

Journal of the Society of Chemical Industry.

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Official Notice.

ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Newcastle-on-Tyne on Wednesday, July 22nd. Full particulars will appear later.

Proceedings of the First General Meeting of the Society under the Charter.

ADOPTION OF BY-LAWS.

Held on Friday, 13th March, 1908, at the House of the Royal Society of Arts, London.

SIR BOVERTON REDWOOD, PRESIDENT, IN THE CHAIR.

THE SECRETARY read the Notice convening the Meeting as follows:—"Take Notice that in pursuance of the provisions of Clause 37 of the Charter, a General Meeting of the Society will be held at the House of the Royal Society of Arts, John Street, Adelphi, London, on Friday the 13th day of March, 1908, at 3.30 o'clock in the afternoon, for the purpose of considering and adopting the Draft By-laws which, after due consideration of the observations received from Members in response to the invitation in that behalf, have been finally settled by the Council; and a print of such finally settled By-laws was sent to each Member of the Society in their January 31st number of the Journal. By order of the Council, Charles G. Cresswell, Secretary."

THE PRESIDENT.—Gentlemen, this is a Meeting arising out of the necessity imposed by Clause 37 of the Charter upon the Society of considering and adopting new By-laws. The clause in question directs that the Council shall, within six months from the date of the Charter, prepare draft By-laws, and shall send these to each Member of the Society, and invite them to offer observations thereon. The date of the Charter being the 17th June, the draft By-laws were sent out on 15th December, which was within the six months provided. In due course, the Council received and took into consideration a number of observations made by Members of the Society, and finally settled the draft By-laws; and, in accordance again with Clause 37, sent a copy of the finally settled draft By-laws to each Member of the Society. This is a Meeting called for the consideration and adoption of the By-laws so sent out—it being provided that this Meeting should be held on a day not less than one month nor more than three months after the expiration of six months from the date of the Charter, and not less than 14 days' notice to be given of the Meeting. Therefore, the latest date on which the Meeting could be held would be the 17th of this month. We are, therefore, in holding the Meeting to-day, within the period.

I have now formally to enquire whether there are any observations on the proposed By-laws which have been so circulated and which we have met here for the purpose of considering and adopting?

If there are no observations, I will proceed to move:—

"That the Society do make By-laws and that the By-laws contained in the print produced at this Meeting, and signed by the President for the purpose of identification, be adopted as the By-laws of the Society to come into operation and take effect so soon as they shall be allowed by the Members of His Majesty's Most Honourable Privy Council."

MR. A. GORDON SALAMON.—I have much pleasure in seconding that Resolution. The care which has been given to the preparation of these By-laws will go far to make them successful in their working. They evidently are received with approval, not only by our Members in this country but across the sea, because they make for the consolidation of the Society and promote the harmonious working of the whole body.

THE PRESIDENT.—I now put to you the Motion which has been duly moved by myself and seconded by Mr. Gordon Salamon. Those in favour of the Motion, please signify the same in the usual way.

On the contrary:—

I declare that it is duly carried unanimously.

That, Gentlemen, concludes the business of the Meeting; and I thank you for your attendance.

MR. A. GORDON SALAMON.—I venture to speak again, and to propose a vote of thanks to you, Sir, for presiding at this Meeting, and for the trouble you have taken in the work of drafting these By-laws and bringing them to their present state of perfection. Perhaps you will permit me to add my appreciation of the great services rendered in that connection by Mr. Markby, our Solicitor.

MR. THOMAS TYLER.—As Treasurer of the Society, I indorse all that Mr. Salamon has said. In this connection, we also owe very considerable thanks to our Secretary for the care and caution he has exhibited in bringing this rather serious matter to a satisfactory conclusion.

THE PRESIDENT.—I would ask Mr. Salamon to put this Motion, and to add to the names which have been already mentioned, the name of Mr. C. C. Hutchinson.

MR. A. GORDON SALAMON.—I will put the Motion; and I also add with great pleasure the names of Mr. Hutchinson and Mr. Cresswell.

The Resolution was carried unanimously.

The President returned thanks.

Birmingham Section.

Meeting held at Birmingham on January 23rd, 1908.

MR. HARRY SILVESTER IN THE CHAIR.

THE REDUCTION OF GOLD CHLORIDE BY CHARCOAL.

BY D. AVERY, M.Sc.

Charcoal is used largely for the precipitation of gold from its solutions in the extraction of this metal from its ores by the "chlorine" or the "cyanide" process. In the former case, the solution of gold chloride is heated, and filtered through tubs filled with wood charcoal. The precipitation is rapid and complete. The charcoal with which the solution first comes into contact soon becomes covered with a beautiful tough yellow deposit of metallic gold; as the solution becomes more dilute no deposit is visible on the charcoal, but the ash left on incineration is coloured pink by the finely-divided metal. In the cyanide process, the filtration through charcoal takes place in the cold. The solutions are much more dilute with respect to their gold content, a longer column of charcoal is used, and no metallic deposit appears. After the charcoal has been in use for some time the precipitation of gold takes place less actively, but it can be renewed by heating the charcoal to redness in absence of air and replacing in the filters. It has also been noticed that if a current of electricity is passed for a short time through the filter, so arranged that the charcoal is the cathode, the gold is afterwards precipitated for some time by the charcoal so treated, even more readily than by freshly-burnt material. The reaction that takes place during this reduction does not appear to have been satisfactorily investigated, although it has been noticed and various

suggestions made regarding it. The reducing action of lamp-black, animal charcoal, bone-black, and coke, on various metallic salts has been described by several investigators, who have attributed the action to impurities in the carbon, to occluded hydrogen, or to the mechanical attraction of carbon for the dissolved salts. In most cases no quantitative work has been attempted. Weppen (*Annalen*, 55, 241) found that the greater part of the acid was present in the free state after the reduction of the salt, and that it was very difficult to recover the last portions of the acid. König (*Chem. News*, 45, 215), working with purified charcoal, found that carbon dioxide was formed. He assumed the reaction to be in accordance with an equation which, expressed in modern form, would be: $4\text{AuCl}_3 + 6\text{H}_2\text{O} + 3\text{C} = 4\text{Au} + 12\text{HCl} + 3\text{CO}_2$; but in his experiment he found only 1/20th of this theoretical amount of carbon dioxide. He concluded that this fraction only of the reduction of gold chloride was due to the charcoal, the rest being due probably to physically absorbed gases.

Experimental.—Attempts were made to prepare charcoal free from all occluded reducing gases, so that the part played by the charcoal alone might be determined. Sugar was first heated to a high temperature in absence of oxygen, but it was found that the light porous mass so obtained was less suitable for manipulation than wood charcoal, and, moreover, that even after prolonged heating it still contained appreciable quantities of hydrocarbons. Ordinary wood charcoal was used, crushed to small size, treated with acids to dissolve out mineral matter, heated in chlorine to decompose hydrocarbons, and then in nitrogen to get rid of the chlorine. It was found that even after prolonged heating in nitrogen, traces of chlorine were still present. Finally a very pure form of charcoal was obtained from coconut shell. This was strongly heated in closed clay crucibles for some hours, then boiled with nitric acid for 5 or 6 hours, and washed thoroughly till the washings were nearly free from acid—the washing taking several days. It was then dried in the air, crushed and sieved, collecting the particles between 1 and 3 mm. diameter. These were dried first in a desiccator over strong sulphuric acid, then over phosphorus pentoxide, and finally heated to redness in a closed platinum crucible and cooled in a desiccator over phosphorus pentoxide. The crucible was heated in a muffle in an oxidising atmosphere to avoid the possibility of absorption of hydrogen or other reducing gases from the furnace. This charcoal was examined, first by heating it for one to two hours in a current of pure nitrogen and finding its loss of weight. It was then heated in a current of oxygen and the amount of water formed, determined. From this the amount of hydrogen still present in the charcoal was calculated. The weight lost when heated in nitrogen was found to be due to water and carbon dioxide. The gas current was carried through another combustion furnace containing copper oxide, but no hydrogen, hydrocarbons, or carbon monoxide were found present.

The results of the analysis of the charcoal were as follows:—

	Heated in nitrogen.	Combustion in oxygen.	
	Loss.	Water formed, calculated as hydrogen.	Ash.
	Per cent.	Per cent.	Per cent.
No. 1	5.0	0.16	0.20
No. 2	0.1	0.25	0.23
No. 3	0.5	0.15	0.20

* No. 1 was dried over phosphoric anhydride but not ignited in platinum crucibles. Nos. 2 and 3 were dried, ignited and cooled over phosphoric anhydride before analysis.

These results show that the amount of absorbed gases in this charcoal is very small—the loss in nitrogen and amount of hydrogen found on combustion (0.25 per cent. at most), being due to a large extent probably, to moisture

still present. In the subsequent work the charcoal is regarded as pure, the small amount of reducing agent other than carbon, possibly present, being ignored.

The gold chloride used, was prepared from pure cornet gold, by dissolving a carefully weighed quantity in *aqua regia* and evaporating three times nearly to dryness on the water bath, care being taken to avoid decomposition of the gold chloride. The remaining excess of acid was nearly neutralised by carefully adding sodium hydroxide, and the solution diluted up to decinormal strength. This value was checked by titration with potassium iodide and decinormal thiosulphate solution in the usual way. It was found that if the gold chloride was too concentrated, the aurous iodide formed by the potassium iodide, slowly decomposed, forming metallic gold and liberating iodine, so that the end-point could not be obtained sharply; but by largely diluting, a very satisfactory titration could be made, the further decomposition taking place only on long standing. The amount of free acid was also determined iodometrically. Ordinary indicators were found to be inadmissible owing to the oxidising action of the gold chloride. The amount of gold was first found by diluting, adding potassium iodide, and titrating, using starch indicator. Potassium iodate was then added, and the acid calculated from the liberated iodine. This was found to give consistent results. All solutions used were standardised against decinormal hydrochloric acid, which in turn was standardised with Iceland spar.

Estimation of hydrochloric acid formed.—It seemed certain that the chlorine present must on reduction form hydrochloric acid. Preliminary experiments showed that the amount of acid present in the reduced solution was less than the theoretical quantity, also that some of the acid formed was retained by the charcoal and could be slowly washed out by repeated treatment with distilled water. It was hoped that by adopting a uniform method of washing, the quantities of acid obtained, might be plotted in a curve, and by extrapolation, the total quantity estimated. It was found that the curve soon became asymptotic and comparison of curves showing the acid obtained from charcoal treated with gold chloride solution, and from charcoal soaked for the same time in free acid of the same equivalent strength, showed that the acid washed out much more rapidly in the latter case. Hence it was not possible to plot another curve showing absorption of acid from fresh lots of N/10 hydrochloric acid, and finding the mean of two asymptotic curves in the usual way.

Determination of hydrochloric acid by the Iceland spar method.—Instead of attempting to separate the acid from the charcoal and determine it in the solution, a weighed piece of pure Iceland spar was introduced into the flask with the gold chloride and charcoal. This was taken out at intervals, washed, dried, and weighed, and then returned to the flask again, the operation being repeated as long as any further loss of weight was found. At first this loss was rapid, but soon became slow, the last traces of acid remaining persistently in the charcoal, even after this was carefully ground and returned to the flask. About 2 grms. of charcoal were used and 150 c.c. of the gold solution (1 c.c. = 0.00686 gm. of gold). The original weight of the Iceland spar was 7.3720 grms.; after 50 days it was 6.6120 grms.; and after 59 days it was reduced to 6.6071 grms. The charcoal was then ground and returned to the flask, and after 60 days the weight was 6.5970 grms., and at the end of 65 days it was 6.5920 grms. Thus, the total loss of weight amounted to 0.78 gm. of calcite, which is equivalent to 0.5686 gm. of hydrochloric acid.

Now 150 c.c. of the solution used contained 0.0054 gm. of free hydrochloric acid, and should form on reduction, 0.5820 gm. (total, 0.5874 gm. of acid). Hence there is $0.5874 - 0.5686 = 0.0188$ gm. of hydrochloric acid not accounted for.

A fresh solution of gold chloride was prepared containing nearly the same amount of gold, but slightly more free acid.

50 c.c. of this solution contained 0.3440 gm. of gold (0.00688 gm. per c.c.) and 0.0097 gm. of free hydrochloric acid, and would form on reduction, 0.1905 gm. of acid. 50 c.c. therefore represented 0.2002 gm. of (total)

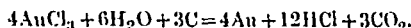
hydrochloric acid. 50 c.c. of this solution were added to the charcoal and calcite of the last experiment. In 12 days the weight was 6.3830 grms., and in 26 days, 6.3265 grms. The flask was corked and stood for about 15 months; the calcite then weighed 6.2863 grms. Thus, the total loss of weight from the beginning is $7.3720 - 6.2863 = 1.0857$ grms. of calcite, equivalent to 0.7904 gm. of hydrochloric acid. Now the total hydrochloric acid value of all solutions used $= 0.5874 + 0.2002 = 0.7876$ gm., or slightly less than that calculated from the loss of weight of the calcite.

It had meanwhile been found that the character of the reaction was not altered by heating the solution, but that the reduction was thereby hastened very greatly. Accordingly 50 c.c. of the same gold chloride solution, about the same amount of charcoal, and a piece of calcite, were heated to boiling, the calcite being weighed at intervals as before.

Original weight of calcite	6.2865 grms.
After boiling 4 hours with reflux condenser	6.0167 "
" " standing overnight, boiling 15 minutes	6.0134 "
" " 3 days " " "	6.0116 "
" " 3 weeks " " "	6.0114 "

The total loss of calcite was 0.2751 gm. $= 0.2005$ gm. of hydrochloric acid, which is just over the calculated amount.

Estimation of carbon dioxide formed.—The equation that has been suggested for the reaction is:—



Charcoal was heated in nitrogen to expel occluded gases, then transferred to a flask containing a measured quantity of the gold chloride solution. The flask was connected, through a reflux condenser, to drying tubes and weighed soda lime tubes, then (in case some of the carbon was oxidised only to the monoxide) to a tube containing heated copper oxide, and again to soda lime tubes. It was found that, on boiling the solution, carbon dioxide was given off, but in rapidly diminishing amounts, and that it was not possible to boil out the amount of gas that should be present according to the above equation—even though the apparatus was connected with a pump and boiled at greatly reduced pressure. No carbon monoxide was found, and in subsequent experiments this part of the apparatus was not included. 100 c.c. of gold chloride containing 0.00688 gm. of gold per c.c. was boiled for periods of usually half an hour; the soda lime tubes were then weighed with the following results:—

Interval.	Total weight of carbon dioxide obtained.	Increase for interval.
1 hour	0.0475	0.0475
2 "	0.0530	0.0055
3 "	0.0705	0.0175
4 "	0.0749	0.0044
5 "	0.0781	0.0032
6 "	0.0803	0.0022
7 "	0.0813	0.0010

Theory requires 0.1150 gm., and although the end was not reached, there seemed to be little hope of obtaining this quantity. Finally the charcoal was treated in the same manner, but after the boiling had been continued from one to two hours, it was rapidly filtered into a short length of combustion tubing, plugged with a wad of asbestos. A current of purified hydrogen was passed through this tube, dried, and the carbon dioxide collected in soda-lime tubes as before. The tube containing the charcoal was slowly warmed to 100°C . and kept at this temperature till no moisture could be seen collecting on the cooled end of the tube. It was then gradually raised to about 300°C ., being kept below the melting point of lead (326°C .) so as to avoid reduction by the carbon of any carbon dioxide present.

50 c.c. of gold chloride solution (1 c.c. $= 0.00688$ gm. of

gold) were introduced into the flask, and the tubes weighed at about half-hour intervals, the hydrogen being displaced by a current of pure air before weighing.

	Total weight of carbon dioxide	Increase.
Boiling for 2 hours	0.0310	0.0310
Heating in hydrogen and weighing at 1-hour intervals...	0.0488	0.0178
	0.0548	0.0060
	0.0568	0.0020
	0.0578	0.0010

Theory requires 0.0575 gm. of carbon dioxide. The equation may therefore be taken to correctly represent the reaction that takes place when gold chloride is reduced by pure charcoal in presence of water.

Occluded gases.—It was found that carbon monoxide reduced gold chloride to metallic gold when simply bubbled through the solution. The monoxide was passed through potassium hydroxide to free it from any carbon dioxide, then through lime water, gold chloride, and finally through lime water again. After a few minutes the gold solution darkened, changing to brownish green, a film of metallic gold appeared on the surface of the solution, and at the same time the second calcium hydroxide solution became milky. On boiling, the gold collected into spongy masses, the solution becoming clear. Free hydrochloric acid was formed in the solution. The reaction is probably expressed by the equation: $3\text{CO} + 3\text{H}_2\text{O} + 2\text{AuCl}_3 = 3\text{CO}_2 + 6\text{HCl} + 2\text{Au}$.

Hydrogen.—On passing hydrogen into the gold solution no reduction of gold was observed either cold or when heated, but a ring of metallic gold was slowly formed on the glass tube at the point of contact of the gas and liquid, the hydrogen bubbling slowly through meanwhile. This was repeated when a freshly-cleaned tube was used. The hydrogen was purified only by being passed through strong sulphuric acid. This reduction might be due to traces of impurities (unsaturated hydrocarbons) in the hydrogen, but seems more probably due to increased concentration of hydrogen owing to surface tension. It has been stated that hydrogen under pressure will reduce silver and platinum from their solutions—probably gold solutions would be reduced similarly.

It seemed probable that occluded hydrogen, at any rate, would cause reduction of gold from its solution. To test this, charcoal was heated and allowed to cool in hydrogen, then heated with gold chloride solution, the carbon dioxide being driven out by boiling and heating as before. The total carbon dioxide obtained, was 0.0555 gm. instead of the theoretical amount 0.0575 gm. The difference, 0.002 gm., is about that which would be caused by the hydrogen which the charcoal used (2 grms.) would absorb.

Reduction of other substances than gold by charcoal has been noticed. Weppen (*Annalen*, 59, 254), amongst others, has noted the reduction, partial or complete, of a number of metallic salts, such as those of copper, zinc, iron (ferric), chromium, nickel, cobalt, silver, and mercury. Some of these were repeated, the action being studied qualitatively only. Carbon dioxide and free acid were found to be formed, and it was assumed that the reaction was of the same character in these cases as with gold chloride.

Conclusion.—The reduction of metallic salts by charcoal in aqueous solution is due to the splitting of the water molecule with formation of the free acid of the salt and oxidation of the charcoal to carbon dioxide, the reaction being hastened by heating. The reaction is correctly expressed by the equation: $4\text{AuCl}_3 + 6\text{H}_2\text{O} + 3\text{C} = 4\text{Au} + 12\text{HCl} + 3\text{CO}_2$.

Occluded gases, such as hydrogen or carbon monoxide, will reduce gold chloride, and possibly also other metallic salts. A large proportion of the products of the reduction is persistently retained by the charcoal, and cannot be removed by simply boiling; but can be removed by the methods described.

DISCUSSION.

Prof. T. TURNER said he knew from experience that the passing of chlorine over charcoal, even at a full red heat, was a very slow process for completely removing the hydrocarbons. He did not know what other suggestion they could accept than that proposed by the author, provided always that the charcoal was pure.

Mr. F. H. ALCOCK said that the charcoal had been purified to such a high degree (there being only 0.25 per cent. of ash) that it would not materially reduce any of the gold.

Mr. H. L. HEATHCOTE said the property of the oxidation of carbon in solution to carbon dioxide seemed at first sight almost incomprehensible, but when one added to the consideration the fact that in all probability all these processes were electrolytic in their origin, and that hydroxyl was deposited on the carbon by local electric currents, he thought the oxidation became a little more comprehensible.

Mr. STACEY JONES said that a firm he acted for, reduced about £40-worth of gold as chloride at once; and the most advantageous process he knew of was to use zinc rods as reducers in dilute solution. He had tried to reduce by dropping the solution through charcoal, but it did not seem that he could remove it completely. Possibly, however, he might be mistaken.

London Section.

Meeting held at Burlington House on Monday, March 2nd, 1908.

DR. J. LEWKOWITSCH IN THE CHAIR.

The Hon. Secretary read an obituary notice of the late R. J. Friswell, Chairman of the Section.

THE PREPARATION OF *para*-TOLUIDINE FROM MIXED TOLUIDINES BY MEANS OF *para*-TOLUIDINE HYDRATE.

BY THE LATE R. J. FRISWELL. REVISED BY THE HON. SECRETARY.

A paper has recently been published (*Trans. Chem. Soc.*, 1907, 91, 1797) by J. W. Walker and Miss Beveridge describing a hydrate of *p*-toluidine. The authors remark that no mention of such a substance is to be found in Beilstein, but that the existence of a hydrate had been indicated by Levy (*Ber.*, 1886, 19, 2728) who observed that crystals of *p*-toluidine from water effloresced in the air.

Since I discovered this hydrate independently, and made many tons of it in 1889 and the following years, I believe that a description of the process of making *p*-toluidine on a large scale through the medium of the hydrate may be of interest.

The manufacture of *p*-toluidine is one of the most difficult economical questions with which a chemical manufacturer has to deal, for it is surrounded by a series of intricate problems which ramify into all departments of an aniline works. It is of course well known that *p*-nitrotoluene can be separated from *o*-nitrotoluene by cooling to 0° C. or below, and this at first sight looks a very promising direction in which to work, but the difficulties are great, and I soon had to abandon the method. In the first place the separation is by no means complete, much *p*-compound remains in the fluid, and some *o*-derivative adheres to the solid. Next the reduction of the nearly pure *p*-nitrotoluene with swarf is difficult and there is the additional difficulty of keeping the condensers at about 70° C. and of handling the separated *p*-toluidine, a liquid near to its solidifying point and therefore most treacherous in passing through pipes, cocks, etc. When, owing to the discovery of primuline, it became necessary to prepare pure *p*-toluidine on a very large scale, these problems were presented to me with great force and demanded an

immediate solution. It may be easily imagined that the separation of the nitro-compounds was abandoned, and here I may digress to say that concurrently with the work to be detailed below I set on foot a series of researches which were largely carried out by the late Dr. T. A. Lawson, then one of my assistants, to endeavour to increase the proportion of *p*-nitrotoluene to *o*-nitrotoluene obtained under ordinary conditions. As is well known, the nitration of toluene under ordinary conditions gives 60 to 65 per cent. of ortho with to 40–35 per cent. of *p*-nitro compounds, with a minute amount of the *meta*-isomeride, and it is remarkable that the sulphonation of toluene gives the same proportions of the corresponding sulphonic acids. To vary the yields, the nitric and sulphuric acids in the nitrating mixture were varied in proportion and in strength, 60 per cent. sulphuric anhydride was used to prepare strong sulphuric acid, also the temperatures were varied by nitrating at or below 0° up to the highest temperature compatible with safety. Nitrating with nitric acid alone was tried, and a long series of experiments were devised on the order and rate of mixing, culminating in a device I constructed where the acids and the toluene were allowed to fall drop by drop into a long inclined tube so that the reaction took place in a thin layer and not in a mass. The result of all this work was to convince us that the ratio of 60–65 per cent. to 40–35 per cent. was unchangeable, and to create a desire, impossible to satisfy in a chemical works, to devote time to an elaborate study of the mechanism of the reaction. The ground having thus been cleared, the only practical alternative consisted in separating the ordinary toluidine into its constituents. I was soon convinced that separating by freezing was the quickest and cheapest method, and began to devise a mode of carrying this out on the large scale, when I noticed great variations in the amount of product obtained at similar temperatures, and found that the presence of water hastened the separation. Carefully pressed specimens showed this to be due to the presence of a hydrate, and a small apparatus was installed in which the mixed toluidines were churned with water and cooled with excellent yields of crystals. Such results were still more easily obtained when a slight modification of the process was made at the suggestion of Lawson, and ice added instead of water.

I now designed and had made the apparatus shown. I had at first intended to have the spindle working inside another hollow spindle or sleeve each provided with nitro wheels gearing into the same driving wheel, so as to get a very thorough beating action as is done in various churns and similar apparatus, but I abandoned this in favour of a single acting beater and an apparatus for reducing the ice to very small fragments or brash. This was carried out as follows:—A heavy disc wheel, 3 ft. in diameter, was provided with four radial slots and mounted on the end of a spindle opposite a tube capable of taking a 2 cwt. prism of ice. Into the slot were fixed blocks of hard metal each having cast into it about twelve strong steel claws made like stout tenterhooks in plan. This disc was revolved at about 600 revolutions per minute, and the prisms of ice forced against its face. The steel hooks tore away the surface, and the ice was thus reduced to small brash or pebbles at the rate of one ton per hour. The beater consisted of strong +-sectioned breaking bars secured to rings surrounding the spindle and the wrought iron body. The arms were skewed, and the spindle stepped in a four-legged cast-iron support which straddled over the opening in the centre of the dished bottom. The top and bottom were of cast iron, the spindle passing through a gland. A manhole and a 4 in. by 6 in. charging hole with slotted cover and two strong hinged bolts served for introducing the crushed ice. The toluidine was forced up by air from an underground receiver into which it was weighed. Everything being prepared, the charge of toluidine was introduced and the agitator set going slowly. 600 lb. of crushed ice was then quickly put into the charging hole, the cover screwed up and the beaters started at about 150 revolutions per minute. The temperature now fell rapidly to about 5°, when the changing of the sound made by the beaters indicated the formation of the hydrate, the whole contents being of the consistency of thick porridge. The air was now