4$ or $C_{16}H_{16}Br_2N_4O_4$.

Ricinine dibromide was found to be soluble in about 200 parts of 95 per cent. alcohol and in about the same quantity of water. It was less soluble in chloroform.

Oxidation of Ricinine.—On treating an alkaline solution of ricinine with potassium permanganate, a substance was obtained melting at 279°—280° C., and becoming black some degrees below the melting point. The aqueous solution of the oxidation product was acid to litmus, and formed crystalline salts with alkalis and with silver nitrate.

—C. A. M.

Helonin. L. Ough. Chem. and Druggist, 1900, **56,** [1014], 158.

Helonin is derived from the rhizome and rootlets of the "devil's bit," *Chamælirium luteum,* Gray, natural order, Melanthaceæ, an indigenous North American plant. The root is frequently found interchanged and mixed with that of *Aletris farinosa,* but its appearance is too characteristic to be readily mistaken for the latter. According to Tilden, the rhizome contains an oleo-resin, a gum, starch, extractive matter, and a bitter principle from which Green in 1878 isolated a glucoside, chamælirin, which is saponaceous in its aqueous and alcoholic solutions. Chamælirin is a yellowish-white, amorphous powder, neutral and bitter, readily yielding, by means of dilute acids, glucose, and an insoluble resinous substance, chamæliretin, which is readily soluble in alcohol and ether, and coloured brown by sulphuric acid.

Helonin, extracted from the rhizome by means of alcohol, forms a golden-brown substance, which, with care, can be reduced to a fine powder. The yield was nearly 13 per cent.—A. S.

Strychnine from Strychnine Hydrochloride Solution; Precipitation of, by Sodium Arsenate. J. Rutherford Hill. Pharm. J. 1900, **64,** [1543], 45.

Dunlop (see Pharm. J. 1899, 604) observed that on mixing solutions of strychnine hydrochloride and of di-sodium hydrogen arsenate, and allowing the mixture to stand, a gradual separation of strychnine occurred. The author discusses the various suggestions which have been made as to the cause of this precipitation, and also gives the results of his own experiments on the subject. He found that the whole of the strychnine is set free; also that quinine is precipitated, in a similar manner, from quinine hydrochloride solution.

The author's theory as to the separation of the strychnine is, that owing to the instability of the di-sodium hydrogen arsenate in solution, and the weak combination of hydrochloric acid in strychnine hydrochloride, double decomposition takes place according to the following equation:—

$$\mathrm{Na_2HAsO_4 + C_{21}H_{22}N_2O_2HCl = NaH_2AsO_4 + NaCl + C_{21}H_{22}N_2O_2}.$$

—A. S.

Antipyrine; Action of Iodine on. J. Bougault. J. Pharm. Chim. 1900, **11**, [3], 97—100.

In a previous communication, the author has shown (J. Pharm. Chim. **7**, 161) that the absorption of iodine by antipyrine in alcoholic solution, in the presence of mercuric chloride, is due to a definite reaction, and may be employed quantitatively for the determination of the base. The iodo-compound formed, is a combination of mono-iodo-antipyrine with mercuric chloride, mercuric iodide, and hydrochloric acid, the formula for which is—

$$\mathrm{(C_{11}H_{11}IN_2O)_4HgCl_2.HgI_2.2HCl}.$$

This body may be obtained in a crystalline form by mixing together 2·54 grms. of iodine, 1·88 grms. of antipyrine, and 3 grms. or more of mercuric chloride, each dissolved in 30 c.c. of 95 per cent. alcohol, heating the mixture to between 70° C. and 80° C., and allowing to crystallise. The iodo-compound separates out in yellowish-white crystals. The reaction may be represented by the equation—

$$\mathrm{4C_{11}H_{12}N_2O + 4I_2 + 3HgCl_2 = (C_{11}H_{11}IN_2O)_4HgCl_2.HgI_2.2HCl + HgI_2 + 2HCl}.$$

If the proportion of mercuric chloride be lessened, a different result is obtained; thus with 2 mols. of $\mathrm{HgCl_2}$ to 4 mols. of antipyrine, the absorption of iodine takes place much more slowly; and the compound obtained, which crystallises in bright yellow crystals, has the composition $\mathrm{(C_{11}H_{11}IN_2O)_4HgI_2.HgI_2.2HCl}$. If the amount of $\mathrm{HgCl_2}$ present be less than 2 mols. per 4 mols. of antipyrine, the iodine absorption is incomplete. It is thus shown that it is necessary that at least 3 mols. of mercuric chloride be present for every 4 mols. of antipyrine, in order to ensure the immediate completion of the iodine absorption.

—J. O. B.

Iodo-Antipyrine. J. Bougault. J. Pharm. Chim. 1900, **11**, [3], 100—102.

When the mono-iodo-mercuric-antipyrine compound—

$$\mathrm{(C_{11}H_{11}IN_2O)_4HgI_2.HgCl_2.2HCl}.$$

described in the preceding abstract, be treated with a solution of potassium iodide made faintly alkaline with a little potassium carbonate, the body is dissociated; $\mathrm{HgI_2.HgCl_2}$ and HCl are removed in solution, and the insoluble residue consists of iodo-antipyrine, $\mathrm{C_{11}H_{11}IN_2O}$, identical with the compound discovered by Dittmar. The same body may be obtained by the action of iodine on antipyrine in the presence of solutions of certain salts, such as sodium acetate; since it is comparatively insoluble, it may serve in certain cases for the gravimetric determination of antipyrine where the iodine absorption method is not applicable. Sodium carbonate may be employed instead of acetate, but since it forms insoluble compounds with iodine and certain phenolic bodies, the use of sodium acetate is preferable.

—J. O. B.

Perfumes. E. Erdmann. Zeits. f. angew. Chem. 1900, [5], 103—116.

In order that a substance may act on the olfactory nerves it is necessary that it shall be volatile, but since the converse does not follow, the author considers that the sense of smell has the nature of a chemical reaction. This is further borne out by the fact that those substances which are most pronounced perfumes belong to the class of aldehydes which, possessing unsaturated valencies, readily form additive compounds. Another instance in support of this view is given by oxygen, which in its ordinary condition is without smell, whilst as ozone it possesses a distinct odour. In considering the sense of smell, a distinction must be drawn between quality and penetrating power. The quality depends entirely upon the chemical constitution of the substance, and those substances possessing similar constitutions have the same or a similar scent. It occasionally, however, occurs that differently constituted substances, *e.g.*, nitrobenzene and benzaldehyde, or bornylacetate and trichlorisobutylalcohol, have an almost identical odour. With regard to the penetrating power of a scent, this also is dependent upon its constitution. The investigations of Valentin, E. Fischer, and Penzoldt (Annalen, **239**, 131) have shown that it is possible to recognise $\frac{1}{600}$ mgrm. of bromine, $\frac{1}{5000}$ mgrm. of sulphuretted hydrogen, and $\frac{1}{20000}$ mgrm. of otto of roses, whilst the 4,600,000th part of a milligram of chlorophenol and the 460,000,000th part of a milligram of mercaptan may be still detected. The condition, however, in which a scent exists, considerably modifies its penetrating power. For instance, the evaporation of a slightly volatile perfume is increased by passing a current of air through it or by dissolving it in alcohol, and if this solution be used in a spray diffuser, the evaporating surface is still further increased and the odour is most fully developed.

Perfumes, according to their chemical constitution and characteristics, may be classed in seven principal groups, which, n descending order of importance, are arranged as follows:—(1) Aldehydes; (2) Alcohols and esters; (3) Ketones; (4) Phenols and phenolic ethers; (5) Acids and acid anhydrides; (6) Nitrogenous substances; (7) Hydrocarbons.

1.—Aldehydes.

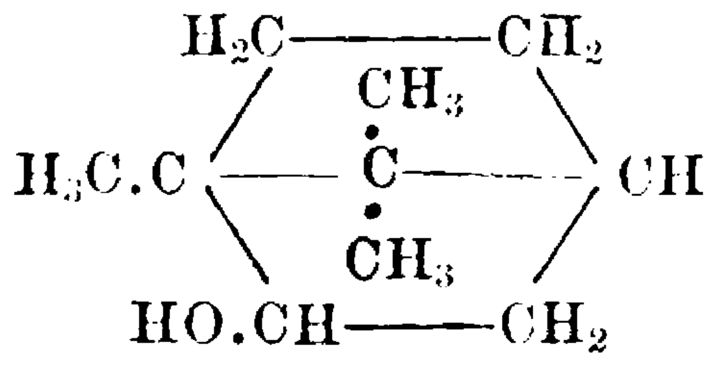
Nos. 1 *and* 2, *open chain, unsaturated; No.* 3, *cyclic; Nos.* 4–11, *cyclic, benzene series.*

Name.	Constitutional Formula.	Occurs Native in
1. Citral	$\mathrm{(CH_3)_2:C:CH.(CH_2)_2.C(CH_3):CH.CHO}$	Oil of lemon, lemon-grass oil.
2. Citronellal	$\mathrm{(CH_3)_2:C:CH.(CH_2)_2.CH(CH_3).CH_2CHO}$	Oil of citronella, lemon oil, eucalyptus maculata.
3. Furfural	$\left.\begin{matrix}\mathrm{CH{-}{-}CH}\\ \mathrm{\ddot{C}H.O.\ddot{C}.CHO}\end{matrix}\right\}$	Clove oil.
4. Benzaldehyde	$\mathrm{C_6H_5CHO}$	Oil of bitter almonds, laurel oil.
5. Phenylacetaldehyde	$\mathrm{C_6H_5CH_2.CHO}$.	.. .. ..
6. Cinnamic aldehyde	$\mathrm{C_6H_5CH:CH.CHO}$	Oil of cassia, oil of cinnamon.
7. Cuminic aldehyde	$\mathrm{1.4.C_6H_4.C_3H_7.CHO}$	Oil of caraway.
8. Salicylic aldehyde	$\mathrm{1.2.C_6H_4.OH.CHO}$	Oil of spiræa, crepis fœtida.
9. Anisic aldehyde (Hawthorn)	$\mathrm{1.2.C_6H_4.OCH_3.CHO}$.	.. .. ..
10. Vanillin	$\mathrm{4.3.1.C_6H_3.OH.OCH_3.CHO}$	Vanilla, gum benzoin, balsam of Peru, beet sugar
11. Heliotropin	$\mathrm{4.3.1.C_6H_3.O.CH_2O.CHO}$	Oil of spiræa.

With regard to the above group, citral can be obtained by oxidising the corresponding alcohol rhodinol (geraniol) or else the essence of linaloes. Furfural, one of the cyclic aldehydes, is always a constituent of clove oil, in which it can be detected by the red coloration given with an acetic acid solution of β-naphthylamine. Benzalde hyde is obtained artificially from toluene, whilst its next higher homologue, phenacetaldehyde (distilling at 90° C. under 15 mm.), which possesses a smell of hyacinths, can be obtained from cinnamic acid. It polymerises very readily. Anisic aldehyde can be produced by methylating *p*-oxybenzaldehyde, but is usually obtained by oxidising anethol. It possesses the odour of the flowering whitethorn (*Crataegus oxyacantha*), and comes into commerce in France as "Hawthorn." The solid sodium bisulphite compound is also a commercial product. Cinnamic aldehyde is obtained technically from benzaldehyde. Vanillin, the essential principle of the vanilla pod, which contains about 2 per cent. is very widely distributed. After its constitution had been determined by Tiemann, it was originally obtained by oxidising coniferin, but at the present time it is produced from eugenol obtained from clove oil. This is first converted into isoeugenol, which is then acetylated and oxidised. No truly synthetic process for obtaining vanillin has been worked commercially, and in view of the low price of clove oil and of vanillin itself, such processes have no prospects. In 1876, vanillin cost 350*l.* per kilo.; in 1897, it fell to 6*l.*, and since that time has fallen still lower. Heliotropin or piperonal has a similar constitution to vanillin, and is obtained in like manner from piperin, the chief principle of pepper, but it can also be produced more economically from isosafrol. It is valued for its use in Cherry Blossom perfume, and costs about 30*s.* per kilo.

2.—ALCOHOLS AND ESTERS.

Nos. 1—4, *open chain, saturated; Nos.* 5—7, *open chain, unsaturated; No.* 8, *cyclic; Nos.* 9—13, *cyclic, terpene series; Nos.* 14 *and* 15, *cyclic, benzene series.*

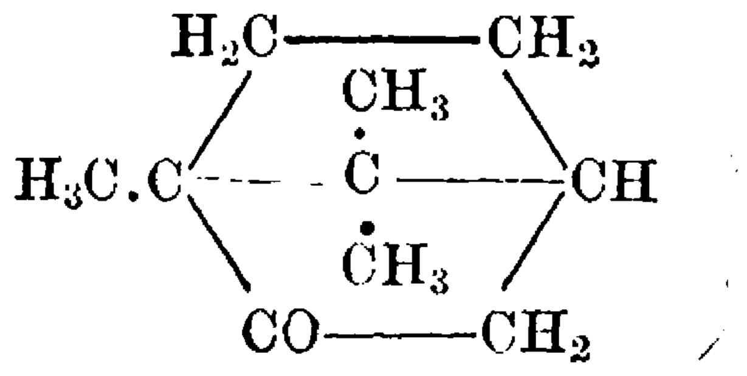
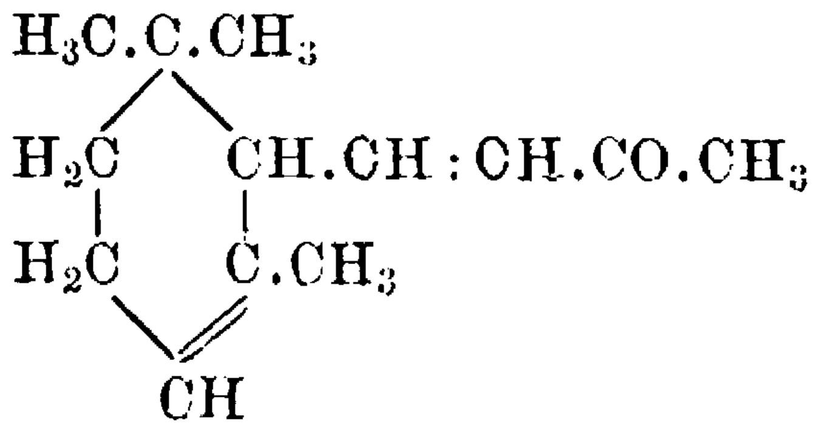
Name and Formula.	Combined with	Occurs Native in
1. Methyl alcohol, $CH_3.OH$	.. ..	Clove oil.
	Benzoic acid (Niobe oil).	.. ..
	Salicylic acid (oil of winter-green)	Gaultheria procumbens, Betula lenta.
2. Ethyl alcohol, C_2H_5OH	Formic acid (essence of rum).	.. ..
	Acetic acid (acetic ether)	White-wine vinegar, Cognac, Magnolia fuscata (?).
	Butyric acid (pineapple ether).	.. ..
	Isovaleric acid (apple oil).	.. ..
	Pelargonic acid (artificial essence of cognac).	.. ..
3. Isobutyl alcohol, $(CH_3)_2:CH.CH_2.OH$	.. ..	Potato fusel oil.
	Isobutyric acid	Camomile oil.
	Angelic acid	
4. Isoamyl alcohol, $(CH_3)_2:CH.CH_2.CH_2.OH$	Acetic acid (essence of pear).	.. ..
	Isovaleric acid (essence of apple).	.. ..
	Caprylic acid Caprinic acid (oenanthic ether)	Oil of cognac.
5. Rhodinol (geraniol)— $(CH_3)_2:C:CH.(CH_2)_2.C(CH_3):CH.CH_2OH$.	.. ..	Otto of roses, geranium oil, citronella, lemon-grass oil.
	Acetic acid	Oil of Neroli.
6. Linalool— $(CH_3)_2:C:CH.(CH_2)_2.C(OH)(CH_3).CH:CH_2$	.. ..	Linaloes, bergamot and lavender oil, Neroli, jasmine.
	Acetic acid	Bergamot oil, Neroli.
7. Citronellol— $(CH_3)_2:C:CH.(CH_2)_2.CH(CH_3).CH_2.CH_2OH$.	.. ..	Otto of roses, citronella oil.
8. Furfuralcohol $\left\{\begin{matrix}CH \;.\; CH \\ \ddot{C}H.O.\ddot{C}.CH_2OH\end{matrix}\right\}$	.. ..	Coffee oil.
9. Borneol, $C_{10}H_{17}OH$	.. ..	Borneo camphor, Ngai camphor, oil of rosemary.
	Acetic acid	Pine-tree oil.
	Isovaleric acid	Oil of valerian.
10. Terpineol, $C_{10}H_{17}OH$	.. ..	Oil of cajeput, cardamom.
11. Menthol, $C_{10}H_{19}OH$	.. ..	Oil of peppermint.
12. Eucalyptol (cineol), $C_{10}H_{18}O$	.. ..	Wormseed oil, eucalyptus oil.
13. Peruviol, $C_{13}H_{22}O$	.. ..	Balsam of Peru.
14. Benzyl alcohol, $C_6H_5CH_2OH$	.. ..	Jasmine oil.
	Acetic acid	Jasmine oil.
15. Cinnamic alcohol, $C_6H_5.CH:CH.CH_2OH$	Benzoic acid	Balsam of Peru.
	Cinnamic acid	Storax.

Of the alcohols and esters, rhodinol, of which the constitution has been determined by Tiemann and Semmler (Ber. **28**, 2132), is the principal constituent of important essential oils, such as otto of roses, geranium oil, and citronella oil. It gives well-defined crystalline compounds such as the diphenylurethane derivative, the silver salt of the phthalic acid ester and the opianic acid ester. Rhodinol in the pure state has, however, a somewhat stale smell, and is an oil which readily alters in the air; the sweet honey-like odour of otto of roses is due to other unknown substances. Linalool is the essential principle of lily of the valley perfume, and occurs native in two different optically active forms usually together with linalyl acetate. as in oil of bergamot, lavender, and orange blossom oil. Of the terpene series, oil of peppermint, of which menthol is the essential principle, is one of the most important. New York and Michigan, U.S.A., Japan, and Mitcham are the principal sources of supply. In 1897, 114,000 kilos. were produced in North America; in 1896, 135,000 kilos. in Japan; whilst Mitcham, which produces the finest quality, gives about 9,000 kilos. annually. Under normal conditions, the world's production of oil of peppermint amounts to 175,000 kilos. which is valued at about 125,000*l.* The percentage of menthol contained in it varies from 55—90 per cent.

Of the ketones, the cyclic compounds of the terpene series are the most important. This sub-group contains camphor, for which Bredt's formula is given. The crude product is obtained by steam distilling the root, stem, and twigs of the camphor tree (*Cinnamomum camphora* or *Laurus camphora L.*), oil of camphor, from which safrol is produced, being obtained simultaneously. Irone, the principle of the violet root (*Iris Florentina*), is isomeric with ionone. The latter is obtained commercially from citral by condensation with acetone, the first product being pseudoionone. This, by the action of dilute sulphuric acid, forms ionone.

3. KETONES.

Nos. 1 *and* 2, *open chain, saturated; Nos.* 3 *and* 4, *open chain, unsaturated; Nos.* 5—12, *cyclic, terpene series.*

Name.	Formula.	Occurs Native in
1. Methylamylketone	$CH_3.CO.C_5H_{11}$	Clove oil.
2. Methylnonylketone	$CH_3.CO.C_9H_{19}$	Oil of rue.
3. Methylheptenone	$(CH_2)_2:C:CH.(CH_2)_2.CO.CH_3$	Linaloes, lemon grass oil.
4. Pseudo-ionone	$(CH_3)_2:C:CH.(CH_2)_3.C(CH_3):CH.CH:CH.CO.CH_3$	.. ..
5. Carvone	$C_{10}H_{14}O$	Kümmel oil.
6. Camphor		Camphor tree.
7. Fenchone	$C_{10}H_{16}O$	Fennel oil, thuja oil.
8. Thujone (tanacetone)	$C_{10}H_{16}O$	Thuja oil, tansy oil, oil of absinthe.
9. Pulegone	$C_{10}H_{16}O$	Polei oil.
10. Menthone	$C_{10}H_{18}O$	Peppermint oil.
11. Irone	$C_{13}H_{20}O$	Orris root (violet root).
12. Ionone		

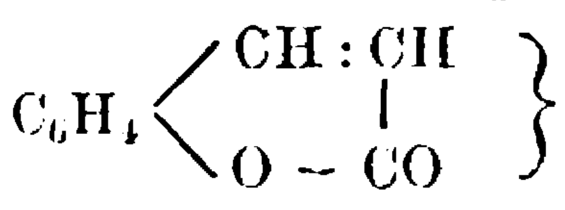
4. PHENOLS AND PHENOL ESTERS.

Nos. 1—9, *benzene series; No.* 10, *naphthalene series.*

Name.	Constitutional Formula.	Occurs Native in
1. *p*-Cresol methyl ether	$1.4.C_6H_4.CH_3.OCH_3$	Ylang-Ylang oil.
2. Guaiacol	$1.3.C_6H_4.OH.OCH_3$	Beech and birch tar oil.
3. Creosol	$1.3.4.C_6H_3.CH_3.OCH_3.OH$	Birch tar oil.
4. Anethol	$1.4.C_6H_4.OCH_3.CH:CH.CH_3$	Oil of anise, oil of fennel.
5. Chavicol	$1.4.C_6H_4.OH.CH_2.CH:CH_2$	Betel oil.
6. Estragol	$1.4.C_6H_4.OCH_3.CH_2.CH:CH_2$	Estragon oil.
7. Eugenol	$4.3.1.C_6H_3.OH.OCH_3.CH_2.CH:CH_2$	Clove oil.
8. Thymol	$1.3.4.C_6H_3.CH_3.OH.C_3H_7$..	Oil of thyme (thymus vulgaris).
9. Safrol	$4.3.1.C_6H_3.O.CH_2O.CH_2.CH:CH_2$	Sassafrass oil, oil of camphor.
10. *β*-Naphthol methyl ether (nerolin)	$C_{10}H_7OCH_3$.	.. ..

5. ACIDS AND ACID ANHYDRIDES.

Nos. 1 *and* 2, *open chain, unsaturated; Nos.* 3—5, *cyclic benzene series.*

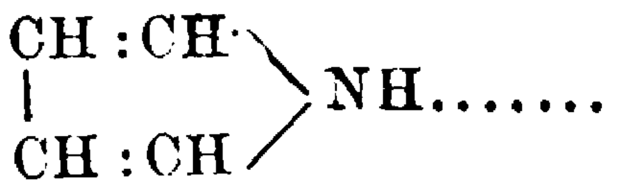
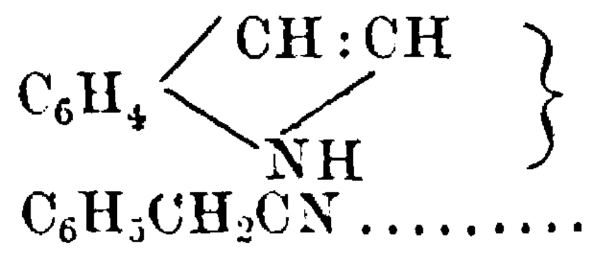
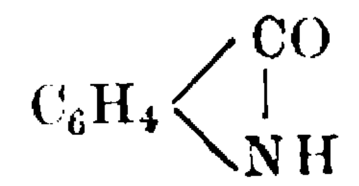
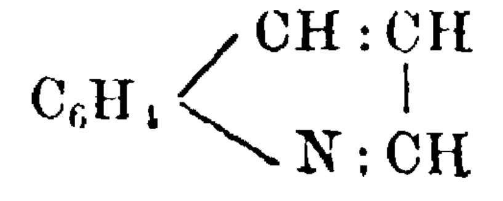
Name.	Constitutional Formula.	Occurs Native in
1. Angelic acid	$CH_2:CH.CH(CH_3)CO_2H$	Ester of camomile oil.
2. Tiglic acid	$CH_3.CH:C(CH_3)CO_2H$	Ester of camomile and croton oil.
3. Benzoic acid	$C_6H_5CO_2H$	Balsam of Peru, gum benzoïn, balsam of Tolu.
4. Cinnamic acid	$C_6H_5.CH:CH.CO_2H$	Balsam of Peru, of Tolu, gum benzoïn (Sumatra storax).
5. Coumarin	 $\left\{ C_6H_4 \left< \begin{matrix} CH:CH \\ \vert \\ O-CO \end{matrix} \right. \right\}$	Tonka beans, woodruff (asperula odorata).

The presence of guaiacol in birch tar oil gives the latter the odour of Russia leather. Pure anethol forms 90 per cent. of oil of Indian anise (badiane), and is the raw product from which anisic aldehyde is obtained. Thymol is principally employed for its disinfecting qualities, whilst eugenol, the essential principle of clove oil, finds an extensive use for the manufacture of vanillin.

The paper also contains tables showing the importance of the spice and perfume industry in Germany. With regard to the total production of certain substances, the author states that the otto of roses annually produced has a value amounting to about 100,000*l.*, the amount of musk shipped is valued at about 300,000*l.*, the export of Sicilian essences (bergamot, lemon, and orange oil) amounts to 300,000*l.*, whilst the value of the oil of peppermint produced is about 125,000*l.*, and of clove oil about 65,000*l.*

6. NITROGENOUS PERFUMES.

Nos. 1--3, *open chain ; No.* 4, *cyclic ; Nos.* 5--12, *cyclic, benzene series.*

Name.	Formula.	Occurs Native in
1. Trimethylamine	$N(CH_3)_3$	Chenopodium oil.
2. Prussic acid	CNH	Oil of bitter almonds, laurel oil.
3. Allyl mustard oil.	C_3H_5NCS	Mustard oil, horseradish, Alliaria officinalis.
4. Pyrrol	 $\begin{matrix}CH:CH\\ \vert\\ CH:CH\end{matrix}\!>NH$	Derivative in orange oil from the unripe fruit.
5. Indole	 $C_6H_4\!<\!\begin{matrix}CH:CH\\ NH\end{matrix}$	Jasmine oil.
6. Benzyl cyanide.	$C_6H_5CH_2CN$	Oil of cress (Lepidium sativum, Tropæolum majus).
7. Mandelic nitrile	$C_6H_5CH(OH)CN$	Almond oil.
8. Nitrobenzene (oil of mirbane).	$C_6H_5.NO_2$.	..
9. Tonquinol (Baur's musk).	$C_6H.(NO_2)_3.C_4H_9.CH_3$	..
10. Anthranilic methyl ester.	$1.2.C_6H_4.NH_2.CO_2CH_3$	Neroli, jasmine oil.
11. Anthranil	 $C_6H_4\!<\!\begin{matrix}CO\\ \vert\\ NH\end{matrix}$	..
12. Quinoline	 $C_6H_4\!<\!\begin{matrix}CH:CH\\ \vert\\ N:CH\end{matrix}$	..

7.--HYDROCARBONS.

Nos. 1—10, *cyclic terpene series ; Nos.* 11 *and* 12, *cyclic benzene series.*

d = dextro ; l = lævorstatory ; i = optically inactive.

Name.	Formula.	Occurs Native in
1. Pinene	$C_{10}H_{16}$	*d*-German turpentine, American turpentine, *l*-French turpentine.
2. Camphene	$C_{10}H_{16}$	*d*-Oil of ginger, oil of spike, *l*-citronella oil, valerian oil.
3. Fenchene	$C_{10}H_{16}$	French turpentine (?).
4. Limonene	$C_{10}H_{16}$	*d*-Orange-peel oil, lemon oil, bergamot oil, *l*-oil of silver-leaved fir, *i*-(dipentene) oil of camphor.
5. Silvestrene	$C_{10}H_{16}$	Swedish, Russian turpentine.
6. Phellandrene	$C_{19}H_{16}$	*d*-Water fennel oil, elemi oil, *l*-Australian eucalyptus oil.
7. Terpinene	$C_{10}H_{16}$	Cardamom oil.
8. Terpinolene	$C_{10}H_{16}$	.. ..
9. Cadinene	$C_{15}H_{24}$	Oil of cade.
10. Caryophyllene.	$C_{15}H_{24}$	Clove oil.
11. Cumene	$1.4.C_6H_4.CH_3.C_3H_7$	Oil of cumin, cyminum, thyme.
12. Styrene	$C_6H_5.CH:CH_2$	Storax.

—T. A. L

Benzoïc Esters; Transformation of Acetic Esters of Phenol, p-Cresol, and Thymol into. F. Bodroux. Bull. Soc. Chim. 1900, **23—24**, [2], 54—55.

DOEBNER obtained phenyl benzoate by the action of benzoyl chloride upon phenyl acetate at 180° C., in the presence of zinc chloride (Annalen. **210**, 255). The author has effected the same transformation at the ordinary temperature by means of zinc dust. After the addition of this substance, the mixture soon becomes warm, then a violent action commences, with the evolution of hydrochloric acid gas. The solid obtained by shaking the reaction product with dilute potash, is dried, and recrystallised from warm 50 per cent. alcohol. The reaction is almost quantitative. The benzoïc esters of *p*-cresol and thymol may be obtained in the same manner. The acetic ester of *β*-naphthol remains unaltered by a similar treatment.—A. C. W.

Oil of Sandalwood, Composition of. M. Guerbet. Comptes Rend. **130**, [7], 417--420.

THE oil, after saponification of the contained esters, and separation of their potassium salts, was fractionally distilled, and yielded two main fractions. From that of lower boiling point were isolated two isomeric hydrocarbons, $C_{15}H_{24}$, *α-santalene*, sp. gr. 0·9134; b.p. 252° C., $\alpha_D = -13°·98$, and *β-santalene*, sp. gr. 0·9139; b.p. 261° C., $\alpha_D = -28°·5$. From the other fraction, no satisfactory separation could be effected; it was a mixture of alcohols $C_{15}H_{26}O$, of rotatory powers varying from − 9°·4 to − 25°·3; very probably there are two alcohols, *α*- and *β*-santalol, corresponding with the two hydrocarbons. The oil also contains the aldehyde $C_{15}H_{24}O$, *santalal*, a colourless oily liquid of peppery odour, which yields on oxidation, *santalic acid*, $C_{15}H_{24}O_2$, a viscous liquid, forming salts with potassium and sodium, which are decomposed by carbonic acid. This acid occurs in the oil, as does *teresantalic acid*, $C_{10}H_{14}O_2$, which crystallises from alcohol in large colourless prisms, melting at 157° C. Acetic and formic acids also occur, the latter in very minute quantity. The odour of the oil (and of the wood) is almost entirely due to more volatile constituents, which, however, are present in such small quantity that no examination of them was possible.—J. T. D.

Lavender, Genesis of Terpene Compounds in. E. Charabot. Comptes Rend. **130**, [5], 257—259.

THE free acids, the linalool, the linalool esters, and the terpenes, were determined in essence of lavender made from plants *a*, in bud, *b*, in full flower, and *c*, when the flowers had faded and were beginning to dry up. The following table gives the results of the determinations :—

——	*a.*	*b.*	*c.*
Density at 15° C.	0·8849	0·8854	0·8821
Rotatory power (*l* = 100 mm.)	− 6° 32′	− 6° 48′	− 6° 50′
„ „ after saponification	− 7° 45′	− 8° 35′	− 9° 10′
Acidity (in grms. of acetic acid per litre).	0·5241	0·4716	0·3846
Esters, per cent.	36 6	40·4	39·75
Free alcohol, per cent.	21·0	16·7	18·9
Total alcohol, per cent.	49·8	48·4	50·25

Thus the acidity diminishes as the plant ages: the esters increase till the plant is in full flower and then diminish: both the free alcohol and the total alcohol diminish up to the period of full flower whilst the esters are increasing, but afterwards the alcohol increases, whilst the esters diminish. Thus, as with bergamot, the esters are formed by the direct action of the acids on the alcohols. During the development of the plant, whilst one part of the alcohol is esterified, another is dehydrated, yielding terpenes: thus not only the free alcohol, but the total alcohol diminishes. Both esterification and dehydration take place in the green or chlorophyllic parts of the plant.—J. T. D.

Geranium, Essential Oils of. Jeancard and Satie. Bull. Soc. Chim. **23—24**, 1900, [1], 37—39.

THE geranium oils have different values according to their origin. The authors have examined samples of known purity. They contained free acid; this constituent was estimated by adding 10 c.c. of decinormal potash and 10 c.c. of alcohol to 3 grms. of the oil, precipitating with water after two minutes, and titrating the excess of alkali. The number of milligrams of potash neutralised by 1 grm. of oil represents the saponification value in the cold. With sufficient accuracy this corresponds to the free acid present. It was found that the acidity of geranium oils showed an increase on keeping in incompletely filled bottles. The accompanying table contains the results of the examination; the esters are calculated as $C_{12}H_{20}O_2$, taking account of the acidity; the alcohols (total) are calculated as $C_{10}H_{18}O$.

The estimation of esters according to the ordinary method gave results varying from 15 to 26 per cent. from the value generally given. It is considered possible to deduce from the table a method of distinguishing geranium oils of different origins.

Origin.	Density at 15° C.	Rotation at 15° C. 100 mm.	Saponification Value.		Esters.	Alcohols.
			Ordinary.	Cold.		
					Per Cent.	Per Cent.
Cannes	0·9872	− 9·40	54·60	26·60	9·80	61·31
Spain.. ...	0·9073	− 7·30	65·80	43·40	7·84	66·23
Corsica	0·9012	− 8·00	60·20	40·13	7·00	68·55
Africa	0·9006	− 8·03	65·80	42·93	8·08	63·19
Bourbon...	0·8905	− 8·20	74·00	56·00	6 65	71·28
India	0·8960	− 0·48	43·00	9·60	11·30	84·62

—A. C. W.

Quinine Alkaloids : Transformation into Derivatives of γ-phenylquinoline. W. Koenigs. J. Prakt. Chem. **61,** 1900, [1], 1—46.

Cerium ; Volumetric Estimation of. P. E. Browning. Chem. News, 1900, **81,** 30.

See under XXIII., *page* 277.

Manganese Oxalate, Formation of Crystalline, in the Permanganic Oxidation of Citric Acid. G. Denigès. J. Pharm. Chim. 1900, **11,** [3], 102.

See under XXIV., *page* 282.

Balsam of Tolu. J. Spilsbury and T. G. Joyce. Pharm. J. **64,** [1545], 93.

See under XXIII., *page* 278.

Morphine ; The Nitrogen-free Decomposition Products of. —VI. E. Vongerichten.

See under XXIV., *page* 283.

Liquorice Pastes, &c. ; Technical Analysis of. M. Trubek. J. Amer. Chem. Soc. **22,** [1], 19.

See under XXIII., *page* 281.

Diazo-Caffeine. M. Gomberg. Amer. Chem. J. 1900, **23,** [1], 51.

See under IV., *page* 235.

Nicotine ; Detection of. J. Schindelmeiser.

See under XXIII., *page* 278.

Alkaloids in Leaves of Datura Stramonium, Hyoscyamus Niger, and Atropa Belladonna ; Quantitative Determination of. E. Schmidt.

See under XXIII., *page* 280.

PATENTS.

Ferruginous Nucleïn Compounds from Blood. A. Jolles, Vienna. Eng. Pat. 2018, Jan. 28, 1899.

The process claimed consists in mixing blood with ten times its volume of brine of 8—10 per cent. strength, shaking the separated corpuscles with an equal volume of water (and with ether), mixing the aqueous solution with an equal volume of 10 per cent. hydrochloric acid, washing the precipitate with absolute alcohol, and drying *in vacuô* at 60°—70° C. The preparation contains about 0·32 per cent. of iron in the form of an absorbable compound not affected by the acid of the stomach.—A. C. W.

Hexamethylenetetramine ; New Compounds of. [*Antiseptics*]. L. Stange, Offenbach-on-the-Main, Germany. Eng. Pat. 4804, March 4, 1899.

Compounds of hexamethylenetetramine (or its derivatives) with halogen pyrrols and aromatic sulphonic acids (or their halogen derivatives), for use as antiseptics, are claimed. The tetra-iodopyrrolhexamethylenetetramine, it is stated, with regard to its antiseptic and bactericidal powers, surpasses iodoform, and it is quite odourless. The processes for their manufacture consist in bringing the components into contact in solution.—A. C. W.

Dimethyl Ether of Sulphuric Acid, Process for Producing. C. D. Abel, London. From Actien Gesellschaft für Anilin Fabrikation, Berlin. Eng. Pat. 7020, April 1, 1899.

By the action of fuming sulphuric acid on methyl alcohol at high temperatures, Müller (Ber. **6,** 1031) obtained oxymethane sulphonic acid. If the reaction be conducted at low temperatures, a good yield of dimethyl sulphate is obtained. The process claimed is for the action of fuming sulphuric acid on methyl alcohol at 30°—40° C., and distillation of the product *in vacuo*.—A. C. W.

Aromatic Aldehydes ; New Derivatives of [*Tannin Protein Compounds*]. H. E. Newton, London. From Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 7524, April 10, 1899.

By stirring salicylic aldehyde (benzoic or resorcylic) with a solution of egg albumin in water for some hours, a thick yellow paste is obtained. This paste combines with tannin in aqueous solution ; the product is washed and dried. It then forms a yellow, tasteless powder, insoluble in water and dilute acids, and soluble with difficulty in alkalis. The thick paste obtained from 1 part of salicylic aldehyde and 2 parts of egg-albumin is slowly added with continuous stirring to a solution of 1·5 parts of tannin in 10 parts of water. The resulting mixture is stirred for about 12 hours at the ordinary temperature. Subsequently it is heated on a water bath until the new compound has separated as a viscous mass at the bottom of the vessel. This reaction product is then washed with water until free from salicylic aldehyde. It is finally dried and pulverised.—A. C. W.

New Lactone, and Phenylacetaldehyde therefrom. H. Erdmann, Halle a/S., Germany. Eng. Pat. 8248, April 19, 1899.

By adding potassium hypobromite to a solution of potassium cinnamate containing boric acid and cooled with ice, and then strong caustic potash, the potassium salt of the β-lactone of α-oxyphenylpropionic acid separates as a thick white precipitate. This is washed with water and ether. The free oxylactone is obtained by adding dilute sulphuric acid to the solution or suspension of one of its salts at 0° C., as a colourless oil which soon solidifies. It melts at 83°—84° C. Phenylacetaldehyde is obtained in better yield than by any other method, it is said, by heating the lactone in a partial vacuum or by mixing with water and blowing in steam ; it is valuable as a perfume. The claims are for the processes described and for the lactone.

—A. C. W.

"Saccharin" ; Impts. in the Manufacture of. H. H. Lake, London. From Chemical Works, formerly Sandoz, Basle, Switzerland. Eng. Pat. 19,629, Sept. 29, 1899.

The process claimed consists in adding slowly *o*-sulphamidobenzoic acid to fuming sulphuric acid (20 per cent. SO_3) at a temperature not exceeding 40° C., allowing to stand 24 hours, and pouring on ice and water. The yield is 95 per cent. of the theoretical, and is almost the same when either chlorosulphonic acid, monohydrated sulphuric acid, or acid of 66° B. is used.—A. C. W.

Quinine and Caffeïne ; Easily Soluble Preparations of. W. P. Thompson. From Schröder and Krämer, Hamburg, Germany. Eng. Pat. 25,167, Dec. 19, 1899.

The production of easily soluble preparations containing quinine and caffeïne "by melting together quinine hydrochloride and caffeïne, with or without the aid of solvents," is claimed.—A. C. W.

XXI.—PHOTOGRAPHY.

Lippmann's Colour Process, New Experiments on. R. Neuhauss. Chem. Zeit. Rep. 1900, **24,** [2], 16. Phot. Rundschau, 1900, **14,** 2.

The author has continued his experiments on Lippmann's interference process (this Journal, 1898, **17,** 181), and has been particularly successful with gelatin-emulsion plates. By washing the emulsion before coating the plates, a great simplification is effected in the manufacture of plates on the large scale, since the emulsion so treated (" die gewaschene Emulsion ") may be kept in a cool place for two or three weeks without deterioration, inasmuch as it only begins to become turbid at 50° C. The perfect reproduction of colours is dependent upon the correct use of suitable dyes to impart sensibility to the plates. Cyanine alone is used to give sensitiveness to the red rays, and for this purpose the author recommends the addition of 3 c.c. of a 1:500 cyanine solution to each 100 c.c. of emulsion. For sensitisation to green (especially blue-green), the Glycin Red of Kinzlberger is found very suitable. With mixed colours, however, yellow-green was not reproduced, and for this purpose Erythrosin is better. The following mixture is recommended for the production of a panchromatic plate :—To every 100 c.c. of emulsion : 3 c.c. of a 1:500 alcoholic Cyanine solution, 2 c.c. of a 1:500 alcoholic Erythrin solution, and 10 c.c. of a saturated alcoholic solution of Glycin Red. A plate so prepared reproduces with absolute uniformity every colour—whether mixed or pure—from red to violet. The author has not tried the mixture for a like purpose with the usual highly sensitive dry plates ; but in applying it in this direction, the formula would have to be modified, because the quantities taken for Lippmann emulsions are necessarily large, owing to the fact that much is removed in the process of washing. The large proportion of alcohol (15 c.c. to 100 c.c. of emulsion) here recommended, so far from being detrimental, is actually advantageous, as it favours the production of fine-grained and clear emulsions.

—W. G. M.

Gelatin Bromide Plates ; Modern Intensifiers for. J. M. Eder. Brit. J. of Photography, **47**, [2074], 68—69.

The author gives the results of his experiments with four different intensifiers ; Lumière's mercuric iodide and Andresen's " agfa " intensifiers are compared with the old method of using a mercuric chloride solution, with or without potassium bromide, followed by a bath of sodium sulphite. The intensifier used by Lumière Frères and Seyewetz consists of a solution of mercuric iodide 1 part, anhydrous sodium sulphite 10 parts, water 100 parts. The reactions which take place are represented as follows :—

$$2HgI_2 + 2Ag = Hg_2I_2 + 2AgI.$$
$$Hg_2I_2 + 2Na_2SO_3 = Hg + HgI_2(Na_2SO_3)_2.$$

After washing, the image is blackened by the reduction of the silver iodide formed to silver, by any ordinary developer.

The " agfa " intensifier of Messrs. Andresen and Leupold depends on the action of double thiocyanates (sulphocyanides).

The tests were made by measuring the opacities of gelatin bromide plates by Hartmann's micro-photometer, before and after intensification, exposure being made by means of the Scheiner sensitometer. Treatment with mercuric chloride solution (1:50), followed, after washing, by a sodium sulphite bath, did not cause very appreciable intensification, and the bleaching of the image was slower than when potassium bromide was used in the first solution ; with this mixture, viz., mercuric chloride 1 part, potassium bromide 1 part, water 50 parts, the intensification was very satisfactory, and it may still be recommended for ordinary work ; both methods showed some loss of density in the finest shadows. Lumière's method gave the greatest possibility of density, agfa being nearly equal in this respect ; red fog may be produced if the washing is not thorough. Agfa intensifier is preferable to the others because there is only one solution, no risk of red fog, and the greatest ease in control.—J. W. H.

Photographic Image ; Transformation of, into a Lamellar State, and Resulting Colour Phenomena. A. Trillat. Comptes Rend. **130,** 170—172.

The conversion of the amorphous silver image in the film of a photographic negative or positive into a lamellar state, is the problem dealt with. The silver deposit is first dissolved and then precipitated in the form required. Its solution in a liquid appeared out of the question on account of the possible deformation of the image or loss of silver, so the author exposes the plate to vapours, which, while dissolving the silver deposit, have no harmful effect on the support. The plate, cleaned, polished, and hardened, is exposed to the fumes of nitric acid until it becomes transparent and the image disappears. This polishing and hardening has some effect on the final results. To precipitate the silver in a lamellar form, for the same reasons as before, the plate is exposed to fumes, in this case, of moist sulphuretted hydrogen. Gradually the image reappears with a metallic aspect, and assumes various colours in its different parts.

Before the colours weaken and lose their vigour, which may happen should the action be too prolonged, the operation is stopped and the plate dried.

On examination of either the film or glass slide by reflected light, a brightly coloured image is seen ; often the colours of the image seen from the two faces are complementary. Although moisture produces temporary changes, the colours are permanent, and may be seen at a much greater angle than the interference effects obtained by Lippmann's method ; the film may be also detached from the glass without losing its colour properties.

Generally there is no relation between the colours obtained and those of the original object ; but required colours may be produced in any spot, since, from the author's experiments, their nature and intensity depend on the thickness of the grains ; the colour develops from white to black, passing through the shades of the rainbow according to a law not yet determined. It would seem possible, in the case where the portions of an image had thicknesses according to their shades, to produce colours more or less true to the original object.

These observations point out clearly the important part the plate must play in the production of colour photographs by an application of this method.—J. W. H.

PATENTS.

Photographic Sensitised Surfaces. L. Hanriau, Paris. Eng. Pat. 5304, March 10, 1899.

This invention relates to the preparation of sensitised paper or other material, by coating it with a solution made by mixing in equal proportions saturated solutions of ferrous sodium oxalate and a salt of either platinum, gold, palladium, iridium, or other metals. For platinum prints, a saturated solution of " protochloride of platinum " is used.

After the image has appeared by exposure under a negative in the usual way, it is fixed by passing it through several baths of 1 per cent. solutions of hydrochloric acid ; the final washing is carried out with water only. The claims are (1) the process for obtaining photographic prints by means of the double salts of platinum or other metal mentioned above, mixed with ferrous sodium oxalate, these prints being obtained by direct printing without developing or toning. (2) a new product, a photographic printing paper as described.—J. W. H.

Photographic Sensitised Materials, Manufacture of. J. E. Thornton, Altrincham. Eng. Pat. 5793, March 17, 1899.

In the manufacture of photographic plates, films, and paper, the coating of emulsion is applied at one operation, with the result that the drying of the film becomes very tedious and troublesome ; after coating, the layer is made to set by cooling, and then transferred to chambers where the drying is accomplished by a current of warm air ; this drying operation takes several hours, and cannot be hastened beyond very narrow limits, without damage to the film, whilst the long exposure of the undried film to the air also may produce disastrous changes. To overcome these difficulties the inventor applies the coating in extremely thin layers, each layer being dried by a blast of heated air immediately, and without setting before the application of the next coat. Heated air applied to a thick layer melts the emulsion, but removes the moisture immediately from a thin one.

For coating films or paper, the material is passed over a roller covered with a thin layer of emulsion, which it has taken up by contact with another roller dipping in the supply trough; immediately on leaving this roller the film passes either vertically or in an inclined position, into the drying chamber, where it meets small blasts of heated air, and is almost instantaneously dried, and ready for the next coating. Plates are carried on an endless travelling band, and when in an inclined position receive a spray of emulsion produced by jets of air directed on them, through the meshes of a rotating silver gauze cylinder, which dips into the supply trough; the drying is carried out as with films. Modifications of these forms of apparatus are given. The 10 to 20 coatings necessary to produce a film of usual thickness may be applied in a few minutes. The claims are—(1) "the formation of a sensitive portion" of photographic plates, films, or papers "by applying to the support a number of very thin coats of the sensitive emulsion, and, without setting, rapidly (almost instantaneously) drying each successive coat by a blast of heated air"; (2) as above, "drying each successive coat by a blast of heated air immediately after coating, and without any immediate setting or chilling of each coat between the coating and drying thereof; (3) the manufacture of photographic material in the manner and by the means and methods described to obtain better results and quicken the process; (4) a photographic film, plate, or paper having its coating made as described; (5) apparatus for the preparation of such plates, films, or paper.—J. W. H.

Photographic Silver Prints, Process for Intensifying. C. D. Abel. From The Actiengesellschaft für Anilin Fabrikation, Berlin. Eng. Pat. 7021, April 1, 1899.

The process depends upon the fact that a solution of the double salts of mercuric thiocyanates will intensify a photographic silver print with a black colour at one operation. The double thiocyanate of mercury and an alkali, alkaline earth, or ammonia can be practically used. A stock solution may consist of 10 parts of mercuric thiocyanate, 8 parts of potassium thiocyanate and 100 parts of distilled water, and must be diluted with 10 parts of water for use.

In the above solution, the alkali thiocyanate is in excess of that necessary to form the double salt, on account of the fact that such a mixture is more readily soluble in water, and keeps better in aqueous solution than the pure double salt. The inventors claim the process of "intensifying silver prints by subjecting them to the action of a solution of a double salt of mercuric thiocyanate with a thiocyanate of an alkali, an alkaline earth, or ammonia, together with an excess of the alkali thiocyanate."

—J. W. H.

XXII.—EXPLOSIVES, MATCHES, Etc.

Lyddite. L. de Belfort de la Roque. Rev. de Chim. industrielle, **11**, 5—12.

The author gives a short review of lyddite (picric acid), claiming it as a French invention, and then with the help of the usual thermochemical and thermodynamical formulæ, he calculates the following constants on the assumption that the explosion of lyddite is represented by the equation—

$$2C_6H_2.OH.(NO_2)_3 = 3CO_2 + 8CO + C + 3H_2 + 3N_2.$$

Glycocoll Nitrate.—The constants obtained for lyddite (trinitrophenol) are then compared with the corresponding constants of the nitric acid salt of amido-acetic acid, the explosive decomposition of which is given by the equation—

$$CH_2(NH_2)COOH.HNO_3 = 2CO + 3H_2O + N_2.$$

——		Lyddite.	Glycocoll Nitrate.
Molecular weight	..	457	138
Heat evolved by the reaction at constant pressure.	Kilogram-calories.	195	145·4
Heat evolved by the reaction at constant volume, and at the existing temperature, for 1 mol.	„	200·2	147·4
Heat evolved per kilo. at constant pressure.	Kilogram-calories	853·4	1,053·7
Heat evolved per kilo. at constant volume.	„	876·0	1,094·1
"Potential" (maximum work capable of being developed by unit weight, assuming complete gasification and adiabatic expansion.)	Kilogram-metres.	372,279·6	464,992·5
Volume of gas produced from 1 grm. mol.	C.c. (at 0° and 760°).	312,338	156,240
Volume of gas from 1 kilo.	„	683,453	1,132,220
Temperature of explosion........	° C.	2,832	2,141
Pressure per sq. cm. developed by the explosion of unit weight (1 grm.) in unit volume.	Kilos.	8,080	10,173

—J. S.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

Asbestos, Retention of Moisture by. G. Auchy. J. Amer. Chem. Soc. 1900, **22**, [1], 46—47.

The author shows, in a series of experimental determinations, that asbestos, or at any rate some varieties of it, retains moisture with great tenacity. For example, a previously ignited and purified sample, after being ignited in a platinum boat in a porcelain combustion tube in a current of oxygen, and then dried for 3 hours at 100° C., weighed 21·1742 grms. On now igniting it for one hour, it lost an additional 1·6 mgrm. Similarly, another sample dried for two hours at 100° C., and then ignited for 15 minutes, lost 1·1 mgrm. in the second process. The conclusion is, therefore, arrived at, that in drying a precipitate in a Gooch crucible, a blank determination should be made at the same temperatures and for the same length of time as is intended with the precipitate. Or, which is more convenient, the amount of moisture retained by the asbestos in the Gooch crucible may be determined once for all, and a correction made for it in each determination.

—C. A. M.

INORGANIC CHEMISTRY.—QUALITATIVE.

Silver; Detection of, in Presence of Mercurous Salts. F. Leteur. Comptes Rend. **130**, [5], 248—250.

When a mixture of silver and mercurous salts is precipitated as chlorides, and the precipitate treated with ammonia, some of the silver chloride always remains, even after prolonged washing with the mercurous chloride; so that if the proportion of silver salt to mercury salt be below one-fifth, it may be altogether overlooked. To ensure the detection of silver in these cases, the residue, after the treatment of the chlorides with ammonia, should be warmed with nitric acid, and enough hydrochloric acid added to convert the mercurous chloride into mercuric. Any silver chloride present will then be left undissolved as a white residue, not blackened, but completely dissolved by ammonia.—J. T. D.

INORGANIC CHEMISTRY.—QUANTITATIVE.

Carbon Monoxide; Volumetric Determination of Small Quantities of. L. P. Kinnicutt and G. R. Sandford. J. Amer. Chem. Soc. 1900, **22**, [1], 14—18.

After pointing out that air containing very small amounts of carbon monoxide may have an injurious effect if breathed for some time, the authors review the methods which have been proposed for the detection and estimation of that gas.

Those based on the formation of carbon monoxyhæmoglobin are, in their opinion, only of value as qualitative tests, whilst of the various methods based on the oxidation of the carbon monoxide to carbon dioxide, they regard that in which iodine pentoxide is used as the oxidising agent as the most satisfactory for the estimation of small quantities.

Nicloux estimated the liberated iodine colorimetrically and Gautier measured the quantity of carbon dioxide formed, but the authors have been unable to obtain accurate determinations of small amounts of carbon monoxide by either method. They have, however, obtained good results by titrating the iodine with a N/1000 solution of sodium thiosulphate in the following manner :—

Twenty-five grms. of iodine pentoxide were placed in a small U-tube which was suspended in an oil bath heated to 150° C. This tube was connected with a Wolff blood-absorption tube containing 0·5 grm. of potassium iodide dissolved in 5 c.c. of water. Its other limb was connected with two small U-tubes, one containing sulphuric acid, and the other fragments of potassium hydroxide, and the air passing through these was freed from all unsaturated hydrocarbons, hydrogen sulphide, sulphur dioxide, and other reducing gases before coming into contact with the iodine pentoxide.

The iodine pentoxide was prepared by the action of nitric acid on resublimed iodine, and after purification in the usual manner was freed from the last traces of iodine by being heated to 150° C. in a U-tube through which a current of air freed from all reducing substances, was passed.

In the authors' test determinations, the air (250 to 1,000 c.c.) was mixed with known quantities of carbon monoxide, and passed through the apparatus at the rate of about a litre in two hours. The results, which are given in tabular form, are in close agreement with the theoretical amounts, and show that the method is capable of determining as little as 0·0025 per cent. by volume of carbon monoxide in air.

It was also proved experimentally that the determination was not affected by the presence of the other constituents of coal-gas, and the authors conclude from their results that if the presence of carbon monoxide in the air be due to a leakage of coal-gas, and the amount of carbon monoxide in the latter be known, an approximate estimation of the amount of leakage may be obtained by their iodometric method.—C. A. M.

Magnesium; Volumetric Determination of. J. O. Handy. J. Amer. Chem. Soc. 1900, **22**, [1], 31—39.

THE magnesium is precipitated in the usual manner as magnesium ammonium phosphate; the precipitate is filtered off, thoroughly washed with 10 per cent. ammonium hydroxide, and after the liquid has drained off, the filter is transferred to a 5-in. filter paper, in which it is left open and exposed until most of the moisture has been absorbed. After about three minutes the foregoing operation is repeated with a fresh filter paper, and the whole is again left to dry. In the case of heavy precipitates this drying with a filter paper would be once more repeated. The filter with its precipitate is finally dried at the temperature of the room, or in an oven at 50°—60° C., until the paper is quite dry for half an inch from the margin inwards, which may be taken as an indication that all free ammonia will have been expelled.

The filter and precipitate are next treated with a measured excess of decinormal sulphuric acid in a small beaker, and stirred until the paper is disintegrated and the precipitate dissolved. Two drops of 0·1 per cent. alcoholic solution of methyl orange are added, and if the liquid does not turn a decided pink, an additional 5 c.c. of the standard acid are added. Finally, the liquid is diluted to 100 c.c. and titrated back with decinormal sodium hydroxide solution.

The author has determined experimentally that 1 c.c. of decinormal sulphuric acid is equivalent to 0·002 grm. of magnesium oxide, and he gives a table of results to show that the method described above is exceedingly accurate.
—C. A. M.

Aluminium; A New Method of Determining. A. Stock. Comptes Rend. **130**, [4], 175—178.

WHEN a mixture of iodate and iodide of potassium is added to a solution of an aluminium salt, a precipitate of aluminium hydrate is formed and a quantity of iodine set free according to the equation—

$$Al_2(SO_4)_3 + 5KI + KIO_3 + 3H_2O = 2Al(OH)_3 + 3K_2SO_4 + 6I.$$

The reaction, although commencing rapidly in the cold, is not complete for some days, especially in dilute solutions. The rapidity is increased if the liberated iodine be got rid of by the addition of sodium thiosulphate, especially when warmed. By heating the solution on a water bath, the reaction is complete in a few minutes, and since this occurs even in very dilute solutions, it may be used for the estimation of aluminium.

The determination of aluminium by means of ammonia of sodium thiosulphate compares unfavourably with this method, both in the washing of the precipitate, and in the ease of working. The solution must be neither too acid nor alkaline. After nearly neutralising with sodium hydrate, the reagent consisting of a mixture in equal parts of a 25 per cent. solution of iodide and a saturated solution of iodate, containing about 6 or 7 per cent. of the salt, is added. After five minutes the solution is decolorised with a 20 per cent. solution of sodium thiosulphate, and a further quantity of the reagent added to make sure of complete precipitation. The flocculent precipitate, after warming on the water bath for half-an-hour, is filtered off and treated in the same manner as that obtained by the ammonia method.

The presence of boric acid does not appear to affect the results, but the method is not available in a solution containing tartaric, oxalic, or phosphoric acids.

When precipitating aluminium with ammonia in presence of sulphate, the aluminium hydrate always retains a little sulphuric acid; this does not occur, however, with this method, unless the quantity of sulphate present be excessive. The absence of sulphur demonstrates that the thiosulphate added does not complete the precipitation.

The method not only lends itself to the estimation of aluminium, but also to that of iron, and possibly of other metals.—J. W. H.

Graphite in Pig Iron; Determination of, by Loss. G. Auchy. J. Amer. Chem. Soc. 1900, **22**, [1], 47—48.

IN this determination the author prefers to use asbestos in a Gooch crucible or Hirsch and Shimer's funnel for the filtration rather than counterpoised filter papers. In the ignition the graphite part of the asbestos is pressed against the wall of the crucible.

The author obtained somewhat varying results in this method of loss on ignition, which he considers probably due to the fact that graphite thus obtained is not always pure carbon, but may contain certain combinations of hydrogen, oxygen, nitrogen, and sulphur.

By combustion in the usual manner, the results obtained were 3·21 and 3·24 per cent., whilst by loss on ignition the amounts found were 3·35, 3·36, 3·38, and 3·20 per cent.

In the author's opinion, however, the method may serve for smaller percentages.

Influence of Gelatinous Silica.—Tamm found that this was not dehydrated in the drying of the graphite, and his statement is confirmed by the author, who found that about 0·13 grm. of gelatinous silica retained 0·0094 grm. of water when dried in the same manner as the graphite is dried in determining the loss upon ignition.—C. A. M.

Alkali Persulphates and Hydrogen Peroxide; Valuation of. B. Grützner. Arch. der Pharm. **237**, 705—706; Chem. Centr. 1900, **1**, [7], 435.

THE author has discovered that arsenious acid is completely oxidised to arsenic acid by alkali persulphates in alkaline solution—

$$2K_2(SO_4)_2 + As_2O_3 + 2H_2O = 2H_2SO_4 + 2K_2SO_4 + As_2O_5.$$

In applying this reaction, about 0·3 grm. of the alkali persulphate is heated gradually to boiling with 50 c.c. of 1/10 normal As_2O_3 solution and a few grms. of potash or soda-lye, then digested for a short time, allowed to cool, the liquid made faintly acid with sulphuric acid, then strongly alkaline with sodium bicarbonate, and the excess of arsenious acid titrated back with 1/10 normal iodine solution. The quantity of hydrogen peroxide may be determined in the same way, 1 c.c. of the commercial product being diluted with 10 c.c. of water. The results are stated to be accurate. The method cannot be used for the examination of sodium peroxide, as the evolution of oxygen, on the addition of water, is too violent.—A. S.

Nitrates; New Method for the Determination of. C. M. van Deventer. Zeits. physik. Chem. **31**, 50—58; Chem. Centr. 1900, **1**, [4], 265.

The author makes use of the brown coloration formed by the reaction between nitrates and ferrous sulphate. This coloration is only permanent when an excess of ferrous sulphate is present.

The tube of a stoppered separating funnel is dipped into a pneumatic trough filled with mercury, and 1 c.c. of bromoform is allowed to flow down the tube to prevent reduction of the nitric acid by direct contact with the mercury. 5 c.c. of the solution containing the nitrate are then run in, the funnel rinsed with a little water, and about 8 c.c. of concentrated sulphuric acid added. The ferrous sulphate solution, which has been standardised by means of a solution containing a known quantity of nitrate, is added from a burette. The outlet tube of the burette is bent upwards, and is introduced into the mercury trough immediately under the opening of the tube containing the nitrate solution. The ferrous sulphate solution is then allowed to rise through the mercury. Towards the end of the reaction, a furthur 8 c.c. of sulphuric acid is added, and then the ferrous sulphate solution in small quantities, till the liquid has acquired a permanent reddish-brown colour. As a confirmation of the end of the reaction, the liquid is tested with potassium ferricyanide for an excess of ferrous sulphate. Six molecules of ferrous sulphate are equivalent to two molecules of nitric acid.—A. S.

Chlorine and Iodine; New Method of Separating. L. Vanino and O. Hauser. Ber. 1900, **32**, [18], 3615—3617.

This method is based on the difference in the behaviour of these halogens towards an alkaline solution of formaldehyde, the chloride being rapidly and completely reduced to metallic silver, whilst the iodide is not attacked.

In making a separation, the solution of the halogens is treated with silver nitrate, and the precipitate washed with hot water, as far as possible by decantation. It is then treated in the beaker with 25 c.c. of a solution of 50 grms. of potassium carbonate in 100 c.c. of water and 5 c.c. of a 42 per cent. solution of formaldehyde, and allowed to stand until bubbles of carbon dioxide cease to rise. Initial warming to 30°—40° C. accelerates the reaction, which is usually complete in about 30 minutes. Any particles of chloride on the filter are reduced to metallic silver, by means of a jet, with the reagent heated to 40° C.

The liquid in the beaker is then filtered off through the same filter, the silver washed with hot water, dissolved in hot dilute nitric acid, and the solution filtered until perfectly clear. The silver iodide left on the filter is washed, dried, and weighed in the usual manner. The silver in the filtrate when precipitated with hydrochloric acid gives the quantity of chloride originally present.

The accuracy of this method is shown by a number of test results, which are in very close agreement.

Action of the Reagent on Bromides.—The authors show that there is too little difference between chlorides and bromides or bromides and iodides as regards their behaviour towards the reagent, to allow of any separation being made. —C. A. M.

Water; Detection and Estimation of Nitrites in Potable, by Means of 1.1′.4.3′-Amidonaphthol Disulphonic Acid [Amidonaphthol K-Sulphonic Acid]. H. Erdmann. Zeits. f. angew. Chem. 1900 [2], 33—35.

The author recommends a method for the determination of nitrites in water containing more than 1 centigrm. of nitrite-nitrogen per cb.m. The method depends upon the property possessed by 1.1′.4.3′-amidonaphthol disulphonic acid of combining in an acid solution with a diazo compound to form a strong azo dyestuff of characteristic shade. The new acid is obtained by nitrating, reducing, and heating with soda lye 1.3.4′-naphthalene trisulphonic acid (Ber. **32**, [16], 3186; this Journal, 1900, 39). The test is carried out as follows:—50 c.c. of the water under examination are mixed with 5 c.c. of a hydrochloric acid sulphanilic acid solution (2 grms. of crystallised sodium sulphanilate per litre), and after 10 minutes about 0·5 grm. of solid 1.1′.4.3′-amidonaphthol sulphonic acid (as acid alkali salt) are added. If nitrous acid be present, a bright Bordeaux red coloration appears which attains its maximum in one hour. This shade is compared with a standard tint obtained from a freshly prepared millionth, hundred thousandth, or ten-thousandth normal nitrite solution, or else with a scale of shades on paper. The new method possesses advantages over those hitherto in use, in that the acid employed does not fluoresce under the conditions of the test, and gives with oxidising agents, a yellow tint which cannot be mistaken for the red produced by nitrites. —T. A. L.

Nitrous Acid; A New Reagent for the Detection and Estimation of Traces of. H. Erdmann. Ber. 1900, **33**, [1], 210—215. (See preceding abstract.)

Iodic Acid in Nitrate of Soda, Estimation of. R. Auzenat. Monit. Scient. 1900, **14**, 72.

"Crude nitrate of soda" often contains as much as 1—2 per cent. of sodium iodate, and the accurate estimation of the latter is therefore a matter of importance.

Rammelsberg's method, although up to the present only applicable where all acids which would react with potassium iodide are absent, has now been somewhat modified so as to admit of the estimation of iodates in the presence of nitrates, &c.

On the addition of sulphuric acid to a mixture of iodate and iodide, the iodic acid set free reacts on the iodide with the liberation of iodine, and the latter may be removed by agitation with such suitable solvents as carbon bisulphide or benzene. But in presence of nitrates, from which the nitric acid is liberated by even dilute sulphuric acid, the potassium iodide is at once decomposed, and potassium chlorate, which is also to be found in crude nitrate of soda, acts in a similar manner.

Acetic acid however has no action on the nitrates, and therefore if it replace the sulphuric acid, the mutual decomposition of iodic acid and iodide takes place as desired, and an iodine solution of a caramel-yellow tint is obtained.

The estimation is carried out as follows:—six test tubes —length, 18 cm.; diameter, 25 mm.; and graduated up to 50 c.c.—are placed over white paper in a filter stand.

Into the 1st, as "standard," 10 c.c. of potassium iodate solution (1 gram per litre) are run; whilst, of crude nitrate of soda solution (33 grams per litre), the 2nd, 3rd, 4th, 5th, and 6th receive 30 c.c., 35 c.c., 40 c.c., 45 c.c., and 50 c.c. respectively. After filling all the tubes up to the mark with distilled water, 2 c.c. of a 10 per cent. solution of potassium iodide are added to each, and finally—with all possible expedition—five drops of glacial acetic acid.

The coloration appears at once, and is uniformly distributed by shaking the tubes slightly. The tubes should be allowed to stand for 10 minutes before comparing the tests with the standard. Should none of them approach the latter in depth, a fresh standard of a suitable strength must be taken and the experiment repeated, care being taken to carry it through under exactly the same conditions as before, since time especially is an important factor in the results, and therefore omissions in, *e.g.*, filling each one of the tubes each time, could modify the result.

Under the above conditions, the estimation may be made as rapidly and as accurately as a determination of ammonia by means of Nessler's reagent.—J. A. P.

Cerium; Volumetric Estimation of. P. E. Browning. Chem. News, 1900, **81**, 30—31 and 41—42. (Amer. J. of Science, 1899, **8**, [48].)

1. *A Modification of Bunsen's Method.* (With G. A. Hanford and F. J. Hall.) Bunsen's method (Annalen der Chemie, **105**, 49) consisted in heating the ignited CeO_2 along with KI and HCl in a flask, from which all air had been expelled previously by means of CO_2, and after all the CeO_2 had dissolved, titrating the liberated iodine. The reaction is the following—

$$2CeO_2 + 8HCl + 2KI = 2CeCl_3 + 2KCl + 4H_2O + I_2.$$

In the authors' experiments, about 0·1 to 0·2 grm. of pure CeO_2, obtained by igniting the oxalate, was placed in a stoppered bottle of about 100 c.c. capacity, along with 1 grm. of pure KI and a few drops of water. Air-free CO_2 was passed into the bottle for five minutes to expel the air, 10 c.c. of pure strong hydrochloric acid were added, the stopper inserted, and the bottle heated gently until all the CeO_2 had dissolved. After cooling, the liberated iodine was determined with thiosulphate. Blank tests in the absence of CeO_2 showed that a deduction of 0·04 c.c. of N/10 iodine solution had to be made. In 18 determinations of CeO_2 in this way, the extremes of error were + 0·98 and − 1·23 per cent.

In a further modification of the test, about 0·1 to 0·2 grm. of CeO_2 was placed, along with 15 c.c. of water, 1 grm. of KI and in the retort of a distilling apparatus, consisting of a Voit flask sealed to the inlet tube of a Drexel wash-bottle, the outlet tube of which was sealed to a set of absorption bulbs, the wash-bottle and bulbs containing dilute KI solution. CO_2 was passed through the apparatus, 10 c.c. of pure strong HCl added, and subsequently the liquid was boiled down to 15 c.c. in volume in a current of air-free CO_2. The free iodine in the receivers, and also the small amount remaining in the distilling flask, were titrated with thiosulphate. The results obtained in 12 determinations were slightly too low, the error ranging from 0·09 to 1·46 per cent.

2. *Action of As_2O_3 upon CeO_2.* (With W. D. Cutter.)—The fact that CeO_2 is reduced by HI as above, suggested that arsenious oxide in acid solution might also reduce it, but the results showed an incomplete reduction of the dioxide by the As_2O_3.

3. *Estimation of Cerium Oxalate by Permanganate.* (With L. A. Lynch.)—Stolba has stated that cerium may be estimated by titration of the oxalate by permanganate solution, as in calcium determinations. The authors have tested the method and find it to give fairly good results; apparently no oxidation of the cerium to the ceric state takes place at the expense of the permanganate.—H. B.

Sulphur; Estimation of Gaseous Compounds of, in Gaseous Mixtures. E. J. Russell. Proc. Chem. Soc. **16**, [219], 41.

In this paper, volumetric methods of analysis are given which have been found to work satisfactorily in the estimation of sulphur dioxide, hydrogen sulphide, carbonyl sulphide, and carbon bisulphide in gaseous mixtures. It is found that, except in the case of sulphur dioxide, absorption methods are not reliable; as a rule, by far the most satisfactory method is to explode with oxygen. In this explosion sulphur dioxide and sulphur trioxide are both produced, the amount of the former being determined directly, and that of the latter calculated from the contraction observed after explosion. Sulphur dioxide is best estimated by absorption with a pellet of lead peroxide, since this substance, if freshly washed, and not too dry, exerts but little of the surface action which tends to cause some of the sulphur dioxide to combine with part of the oxygen to form sulphur trioxide.

ORGANIC CHEMISTRY.—QUALITATIVE.

Caro's Reagent; Action of on Ketones. A. Baeyer and V. Villiger. Ber. **33**, [1], 124—126. (See also this Journal, 1900, 179.)

The active principle of Caro's reagent appears to be a compound of sulphuric acid and hydrogen peroxide, since a solution having all the properties of this reagent, is obtained by adding concentrated sulphuric acid to an aqueous solution of hydrogen peroxide, or by dissolving sodium or barium peroxide in concentrated sulphuric acid. The authors term the acid so obtained "Caro's acid," and consider it to be an analogue of chamber crystals. For its preparation, commercial hydrogen peroxide (containing about 5 per cent.), is well cooled and treated with 5 times its weight of concentrated sulphuric acid. On adding 1 drop of acetone to about 1 c.c. of the reagent cooled with ice, an immediate precipitate of acetone peroxide, melting at 133° C. is obtained. The reaction is sufficiently delicate to serve as a test for acetone, and gives a precipitate with 1 drop of a solution of 5 parts of acetone in 100 parts of alcohol. Tetrahydrocarvone is converted by the new reagent into the corresponding lactone. With regard to the action of the reagent on higher homologues of acetone, the authors treated diethyl- and dipropyl-ketone in a similar manner, and find with these compounds also, that whilst evidently the first products of the reaction are simple peroxides, these polymerise more or less readily, but by the action of concentrated sulphuric acid, can again apparently be converted into simple peroxides. Thus, to put the matter briefly, by the action of Caro's reagent on ketones with open chain, the simple ketone peroxides are produced, which act on potassium iodide. These then, more or less easily pass into polymeric forms, which (polymeric ketone peroxides) by the action of concentrated sulphuric acid are apparently re-converted into the simple forms.—T. A. L.

Nicotine; Detection of. J. Schindelmeiser. Pharm. Centr.-H. **40**, 703; Chem. Centr. 1900, **1**, [1], 67.

The author has discovered a colour reaction by means of which 0·0005 grm. of nicotine can be detected. If nicotine, which has not been resinified, be treated with a drop of 30 per cent. formaldehyde free from formic acid, and then a drop of concentrated nitric acid be added, the solution is coloured an intense rose-red. If the mixture of nicotine and formaldehyde be allowed to stand for a few hours, a solid residue is obtained, which, on the addition of a drop of nitric acid, exhibits the colour reaction better than the solution. 0·005—0·1 grm. of nicotine gives a dark red, whilst nicotine which has been resinified gives a blood-red coloration. Only a small quantity of formaldehyde should be taken, otherwise the solution, after some time, becomes green and explodes. The mixture of nicotine and formaldehyde must not be evaporated on the water bath, owing to the danger of decomposition with explosion. Concentrated formic acid may be used in place of formaldehyde. The reaction is not so sharp with acetaldehyde or concentrated acetic acid.

Piperidine, trimethylamine, pyridine, quinoline, picoline, and aniline do not give the reaction.—A. S.

Balsam of Tolu. J. Spilsbury and T. G. Joyce. Pharm. J., **64**, (1545), 93—94.

Although the authors confirm the statement of Braithwaite, that treatment with carbon bisulphide and the determination of the amount and saponification number of the matter removed by that solvent will differentiate between spurious and genuine Tolu balsam, they point out that this method will not suffice for the detection of "exhausted" balsam, which has been treated with water. They suggest that, instead of calculating the saponification number, the acidity of the residue should be expressed as percentages of cinnamic acid on the original balsam. The number of samples examined by them is not, as yet, sufficient to permit of the fixing of a minimum limit, but the experiments show that this should probably not fall lower than 18 per cent.—J. O. B.

ORGANIC CHEMISTRY.—QUANTITATIVE.

Cresols; Application of the Bromine Substitution of, for their Quantitative Determination. R. Clauser. Oesterr. Chem. Zeit. **2**, 585—588. Chem. Centr. 1900, **1**, [2], 118.

The author has investigated the sources of error in the determination of the cresols by Koppeschaar's method. According to his experiments, *o*-cresol may be determined with a maximum error of + 0·5 per cent., if the cresol be used in a dilution of from $\frac{1}{30000}$ to $\frac{1}{40000}$, and so much bromide-bromate solution be added that 6 atoms of bromine are present for each molecule of cresol, the reaction being stopped by the addition of potassium iodide, as soon as the precipitate begins to be coloured yellow, and the solution being then allowed to stand for half an hour.

—A. S.

Vinegar; New Method for the Acetimetric Valuation of. Durieu. J. Pharm. Chim. **11**, [1], 22—23.

The method described is advocated as being applicable to coloured vinegars. Two solutions are prepared, the first a

5 per cent. solution of sodium bicarbonate; the second, a 7 per cent. solution of acetic acid. The apparatus requisite consists of a tube, graduated in 0·1 c.c. such as is used in the determination of urea by Esbach's method, and a 1 c.c. pipette. 6 c.c. of the bicarbonate solution are first introduced into the tube, followed by 6 c.c. of 95 per cent. alcohol, so added that the two liquids do not mix, and 1 c.c. of the dilute acetic acid solution is then added, and the whole well shaken. When reaction is complete, the opening of the tube is placed under water, and the volume occupied by the gas read off, as in making a determination of urea. The volume is noted, and a second experiment is made with a similar quantity of the vinegar to be examined. The volume of gas evolved by this, compared with the volume given by the standard acetic acid solution, will give the amount of acetic acid in the sample. It is of course first necessary to determine that the whole of the free acid in the vinegar is acetic acid.—J. O. B.

Tanning Materials and Basic Dyestuffs; Ullmann's Method for the Valuation of. A. Heinemann. Chem. Zeit. **24**, [7], 58.

Ullmann proposes (this Journal, 1900, 76) to utilise the property which various tanning materials possess, of precipitating the basic dyestuffs as insoluble tannin "lakes," for the valuation of both classes of bodies (tannins and basic dyestuffs), corrections, depending on colorimetric examination of the filtrate for unprecipitated dyestuff, being subsequently made.

It is much to be doubted whether the cotton-mordanting and "lake"-forming tannins are identical. The relative affinities, for the vegetable fibres, of a commercial tannin containing, say, 14 per cent. of "non-tannin" (soluble matter not removable by hide-powder) and of the so-called "pure tannic acid" containing 95 per cent. of precipitable tannin, are by no means as 86 : 95, the commercial tannin often yielding a better result than the pure article.

On the other hand, where the tannin is to be fixed on an iron mordant, *i.e.*, as an insoluble iron "lake," the pure tannin gives the better result. This apparent contradiction is, however, easily explained on the assumption that tannin is no uniform substance, but owing to partial decomposition, a mixture of complex substances with simpler ones, products of the subdivision of their molecules. The simplest of these, gallic and digallic acids, possess absolutely no affinity for cellulose, but are still capable of producing "lakes," and of dyeing on an iron mordant.

Again, the different commercial tannins contain gallic acid, but in very varying proportions.

Therefore, since gallic acid is generally recognised as valueless as a cotton-mordant, it seemed advisable to determine its behaviour, when submitted to the tannin tests of Ullmann. The experiments proved, however, that gallic acid possesses lake-forming powers, hence Ullmann's method based upon this property as an index of mordanting value, is deceptive.

An attempt of the author's to effect separation of tannic and gallic acids by means of basic dyestuffs proved unsuccessful; in one case, owing to retention in solution of a portion of the tannin, by acid set free from the dyestuff; in the other, because subsequent neutralisation of this acid resulted in precipitation of an alkali-gallate.

In the colorimetric examination of the filtrate after precipitation, a neutral "standard solution" is compared with a sample of the filtrate, necessarily acid (acid being liberated from the dyestuff during precipitation); and it is only when carried out under the same conditions that such colorimetric tests can be considered accurate, a mere trace of acid being sufficient to seriously affect the tint of the necessarily weak solution examined.

In any case where using the "Ullmann Method," it would at least be necessary to acidify the "standard" to the same degree as the filtrate compared with it.

It must also be remembered that any precipitation-method of tannin-analysis reveals nothing as to the effect on the ultimate dyed shade of the tannin in question.

For all the above reasons, the original method of mordanting and dyeing on the small scale, as far as possible on the same lines as in the factory, seems by far the more preferable.—J. A. P.

Glue; Examination of, and Determination of the Gelatin in. A. Ruffin. Ann. Chim. anal. appl. **4**, 401—404. Chem. Centr. 1900, **1**, [3], 233.

The author has found, in practice, that the best method for the determination of gelatin in glue, is that of Jean (this Journal, 1897, 364), in which the gelatin is precipitated with a known quantity of tannin, the excess of the latter being titrated back with iodine. The methods of Groeger—weighing the precipitated gelatin tannate; and of Beugnat—titration of the solution of glue with a standardised tannin solution, yield only approximate results.—A. S.

Mechanical Soil Analysis; Rapid Method of [*Centrifugal*]. C. G. Hopkins. 15th Annual Conv. of Official Agricult. Chem., Washington, 1898. U.S. Dept. Agric. Bull. 1899, [56], 67—68.

Ten grms. of soil are placed in a small sieve with circular perforations of 0·32 mm. diam. The sieve is then placed in a porcelain basin, and the fine earth washed through with successive portions of water with the aid of a rubber-covered rod or pestle. The gravel and coarse sand are then removed, separated, and weighed. The washings containing the fine earth, amounting to 300—400 c.c., are placed in a bottle and agitated in a shaking machine until the earth is completely disintegrated. About 125 c.c. of the contents of the bottle are transferred to a flat-bottomed cylinder (40 mm. diam. and 160 mm. high), and whirled in a centrifugal until all particles coarser than 0·001 mm. diam. have been deposited. The time and speed necessary for this must be determined for each machine by microscopic tests. The water and suspended clay are siphoned into a beaker without disturbing the sediment. The sediment is then broken up with a rubber-covered rod, and the operation is repeated with the further contents of the bottle, and subsequently with sufficient water to wash all the clay out of the sediment. The clay is determined by evaporating the water in which it is suspended, and the sediment is then again shaken up with water and whirled for a shorter time at a reduced speed, so determined that particles coarser than 0·0032 mm. shall remain behind. When nothing more remains in suspension at this latter speed, a further fraction, from 0·01—0·0032 mm., can be obtained at a still lower speed. Further fractionation is effected by gravitation. The results afforded by the above method are very accurate as shown by tables, and an analysis can be completed in two days. —J. F. B.

Sugar Analysis [*Beet Molasses*], *Report on.* R. S. Hiltner. 15th Ann. Convention of Offic. Agricul. Chem. Washington, 1898. U.S. Dept. Agric. Bull. 1899, [56], 87—96.

The author reports on comparative analysis by various chemists of a sample of beet molasses, supplied to them with instructions as to the methods to be employed.

Moisture.—The provisionally official method of Carr and Sanborn, and the method of drying down with quartz sand were prescribed. The results were equally good, and the averages were identical; it is recommended that Carr and Sanborn's method be made official and the quartz method be made optional. The time required for desiccation bears no direct relation to the quantity of molasses used, but depends on the nature of the drying oven, and on the proportion of pumice or quartz employed. It is pointed out that the moisture cannot be determined accurately by dilution and specific gravity in the case of low-grade products.

Ash.—No modification is suggested in the standard methods in use at the present time. In the case of the method by incineration with zinc oxide, the author points out that the official directions provide for only one-tenth of the proportion of zinc oxide recommended by Lucien in his original description of the method, and suggests a typographical error.

Reducing Sugars.—Only "gravimetric" methods are permissible; the cuprous oxide may be determined either by electrolytic deposition by Formanek's method or may be titrated by standard permanganate, the results being strictly identical.

Saccharose and Raffinose.—The results of the various chemists are sufficiently concordant for the direct polarisation, but vary in a most inexplicable manner in the case of the polarisation after inversion. The author suggests that small irregularities in carrying out the inversion have very great effects on the results. Clerget's formula is not applicable in presence of raffinose, and Creydt's formula should be adopted.—J. F. B.

Nicotine in Tobacco Powders; and Extracts; Report on Methods for Determination of. E. A. de Schweinitz, J. A. Emery, and F. K. Cameron. 15th Ann. Conv. of Offic. Agricult. Chem., Washington, 1898. U.S. Dept. Agric. Bull. 1899, [56], 113—126.

After exhaustive experimental investigations the authors state the following conclusions:—Up to the present time no reliable method for the separation of nicotine from ammonia and other organic bases by precipitation has been suggested. A complete separation of nicotine by ether and some other solvents is readily accomplished; evaporation of the ethereal extract affords a practicable separation from ammonia alone, but not from other organic bases. In a separation by steam distillation, it is preferable to keep the volume in the distilling flask approximately constant, and a much larger quantity of distillate being really required than is generally supposed. No method of separation by decomposition of accompanying bodies has been found practicable. A separation of nicotine from ammonia by means of alcoholic oxalic acid can be effectively accomplished. The presence of tertiary amines and possibly some pyridine derivatives in tobacco extracts, is as yet an insurmountable difficulty in the separation or estimation of nicotine. Prof. Winton has reported an abbreviation of the Kissling method for the analysis of tobacco extract for commercial purposes. From 1 to 5 grms. of the extract are weighed out into a flask of 400 c.c. capacity, having a neck 3 cm. in diameter. If the solution be too viscous to flow readily, it may be diluted with 1 c.c. of water. One c.c. of saturated caustic soda solution is then added and mixed as intimately as possible with the extract by gentle agitation. Experiments have proved that under the above conditions the separation of the ammonia by evaporation into the air is sufficiently complete for practical purposes, if the flask be exposed for 24 hours at the ordinary temperature without a cork; errors are stated to be due to pyridine derivatives not to ammonia.—J. F. B.

Syrups and Similar Products; New Method of Determining the Water in. O. Molenda. Oesterr.-Ung. Zeits Zucker. Ind. u. Landw. 28, 621—625. Chem. Centr. 1900, 1, [1], 71.

The author proposes a new method of determining water in syrups, &c., based on the formation of acetylene from calcium carbide by the action of the water. It proved impracticable to determine the amount of acetylene by weighing, and the plan of reading off the volume was therefore adopted. The operation may be carried out in Scheibler's apparatus for the determination of calcium carbonate in animal charcoal. The apparatus must be carefully dried before being used. In applying the method, 0·09—0·12 grm. of the syrup, of a strength of from 20 to 27 saccharometer degrees, is taken. The calcium carbide is weighed off in special weighing glasses, and then introduced into the apparatus.

The method of calculating the percentage of water from the volume of gas read off is described in the original.

—A. S.

Wine; Detection and Estimation of Aldehydes in. M. Ripper. Zeits. Landw. Vers. Wes. Oest. 3, 26—34. Chem. Centr. 1900, 1, [7], 436.

In continuation of his former communication on the analysis of wines (this Journal, 1899, 387), the author gives the following method for the detection of aldehydes in wine. From 200 c.c. of the wine in question, about 20 c.c. are distilled off, and tested in turn, with the following reagents: phenylhydrazine hydrochloride, Guyon's reagent, *m*-phenylenediamine hydrochloride, ammoniacal silver solution, and resorcinol and hydrochloric acid. With all the wines examined by the author, the reactions peculiar to aldehydes or ketones were obtained, but it is undecided whether the aldehydes are present as such in the wine, or are formed as decomposition products on heating.

For the quantitative determination of aldehydes in wine, the following method is employed:—50 c.c. of the wine are treated with 5 c.c. of sulphuric acid (1:3), and titrated with $\frac{1}{50}$ normal iodine solution, with starch as indicator, to a permanent blue coloration, as in the determination of aldehyde-sulphurous acid. Then 50 c.c. of the wine are treated in a flask with 50 c.c. of $\frac{1}{50}$ normal potassium bisulphite solution, the flask corked up, and allowed to stand for 15 minutes. 5 c.c. of sulphuric acid (1 : 3) are then added, and the solution titrated with $\frac{1}{50}$ normal iodine solution. The difference between the amounts of iodine consumed by 50 c.c. of the wine before and after treatment with 50 c.c. of the potassium bisulphite solution, is obtained, and then the difference between this number and the amount of iodine required for 50 c.c. of the potassium bisulphite solution alone, indicates the proportion of aldehydes contained in the wine.—A. S.

Margarine; Detection and Estimation of Cane Sugar in. Mecke. Zeits. öffentl. Chem. 5, 496. Chem. Centr. 1900, 1, [7], 435.

In order to render margarine similar to natural butter, it is mixed with cane sugar and yolk of egg (this Journal, 1899, 948). The detection of cane sugar in presence of milk sugar is difficult, but may be accomplished by inversion with citric acid and polarimetric determination according to Dowzard's method (this Journal, 1899, 180). A polarimetric examination of margarine is not practicable, as clear solutions are difficult to obtain, and only a small amount of sugar is present. The author determined the amount of the two sugars gravimetrically before and after inversion with citric acid.—A. S.

Alkaloids in Leaves of Datura Stramonium, Hyoscyamus Niger, and Atropa Belladonna; Quantitative Determination of. E. Schmidt. Pharm. J. 1900, 64, [1542], 22. From Apoth. Zeit.

For the determination of alkaloids in drugs containing considerable amounts of chlorophyll, and yielding deep green coloured extracts to chloroform, the author recommends the following modification of Keller's method:—10 grms. of the finely powdered drug are dried to constant weight over caustic lime, then mixed with 90 grms. of ether and 30 grms. of chloroform, the whole well agitated, and 10 c.c. of 10 per cent. caustic soda solution added, whilst the shaking is continued. After standing for three hours, 10 c.c. of water is added, or enough to make the powder aggregate together, and then after well shaking, the mixture is allowed to settle, till the ether-chloroform solution separates clear on the surface. After about one hour, 60 grms. of the clear solution (= 5 grms. of leaves) is filtered, and the ammonia expelled by distilling to half the original volume; further distillation must be avoided, as it would be attended with formation of hydrochloride, in consequence of reaction with chloroform. The dark green liquid is well shaken with 10 c.c. of $\frac{1}{100}$ normal hydrochloric acid; if necessary, enough ether is added to make the ether solution float, then the acid layer is filtered through a small wetted filter. The ether-chloroform solution is washed with 10 c.c. of water, three times in succession, each washing being passed through the same filter, and the filter is washed with water till the acid solution measures about 100 c.c. Enough ether is added to give a layer of about 1 cm., then 5 drops of alcoholic iodeosin solution (1 in 500), and $\frac{1}{100}$ normal potash solution run in, with vigorous shaking after each addition, until the aqueous liquid acquires a pale red colour. It is advisable to add only 1 c.c. of potash solution at a time, and to shake well after each addition, until the aqueous liquid begins to appear faintly but distinctly red when observed on a white background, while the ether layer is, if necessary, covered with a piece of black paper; 1 c.c. of $\frac{1}{100}$ normal acid should then be added, and after shaking well, $\frac{1}{100}$ normal potash solution added in successive quantities of 0·1 c.c. until the aqueous liquid again acquires a faint red colour. One c.c. of $\frac{1}{100}$ normal acid is equivalent to 0·00289 grm. of atropine or hyoscyamine.

The iodeosin used as an indicator should be absolutely insoluble in water mixed with a trace of hydrochloric acid; also, the alcoholic solution of the iodeosin should be tested, in a blank experiment, as to its sensitiveness. The ether-chloroform solutions should be saved, and can be purified for further use by shaking with excess of dilute sulphuric acid, distilling from a water bath, drying the distillate with calcium chloride and again distilling. This second distillate is brought to a specific gravity of 0·850 by addition of ether or chloroform, and is then ready for use.

The following results were obtained by this method, the amounts of alkaloid in henbane leaves and stalks being very high as compared with the amounts found by other observers:—

	Per Cent. of Alkaloid.
Belladonna leaves:—	
Wild	0·40
Cultivated	0·26
Stramonium leaves grown in the Marburg Botanic Gardens	0·40
Hyoscyamus leaves (Cæsar and Loretz):—	
Leaves without stalk, I.	0·2762
" " " II.	0·2861
Leaf stalks, I.	0·363
" " II.	0·365

—A. S.

Aromatic Quinones and p-Diphenols, Volumetric Estimation of. A. Valeur. Bull. Soc. Chim. 1900, 23—24, [2], 58—61.

The method is founded on the instantaneous reduction of quinones by hydriodic acid, and titration of the liberated iodine, $C_6H_4O_2 + 2\ HCl + 2\ KI = C_6H_6O_2 + 2\ KCl + I_2$. A quantity of quinone capable of liberating 0·2—0·5 grm. of iodine is dissolved in a little alcohol, 20 cc. of 10 per cent. potassium iodide solution are added to a cooled mixture of 20 cc. of strong hydrochloric acid and 20 cc. of alcohol. This mixture is then added to the quinone solution, and the liberated iodine at once titrated by $\frac{1}{10}$ N. thiosulphate. The method appears to be applicable to the majority of the true quinones, the results quoted for ordinary quinone, 2·6-dichloroquinone, toluquinone and thymoquinone are satisfactory.

p-Diphenols may be estimated by the reverse reaction. In alkaline solution iodine oxidises them to quinones. The *p*-diphenol is dissolved in water, starch paste added, and then standard iodine solution with constant shaking, until the coloration is permanent.—A. C. W.

Liquorice Pastes; Technical Analysis of. M. Trubek. J. Amer. Chem. Soc. 1900, 22, [1], 19—21.

After several experiments the following method was worked out and found to give satisfactory results:—

Starch, Gum, &c.—Two grms. of the extract to be examined are mixed with 5 c.c. of water and stirred into an even mass on a warm plate. To this mixture 20 c.c. of 96 per cent. alcohol are added, and, after being left to settle, the precipitate is transferred to a weighed filter, and washed with a mixture of water and 96 per cent. alcohol (1 : 4) until the filtrate is colourless. The filter and its contents are dried for three hours at 105° C. and weighed, giving the gummy substances, starch, &c.

Glycyrrhizin.—The filtrate and washings are distilled or evaporated to a thick syrup of from 1 to 1·5 c.c. This is dissolved in 2 c.c. of glacial acetic acid and 30 c.c. of absolute alcohol added, with constant gentle stirring. The liquid is allowed to settle, filtered through a weighed filter, and the precipitate washed with absolute alcohol until free from acid, and dried at 105° C. and weighed. The glycyrrhizin salt thus obtained is a light yellow amorphous powder, with a very sweet taste. It is soluble in glacial acetic acid, hot and cold water, and dilute alcohol. In order to obtain the amount of the pure glycyrrhicic acid, an aliquot portion of the precipitate is incinerated with the aid of the blast flame at the end, and 0·7 of the percentage of the ash deducted from the percentage of the salt found. The ash consists principally of calcium oxide, with small amounts of alumina and magnesia.

Extractive Matters.—The filtrate from the glycyrrhizin is distilled, and the aqueous residue diluted and evaporated until the acetic acid has been expelled. The residue, which is dried at 105° C. for three hours and weighed, contains the saccharine matter originally in the root, or subsequently added to the paste, together with some tannin, resin, &c.

Ash and Total Solids.—Two grms. of the paste are incinerated in a platinum dish in order to obtain the ash. For the total solids about 5 grms. are dissolved in hot water, the solution made up to 500 c.c. when cold, and well shaken, 50 c.c. of the liquid are evaporated, and the residue dried for three hours at 105° C.

Solids Soluble in Cold Water.—These are determined by evaporating 50 c.c. of the clear filtrate, and drying the residue.

Moisture.—This is taken as the difference between the total solids and 100.

Reducing Substances are estimated by adding to the solution basic lead acetate, and titrating the filtrate with standardised Fehling's solution.

Representative Analyses.—The following results were obtained by the above method with different samples of liquorice extract:—

—	1.	2.	3.	4.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	24·18	25·14	19·58	23·99
Residue insoluble in cold water.	3·95	6·79	14·35	2·54
Residue insoluble in mixture of 1 water and 4 alcohol.	27·27	25·20	36·58	24·65
Glycyrrhizin	22·78	21·97	18·51	19·90
Extractive matters	25·02	25·05	26·02	28·91
Ash	6·25	6·02	5·60	5·59
Reducing substances as glucose.	..	..	..	9·76

Composition of Liquorice Root.—The subjoined results were obtained in the analysis of fresh and spent liquorice root—

—	Fresh.	Spent.
	Per Cent.	Per Cent.
Moisture	8·40	10·00
Total solids	33·18	10·86
Substances soluble in cold water	29·65	..
Gum, starch, &c.	8·07	3·56
Glycyrrhizin	11·21	2·25
Extractive matters	14·77	5·60
Ash	2·68	0·55
Reducing substances	0·52	..

—C. A. M.

XXIV.—SCIENTIFIC & TECHNICAL NOTES.

Silica, Optically negative form of Anhydrous. A. Lacroix. Comptes Rend, 130, [7], 430—432.

This mineral, found in the Upper Lutetian beds of Paris, in *amygdalites* from Madagascar, &c., is associated with crystalline quartz, and with calcedony, quartzite, and lutecite, but differs from all these in its optical characters. Its double refractive power (0·0045) is only half that of other forms of quartz, and it is optically negative. The author proposes to call it *pseudo-calcedonite*.—J. T. D.

Peroxides, and Conditions as to the Formation of Hydrogen Peroxide. S. Tanatar. Ber. 33, [1], 205—208.

All peroxides of the H_2O_2 type yield H_2O_2 on decomposition by acids. These have been called "true peroxides." Others, which may be called "pseudo peroxides," yield no H_2O_2, but mostly decompose it energetically. The former class all reduce, in acid solution, certain compounds rich in oxygen, such as MnO_2, PbO_2, $KMnO_4$; none of them can be produced by oxidation by means of nitric acid or hypochlorous acid. The classification of persulphuric acid presents difficulties, as it does not reduce $KMnO_4$, but under certain circumstances can produce H_2O_2.

The author questions whether it is necessary to assume a constitutional difference between the two kinds of peroxides, and suggests that perhaps thermochemical considerations alone can explain the differences in their reactions. Certain peroxides—those of Mn, Pb, Co, and Tl—could not yield H_2O_2 on treatmant with acids, as the heat of reaction is insufficient for the formation of H_2O_2. Of the "pseudo peroxides" only nickel peroxide could yield H_2O_2; it dissolves quickly in sulphuric acid, but as the slightest quantities of it decompose H_2O_2 readily, very little H_2O_2 could be expected to remain in the liquid. Nevertheless, if freshly prepared, nickel peroxide free from chlorine is suspended in water at 60°, and a little H_2SO_4 is added, the solution, after standing some time, will liberate some iodine from KI, as may be proved by the starch test. The solution will also decolorise a small quantity of $KMnO_4$. Though the formation of H_2O_2 from "pseudo peroxides" is precluded on thermochemical grounds, they can yield other peroxides of the H_2O_2 type when alkaline solutions are employed. Again, while it is true that "true peroxides" are not formed by means of oxidation with hypochlorous acid, one reaction apparently requires the assumption that the latter does oxidise $Ba(OH)_2$ to BaO_2; thus, if an alkaline solution of sodium hypochlorite is treated with $KMnO_4$ and $Ba(OH)_2$, the $KMnO_4$ is decolorised.—H. B.

Manganese Oxalate, Formation of Crystalline, in the Permanganic Oxidation of Citric Acid. G. Denigès. Journ. Pharm. Chim. 1900, **11**, [3], 102—104.

The formation of crystalline manganese oxalate by the oxidation of citric acid with potassium permanganate is shown by the following experiment. 90 grms. of potassium permanganate are dissolved in 3 litres of warm water, and the solution cooled to 15°—16° C. 300 grms. of citric acid are dissolved in 600 c.c. of warm water, and also cooled to the same temperature. This solution is added to the permanganate, the vessel in which mixing takes place being surrounded by water at 10° to 12° C. The mixture rapidly changes colour, and the temperature rises to 30°—35° C. and should not be allowed to exceed 40° C. When the evolution of gas begins to slacken, the mixture should be rapidly cooled at 25—28° C., and set aside.

In four or five days the sides of the vessel will be covered with well formed crystals. The mother liquor is decanted, the crystals washed and dried over H_2SO_4. They will be found to consist of two forms; the larger, rose coloured and prismatic, may be picked out from the smaller white hexagons, which occur in groups. The former on analysis will be found to contain 3 molecules of water of crystallisation, $MnC_2O_4 + 3H_2O$, the latter 2 molecules, responding to the formula $MnC_2O_4 + 2H_2O$. The author has also obtained the prismatic form by the slow double decomposition, in the cold, of a mixture of manganese sulphate and ammonium oxalate, and the white hexagonal form by the same means, with boiling solutions. From the solution resulting from the manganic oxidation of citric acid, the latter form with 2 mols. of H_2O. The salt is deposited whilst the mixture is warm; the larger prismatic crystals, with 3 mols. H_2O, are slowly formed in the cold, as the acetone-dicarboxylic acid at first formed, decomposes.

—J. O. B.

Rhodicyanides. E. Leidié. Comptes Rend. **130**, [2], 87—90.

Freshly precipitated rhodium hydroxide is dissolved in 30 per cent. solution of potassium hydroxide, the solution diluted with water till a permanent turbidity appears, and then poured into a quantity of 25 per cent. hydrocyanic acid more than sufficient to convert the whole of the hydroxides into cyanides. The liquid is kept for a while in a closed vessel, filtered, and allowed to evaporate *in vacuo* over sulphuric acid. The crystals so obtained, after two recrystallisations, are faintly yellow monoclinic prisms, agreeing closely in their crystallographic characters with ferricyanide or cobalticyanide of potassium. They are anhydrous, very soluble in water, decomposed by alkalis or strong hydrochloric acid, and give with iron or copper salts, precipitates resembling those of other complex cyanides. Analysis gives figures agreeing with the formula $K_3RhC_6N_6$

—J. T D.

Fatty Aldehydes (Formaldehyde) Action of Potassium Cyanide on [preparation of Glycollic Acid]. L. Kohn. Monatsh. für Chem. 1899, **20**, [10], 903—908.

Potassium cyanide reacts energetically with formaldehyde. If the solid cyanide be added to commercial aldehyde solution, keeping the temperature below 30° C. by external cooling, a thick colorless solution is obtained. This contains potassium glycollate and hexamethylenetetramine. The reaction proceeds according to the equation—

$$10CH_2O + 4KCN + 2H_2O = (CH_2)_6N_4 + 4CH_2OH.COOK.$$

Pure glycollic acid may be conveniently isolated and prepared from the solution by precipitating with lead acetate, collecting on a filter, and decomposing with hydrogen sulphide. No intermediate product has yet been isolated. If a solution of equivalent quantities of potassium cyanide and calcium chloride be dropped into formaldehyde solution, an energetic reaction occurs, and a white precipitate separates, this is a very unstable compound free from nitrogen. From the residual solution, calcium glycollate separates with evolution of ammonia. In this case hexamethylenetetramine is not formed.—A. C. W.

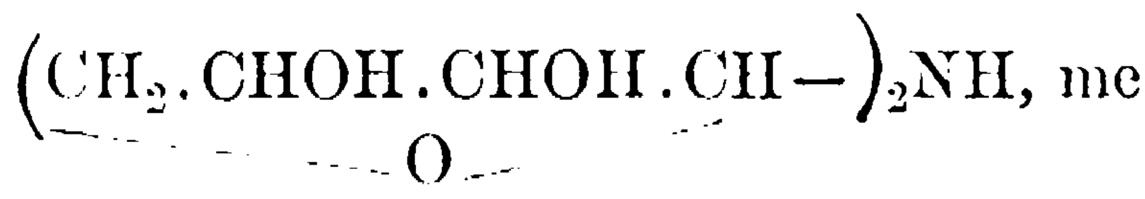
d-Erythrose. O. Ruff. Ber. 1899, **32**, [18], 3672—3681.

The passage from pentose derivatives to tetrose was effected by the author's general method, by oxidation of calcium *d*-arabonate with hydrogen peroxide in presence of ferric acetate. The crude erythrose was purified by the benzylphenylhydrazine method (see this Journal, 1899, 1141). The pure syrup was dried, but could not be induced to crystallise. The sugar showed multirotation with $[\alpha]_D^{20} = +1°$ at first, changing to a final value of $-14 \cdot 5°$; it reduced Fehling's solution faintly in the cold and strongly on heating; it was unfermentable by yeast. The osazone, crystallising from benzene in flexible needles, melted at 164° C. The crude *d*-erythrose syrup on reduction with sodium amalgam yields *i*-erythrite, which, after purification with alcohol or distillation under reduced pressure, melts at 120° C. This product was proved to be in every way identical with natural *i*-erythrite. Crude *d*-erythrose was oxidised with bromine in presence of water, the hydrobromic acid removed with lead and silver, and the *d*-erythronic acid isolated as the brucine salt, which had $[\alpha]_D^{20} = -23 \cdot 5°$. The free acid was liberated from the brucine salt by treatment with barium hydrate; it is strongly lævo-rotatory. When the free acid is heated, *d*-erythronic acid lactone is produced, obtainable in prismatic crystals, m.p. 103° C., and $[\alpha]_D^{20} = -73 \cdot 3°$. The calcium and barium erythronates, both crystallising with 2 mols. of water, are described. The free acid and the lactone both yield the phenylhydrazide, m. pt. 128° C., and $[\alpha]_D^{20} = +17 \cdot 5°$. Ruff and Neusser have isolated a trihydroxybutyric acid from the products of oxidation of fructose with mercuric oxide and baryta, which they prove to be identical with the *d*-erythronic acid described above.—J. F. B.

l-Arabinose; Degradation Products of [Pentose to Tetrose]. A. Wohl. Ber. 1899, **32**, [18], 3666—3672.

The stages through which the author proceeded from the pentose *l*-arabinose to the tetrose *l*-erythrose were as follows:—(1.) Preparation of *l*-arabinoseoxime by the action of hydroxylamine, the yield being almost quantitative. (2.) Acetylation of the oxime by acetic anhydride and sodium acetate, with formation of tetra-acetylarabonic nitrile, which may be formulated as a tetrose derivative—

$$CH_2(C_2H_3O_2).[CH(C_2H_3O_2)]_2CH\left\langle\begin{matrix}CN\\(C_2H_3O_2)\end{matrix}\right.$$

(3a.) On saponification with silver oxide and traces of ammonia, the nitrile yields *l*-triacetylerythrose, from which the free sugar might be prepared, but the yields in these two saponifications are so very poor that the method is not available. (3b.) If an alcoholic solution of the nitrile be treated with a solution of silver oxide in excess of ammonia under certain conditions, *l*-erythrosediacetamide is one of the principal products of the reaction. (4.) *l*-Erythrose can then be prepared from the acetamide by treating with normal sulphuric acid in a boiling water-bath. The author has not succeeded in obtaining the sugar in the crystalline state. *l*-Erythrosazone is prepared in the usual way by heating with phenylhydrazine acetate at 70°—80° C.; it crystallises directly, and can be recrystallised from water or benzene; it melts at 163°—164° C. (corr.). The products of saponification of the acetamide compound, after removal of the sulphuric acid by barium carbonate, contain sugar and ammonium carbonate. In the arabinose group the latter may be removed by long boiling, but in the erythrose group the two bodies combine, forming a crystalline compound, *l*-dierythroseimide  $\big(\underbrace{\text{CH}_2.\text{CHOH.CHOH.CH}}_{\text{O}}-\big)_2\text{NH}$, melting at 155° C., and strongly dextro-rotatory.—J. F. B.

Ferment, Hydrogenising, in the Animal Organism; Transformation of Nitrobenzene into Aniline by. E. Abelous and E. Gérard. Comptes Rend. **130**, [7], 420—422.

A clear aqueous extract of horse's liver, in presence of chloroform, reduced nitrobenzene, in an atmosphere of hydrogen, to aniline, while the same extract, previously boiled, was without action. The authors have previously shown (this Journal, 1899, 871) the deoxidising action of this ferment, but have had no instance of hydrogenation under its influence. No doubt the formation of organic bases in the animal body is attributable to the action of this ferment, and their disappearance or accumulation is connected with the greater or less energy of oxidising actions in the body, as respiration is vigorous or feeble.—J. T. D.

Morphine; The Nitrogen-free Decomposition Products of.—VI. E. Vongerichten. Ber. **33**, 1900, [2], 352—359.

Morphenol and acetylmorphenol do not give phthalic acid on oxidation. This is an argument against the author's formula for morphenol. The quinone formed by the oxidation of acetylmorphenol by chromic acid has now been obtained in some quantity. It unites with *o*-toluylenediamine to form the compound $C_{23}H_{14}N_2O_3$, which crystallises in small needles melting at 231—232° C. Morphenol is reduced by zinc dust to phenanthrene, and by sodium is converted into morphol, which may be oxidised to morpholquinone, and this converted into a body related to alizarin, and acting as a polygenetic dyestuff on mordanted fibres.

Benzoylmorphenol may be obtained by Schotten and Baumann's method in almost quantitative yield; it crystallises from glacial acetic acid in small colourless wart-like masses, melting at 123° C.

Bromacetylmorphenol melting at 208° is obtained by the action of bromine on acetylmorphenol in chloroform solution. *Morphenol* is obtained from its methyl ether by heating in the oil-bath at 130°—135° C. for 1—1½ hours with acetic and hydriodic acids, treating with water containing bisulphite, filtering, washing, dissolving in caustic soda, filtering, acidifying, and extracting. Yield about 90 per cent. of the methyl ether.—A. C. W.

Plants, The presence of Vanadium, Molybdenum, and Chromium in. E. Demarçay. Comptes Rend. **130**, [2], 91—92.

The ashes of various woods were extracted by water, the extract saturated with hydrogen sulphide, and acidified after standing a day. The precipitate was collected and burnt off in a crucible at a low temperature. The residue, treated with hydrochloric acid and examined by the spectroscope, with the aid of photographic plates, gave clear indications of molybdenum, chromium, and vanadium.—J. T. D

New Books.

Mesure des Températures Élevées. Par H. Le Chatelier, Professeur de Chimie Minérale au Collège de France, &c.; et O. Boudouard. Georges Carré et C. Naud, 3, rue Racine, Paris. 1900. Price 5 frs.

8vo volume containing 215 pages of subject-matter, illustrated with 52 engravings, and closing with a bibliographic index. The work commences with a table of contents, and a frontispiece illustrating the Wedgwood Pyrometer. Following chapters devoted to thermometric scales, fixed points, &c., the following instruments or classes of instrument are described:—Pyrometers. I. Normal Thermometric Scales. II. Normal Thermometer. III. Gas Pyrometers. IV. Calorimetric Pyrometer. V. Electrical Resistance Pyrometer. VI. Thermo-electric Pyrometer. VII. Calorific Radiation Pyrometer. VIII. Luminous Radiation Pyrometer. IX. Shrinkage Pyrometer (Wedgwood). X. Fusible Indicators (Seger). XI. Registering Pyrometers.

Die künstliche Seide: Ihre Herstellung, Eigenschaften und Verwendung. Unter besonderer Berücksichtigung der Patent-Litteratur. Bearbeitet von Dr. Carl Süvern. Verlag von Julius Springer, Monbijouplatz 3, Berlin, N. 1900. Price M. 7.

This work contains 121 pages of subject-matter, illustrated with 25 wood engravings and two sheets of specimens, a table (2 pages) of Reactions for distinguishing Natural from Artificial Silk, and a short chapter (2 pages) on the Employment of Artificial Silk, and it concludes with indexes of names and subject-matter. The text is subdivided as follows, each chapter treating of a specific process being concluded with a list of patents thereon. I. Preparation of Artificial Silk. (*a.*) From Nitrocelluloses. (*b.*) From Non-nitrated Vegetable Materials. (*c.*) From Animal Substances. (*d.*) The Processes for Silk-making. II. Properties of Artificial Silk. (*a.*) Chardonnet Silk. (*b.*) Vivier Silk. (*c.*) Lehner Silk. (*d.*) Pauly Silk. (*e.*) Viscose Silk. (*f.*) Vanduara Silk. (*g.*) Behaviour of the Silks towards Dyestuffs. III. Uses of Artificial Silk.

Glue and Glue Testing. By Samuel Rideal, D.Sc. (Lond.). Scott, Greenwood and Co., 19, Ludgate Hill, London, E.C. 1900. Price 6*s.* 6*d.*

8vo volume, containing preface, in which the author acknowledges the assistance of C. G. Stewart; table of contents, and 140 pages of subject-matter, followed by an alphabetical index. The text is illustrated with 14 engravings, and the general subject is sub-divided as follows:—I. Constitution and Properties. II. Raw Materials and Manufacture. III. Uses of Glue. IV. Gelatin. V. Glue Testing. VI. Commercial Aspects.

Lectures on the History of the Development of Chemistry since the Time of Lavoisier. By Dr. A. Ladenburg. Translated from the Second German Edition by Leonard Dobbin, Ph.D. William F. Clay, 18, Teviot Place, Edinburgh. Simpkin, Marshall, Hamilton, Kent and Co., Ltd., London. 1900. Price 6*s.* 6*d.*

This subject is treated of in sixteen chapters, commencing with the Phlogiston Theory and ending with the Doctrine of Phases, Stereo-Chemistry, Iodoso-Compounds, Terpenes, and Perfumes. The work is one of 356 pages, and it concludes with ample indexes of names and subject-matter.

Practical Chemistry. Part I. By William French, M.A. (Cantab.). Methuen and Co., 36, Essex Street, London, E.C. 1900. Price 1*s.* 6*d.*

Graduated treatise of elementary practical chemistry, containing 134 pages and 57 illustrations.

Trade Report.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

United States.

Customs Decisions.

The Board of Trade have received copies of the following decisions of the United States Treasury Department :—

Nitro-benzol.—Nitro-benzol, known as "artificial oil of bitter almonds" and "oil of mirbane," which is a product of coal tar, and not medicinal, nor a colour, nor a dye, is admitted free of duty, under paragraph 524 of the tariff.

Coverings for Chloride of Ethyl.—Chloride of ethyl imported in glass coverings, with caps similar to those on paint tubes and cologne bottles, largely used by dentists and surgeons, the removal of the cap permitting escape of the substance from the tube by the pressure and warmth of the hand: *Held*, that the tubes are not unusual coverings.

Imports of Chemical Products into Turkey.

Bd. of Trade J., March 15, 1900.

The *Levant Herald* of the 6th inst. reports that the Sultan has confirmed the decision of the Imperial Ottoman Government to suppress the tax of 2 paras levied on every label affixed at the Custom Houses on chemical products imported from abroad, but has commanded that, in the interests of public health, the Customs authorities should continue to apply the labels. The Ministry for Foreign Affairs, it is stated, has communicated the Sovereign's decision to the foreign missions, informing them at the same time that the analysis of chemicals will henceforth be made at the Custom Houses, to each of which a competent analyst and a laboratory provided with the latest appliances will be attached.

EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

Maccaroni Manufacture in Italy.

Bd. of Trade J., March 8, 1900.

Her Majesty's Consul at Naples gives the following account of the manufacture of maccaroni in that district:—

Maccaroni is made of hard red wheat from the Black Sea, mixed with Italian wheat, grown mainly in the plains round Foggia. This is ground into semolina (not flour), the bran and husks are removed, and the semolina kneaded in hot water till it has the appearance and consistency of dough. The dough is then placed in a vertical brass cylinder, about 8 or 9 ins. in diameter, the bottom of which is a plate like the rose of a watering-pot, which is fine or thick, according to the maccaroni required.

Thus, for making vermicelli and all kinds of solid maccaroni, the holes are very small, while for making the tube maccaroni the holes are much larger. In the latter case also a conical blade is fixed in the middle of the hole to form a tube. The dough being placed at the top of the cylinder, it is driven down by hydraulic pressure through the perforated plate and cut off by hand in lengths of about 3 ft. It is then hung on canes in the sun to dry. In the case of the solid maccaroni there is no difficulty in grasping the process. In the case of the tubular maccaroni the conical blade and its attachment cut through the dough and the maccaroni issues with a slit all along it. This, however, shrinks together at once and forms a perfect tube, the join being practically invisible.

No maccaroni is now made by the laborious hand process. There was for a long time a prejudice against machinery, but this has been overcome.

The best maccaroni is made at Gragnamo and Torre dell'Annunziate. A little also of the best quality is made at Amalfi, Alfonso Garofalo, of Gragnano, being the most important manufacturer.

About a million boxes are sent annually to the United States, and about ten thousand to London. The remainder is sold in Italy.

Trade of Servia for 1898—1899.

Foreign Office Annual Series, No. 2383, *Feb.* 1900.

Chemicals.—Drugs, chemicals, and colours were imported in 1898 to the value of 52,814*l.*, a sum which is less than the importation during 1897 by 5,035*l.* Of the total, Austria-Hungary furnished to the value of 39,174*l.*; Germany, 8,247*l.*; and Great Britain, 1,433*l.*

Chemicals and drugs amounted to 17,844*l.*, or 2,184*l.* less than in 1897. Made-up drugs and medicines, 18,870*l.*, an increase, as compared with the preceding year, of 2,146*l.* Dyes, 16,100*l.*, a decrease of 4,997*l.* The importation of raw colours in 1898 fell off by half, the total value being reckoned at 4,388*l.*

During the first nine months of 1899 the importation under the general head of drugs, chemicals, and colours, was valued at 66,733*l.*, an improvement, as compared with the corresponding period of 1898, of 29,426*l.*

Patent Medicines.—Attention was drawn in last year's report to the fact that the sale of patent medicines and made-up drugs is regulated by the internal sanitary laws of Servia, which require an official analysis and licence. It is further necessary to point out that the Servian Government claims the right to fix the price of all patent medicines sold in the country.

Fats and Oils.—Articles classed under the head of fatty produce were imported in 1898 to the value of 59,313*l.*, a slight falling-off as compared with 1897. These included olive and other edible oils at 22,887*l.*, of which British cotton-seed oil was valued at 7,527*l.*; linseed, palm, cocoa, and colza oils were estimated at 6,927*l.*, and petroleum at 7,652*l.* Other importations consisted of candles, 4,886*l.*; scented soap, 6,570*l.*, and unscented, 1,985*l.*

During the first nine months of 1899, importations of fatty produce was valued at 37,060*l.*, an improvement, as compared with the corresponding period of 1898, of 6,444*l.*

Glass Bead Manufacture at Venice.

Foreign Office Annual Series, No. 2380, *Feb.* 1900.

There are more than 3,000 men employed in this industry. Formerly Venice was the only place where beads were made. It was asserted that there was no possibility of making them elsewhere owing to some climatic influence, and the chemical composition of the local sweet and salt sands. Manufactories, however, now exist in France, Bohemia, and Antwerp. Some years ago a factory was also opened in India. The exportation in 1898 was 639 tons, of the total value of 144,362*l.*

The process of making glass beads is as follows:—(1.) The vitreous paste is composed, and is then fused in the furnace. (2.) The "canna," or long, thin, perforated tube, is made by the "Margaritai" for producing the round small globes of glass of different colours, or imitation of pearls, coral, and precious stones. (3.) The rounding and working of glass pearls is done at the flame of a lamp. The first operation is considered the most important, as it provides the material necessary for making all kinds of beads, and requires some technical knowledge, and great practice, as the preparation and composition of the various pastes are still jealously kept secret.

It may perhaps be of interest to know something of a bead factory. The furnace contains five or six large earthen vases, divided one from the other, so that they may be differently heated, according to the various compositions which are poured into them. The operations for making the "canna forata," or long hollow tubes, to be converted into beads, and the "canna massiccia," to be reduced into pearls, are these: the vitreous paste is reduced into long glass tubes, more or less thin, according to the different thickness of the beads to be made, but in such a way that the hole in the middle of the tube is always maintained. The work is executed by the foreman, who has under his orders two assistants and four workmen, called "tiradori." One of the assistants dips the end of an iron rod, about 4 ft. long, into one of the vases containing the molten paste of the required colour. He then rolls it on an iron table to reduce it in a cylindrical form, and makes a round hole on the upper part of the paste. After this the foreman takes the rod in his hand and heats in the furnace the portion of paste attached to its end by giving it a few turns, and sees that the hole made is exactly in the centre. He then promptly attaches another rod to the upper part of the paste. The two rods are at once delivered to two "tiradori," who, running speedily in two opposite directions, reduce the molten material into a very long thin tube, which preserves the hole in its centre for all its length. The glass tubes are then divided according to their thickness, and cut in small pieces. Such pieces are then sifted and put in iron tubes with sand and coal powder, and by turning them in the furnace the pieces are made round. The pearls are then polished by placing them in a bag containing some sand and shaking them for some time. They are then separated from the sand by a sieve and put in another bag containing a portion of white bran, and again shaken, when they become extremely brilliant, and, after being sifted, are ready for sale.

OIL IMPORTS OF VENICE.

Foreign Office Annual Series, No. 2380, *Feb.* 1900.

Russian oils extracted from naphtha are imported from Batoum, and American oils also come here for lubricating purposes.

It will be important for exporters in England to know that the oils, in order to be admitted to the favourable duty of 8 frs. per 100 litres, must exceed a sp. gr. of 0·8745 at a temperature of 150° C.; that they must be coloured red more densely than the type prescribed by the Ministry of Finance (samples might be procured by the Consulate), and that the colour of the oils must be separable from them only by distillation; they must have a specific density of $20\frac{1}{2}$ as compared with water; and must not contain more than 8 per cent. of light distilled oils up to the temperature of 310° C.

Medicinal cod-liver oils come from Great Britain and Sweden.

Fish oils for industrial purposes are largely imported from Great Britain. The Scotch marks are in demand. Fish oils are said to be produced in great abundance in Japan, and British firms might perhaps find it advantageous to obtain them there for re-exportation. Linseed oils were formerly much in demand, but have now to compete with the oils produced in Italy. Palm and cocoanut oils are also imported for soap-making; the former from England and the latter from France. The average export of olive oil from Italy is about 50,000 tons a year. The principal countries to which it goes are England, France, Russia, America, Austria, and Germany.

COPPER SULPHATE IN ITALY.

Foreign Office Annual Series, No. 2380, *Feb.* 1900.

The rise in the price of copper sulphate which took place during the last year has created a sensation among agriculturists here, as it is much used for the vines. Several substitutes have been suggested, but they appear to have all proved either more expensive or inefficient. A mixture of sulphate of copper and lime, as well as a mixture of equal parts of sublimed and ground sulphur, are stated to have given satisfactory results. Agriculturists, however, do not feel much inclined to adopt them. There are some manufactories of sulphate of copper in Italy, but the demand is so great that a very large quantity is imported every year, chiefly from Great Britain.

TRADE OF HIOGO AND OSAKA FOR 1898.

Foreign Office Annual Series, No. 2379, *Feb.* 1900.

Matches.—For the third year in succession matches occupy the second place amongst exports. In 1898 over 21,500,000 gross, value 625,000*l.*, were sent out, being an advance of 2,500,000 gross in quantity, and 65,000*l.* in value over 1897. The demand from India, which takes chiefly sulphur matches, has somewhat fallen off owing to the fact that the Japanese match is hardly good enough to stand the moist climate of that country. Most of the shipments were made to China, Hong Kong, and the Straits Settlements, and consisted nearly entirely of safety matches.

TABLE I.

Return of Principal Articles of Import to the Ports of Hiogo and Osaka during the Years 1898–97.

Articles.		1898.		1897.	
		Quantity.	Value.	Quantity.	Value.
Chemicals and drugs:—			£		£
Alcohol	Tons	2,271	37,471	997	14,738
Dynamite	„	366	30,646	117	12,637
Gunpowder	„	197	4,274	315	7,649
Musk	..	..	9,061	..	5,232
Phosphorus amorphous.	Lbs.	240,989	26,034	197,897	25,209
Potash, chlorate of.	„	4,291,909	55,062	2,857,093	43,759
Salicylic acid	„	234,239	20,109	129,202	10,687
Soda:—					
Bicarbonate of.	Tons	1,219	7,490	917	5,875
Caustic	„	4,244	26,028	1,771	10,775
Sundry	..	..	154,444	..	99,685
Dyes and paints:—					
Aniline dyes	Lbs.	988,173	64,107	665,681	38,568
Indigo, dry	„	461,467	45,514	127,550	10,028
Lacquer	„	675,358	20,200	818,489	24,546
Logwood, extract of.	„	640,177	10,212	770,070	12,486
Paint in oil	„	1,300,207	10,604	1,424,322	13,134
Vermilion	„	68,507	6,213	56,678	5,454
Sundry	..	..	28,838	..	22,515
Hides	Tons	1,568	57,529	1,127	34,117
India-rubber ware.	..	..	13.479	..	11,278
Leather:—					
Sole	Lbs.	732,579	24,732	515,014	16,573
Other	„	548,189	38,831	494,468	30,724
Glass and glassware	..	..	49,416	..	38,547
Lead and leadware.	..	..	30,511	..	30,155
Steel and steelware	..	..	77,659	..	36,637
Tin	Tons	226	14,638	174	10,357
Tinplates	..	..	15,032	..	14,906
Zinc	Tons	3,139	62,602	3,783	75,228
Sundry	..	..	73,399	..	32,842
Oilcakes	Tons	78,280	305,483	65,205	224,547
Oil and wax, sundry non-medicinal.	..	..	35,546	..	32,839
Paraffin wax	Tons	3,677,171	19,979	4,257,294	26,628
Portland cement	„	6,271	9,711	30,517	46,187

TABLE II.

Return of Principal Articles of Export from the Ports of Hiogo and Osaka during the Years 1898–97.

Articles.		1898.		1897.	
		Quantity.	Value.	Quantity.	Value.
			£		£
Camphor	Tons	1,434	118,837	1,524	132,472
„ oil	„	368	8,158	648	18,435
Drugs, sundry	..	..	35,858	..	25,639
Gallnuts	Lbs.	627,612	11,149	321,562	8,881
Gentian	„	82,603	6,004	105,149	6,175
Ginseng	„	146,207	14,763	162,751	19,671
Glassware	..	..	29,078	..	38,393
Hides, undressed	Tons	391	9,538	396	8,718
Kanten (isinglass).	Lbs.	1,413,357	53,422	1,634,926	55,059
Leather	Tons	45	7,422	109	17,498
Lacquer ware	..	..	15,709	..	17,500
Matches	Gross	21,515,867	624,663	19,002,004	561,688
Metals:—					
Antimony	Tons	1,328	22,111	1,553	27,177
Brass:—					
Ware	..	..	1,976	..	6,649
Wire	Tons	215	11,439	145	7,354
Bronze ware	..	..	13,648	..	10,391
Copper:—					
Ingot	Tons	381	16,386	447	18,460
Slab	„	..	..	494	22,454
Ware	..	..	30,039	..	21,168
Other manufactured.	..	..	398,212	..	278,727
Ironware	..	..	13,010	..	11,787
Sundry	..	..	23,749	..	34,107
Total		..	530,570	..	438,274
Oil and wax, sundry non-medicinal.	..	..	6,530	..	6,513
Porcelain ware	..	..	132,387	..	117,381
Rags	Tons	5,305	21,179	3,361	12,849
Rapeseed oil	Lbs.	329,273	2,584	755,328	6,337
Soap	..	..	10,920	..	9,848
Wax, vegetable	Tons	2,167	59,817	2,390	71,347

The bulk of the business is in the hands of Chinese merchants. No improvement is perceptible in the quality of the manufacture, and claims against the shippers are frequently made on the grounds of inferiority of quality and of careless packing. This annoyance is due in a great measure to the difficulty of inspecting before shipment. The cases are brought ready to be put on board the vessel; and if opened, the packages would be more or less injured, or else expenses would have to be incurred which the trade could not stand. Prices were a little higher than previously owing to the general rise in wages and in the price of wood. This branch of business supplies a large amount of tonnage to steamers, but the rate of freight charged is too low to be remunerative.

Copper. — The export of this metal shows a steady increase, over 444,000*l.* worth in 1898, as against 340,000*l.* the previous year. The prices ranged from about 2*l.* 16*s.* per picul at the beginning of the year to 3*l.* 5*s.* at the end; the buying being very brisk during November and December. Of over 8,400 tons exported, Europe took nearly two-thirds and China the most of the remainder.

Sundries.—Porcelain-ware is the only other export worth notice which shows an increase. Camphor, vegetable wax, isinglass, and coal all show a falling-off.

Whilst the trade with the British Empire as a whole continues to advance, that with its two great commercial rivals, the United States and Germany, is advancing at a much more rapid rate.

Nickel Mining in Ontario.

U.S. Cons. Reps., Feb. 1900, 235.

The Toronto *Globe* of November 24 states that the Ontario Government has issued an Order in Council which will have a most far-reaching effect upon the industries of the province, and which must result in the establishment in Ontario of extensive works for the production of nickel, to be used in the building of war ships for the British Navy. The Government intends to reopen negotiations with the imperial authorities, under which the Lords Commissioners of the Admiralty may obtain an interest in the still unpatented nickel lands of the province, and by means of their development secure an unlimited supply of nickel for the manufacture of armour plate for war vessels. The Order in Council also provides for the refining in Ontario of nickel ores produced from lands that may hereafter be patented, and, if necessary to the development of the industry, prohibits the export from the province of nickel ores and matte.

In the seven years 1892–1898, the quantity of ore smelted and reduced to matte in the Sudbury district was 591,852 tons, and the estimated metallic contents were 29,705,000 pounds nickel and 34,570,500 pounds copper. At the selling price of matte at the furnaces, which is the form in which it is exported to the refineries, the total value of the nickel product for the seven years was 3,294,060 dols., and of copper, 1,302,805 dols., or a total of 4,596,865 dols. But at the average selling price of the metals during the seven years, the value of the refined metals would be 10,396,750 dols. for nickel and 3,975,607 dols. for copper, or a total of 14,372,357 dols. The total amount paid for wages in Ontario during the seven years was 1,929,894 dols., and this makes up a large proportion of the expenditure for all purposes in our province for the production of matte. It is certain that the share of the value of the refined metals distributed outside of Ontario for wages, services, and profits has been not less than 10,000,000 dols., or about two-thirds of the whole.

Magnesite in Greece.

U.S. Cons. Reps., Feb. 1900, 168.

Magnesite is found in the northern part of Eubœa, in Limnos, near Megara, and as far west as Perachora, and in Locris and Phthivdis.

At Mondondi it covers a large area near the surface, but they have sunk galleries there to the depth of 50 metres (164 feet). The veins of magnesite sometimes extend 100 metres or more below the surface of the ground. The mineral is generally sold in a crude state, but sometimes the Society of Public Works of Athens calcines it and manufactures refractory brick.

The largest owners of magnesite deposits in Greece are M. B. Boudouri, the present Minister of Marine; the Society of General Enterprises; and the Society of Public Works, Athens. There are a number of smaller owners of such lands or mines, among whom are Messrs. Ziller and Christomanos, who have valuable deposits at Pyli, Eubœa.

Greek magnesite is exported at the rate of 25,000 to 30,000 tons per annum. It has been exported from Greece since 1870, and is used to line metallurgical furnaces, for the fabrication of artificial marble, &c.

The official analysis of the magnesite of Mondondi and of Pyli is as follows:—

	Per Cent.
Carbonate of magnesium	95·12
Carbonate of calcium	4·02
Silicic acid	0·52
Moisture	0·31
Oxide of iron	Traces.

The mineral is sold in the crude state, on board of vessels at the nearest harbour, at 23 francs per ton. The cost of transporting the crude mineral to Liverpool and Hamburg is from 17 to 20 francs per ton, and to New York but little more. There is an export duty of 5 per cent. *ad valorem.*

The above-named firms are the best parties from whom to buy. A firm desirous of shipping large quantities of the mineral to home markets would do well to purchase a mine outright. There is a large tract of this mineral land for sale in Eubœa, and interested parties can learn the terms of sale by addressing Anast. K. Christomanos, professor of chemistry in the University of Athens. Magnesite is also found in Asia Minor.

Trade of the Somali Coast for 1898 and 1899.

Foreign Office Annual Series, No. 2384, *Feb.* 1900.

Gum and Resins.—A considerable decrease in gums and resins is reported at Berbera, due to a falling-off in prices at the coast and to less demand in the markets. The trade varies according to the season and the demand in Europe. But little business is done in gums at Zaila.

The value of the exports for the year was, Zaila, Rs. 6,992; Berbera, Rs. 1,21,703; and Bulhar, Rs. 62,194.

Samples of the various gums and resins produced in the protectorate are kept at Berbera, and can be inspected, or small samples forwarded on application to the Superintendent of Customs.

Sanseveria Fibre.—There is an increase in the value of fibre at Berbera of Rs. 5,613, the exports for the year being Rs. 10,355, as against Rs. 4,742 in the preceding year.

This is the produce of a species of aloe, the *Sanseveria Ehrenbergii*, which grows in profusion over many parts of the protectorate. It is used all over the country by the natives, who manufacture from it the ropes they require for loading their camels. The qualities of this fibre have attracted attention in the United Kingdom, and now that a British firm has taken the matter in hand there is every hope that the industry will be developed.

Phosphate Rock at Pensacola, U.S.A.

Foreign Office Annual Series, No. 2382, *Feb.* 1900.

One of the principal items of Pensacola's increasing export business is phosphate rock. This comes from Tennessee, and has already assumed large proportions, promising to still further increase in the near future. The quantity shipped during 1899 was 139,816 tons, valued at 145,642*l.*, against 62,620 tons at 65,229*l.* during 1898.

GENERAL TRADE NOTES.

Naphtha Production in Russia.

The *Reichs-Anzeiger* of 16th February reports that the total production of naphtha in Russia in 1899 amounted to 524,400,000 pouds, against 486,000,000 pouds in 1898, (poud = 36 lb. avoirdupois). The production of the most important Russian naphtha undertakings was as follows :—

——	1898.	1899.
	Pouds.	Pouds.
Nobel Bros.	45,000,000	95,000,000
A. J. Mantashev and Co.	52,300,000	46,418,000
Caspian Co.	33,230,000	32,900,000
Baku Co.	19,500,000	18,652,000
Tagier	56,350,000	33,300,000
Mirsayev	26,206,000	24,500,000

PRODUCTION OF GOLD AND PLATINUM IN THE URAL DISTRICT.

Le Journal de la Chambre de Commerce de Constantinople of the 10th January, states that 651 pouds of gold, valued at 750,000 roubles, were produced in the Ural District in 1899, as against 611 pouds in 1898. The yield of platinum was 380 pouds in 1899, as against 364 in 1898. The platinum bearings belonging to Count Schouvaloff yielded a third of the whole production, the works of the Prince de Donato supplying 70 pouds, while a foreign joint-stock company produced from 100 to 120 pouds. Owing to the greatly increased price of platinum, additional operations are being undertaken in the platinum-bearing soils.

OIL OF ROSES IN THE CAUCASUS.

The *Reichs-Anzeiger* of 12th February, states that the administration of State Domains in the Caucasus has for some years been making experiments in the cultivation of different foreign plants of economic use, as for example, the large tea plantations laid out at Chakva, near Batoum. The result of these has been to show that tea can be successfully cultivated on the Eastern Coast of the Black Sea. Last year several young rose trees were obtained from Kasanlyk, in Bulgaria, and planted at Kachetien, in the Tiflis Government. These have all grown well, and show for the first year's yield about 5½ oz. per acre, as well as a quantity of rose water. The result of the analysis of the oil of roses was eminently satisfactory; it showed that the Caucasian oil was of even better quality than the Bulgarian. In consequence of the satisfactory results of this experimental cultivation, the Administration of Domains is laying out fresh plantations in the current year.

EXPORT TRADE OF SUMATRA.

The following table, taken from the *Bulletin Commercial*, of Brussels, of the 10th February, shows the principal exports from Sumatra during recent years. The quantities are stated in piculs (133⅓ lb. avoirdupois), except in the case of skins, which are recorded by number :—

——	1889.	1893.	1897.	1898.
	No.	No.	No.	No.
Skins	32,018	27,898	43,360	51,985
	Piculs.	Piculs.	Piculs.	Piculs.
Copra	61,645	52,160	43,360	89,297
Coffee	115,467	69,036	71,882	48,875
Rattan	29,734	13,620	20,696	12,945
Damar gum	6,490	4,980	7,200	9,519
Cassia vera	7,054	11,697	16,116	8,808
Gambier	5,045	5,370	8,118	7,622
Tobacco	3,433	6,681	8,467	7,316
Gutta-percha	1,252	1,893	1,961	4,087
Benzoin gum	5,033	6,011	5,156	3,643
Nutmegs	3,484	5,511	3,492	3,014
Mace	659	884	1,060	1,005
Buffalo horns	196	287	409	518
Black pepper	3,062	155	25	25

THE FISH GUANO INDUSTRY IN NEWFOUNDLAND.

Chem. Trade J., March 3, 1900.

This industry turns to account the waste fish offal of the extensive Newfoundland fishing grounds. At Balena Harbour, in Macullum's Bay, on the south of the island, there has been erected, alongside of the works of the Cabot Steam Whaling Company—a local undertaking—a large factory, where machinery is being put up capable of dealing, not only with fish and fish offal, but with whales and carcases of whales—machinery which, on completion, will be capable of treating a very large quantity of raw material. Arrangements have been entered into whereby the Cabot Company capture the whales and remove the blubber, which is their perquisite, and hand over the remaining carcase to the factory for the manufacture of whale oil and whale guano. The capture of whales by the Cabot Company last year at their station at Snook's arm, on the east coast, amounted to 92 whales, some weighing upwards of 80 tons apiece. At their new station on the south of the island, where their factory is situated, it is anticipated a minimum catch of 150 during the season will be made, at an estimated weight of 50 tons apiece, and representing a total quantity of 7,500 tons of raw material available for treatment. The whale guano produced at Balena is an exceptionally good fertiliser, containing nitrogen equal to 15½ per cent. of ammonia, whilst the whale oil is of very superior quality. In addition to the whale oil and guano factory at Balena, offal from the cod fishery is available, and its utilisation will be effected by the erection of small works around the coast, with machinery on a sufficiently large scale to deal with the local supplies.

STATISTICS OF COPPER.

Henry R. Merton and Co., Ltd., 2, Metal Exchange Buildings, Leadenhall Avenue, E.C.

——	15th Feb. 1900.	31st Jan. 1900.	15th Jan. 1899.	31st January		
				1899.	1898.	1897.
Stocks in England and France.	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Standard copper :—						
Liverpool and Swansea, Chili bars and ingots	2,059	2,131	2,655	13,399	17,743	20,814
„ „ English standard copper	7,338	7,864	8,199	536	208	900
„ „ other standard copper	445	657	726	1,872	3,060	2,933
London (including landing) and Newcastle-on-Tyne	1,289	1,185	1,184	618	2,007	1,631
	11,131	11,837	12,764	16,425	23,018	26,378
Liverpool and Swansea, furnace material (fine)	2,849	1,940	1,981	1,777	731	690
Stocks of fine copper in Havre, Rouen, Bordeaux, and Dunkirk	2,660	2,450	2,265	2,463	2,497	1,939
	16,640	16,227	17,010	20,665	26,246	28,907
Advised from Chili by mail and cable, fine copper	3,275	2,350	2,400	3,000	2,000	2,400
„ Australia „ „ „	2,825	2,750	2,625	2,350	1,500	1,000
	22,740	21,327	22,035	26,015	29,746	32,307
	£ s. d.	£ s. d.	£ s. d.	£ s. d.	£ s. d.	£ s. d.
Price of G.M.B.'s and standard copper, per ton	74 17 6	71 5 0	70 10 0	69 10 0	49 0 0	51 2 6

Mining Industy in Bolivia.

Bd. of Trade J., March 15, 1900.

The Bulletin of the Bureau of American Republics says that the high price of tin has stimulated that industry in Bolivia, especially in the Uyuni district. The exports of tin ore for the year 1899 amounted to more than 6,000 tons, and an increase is anticipated during the year 1900. Difficulties of transportation alone prevent the Bolivian tin product from being 100 per cent. greater.

Although copper veins containing almost pure ore have been traced throughout the Republic, the cost of conveying the ore to the sea-shore has hitherto limited production. At present copper is only worked in a very primitive fashion at Corocoro, near Lake Titicaca. Other great drawbacks are lack of fuel, and scarcity of capital which prevent the opening up of the mines in a scientific manner. In 1897 the four companies working in the Corocoro district produced 2,912,500 kilogs. of copper, and the annual exportations for several years past has averaged 3,000 tons. The shipments went to Europe, passing through the port of Mollendo, Peru. Foreign capitalists should interest themselves in its exploitation.

Antimony, borax, and bismuth are to be found in large quantities in Bolivia, but in these, as well as in other products, the absence of transportation facilities interfere with their production and exportation, except in very limited amounts. They are mined in the primitive methods employed a century ago.

German Carbide Consumption.

Ironmonger, March 17, 1900.

The February report of the Allgemeine Carbid Ges., of Berlin, contains several interesting statistics regarding the spread of acetylene-lighting in Germany. In 1898, excluding the carbide used by the railway companies for the manufacture of their mixed-oil gas, but including the material imported from America, the total calcium carbide bought and sold amounted to 1,200 or 1,400 tons. As the "famine" was so severe in the winter that many installations were temporarily unworkable, it is estimated that the consumption should have been 1,600 or 2,000 tons. In 1899 the corresponding figures were 6,000 or 7,000 tons—three or four times as much. During 1900 the eighteen German works should turn out about 42,000 tons, of which the railways will require about 5,000, leaving 37,000 available for general purposes. Deducting a probable export of 15,000 or 20,000 tons, the home demand should reach 17,000 or 22,000—again three or four times as much as in the previous year. Spot carbide in Berlin last month was quoted at 34m. to 36m. per 100 kilos.; ex ship Hamburg, by truck-loads, 31·75m.

Iodine Manufacture in Java.

Chem. and Druggist, March 24, 1900.

An application has been made to the Netherlands Indian Government by a Mr. K. Ellinger for a concession for the manufacture of iodine compounds from saline springs near Sourabaya, Java. In accordance with the law, however, no such concession is required, so that the manufacture will commence at an early date.

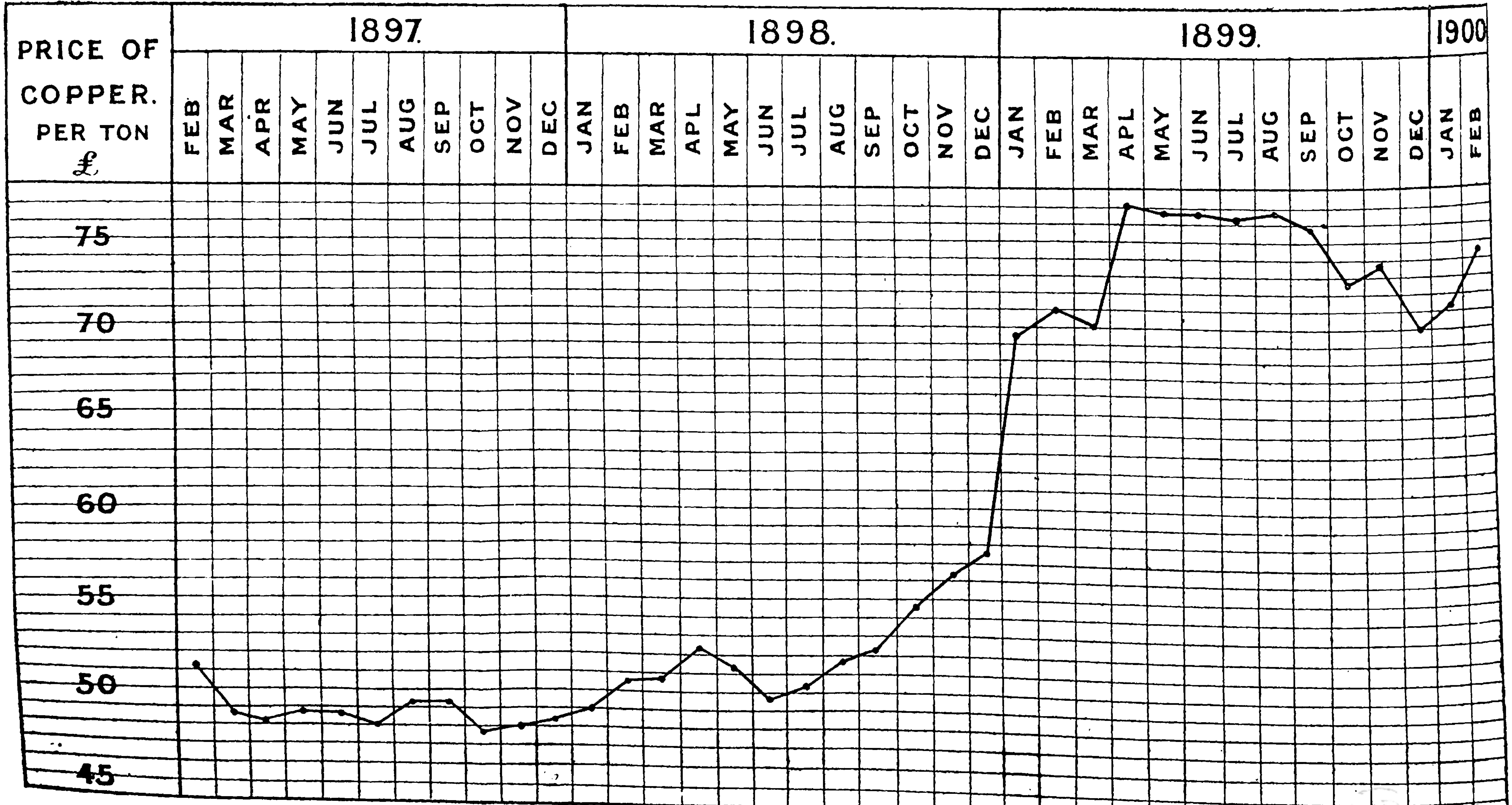
Patent Law Reforms.

Ironmonger, March 17, 1900.

In the course of a speech at the annual banquet of the Associated Chambers of Commerce, the president of the Board of Trade referred to the fact that under the present patent law system, abridgments of patents are kept, which can be consulted by inventors before asking for protection for their inventions, but those abridgments are of a very limited character, and only extend a few years back. He stated, however, that he had appointed a committee on the subject, and that he believed that this committee would recommend that the preparation of the abridgments shall go many years further back than the existing ones. It is also understood to be intended that all the various abridgments in connection with any existing class of patents shall be collected into volumes, so that anyone who wishes to get information, shall, for a small payment, be able to obtain the volume containing the particulars with respect

FLUCTUATIONS IN THE PRICE OF COPPER DURING THE PAST THREE YEARS.

CHEMICAL TRADE JOURNAL 24.2.1900.



to all the British patents in existence bearing on the invention he is desirous of protecting. This, moreover, is only a stepping-stone to another committee which Mr. Ritchie said that he is about to appoint, and over which Sir Edward Fry has consented to preside. One of the functions of this second committee will be to devise means by which a search can be made, so that applicants may be advised whether or not they are applying for something already patented, instead of as at present, leaving them to find out for themselves whether they have been forestalled, while the Government simply take the fees. It is also anticipated that the committee will deal with the system under which a foreigner who takes out a patent in his own country and in this, can prevent anyone here from taking out a similar patent in the United Kingdom, and then dictate the terms on which he is prepared to sell the British rights for the patented article.

Quantities and Values of Undermentioned Articles Imported into the German Empire during the Years 1898 and 1899.

Articles.	Quantities.		Value.	
	1898.	1899.	1898.	1899.
	100 Kilos.	100 Kilos.	1,000 Marks.	1,000 Marks.
Caoutchouc and gutta-percha.	100,977	139,164	54,528	75,149
Coal	58,203,322	62,204,885	68,950	73,402
Petroleum, refined	8,896,747	8,971,750	60,774	61,227
Copper, raw	732,905	700,914	78,846	75,699
Chemicals:—				
Indigo	10,362	11,078	8,290	8,862
Nitrate of soda	4,250,000	5,269,000	61.633	76,407
Turpentine	260,000	239,000	12,600	11,589
Lead	475,000	556,000	12,999	15,227
Tin	146,233	122,529	21,204	17,767
Hides and skins	1,165,289	1,025,285	173,348	155,553
Leather manufactures	124,848	110,390	57,915	55,330
Oilcake	4,795,082	4,806,342	53,164	53,302
Oleomargarine	221,797	185,329	14,707	12,287
Rags	446,000	446,000	8,922	8,928

Quantities of Imports and Exports of Holland for 1898 and 1899.

Articles.	Imports.		Exports.	
	1898.	1899.	1898.	1899.
	1,000 Kilos.	1,000 Kilos.	1,000 Kilos.	1,000 Kilos.
Animal fats	35,861	38,543	20,156	25,651
Peruvian bark	5,282	5,700	5,388	5,577
Cement	..	..	41,640	42,404
Coal	5,080,828	5,212,850	616,064	613,058
Copper	50,720	47,718	46,829	41,261
Hides and skins	18,316	19,373	18,718	20,347
Indigo	1,303	899	..	..
Lead:—				
Pig	..	..	2,864	6,930
White lead	..	..	9,711	10,927
Margarine	..	..	42,804	50,478
Oil:—				
Linseed	..	..	24,376	21,857
Palm	18,317	21,050	..	..
Oleomargarine	47,710	44,449	22,159	18,803
Paper	..	..	9,998	10,766
Petroleum	161,463	157,856	..	..
Potash and pearlash	7,419	5,218	..	..
Rags	..	..	35,194	38,009
Salt	82,786	78,157	..	..
Saltpetre	84,711	99,327	76,288	77,368
Soda	30,692	29,696	..	..
Sugar:—				
Raw	53,982	42,334	10,883	13,204
Refined	..	..	125,432	117,251
Sulphur	5,782	8,003	..	..
Tallow	32,002	33,389	12,686	12,042
Tin	20,884	20,212	16,348	17,104
Zinc	43,431	42,304	..	..

Quantities and Values of the Undermentioned Articles of Domestic Produce Exported from the German Empire during 1898 and 1899.

Articles.	Quantities.		Value.	
	1898.	1899.	1898.	1899.
	100 Kilos.	100 Kilos.	1,000 Marks.	1,000 Marks.
Caoutchouc manufactures.	28,211	31,683	30,486	34,413
Cement, Roman	4,978,000	5,287,000	18,667	19,825
Chemical products:—				
Aniline and other coal tar dyes.	197,123	227,046	71,950	82,872
Chloride of potassium	962,358	1,010,450	13,473	14,146
Quinine, &c.	2,009	2,019	6,630	6,960
Carbolic acid	..	..	872	1,425
Indigo	9,180	13,643	7,574	11,255
Coal	139,892,000	139,432,000	150,702	158,952
Coke	21,332,000	21,380,000	40,791	40,836
Copper manufactures	309,959	340,347	64,762	70,182
Glass and glassware	767,547	838,229	18,725	20,131
Gunpowder	24,921	26,517	5,233	5,569
Hides and skins	349,826	386,673	68,390	68,498
Lead	248,668	244,909	6,548	6,448
Leather manufactures	122,450	145,686	128,517	142,637
Precious metals manufactures.	758	844	36,743	40,914
Oilcake	1,201,161	1,403,538	14,540	16,983
Paper, gilt, and fancy	69,381	69,270	24,977	24,937
Pulp (paper-making)	566,956	582,941	13,607	13,991
Rags	476,785	503,562	7,152	7,553
Sugar	10,310,887	9,385,877	212,063	191,974
Zinc	494,712	450,309	20,047	18,247

BOARD OF TRADE RETURNS.

Summary of Imports.

Articles.	Month ending 28th February	
	1899.	1900.
	£	£
Metals	1,982,379	2,122,573
Chemicals and dyestuffs	744,967	546,629
Oils	739,383	602,070
Raw materials for non-textile industries.	2,926,154	3,556,351
Total value of all imports	35,539,109	37,604,808

Summary of Exports.

Articles.	Month ending 28th February	
	1899.	1900.
	£	£
Metals (other than machinery)	2,587,965	3,856,669
Chemicals and medicines	755,278	869,278
Miscellaneous articles	2,669,284	2,819,871
Total value of all exports	19,382,406	23,219,849

Imports of Oils for Month ending 28th February.

Articles.		Quantities.		Value.	
		1899.	1900.	1899.	1900.
				£	£
Cocoa-nut	Cwt.	29,297	40,335	32,864	47,586
Olive	Tuns	918	1,196	33,979	44,826
Palm	Cwt.	56,351	71,813	62,698	81,884
Petroleum	Gall.	18,835,549	11,840,787	357,317	246,162
Seed	Tons	6,572	2,470	106,874	53,860
Train, &c.	Tuns	468	441	9,717	7,729
Turpentine	Cwt.	42,796	34,786	63,796	66,473
Other articles	Value £	..	..	72,138	53,551
Total value		..	..	739,383	602,070

IMPORTS OF METALS FOR MONTH ENDING 28TH FEBRUARY

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900.
			£	£
Copper :—				
Ore .... Tons	2,590	14,240	30,459	125,579
Regulus .... „	2,693	6,256	94,071	204,378
Unwrought ..... „	4,135	4.484	283,739	332,659
Iron :—				
Pig and puddled „	17,732	6,264	47,231	28,284
Ore .... „	550,158	533,208	406,793	452,210
Bolt, bar, &c. ... „	3,524	3,922	25,278	30,765
Steel, unwrought.. „	5,626	1,496	28,285	14,256
Lead, pig and sheet „	15,607	10,076	216,301	166,035
Pyrites .... „	50,820	60,431	86,224	109,294
Quicksilver .... Lb.	1,515,160	57,500	161,616	5,755
Silver ore....... Value £	..	..	65,407	72,916
Tin .... Cwt.	46,885	44,186	248,875	284,191
Zinc............... Tons	5,957	5,707	133,801	126,086
Other articles ... Value £	..	..	154,299	179,165
Total value.........	..	..	1,982,379	2,122,573

IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900
			£	£
Alkali............. Cwt.	10,452	27,152	5,340	12,783
Bark (Tanners', &c.) „	10,236	16,907	3,459	6,204
Borax .... „	287	47,797	273	24,580
Brimstone .... „	25,813	29,020	6,007	6,833
Chemicals...... Value £	..	..	126,127	109,240
Cutch and gambier Tons	3,619	1,318	55,871	26,858
Dyes :—				
Alizarin.......Value £	..	..	15,763	17,194
Anilin and other „	..	..	35,304	37,189
Indigo .... Cwt.	19,737	10,152	352,305	166,669
Nitrate of potash.. „	18,747	29,047	15,441	24,903
Valonia .... Tons	2,274	1,131	25,860	11,713
Other articles ...Value £	..	..	103,217	102,472
Total value.........	..	..	714,967	546,629

IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900.
			£	£
Bark, Peruvian .. Cwt.	2,342	4,886	4,621	10,228
Bristles.......... Lb.	266,980	246,319	36,475	28,921
Caoutchouc ...... Cwt.	32,718	45,730	441,107	682,025
Gum :—				
Arabic .... „	3,700	3,926	11,642	10,204
Lac, &c. .... „	8,728	12,344	30,691	39,660
Gutta-percha .... „	3,509	8,934	48,938	125,609
Hides, raw :—				
Dry............ „	28,017	53,337	75,128	142,858
Wet .... „	64,219	51,820	130,957	122,347
Ivory .... „	697	694	31,441	33,266
Manure :—				
Guano .... Tons	1,431	2	10,492	12
Bones.......... „	9,103	6,252	41,477	27,823
Nitrate of soda... „	7,240	2,832	57,229	24,117
Phosphate of lime „	38,688	37,758	70,811	67,540
Paraffin.......... Cwt.	87,469	109,902	80,385	119,360
Linen rags .... Tons	1,166	1,534	12,233	14,209
Esparto .... „	23,361	20,248	83,740	79,975
Pulp of wood..... „	30,123	39,207	149,445	172,631
Rosin............ Cwt.	92,566	51,749	19,988	15,457
Tallow and stearin „	136,020	129,285	147,376	175,184
Wood :				
Hewn .... Loads	125,155	153,690	236,018	276,360
Sawn .... „	78,223	83,073	201,259	245,990
Staves .... „	9,139	6,786	58,788	48,979
Mahogany ..... Tons	5,479	5,471	51,050	54,347
Other articles....Value £	..	..	894,863	481,716
Total value	..	..	2,926,154	3,556,351

Besides the above, drugs to the value of 90,339*l*. were imported, as against 91,972*l*. in February 1899.

IMPORTS OF MANUFACTURED ARTICLES FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900.
			£	£
Cement.......... Tons	..	7,018	..	22,939
China and earthenware. Cwt.	..	27,768	..	128,953
Glass :—				
Sheet .... „	120,822	140,319	59,597	72,323
Plate .... „	24,714	44,658	27,369	46.238
Flint .... „	..	33,077	..	62,380
Bottles .... Gross	89,305	123,656	37,303	49,019
Other.......... Cwt.	56,735	23,889	94,701	41,491
Glue, size, and gelatin. „	..	18,229	..	36,437
Leather, unmanufactured. „	99,405	93,481	632,475	632,873
Paints and pigments. Value £	..	..	..	123,536
Paper, pasteboard Cwt.	391,714	479,832	267,851	330,373
Scientific instruments. Value £	..	..	..	43,916
Soap and soap powder. Cwt.	..	16,730	..	20,596
Zinc manufactures. „	39,924	31,320	55,001	39,727
Total of manufactured articles..	..	..	6,808,760	7,503,659

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900.
			£	£
Alkali .... Cwt.	251,609	324,542	70,743	93,767
Bleaching materials „	96,174	131,362	23,345	37,700
Copper sulphate .. Tons	7,636	7,026	149,658	165,252
Chemical manures „	31,119	31,234	194,541	242,809
Medicines....... Value £	..	..	87.217	94,713
Other articles... „	..	..	229,774	235,537
Total value	..	..	755,278	869,278

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900.
			£	£
Gunpowder...... Lb.	792,000	435,300	17,578	11,580
Military stores.. Value £	..	..	146,064	120,385
Candles......... Lb.	2,107,200	1,865,200	29,317	32,134
Caoutchouc..... Value £	..	..	101,803	115,708
Cement......... Tons	21,010	28,814	41,595	53,516
Products of coal. Value £	..	..	115,819	149,277
Earthenware ... „	..	..	120,264	145,604
Stoneware ...... „	..	..	9,266	13,819
Glass :—				
Plate .... Sq. Ft.	153,803	124,134	7,248	6,229
Flint .... Cwt.	5,816	6,151	14,613	15,800
Bottles .... „	54,632	54,380	25,209	26,066
Other kinds ... „	14,444	14,435	12,209	15,088
Leather :—				
Unwrought.... „	12,418	12,453	116,577	144,631
Wrought......Value £	..	..	33,197	33,197
Seed oil .... Tons	2,922	3,348	52,898	78,282
Floorcloth.......Sq. Yds.	2,544,600	2,996,300	101,644	127,933
Painters' materials Val. £	..	..	131,722	161,296
Paper............. Cwt.	71,088	75,379	112,278	118,109
Rags.............. Tons	3,629	5,782	20,323	36,548
Soap .... Cwt.	64,135	66 552	66,116	63,685
Total value	..	..	2,669,224	2,819,871

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Value.	
	1899.	1900.	1899.	1900.
			£	£
Brass............ Cwt.	6,852	9,750	32,321	50,795
Copper:—				
Unwrought.... „	49,968	38,872	160,189	147,494
Wrought...... „	25,900	16,934	93,923	73,715
Mixed metal... „	20,189	16,085	55,419	52,230
Hardware ..... Value £	..	..	114,503	118,806
Implements...... „	..	..	99,492	110,575
Iron and Steel ... Tons	224,257	273,574	1,727,181	2,516,552
Lead............. „	2,905	2,487	43,740	48,419
Plated wares... Value £	..	..	24,048	26,496
Telegraph wires.. „	..	..	59,424	502,816
Tin.............. Cwt.	7,106	8,233	38,565	32,395
Zinc........... „	11,785	22,617	13,523	22,683
Other articles... Value £	..	..	85,665	96,079
Total value.......	..	..	2,587,965	3,856,669

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.

3301. C. S. Whitelaw. Agitator for agitating explosive and other liquid material. Feb. 20.

3368. C. C. Sahlström. *See* Class XII.

3553. R. Thomson. Improvements in kilns. Feb. 23.

3748. E. G. Scott. Improvements in or connected with vacuum evaporators. Feb. 26.

3839. A. Martini. Improvements in gas and liquid mixing apparatus. Feb. 27.

4027. I. Davis. Improvements in and relating to apparatus for sterilising liquids. March 2.

4394. J. E. L. Ogden. Improvements in and relating to apparatus for evaporating liquids, and for like purposes. March 7.

4415. J. C. Johnson. Furnace. March 8.

4556. W. P. Thompson.—From V. Karavodine, France. Improvements in or relating to the solidification of crystalline bodies containing water. March 9.

4670. A. G. Brookes.—From the firm of Breda and Holzt, Germany. *See* Class XII.

4721. J. V. Musgrave. Improvements in humidifying apparatus. March 13.

4875. M. Sachs. Process and apparatus for the utilisation of the heat of vapours and gases. March 14.

5033. A. J. Van Vriesland. Improvements in apparatus for compressing gas. March 16.

COMPLETE SPECIFICATIONS ACCEPTED.*

1899.

5742. H. H. Lake.—From Schäffer and Budenburg, Germany. Pressure reducing apparatus. Feb. 28.

8414. E. Makin, jun. Apparatus for heating, evaporating, and condensing. March 7.

8486. W. Foulis. Apparatus for charging retorts and for stirring and withdrawing the charge of retorts. March 14.

8615. J. Lehmann. Water filtering apparatus. March 7.

8814. J. McNeil and C. McNeil. Evaporating or concentrating apparatus. March 7.

9264. F. O. Nilsson. Centrifugal apparatus. March 7.

9329. H. F. Swan.—From J. D. Swensson, Russia. Apparatus for spraying liquid fuel. March 14.

17,233. H. Pahaut. Furnaces. March 14.

19,196. A. Wall. Method of and apparatus for carbonating liquids. Feb. 28.

25,094. A. J. Boult.—From The Société Anonyme de Produits Chimiques de Droogenbosch, Belgium. Method of rendering iron vessels resistant to acids. Feb. 28.

1900.

701. J. Grouvelle and H. Arquembourg. Manufacture of coolers or condensers. March 14.

2091. J. G. Lorrain.—From The Keyser Manufacturing Company, United States, Refrigerators. March 14.

II.—FUEL, GAS, AND LIGHT.

APPLICATIONS.

3332. W. G. Hicks. Improvements in and relating to incandescent gas burners. Feb. 20.

3335. F. W. C. Schniewind. Improvements in regenerative coke ovens. Complete Specification. Feb. 20.

3429. R. Harrison. Improvements in and relating to the production of combustible gases from peat, spent tan, and similar substances, and in appliances in connection therewith. Feb. 21.

3465. F. W. Jeffrey. Improvements in or appertaining to incandescent gas burners. Feb. 22.

3514. H. K. Hess. *See* Class VII.

3530. J. Janz. Improvements in incandescent bodies for illuminating purposes. Complete Specification. Feb. 22.

3631. E. J. Duff. Improvements in gas producers. Feb. 24.

3674. J. St. C. Legge. Improvements in and relating to vapour incandescent lighting. Feb. 24.

3734. J. P. Dalby. Improvements in fuel briquettes or fire-lighters. Feb. 26.

3735. P. Heinroth and G. Tresenreuter. Improvements in incandescent gas burners. Complete Specification. Feb. 26.

3745. A. Messer. Improvements relating to acetylene gas generators. Feb. 26.

3840. J. H. H. Duncan. Improvements in the manufacture of incandescence bodies. Feb. 27.

3844. E. W. Lancaster. A new or improved apparatus for stirring or agitating the contents of and cleaning acetylene generators. Feb. 27.

3874. T. P. R. Bradshaw. Improvements in artificial lighting. Feb. 28.

3925. H. Helmecke. Improvements in the manufacture of mantles for incandescent gas lighting. Complete Specification. March 28.

3995. J. Place. Improvements in the construction of mantles for incandescent gas light. March 1.

4134. C. B. Tully. Improvements in and relating to processes for enriching or carburetting coal and other gases for illuminating purposes, and in appliances connected therewith. March 5.

4191. S. H. Berry and G. Seagrave. Improvements connected with apparatus for generating acetylene gas. March 5.

4195. W. Widmann. An improved calcium carbide admission valve for use with acetylene gas generators. Complete Specification. March 5.

4200. L. F. Bergdolt. Improvements in acetylene gas generators. Complete Specification. March 5.

4229. J. Liddle.—From H. Luckenbach and W. McPhee, United States. Apparatus for volatilising and burning oil, &c. Complete Specification. March 6.

4260. W. G. Hicks. Improvements in and relating to automatic gas lighting. Complete Specification. March 6.

4407. G. Stevenson. Improvements in apparatus for the generation and storage of acetylene gas. March 8.

4641. W. F. Richards. Improvements in the production of gas for regenerative gas furnaces. March 12.

4713. W. H. Parnall and L. J. Ruth. Improvements in acetylene gas apparatus. March 13.

4736. A. H. Arzt. Improvements in or relating to gas for illuminating and heating purposes. March 13.

4758. E. A. McAllister and J. W. Gilroy. Process of carburetting aeriform fluids. Complete Specification. March 13.

4759. E. A. McAllister and J. W. Gilroy. Improved carburettor. Complete Specification. March 13.

4786. C. T. Morelle. Improvements in apparatus for generating acetylene gas. March 13.

4791. J. H. H. Duncan and H. T. Barnett. Improvements in incandescence gas lighting apparatus. March 13.

4847. Welcome and Co. and G. C. Fowler. An improved method of and means for incorporating alcohols, ketones, volatile liquid, hydrocarbons, and other similar liquids, with solid substances for the production of a solid portable fuel. March 14.

4877. J. F. Wittemann. Improvements in and connected with the treatment and the utilisation of fermentation gas. March 14.

Complete Specifications Accepted.

1899.

4138. G. Mugna. Apparatus for washing or purifying smoke, or the products of combustion from boiler or other furnaces or fireplaces. March 7.

4589. O. Hentze and H. Müller. Method of producing hard mantles or hoods for incandescent light. Feb. 28.

5564. B. Hoffman. Process for solidifying petroleum and manufacturing briquettes therefrom. March 14.

6225. K. Hulett and Chandler, Ltd.—From F. Marshall, Denmark. Condensers chiefly designed for use in the manufacture of gas. March 14.

6340. H. R. Romney, J. Thame, and The Fibrous Material Syndicate. Composite fuel. March 21.

6525. G. Wanderpepen and A. Van Berckelaer. Manufacture of gas. March 14.

7443. E. Füller. Apparatus for mixing air and gas. March 14.

7556. C. Percy. Automatic acetylene gas generator. Feb. 28.

7984. R. Brunck. Coke-ovens with recovery of by-products. March 21.

8058. H. Helmecke. Process for manufacturing durable incandescent gas mantles. March 7.

8239. F. Clauss. Process of generating water-gas and apparatus employed therein. March 14.

8257. R. B. Ransford. — From The Société l'Aster, France. Apparatus for vaporising hydrocarbons and carburetting air. March 14.

8545. A. J. Boult.—From G. Peters, Belgium. Carburation of gas. Feb. 28.

8648. W. P. Thompson.—From A. Wegmann-Hauser, Switzerland. Acetylene gas generators. Feb. 28.

9035. E. Pestel. Acetylene gas apparatus. March 14.

9126. W. T. Sugg. Incandescent gas lighting. March 14.

9322. W. Ellen and W. I. Davies. Acetylene gas generators. March 21.

9393. P. R. J. Willis.—From J. A. Hebb, United States. Apparatus for drawing coke. Feb. 28.

9493. A. G. Glasgow. Gas holders for equalising intermittent gas production and other purposes. March 14.

10,506. W. T. Sugg. Gas lighting. March 21.

10,748. J. W. Powers. Apparatus for generating and storing acetylene gas. March 21.

11,041. O. Ernst and A. Philips. Manufacture and production of material for cleansing acetylene gas. March 7.

16,166. A. J. Boult.—From F. Isitt, New South Wales. Manufacture of mantles to be used in incandescent gas lighting. March 21.

17,053. E. Blass. Process and apparatus for the production of heating gases poor in nitrogen from combustible materials containing hydrocarbons. March 7.

20,884. F. Trendel. Process for the production of solid igniting bodies for automatic gas igniters. March 7.

25,374. H. J. Robus. Apparatus for use in the manufacture of coal gas. March 14.

1900.

56. H. M. V. Etten and H. Greenfield. Manufacture of artificial fuel. Feb. 28.

1356. E. Andreoli. Production of ozone. Feb. 28.

2337. M. Le Mat. Acetylene gas apparatus. March 14.

2672. R. A. Neilsen. Manufacture of incandescing media for incandescence lighting. March 14.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Complete Specification Accepted.

1900.

243. E. Sorel. Extraction of benzols dissolved by heavy oils and separation of their constituents. March 7.

IV.—COLOURING MATTERS and DYES.

Applications.

3265. G. B. Ellis.—From Chemische Fabrik von Heyden Actiengesellschaft, Germany. Manufacture of new colouring matters from β-naphthoquinone and its derivatives. Complete Specification. Feb. 19.

3615. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new azo-colouring matters and of new intermediate products for the production of such colouring matters. Feb. 23.

3673. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new trisazo colouring matters. Feb. 24.

3730. G. B. Ellis.—From Chemische Fabrik von Heyden Actiengesellschaft, Germany. Improvements in the production of indigo and substituted indigos. Complete Specification. Feb. 26.

3915. A. Bischler. An improvement in iodochloroxyquinoline. Complete Specification. Filed Feb. 28. Date applied for Sept. 7, 1899, being date of application in United States.

4115. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture and application of alizarin products for directly dyeing vegetable fibres. March 3.

4175. G. W. Johnson. — From C. F. Boehringer and Soehne, Germany. Improvements in the production of amines from the corresponding nitro compounds. March 5.

4329. J. E. Bedford and C. S. Bedford. Improvements in the preparation of hematine. March 7.

4677. R. B. Ransford. — From L. Cassella and Co., Germany. Improvements in the production of monoazodyes derived from amidonaphtholsulpho acids. March 12.

4792. A. G. Green, A. Meyenberg, and The Clayton Aniline Company, Ltd. Improvements in the preparation of intermediate products for colouring matters. March 13.

4799. G. W. Johnson. — From C. F. Boehringer and Soehne, Germany. The manufacture of para-amidophenylglyoxylic acids. Complete Specification. March 13.

5040. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new aromatic compounds and colouring matters therefrom. March 16.

5122. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Berlin, Germany. Manufacture of new organic bromo-compounds. March 17.

5123. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Berlin, Germany. Manufacture of new organic iodo-compounds. March 17.

Complete Specifications Accepted.

1899.

8051. B. Willcox.—From The Badische Anilin und Soda Fabrik, Germany. Manufacture and production of colouring matters of the anthraquinone series and intermediate products relating thereto. Feb. 28.

8532. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of dyestuffs for cotton. Feb. 28.

9413. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of cotton dyestuffs. March 14.

9498. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of azo-colouring matters. March 14.

9514. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of a red-brown dyestuff containing sulphur directly dyeing cotton. March 7.

9997. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of ortho- and para-nitro-dibenzlaniline sulphonic acids and their homologues. March 14.

9998. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of para-oxy-para-amido-ortho-oxydiphenylamine by heating para-oxy-para-amido-diphenylamine ortho-sulphonic acid with caustic alkalies. March 14.

10,054. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of rhodamine dyestuffs. March 21.

10,407. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of anthraquinone dyestuffs. March 14.

10,775. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of a black colouring matter directly dyeing cotton. March 7.

10,872. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of anthraquinone derivatives. March 14.

11,343. C. D. Abel.—From Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of methyl α-sulphonic acids of primary aromatic amines and of mixed amido-azo compounds resulting therefrom. March 21.

1900.

1007. O. Imray.—From The Society of Chemical Industry in Basle, Switzerland. Manufacture of colouring matters which dye unmordanted cotton. Feb. 28.

1088. G. W. Johnson.—From C. F. Boehringer and Söhne, Germany. Manufacture and production of amido-phenyltartronic acids and substitution products thereof. March 21.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Applications.

3300. H. W. Kearns. Improvements in the method of producing a silky lustre on cotton yarns and fabrics. Feb. 20.

3416. H. W. Kearns. Improvements in the method of producing a silky lustre on cotton yarns or fabrics. Feb. 21.

3644. W. Tattersall. Improvements in the means or method of humidifying yarn during the process of spinning. Feb. 24.

5104. H. E. Aykroyd and J. Graham. Improvements in or in connection with mercerising apparatus. March 17.

Complete Specifications Accepted.

1899.

5476. A. Rom. Machine for extracting fibre from turf. March 21.

5703. A. Fischer. Process for lustering mercerised yarn. March 14.

6315. L. Schreiner. Machine for mercerising and otherwise treating hanks or skeins under tension. March 21.

7315. J. E. Bedford and C. S. Bedford. Manufacture of linoleum. Feb. 28.

9044. J. A. J. Florin and H. L. Lagache. Process for softening unshrinkable wool. March 21.

9051. B. H. Saillant. Treatment of ramie or china grass. March 21.

9452. A. R. Miller. Mercerising cotton yarns, and apparatus therefor. March 7.

13,945. E. J. de Courcy and R. Crawford. Machinery for scutching and cleaning flax. March 21.

1900.

486. R. Weiss. Apparatus for treating textile materials with circulating fluids. March 7.

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

Applications.

3288. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in or connected with the discharging of dyed textile fabrics. Feb. 19.

3519. J. Y. Johnson.—From The Badische Anilin und Soda Fabrics, Germany. Improvements in dyeing paper, and new materials for use therein. Feb. 22.

3695. J. Kershaw. Improvements in the means or method of dyeing fibres and yarns two, three, or more colours or shades. Feb. 26.

4003. J. T. Pearson. Improvements in and relating to bleaching, mercerising, lustering, dyeing, and otherwise treating fibrous substances and fabrics. March 2.

4534. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in or connected with printing on raw silk or textile material containing raw silk. March 9.

4979. J. C. Chorley. Improvements in colouring vegetable textile fabrics. March 16.

5056. F. M. Spence, D. D. Spence, and T. J. I. Craig. Improvements in the manufacture of certain products used in mordanting, and for other purposes. March 16.

5057. F. M. Spence, D. D. Spence, and A. Shearer. Improvements in the manufacture of certain products used in mordanting and for other purposes. March 16.

5066. H. Hadfield. Improvements in the process and apparatus for bleaching textile fabrics. March 17.

Complete Specifications Accepted.

1899.

8440. J. C. Hamer. Apparatus for dyeing, scouring, bleaching, and otherwise treating yarn in cop or other similar compact form. Feb. 28.

18,498. J. Pfister, H. Paetz, and L. Lewin. Dyeing of wood, and apparatus for use in connection therewith. Feb. 28.

VII.—ACIDS, ALKALIS, and SALTS.

Applications.

3313. W. Bramley. Improvements in the manufacture of baryta. Feb. 20.

3514. H. K. Hess. Improvements relating to the decomposition and formation of metallic chlorides in the manufacture of hydrogen gas, and to apparatus therefor. Complete Specification. Feb. 22.

3866. A. W. Lawton. Improvements in or connected with the manufacture of common salt. Feb. 28.

3918. W. H. Woodcock. Improvements in the method of and apparatus for extracting nitrate of soda from the raw material "caliche" and from refuse thereof. Feb. 28.

3987. E. G. Scott. Improvements in the treatment of ammonia liquors and in the manufacture of sulphate of ammonia. March 1.

4112. W. C. Sellar. Improved treatment of calcium fluoride for the production of silico-fluorides. March 3.

4269. H. H. Lake.—From H. K. Moore, E. A. Allen, G. W. Ridlon, and J. Quincy, United States. Improvements relating to the production of chlorine and caustic soda or other hydrate, and to apparatus therefor. March 6.

4272. J. Potut. Improved means of and apparatus for the manufacture of sulphuric acid. March 6.

4712. J. Hargreaves. Improvements in the manufacture of chlorides and oxides. March 13.

COMPLETE SPECIFICATIONS ACCEPTED.

1899.

4643. A. McDougall. Manufacture of nitric, nitrous, and sulphuric acids, and oxides of nitrogen. March 7.

6777. W. Garroway. Manufacture of nitric acid, sulphate of soda, ammonium nitrate, carbonate of lead, and such like. March 7.

9145. J. W. Kynaston. Manufacturing caustic alkali from alkali metal amalgams, and apparatus therefor. March 7.

9432. H. W. Hemingway. Desulphurisation of sulphuretted hydrogen. March 21.

9806. H. Palas and F. Cotta. Process of manufacturing copper sulphate or other metallic sulphate. March 21.

11,434. The United Alkali Company, Ltd., and M. Muspratt. Manufacture of acetic acid. March 14.

25,161. A. Wenck. Process for the production of strontium carbonate. March 7.

VIII.—GLASS, POTTERY, AND ENAMELS.

APPLICATIONS.

3611. I. H. Storey and W. E. McCalla. Improvements in the manufacture of plates or tablets of glass, metal, earthenware, porcelain, and like articles of similar substances for various purposes, and having a surface capable of adhering to plaster or cement. Feb. 23.

4351. E. H. L. Bolle. Improvements in and relating to the finishing of ceramic and similar ware. March 7.

4678. H. Kjaertinge. Improvements in the manufacture of glass vessels or reservoirs. Complete Specification. March 12.

COMPLETE SPECIFICATIONS ACCEPTED.

1899.

4334. A. A. Marchand. Manufacture of articles of glass by moulding, casting, or blowing, and apparatus therefor. Feb. 28.

6609. G. E. Holloway and B. Dellagana and Co. Means and their manufacture for effecting the coloured decoration of pottery. Feb. 28.

7616. The Worcester Royal Porcelain Company, Ltd., and S. Ranford. Decoration of articles of porcelain and other earthenware, glass, and the like. March 7.

1900.

1273. L. L. Grimwade. Method of decorating china and earthenware. March 7.

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

APPLICATIONS.

3262. H. H. Lake.—From American Wood Fire-Proofing Co., Ltd., United States. Improvements in and relating to the treatment of wood and other combustible substances to render them fire-proof, and for other purposes. Feb. 19.

3556. J. Lander. Improvements in and relating to the fabrication of artificial stone. Feb. 23.

3657. H. Higgins. Improvements in or relating to the impregnation of wood. Feb. 24.

3749. A. C. F. P. Thaarup and S. F. A. Dohlmann. Improvements in the manufacture of artificial stone. Complete Specification. Feb. 26.

4244. R. Fimmen. Process and apparatus for the manufacture of artificial stone. Complete Specification. March 6.

4355. C. E. Challis. Artificial stone and appliances used in connection therewith. March 7.

4923. J. Doyle. Artificial stone. March 15.

5108. A. Haacke. An improved process of manufacturing building and non-conducting material from cork cuttings or granulated cork. Complete Specification. March 17.

COMPLETE SPECIFICATIONS ACCEPTED.

1899.

5603. E. Coulon. Process and apparatus for the manufacture of artificial stone. March 21.

8210. W. E. Hopps. Manufacture of Portland cement. March 21.

9441. H. Alexander. Drying chamber for the hardening of bricks, artificial stone, and the like. March 21.

21,779. C. Straub. Process of treating lime in the manufacture of plaster, cement, and the like. Feb. 28.

X.—METALLURGY, MINING, ETC.

APPLICATIONS.

3421. A. James. Improvements in apparatus for precipitating gold and silver from their solutions. Feb. 21.

3462. J. Armstrong. Improvements in treating zinc ores and apparatus therefor. Feb. 21.

3500. J. B. de Alzugaray. Improvements in or relating to the extraction of gold, silver, and other metals from ores and the like. Feb. 22.

3531. M. Wiszniewska and J. H. Strzelecki. Improvements in process of welding aluminium. Complete Specification. Feb. 22.

3598. T. Barclay, E. G. Barclay, and L. W. Winship. A new metallic alloy. Feb. 23.

3670. W. P. Thompson. From J. H. Darby, United States. Improvements in or relating to the manufacture of steel. Feb. 24.

3713. A. James. Improvements in apparatus for precipitating gold and silver from their solutions. Feb. 26.

3782. S. Cowper-Coles. Improvements in aluminium alloys. Feb. 27.

3910. D. A. Péniakoff. Improvements in the treatment of ferro-aluminous ores. Complete Specification. Feb. 28.

4225. S. Lewis. Improvements in or relating to the charging of blast furnaces. March 6.

4478. J. Keith. An improvement on the manufacture and application of solder. March 9.

4629. C. J. M. Marrier, Baron de Lagatinerie. A process for fusing copper and other metals. March 10.

4771. F. W. Hawkins and E. J. Lynn. Improvements relating to the refining of metal. Complete Specification. March 13.

4809. L. C. Aguirre. Improvements in gas furnaces for calcining lead ores. Complete Specification. March 13.

4896. T. Twynam. Improvements in the direct production of iron and steel. March 15.

4941. C. L. Bell. Improvements in apparatus for casting metals. March 15.

4961. R. Weidner. An improved alloy and process of producing the same. March 15.

COMPLETE SPECIFICATIONS ACCEPTED.

1898.

25,431. T. Twynam and F. W. Harbord. Treatment of zinc-lead sulphide ores. Feb. 7.

1899.

7968. H. Maton. Method of and composition for hardening and strengthening steel. March 14.

8043. G. de Bechi. Method of treating complex tin ores. Feb. 28.

8125. A. Tropenas. Gas furnaces for the manufacture of steel. Feb. 28.

9806. H. Palas and F. Cotta. *See* Class VII.

24,699. W. D. Allen. Method of and means for making metallic castings. March 7.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

APPLICATIONS.

3361. J. G. Hathaway. Improvements in the manufacture of secondary batteries. Complete Specification. Feb. 20.

3524. W. P. Thompson.—From H. Becker, France. Improvements in features for the anodes in electrolytic apparatus. Feb. 22.

3693. H. Korten. Improvements in secondary batteries. Feb. 26.

3753. E. N. Commelin and R. A. Viau. Improvements in and relating to electrical secondary batteries. Feb. 26.

4190. L. Boulard. Improvements in solution for galvanic batteries. March 5.

4262. P. A. Newton.—From The Knickerbocker Trust Company, United States. Improvements in storage batteries, and in the method of making electrodes therefor. Complete Specification. March 6.

4376. E. Emerson. Improvements relating to the electrolytic production of metal bars for the manufacture of wire. March 7.

4489. A. M. Young. Improvements in primary batteries. March 9.

4844. J. B. de Alzugaray. Improvements in obtaining metals from their fused ores, oxides, and salts, by electrical action. March 14.

5016. T. J. Holland and A. P. Laurie. Improvements in porous diaphragms for electrolytic apparatus. March 16.

Complete Specifications Accepted.

1899.

4865. B. J. Round, B. J. Round, and A. Round. Electro-gilding process and apparatus to be used in connection therewith.

5100. V. B. D. Cooper and E. Ridley. Methods for charging accumulators. March 21.

6873. P. Prestwich. Accumulators or secondary batteries. March 21.

8358. J. T. Niblett and M. Sutherland. Electric storage or secondary batteries. Feb. 28.

8522. R. Welford. Storage batteries. March 14.

9351. H. H. Strecker. Manufacture and production of elements for secondary batteries or electric accumulators. March 21.

9456. J. T. Niblett and M. Sutherland. Electric storage or secondary batteries. March 14.

9512. C. D. Abel.—From La Société d'Etude des Piles Electriques, France. Voltaic batteries. March 14.

9741. E. Andreoli. Ozonisation of water and substances mixed with liquids. March 14.

11,424. P. Marino. Accumulator batteries. March 14.

18,350. Q. Marino. Electrolytes. March 14.

25,491. P. Marino. Accumulator batteries. March 21.

1900.

2077. V. Cheval and J. Lindeman. Accumulator battery. March 14.

XII.—FATS, OILS, and SOAP.

Applications.

3363. P. Magnier, P. A. Brangier, and C. Tissier. A new or improved process for saponifying fatty substances and converting oleic acid into solid fatty acid. Feb. 20.

3368. C. A. Sahlström. Improvements in the process for deodorising, bleaching, and purifying. Feb. 20.

3582. P. M. Villain. Improvements in the manufacture of soap. Feb. 23.

3608. H. Beckmann. An improved apparatus for filtering and clarifying oils and the like. Complete Specification. Feb. 23.

3716. S. von Greave and A. Reinecken. Process of manufacturing oxidising agents from fats or fatty acids for converting hydrocarbons into fats or fatty acids. Feb. 26.

4379. C. G. Sudre and C. V. Thierry. Improvements in extracting glycerin from impure glycerin lyes or solutions. March 7.

4506. J. Crichton and P. H. Joselin. Improvements in refining oils, fats, and waxes. March 9.

4670. A. G. Brookes.—From the firm of Breda and Holzt, Germany. A new or improved process for separating oil, fatty substances, and the like from water. Complete Specification. 12 March.

Complete Specifications Accepted.

1899.

7044. J. H. Tynan. Composition for washing (laundry) purposes. March 7.

8872. G. E. Davis and A. R. Davis. Process of and apparatus for extracting oil from seeds or other suitable oil-containing substances by means of solvents. March 7.

9157. L. O. Helmers. Odourless and tasteless sulphur compounds of mineral oils and similar hydrocarbons. March 7.

1900.

624. C. J. Röhr. Manufacture of resin soaps suitable for sizing paper. Feb. 28.

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

Applications.

4484. H. M. Chalmers and J. F. Govan. Improvements in paints. March 9.

4615. E. Schaal and M. Schaal. Manufacture of a substitute for gum, copal, and amber. March 10.

4958. J. J. Pearson. Resilised rubber. Complete Specification. March 15.

Complete Specifications Accepted.

1899.

9652. J. R. Hatmaker.—From J. A. Just, United States. Paint. March 14.

1900.

371. F. Schmidt. Manufacture of graphited carbon. March 7.

XIV.—TANNING, LEATHER, GLUE, and SIZE.

Application.

3383. M. R. Mindus and K. F. F. G. Sommer. A rapid tanning process. Complete Specification. Feb. 20.

XV.—AGRICULTURE and MANURES.

Application.

3245. E. O. Blackwell. An improved curative and fertilising compound, especially for trees affected with mussel blight. Complete Specification. Feb. 19.

Complete Specification Accepted.

1899.

25,150. A. Wenck. Manufacture of artificial manure. March 21.

XVI.—SUGARS, STARCHES, GUMS, Etc.

Applications.

3648. F. R. Boardman. Perforating sugar or other material and compounds. Feb. 23.

4661. C. Steffen. Improved process for the rapid crystallization of impure masses of sugar. Complete Specification. March 12.

4807. C. G. Redfern.—From E. Casper, Belgium. Improvements in the manufacture of cube sugar. March 13.

Complete Specifications Accepted.

1899.

7985. A. Olivier. Production of glucose. Feb. 28.

9577. O. Imray.—From E. G. P. Thomas, J. Bonavita, and M. Olivier, France. Manufacture of viscose. March 7.

1900.

259. A. Classen. Process for converting wood into fermentable sugar. March 7.

2459. C. B. Duryea. Manufacture of starch. March 21.

XVII.—BREWING, WINES, SPIRITS, Etc.

Applications.

3522. C. G. Sudre and C. V. Thierry. Improvements in the treatment of distillers spent residues or wash. Feb. 22.

3672. J. A. Ruckdeschel. An improved apparatus for making coloured malt and for pasteurizing beer in large quantities. Complete Specification. Feb. 24.

3919. A. Gough, jnr. Improved apparatus for germinating grain and withering of malt. Feb. 28.

4692. A. Bernstein. Improvements in the manufacture of alcoholic beverages. March 12.

Complete Specifications Accepted.

1899.

6272. W. Naylor. Methods for the purification of waste liquors from manufacturing processes, such as distilling, brewing, or any others containing organic matters in solution or suspension. March 21.

8780. E. de Meulemeester. Process for the extraction of the protoplasm from yeast. March 21.

8781. E. de Meulemeester. Process for the purification or refining of protoplasm extracted from yeast. March 14.

8782. E. de Meulemeester. Process for clarification of yeast extracts. March 14.

8821. W. S. Squire. Process for purifying waste distillery liquors and extracting therefrom valuable products. March 7.

1900.

828. G. Ulbrich. Apparatus for heating or cooling and mixing brewers' mash and similar materials. March 14.

XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

Applications.

A.—Foods.

3239. C. A. Allison.—From The American Compressed Food Company, United States. Improvements in food products. Complete Specification. Feb. 19.

B.—Sanitation.

3415. T. W. H. Garstang. Improvements in the bacteriological purification of sewage and other foul liquids, and in apparatus therefor. Complete Specification. Feb. 21.

4067. B. R. Phillipson. Improvements in apparatus for the purification of sewage. March 3.

4173. D. Cameron, F. J. Commin, and A. J. Martin. Improvements in and connected with apparatus for the treatment and disposal of sewage. March 5.

4735. A. J. Boult.—From C. L. Kennicott and C. J. Blair, United States. Improvements in or relating to water purifying apparatus. Complete Specification. March 13.

Complete Specifications Accepted.

A.—Foods.

1900.

1370. C. Arendt and W. Hönicke. Manufacture of a nutritious extract from the flesh of edible crustaceæ. March 21.

1916. W. Bredon. Process and apparatus for manufacturing nutritious coffee compound. March 7.

B.—Sanitation.

1899.

4907. M. J. F. Thirault. Treatment of sewage. March 7.

6272. W. Naylor. *See* Class XVII.

C.—Disinfectants.

1899.

8259. C. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Preparation of formic aldehyde and disinfecting thereby. Feb. 28.

20,712. H. H. Lake.—From Stackmann and Retschy's Chemical Works, Germany. Process for the production of disinfecting powder. Feb. 28.

XIX.—PAPER, PASTEBOARD, Etc.

Applications.

3595. F. A. Fletcher. An improved process for the manufacturing of paper. Complete Specification. Feb. 23.

4309. F. E. Keyes. An improved method of making fire-proof pulp board. Complete Specification. March 6.

Complete Specification Accepted.

1899.

22,867. A. N. Petit. Method of and solvent materials for treating surfaces of celluloid. March 21.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

4239. W. Smith and O. P. Macfarlane. A method of forming a solution in glycerine of eucalyptus and other camphoraceous and essential oils, and extracts therefrom. March 6.

4525. O. Imray.—From The Basle Chemical Works, Switzerland. Improvements in the manufacture of orthotoluenesulphonic chloride and of saccharine therefrom. March 9.

Complete Specification Accepted.

1899.

9123. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of acidyl-salicylic acids. March 7.

XXI.—PHOTOGRAPHY.

Application.

3299. J. E. Thornton. Improvements in daylight loading photographic film rolls. Feb. 20.

Complete Specification Accepted.

1899.

11,025. C. D. Abel.—From Actiengesellschaft für Anilin Fabrikation, Germany. Process for intensifying photographic silver prints. March 21.

XXII.—EXPLOSIVES, MATCHES, Etc.

Applications.

3301. C. S. Whitelaw. *See* Class I.

3529. H. S. Maxim. Improvements relating to the manufacture of perforated explosive or other plastic material and to apparatus therefor. Feb. 22.

4204. W. E. Rowlands and W. G. Hay. Improvements in and connected with explosive shells. March 6.

4593. W. D. Borland. Improvements in the manufacture of nitro-explosives. Complete Specification. March 10.

Complete Specifications Accepted.

1898.

24,662. A. Luck. Explosives. Feb. 28.

1899.

4507. O. Beneké. Manufacture of explosive compounds. March 7.

6419. F. L. Van Dusen. Match-making machines. March 14.

6723. W. H. Akester. Manufacture of matches. March 21.

10,071. H. S. Maxim. Manufacture of explosives. March 7.

PATENTS UNCLASSIFIABLE.

Applications.

4114. A. Denaeyer. A process for chemically agglomerating powdered materials. March 3.

4166. E. W. Engels. Process for the production of fire and acid proof materials and coatings. Complete Specification. March 5.

4676. M. Otto. An improved product obtained by the solution of ozone in petroleum. March 12.

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