

to the production of the compound by a "self-thickened" cottonseed oil.

The high cost of oleo-stearine makes the method an attractive one and the hydrogenated product from cottonseed oil has the advantage, if properly made, of being very stable in character.

We believe, however, that for best results, it is desirable to hydrogenate the entire body of oil to a fatty acid titer of 36 or 38, or whatever consistency may be required, rather than to take 20 per cent. or so of the oil and harden it to a titer of 52 or thereabouts and incorporate with unhydrogenated oil.

It appears that the hydrogenation of the total body of the oil, by transforming the linoleic and linolenic compounds and the like, has a tendency to improve the oil as regards its edibility and certainly gives it greater stability.

EDIBILITY OF HYDROGENATED OILS

It seems to be generally accepted by those who have investigated the matter carefully that the hydrogenated oils have as desirable a degree of edibility as the oils from which they are derived. It is even claimed that by destroying traces of certain unsaturated bodies thought to be slightly toxic in nature, hydrogenation renders the oil better adapted for human consumption.

A question of serious import has, however, arisen in the use of nickel catalyzer. Aside from the fact that by careless filtration, traces of the suspended nickel may be present in the product, there is the more serious problem of the actual solution of nickel to form nickel soaps which cannot be easily removed.

According to Bömer,¹ nickel is dissolved by oils during the hydrogenation treatment only when the oil contains free fatty acid in considerable amounts. A sample of hydrogenated sesame oil containing 2½ per cent. of fatty acid was found to contain 0.01 per cent. ash with 0.006 per cent. nickel oxide. Whale oil, containing 0.6 per cent. fatty acid, yielded 0.006 per cent. ash and 0.0045 per cent. nickel oxide. Such an amount of nickel presumably would not be tolerated in a product intended for edible purposes.

The Bureau of Animal Industry of the Department of Agriculture is now investigating the matter and apparently intends to determine the relative degree of toxicity of the traces of nickel in the form existing in improperly made hydrogenated oil. We may add that, so far as can be ascertained, the Department looks kindly upon the advent of hydrogenated oil in view of the likelihood that it is destined to prove a very acceptable substitute for higher priced animal fats and does not propose, according to my understanding, to venture any ruling until the matter has had protracted scrutiny.

It is our belief that the use of nickel in the form of an oxide, or the use of nickel catalyzer containing more than traces of oxide, is undesirable from the point of view of solubility in oil. Nickel, in the metallic state, cannot combine with a fatty acid to produce a soap, except with the elimination of hydrogen, and in the presence of an atmosphere wholly of hydrogen,

because of mass action, such reaction would not be likely to take place. On the other hand, nickel in the form of oxide would yield water on combining with fatty acid which would be yielded practically into a vacuum as regards the vapor pressure of water.

Hence it seems to the writer that in the manufacture of products intended for edible purposes, care should be taken to maintain conditions such that the catalyzer, if of the nickel type, is preserved almost wholly in the metallic state. Also it is desirable to not force the reaction too rapidly with the consequent danger of breaking down the carboxyl group and setting free water which would react to produce fatty acid.

Finally, it may be stated, by partial saturation of glycerides, we have the possibility of preparing from tri-olein the oleo-distearine or the dioleostearine. Dioleopalmitin would give either oleostearopalmitin or distearopalmitin. From tri-olein we may have the two isomeric oleo-distearines, α - and β -oleo-distearine as well as α - and β -dioleostearine. Which of these we may be able to produce controllably and which may prove best from the edible standpoint are problems for the future to solve.

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BORON: ITS PROPERTIES AND PREPARATION¹

By E. WEINTRAUB

To the study of the chemical elements attaches a twofold interest. Their differences and similarities are a constant source of speculation, and any increase in the knowledge of their properties is an advance toward the discovery of the underlying laws of the constitution of matter. To the practical man the elements have the still further interest that they may have some unusual properties capable of technical application. All would like to learn the properties of such elements as uranium, titanium, and a number of others, and to know what they are good for.

The element boron is interesting from both points of view. It has exceptional characteristics—characteristics almost unique among elements, and it is also capable of practical uses of considerable importance.

In an article published in the 1909 Transactions of the American Electrochemical Society, Vol. 16, p. 165, the methods for the isolation of the element boron and the main properties of the element were described. The work has been going on steadily since then, mainly in the direction of developing the practical applications.

SPECIFIC RESISTANCE OF BORON

Among the characteristics of boron, the most interesting are those pertaining to its electrical resistance. The specific resistance of pure fused boron at ordinary temperature is very high, as compared to that of other elements which have any appreciable conductivity at all. Thus a piece of boron, regular in shape, two one-hundredths of a centimeter long and two one-hundredths of a centimeter square in cross section, has a resistance at 0° C. of nearly two and a half million ohms, or, in other words, 10¹² larger than

¹ Abstracted by W. R. Whitney from a lecture presented by Dr. Weintraub before the Eighth International Congress of Applied Chemistry, New York, September, 1912.

¹ Chem. Rev. Fett.-Harz.-Ind., 1912, 221.

that of copper at ordinary temperatures. The element silicon, which comes next to boron, in resistivity, is still separated by a very considerable gap from it. The specific conductivity of boron at 0° is equal to about $0.5-0.6 \times 10^{-6}$ reciprocal ohms per cm./cm²., the conductivity of silicon is 1.8, or nearly a million times as large as that of boron.

NEGATIVE TEMPERATURE COEFFICIENT OF RESISTANCE

This high specific resistance of boron is accompanied by a negative temperature coefficient of resistance, which is also unprecedented among elements. The resistance of boron drops very markedly as the temperature is increased, and it does this with exceptional rapidity at ordinary room temperature. At this temperature the conductivity of boron doubles in value for every seventeen degrees centigrade, as is illustrated by Fig. 1, where the temperature interval is 27°-450° C. The change in resistance is so great that it is difficult to represent the measurements on one curve, so two curves are used. In the first one the temperature interval is 27°-200° C. and the unit of resistance is a megohm; in the second one the tem-

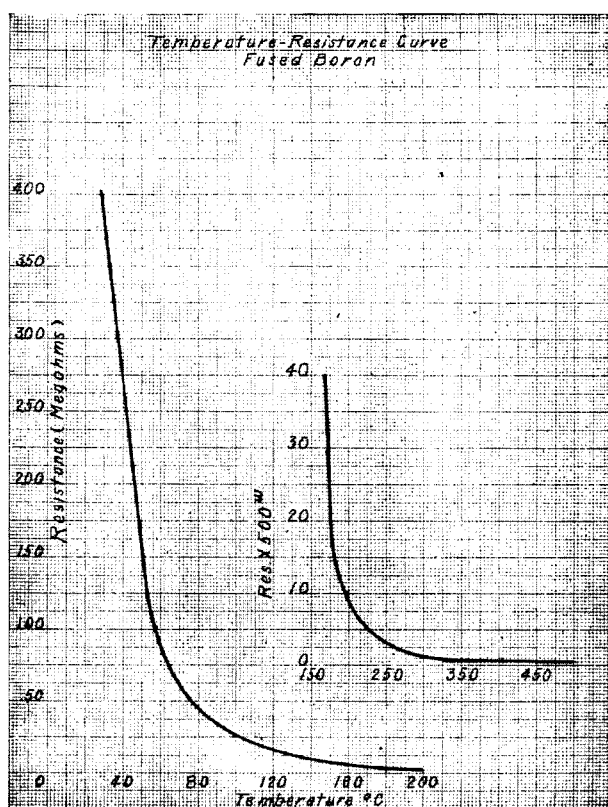


FIG. 1

perature interval is 200°-450° C., the unit of resistance is 500 ohms.

The following table gives measurements made on a very small piece of boron, which are plotted in Fig. 2.

Three ordinate scales are used, the resistance units being 10,000, 1,000 and 50 ohms, respectively, and the total temperature interval is room temperature to 650° C.

The cold resistance is 9,000,000 ohms and the re-

sistance at 650° is too small to be represented even on the smaller scale of the last curve. At tempera-

Temperature Degrees Centigrade	Resistance Ohms
27	775,000
100	66,000
170	7,700
320	180
520	7
600	4

tures above 1000° the resistance is but a fractional part of an ohm.

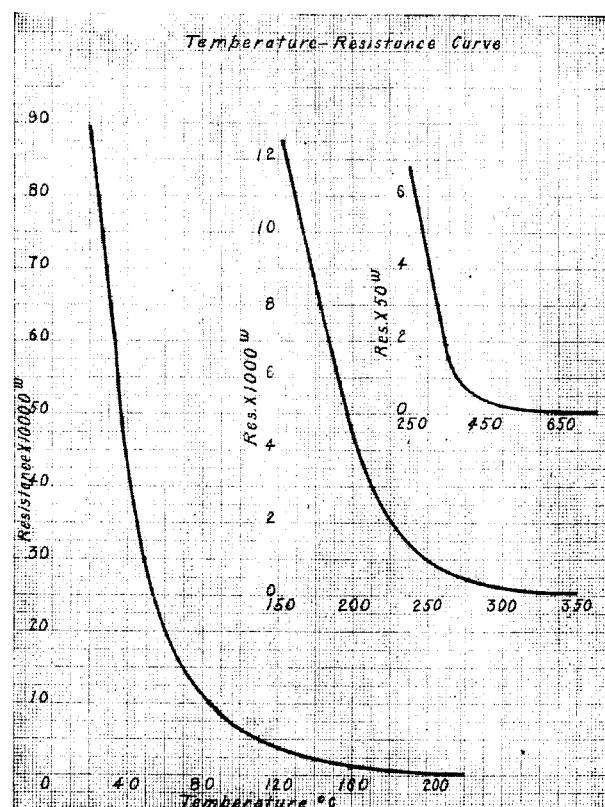


FIG. 2

The relation between temperature and resistance is not a simple one, but for small intervals of temperature it can be assumed that the drop in resistance is proportional to the resistance and the change in temperature, that is,

$$\Delta R = -\alpha R \Delta T.$$

At ordinary temperature the temperature interval, for which the resistance drops to half value is, as stated above, about 17°; this interval of temperature increases as the temperature rises, and at 600° it is about 80°. This leads to an exponential expression for the resistance which can be used for small intervals of temperature of about 50° with only a small error.

A comparison of this behavior of boron with that of other materials is instructive. It is generally known that metals have a *positive* temperature coefficient of resistance; in other words, their resistance increases with temperature. The metalloids have a negative temperature coefficient of resistance. Carbon, in its

amorphous form, has a slight negative temperature coefficient, its resistance dropping in the ratio of two to one between ordinary temperature and white heat. Silicon again comes nearest to boron, but the gap separating the two is again very considerable. Resistivity measurements on specimens of chemically pure silicon have never been made. It is, moreover, difficult to make consistent measurements on silicon on account of its tendency to crystallize in grains of different size and shape, but some specimens will show a drop of resistance as large as one hundred to one between ordinary temperature and the temperature near its melting point, which is about 1400°C .

Roughly speaking, therefore, between 0° and a good red heat the resistance ratio is for carbon, two to one, for silicon, one hundred to one, and for boron 10^6 or 10^7 to one.

Among compounds there are a large number with negative temperature coefficients of resistance. A familiar example is the Nernst Glower, which is a mixture of oxides and shows a very great drop in resistance with rise of temperature, which drop, however, begins, or at least becomes measurable only at high temperatures, 500° to 600°C . The same holds true for pure oxides. The drop of resistance of calcium oxide and of copper oxide from measurements made by Horton, are given in the following tables:

	Temperature	Resistance
Calcium oxide.....	763°	7×10^6
	1011	2×10^6
	1468	91
Copper oxide.....	12	462,400
	59	91,560
	134	12,360
	1038	0.02

While calcium oxide shows a drop of resistance only at high temperatures, copper oxide shows a rapid drop of resistance around room temperature, and in this way is very similar to boron.

The element boron then approaches in its electrical behavior more nearly certain compounds than the elements. As was pointed out in another place, it is the magnitude of resistance that determines the sign and value of the temperature coefficient of resistance and no fundamental difference exists in that respect between elements and composite substances.

Fig. 3 shows the relation between the resistance of boron and the watt input. This curve has not the theoretical importance of the temperature resistance curve, but is useful in connection with practical applications. The general course of the curve is the same as that of the temperature-resistance curve. The drop of resistance with watt input is so rapid that it is hard to represent it on one scale even with such a small variation of wattage as is embraced by the curve, namely, between 7 and 28 watts. The curve refers to a small piece of boron in air.

INFLUENCE OF DISSOLVED ELEMENTS

The influence of the addition of other elements dissolved in boron is no less interesting. This influence is of extraordinary magnitude, so that even as small an amount as 0.1 per cent. of carbon, for instance, will increase the conductivity of boron many fold.

When 7-8 per cent. of carbon are dissolved in boron the remarkable characteristics of boron disappear altogether and the material has a conductivity comparable to that of silicon or even carbon. The law of variation of the conductivity of carbon with the content of dissolved material is not established, but the experiments, as far as they have been carried out, point to an exponential curve. These experiments are as difficult as they would be interesting. One of the difficulties is the necessity of very accurate chemical analysis as a small error in the analysis would produce an enormous change in the curve.

It is again interesting to compare this behavior with that observed in other substances.

With metals the opposite case is true; if one metal is dissolved in another the resistivity increases, not

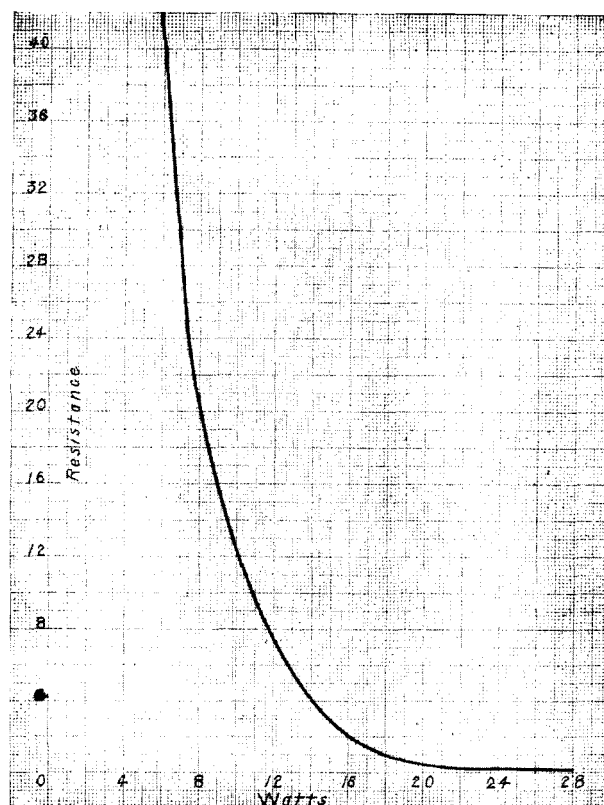


FIG. 3

the conductivity. Silver is a better conductor than copper, but addition of silver to copper increases the resistivity of the latter. With "poor" conductors, however, the conductivity of mixtures is greater than that of pure substances. This general behavior of poor conductors has been pointed out by the author previously.

Hand in hand with this diminution of resistivity goes the decrease in the value of the temperature coefficient of resistance. The temperature coefficient of resistance of solutions of elements in boron is still negative, but less so; so that while the difference in specific resistance at ordinary temperatures between boron and its solutions is very great, this difference becomes less and less as the temperature increases.

Finally, at high temperatures, in the neighborhood of 1000° there is very little difference in the conductivity of boron and the conductivity of dilute solutions of other elements in boron; all the resistance temperature curves seem to come together at high temperatures.

The reverse phenomenon has been observed with metals, but leading to the same final result. A solution of silver in copper is a poorer conductor than copper, but the temperature coefficient of resistance which is positive in this case is smaller than that of copper. At high temperatures the two curves will probably come very near together.

The rise in resistance, which takes place with good conductors, the drop in resistance which takes place in poor conductors, the above-described connection between the temperature coefficient and the magnitude of resistance all suggest the possibility that at a certain very high temperature all materials if preserved in solid or liquid form would show the same conductivity. What the value of that temperature is and what the theoretical significance of it may be are questions which will not be discussed here.

For the practical use of boron this influence of dissolved substances is very important, because one has it in his hand to change the specific resistance within very wide limits and also to change the temperature coefficient from the enormous value it has in boron to a very small value.

VOLT-AMPERE CURVE AND BREAKDOWN VOLTAGE

A consideration of the nature of the curve representing the relation of the *current and voltage* across a piece of boron is of importance. The volt-ampere curves of solids have not been used very much in theoretical discussion, for the reason that they have not the fundamental importance of the tempera-

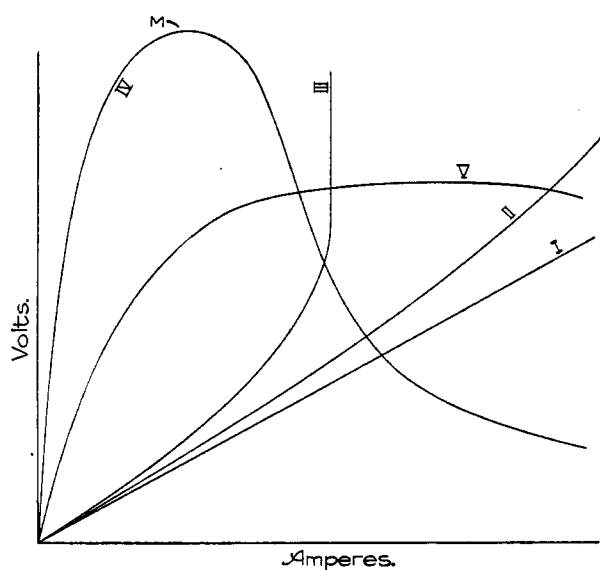


FIG. 4

ture resistance curve. The temperature resistance curve is perfectly definite; the volt-ampere curve will vary with the way in which the heat is dissipated. This is because a certain volt-ampere product means a certain watt input which may give any temperature

according to conditions. These curves have, however, been used in the study of gaseous discharges to a great extent (Kaufman) for the simple reason that in this case the volt-ampere curve is the only thing that can be determined. In the study of the volt-ampere curve of solids or liquids three cases can be distinguished (see Fig. 4). If the resistance does not vary with the temperature, then the volt-ampere curve is a straight line and the tangent is a measure of the resistance (Curve I). For a material with a positive temperature coefficient of resistance (metals, etc.) the curve will be convex to the axis of abscissa (Curve II); in some cases when the temperature coefficient is large enough a part of the curve may run almost parallel to the axis of ordinates (Curve III). In other words, there may be a region in which a very large variation in volts corresponds to a very small variation in the current. This case is exemplified by iron, within a certain narrow interval, and also by tungsten. Placing these materials in hydrogen is favorable to this result, as the heat dissipation (conduction) at lower temperature is relatively more favored than at high temperatures (mostly radiation). For a material with a negative temperature coefficient the general form of the volt-ampere curve is represented by Curve IV. The resistance corresponding to 0 current is represented by the tangent of the angle and the whole curve will lie between this tangent and the axis of abscissae. This is because the volts rise more slowly than if the resistance remained constant. A few simple considerations will show that the curve will, in general, possess a *maximum*. From $E = RI$ it follows that

$$dE = RdI - I[dR].$$

$[dR]$ indicates the absolute value of the resistance change. For $I = 0$ one has, of course,

$$\frac{dE}{dI} = R.$$

For low values of current dE is represented essentially by RdI which is *positive*; therefore, the voltage first increases with the current. However, as the current increases the second factor, $I[dR]$ becomes more and more important; since the first factor containing R becomes smaller as the resistance drops, a point is finally reached where the two are equal. At this point $dE = 0$ and the voltage is a maximum. From this point on, $I[dR]$ will predominate and the volts will drop with increasing current. The general form of the curve is, therefore, that given by IV. It is seen that for the same value of voltage there correspond in general two values of current and that the curve consists essentially of two different parts, a rising part in which the conditions correspond to stability and a falling part which is essentially unstable. On this falling part no equilibrium is possible (unless there is external resistance in the circuit with a constant or rising characteristic). On constant voltage the current and the energy input (represented by the area of the curve) would increase until a condition equivalent to a short-circuit would take place. The point M which separates the two branches of the curve and which corresponds to the maximum voltage possible

across the resistor will, on this account, be called the "breakdown" voltage.

It is at first sight somewhat difficult to see why the breakdown does not occur with ever so small a voltage since the energy introduced into the resistor should cause a drop in resistance; this in its turn causes an increased energy input, and so on. It will, therefore, be instructive to derive the existence of a breakdown point on the basis of energy relations. It will suffice to consider the very simple case in which constant voltage is applied, E . The energy input is equal to

$$W = \frac{E^2}{R},$$

and assuming that R is an exponential function of the temperature the energy input has the value

$$W = \frac{I}{C} E^2 e^{\alpha T}$$

If the temperature at which the breakdown occurs is low (this is true of boron), one may assume that the dissipation of energy takes place only by heat conduction and therefore is proportional to the temperature difference and is equal to AT . At equilibrium, energy input and output must be equal. Hence, equation

$$\frac{E^2}{C} e^{\alpha T} = AT.$$

By simple reasoning it is easy to see that this equality is possible only when E is below a certain critical value. When the volts exceed this critical value the equality is no longer possible at any temperature and the input will always be larger than the output. This particular voltage will correspond to the breakdown voltage. If the breakdown occurred at higher temperatures, the second part of the equation would contain a term corresponding to radiation, but the general conclusion would not be changed. Nor will it be materially affected if the resistance-temperature function is not exactly exponential.

The form of the curve discussed up to the present is characteristic of boron itself and all similar materials which have a very large negative coefficient of resistance. According to the value of this temperature coefficient and also to the values of the constants C and A (nature of the surface, of heat insulation) the curve may assume different forms. After the maximum voltage corresponding to M is reached the voltage may drop only very slightly or move along a horizontal part giving a constant voltage with varying current (Curve V) and finally the maximum voltage may lie at very high temperatures so that the falling part of the curve is not reached at all in ordinary work. In this case the curve is constantly rising, always remaining concave toward the X axis. The same form of volt-ampere curve may be obtained by combining a resistor with a positive temperature coefficient and one with a negative temperature coefficient of resistance, in series or in multiple.

These theoretical conclusions are illustrated by the following curves which are the result of actual measurements made on boron and on boron with a small percentage of carbon. Fig. 5 gives a volt-ampere curve obtained from measurements on a small piece

of pure boron in air. The contents were made by fusing on platinum wires. The first part, from 0 up to the maximum, rises very rapidly. The tangent of the angle this part forms with the axis of abscissa, gives the resistance of the material at room temperature. At 400 volts the breakdown voltage is reached.

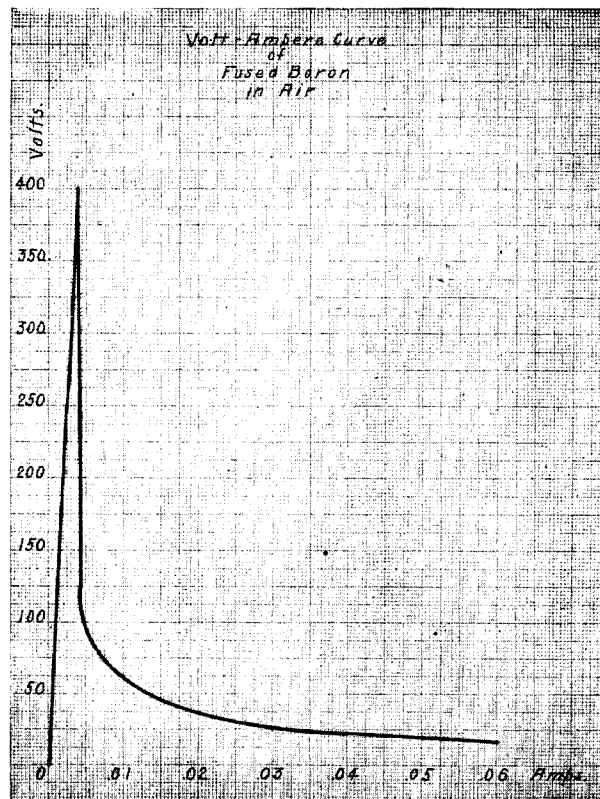


FIG. 5

The current corresponding to this part is only about 0.004 of an ampere. After the breakdown occurs the current rises and the voltage drops very rapidly.

In Fig. 6 the same measurements are represented but the part of the curve around the breakdown point is shown on a different scale.

Fig. 7 shows the volt-ampere curve of boron containing a small percentage of carbon. The breakdown voltage is lower, only about 80 volts in this particular case, but the general course of the curve is the same, except that the whole curve is lower.

Fig. 8 shows the *time interval* before the material breaks down at different impressed voltages. With this particular piece the breakdown voltage is about 85. As the voltage is gradually raised above this value the material breaks down after an interval which is the smaller the higher the voltage applied, until at 140 volts the breakdown is practically instantaneous.

OTHER PROPERTIES OF BORON

Boron is very hard. That would be expected, from the fact that boron carbide is a very hard material. Moreover, both boron and boron in which small percentages of other elements are dissolved can be prepared in a form in which they are tough to a certain extent, and in that way they differ from carborundum and

other artificial hard materials. The combination of these properties make possible their use for small bearings, such as meter jewels and for similar applica-

perfect polish. These methods have now been developed and meter jewels obtained, a large percentage of which are equal to diamond as to life and as to constancy of friction.

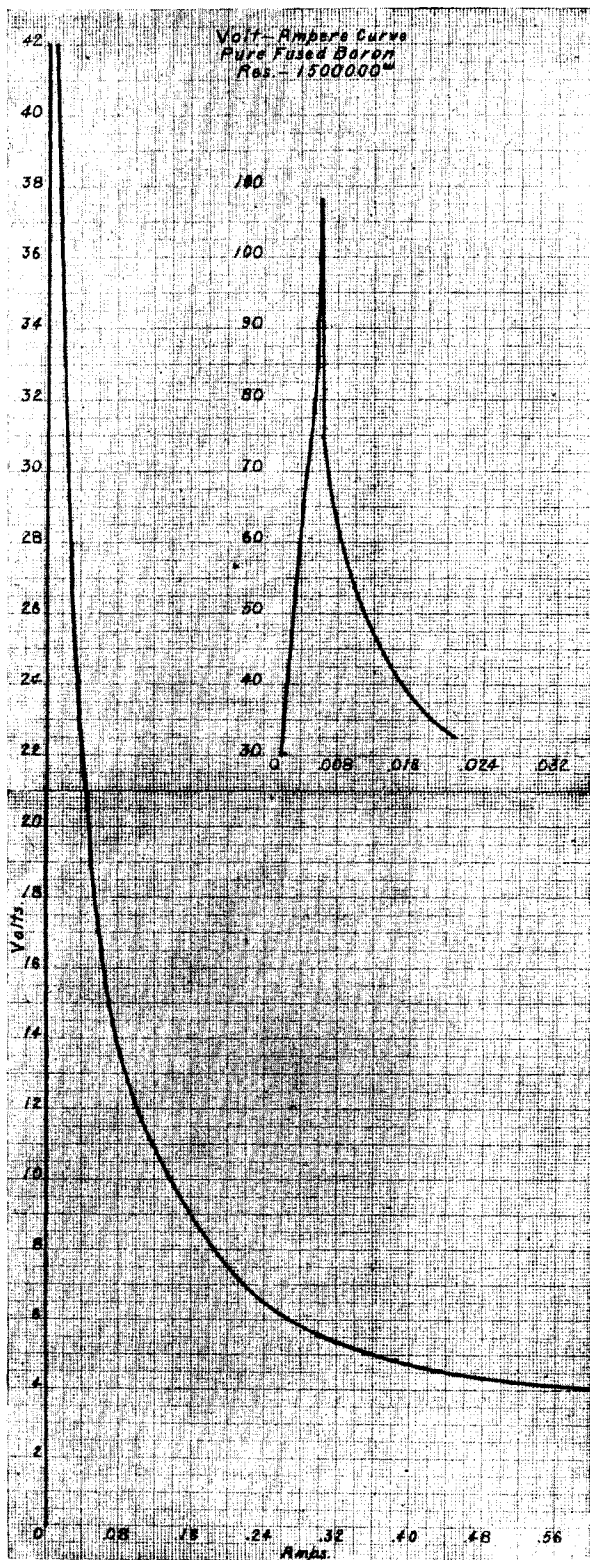


FIG. 6

tions. It has been, however, necessary to develop methods which would make a perfectly homogeneous material devoid of porosity and capable of taking a

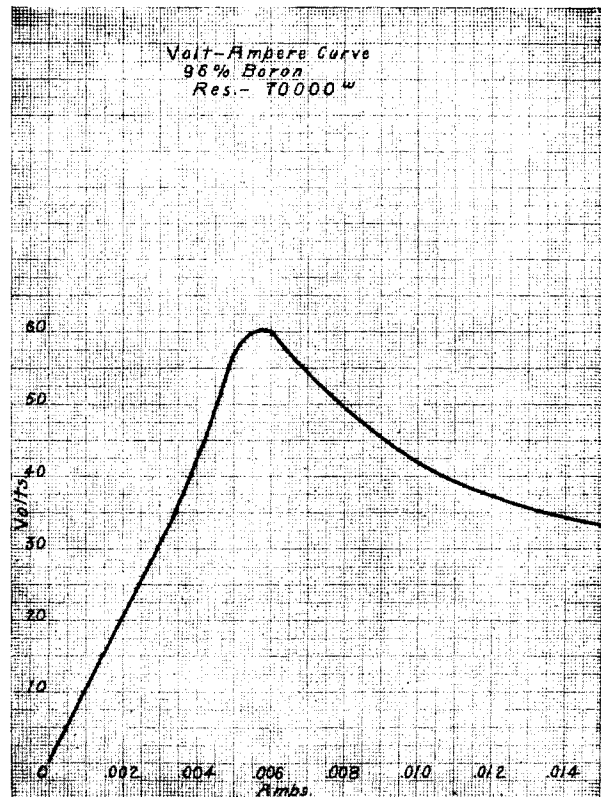


FIG. 7

CAST COPPER

Boron sub-oxide, a by-product obtained in the manufacture of boron, can be used for obtaining high conductivity cast copper. Copper cast without additions is full of pores and blow-holes, and therefore, mechanically unfit and of very low electric conductivity; the removal of the gases from copper by the known deoxid-

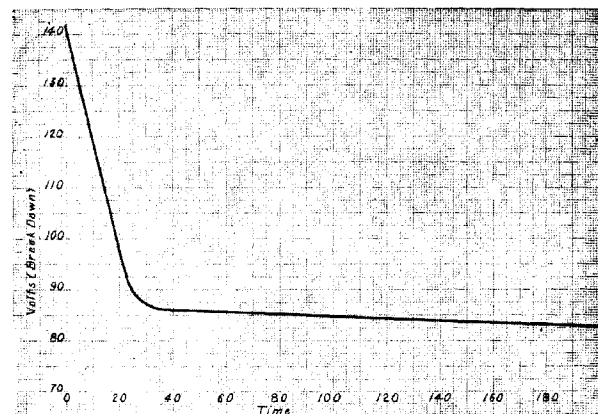


FIG. 8

izers is liable to give an alloy containing a small amount of deoxidizer, an amount sufficient, however, to lower the conductivity of the copper very consid-

erably. Boron sub-oxide, however, has the property of deoxidizing copper without combining with it, as boron sub-oxide has no affinity for copper. Tons of copper are cast now by this process, improving the quality of the product and at the same time cheapening it.

METHODS OF PREPARATION OF BORON

A brief description of the methods used to produce pure fused boron, also technically pure materially in quantities, and to bring boron into definite shape, may be of interest. The methods are novel to a large ex-

Electrochemical Society, Vol. 16, p. 165 (Oct., 1909). This boron sub-oxide or magnesium boride compound is decomposed and the resulting boron melted in an arc. As boron sub-oxide is practically an insulator a high potential arc is used. Boron sub-oxide in a copper cup forms one electrode, the other is formed by a water-cooled copper electrode. The fusion takes place in an indifferent atmosphere, such as hydrogen. The arc is started with the two electrodes a certain distance apart by means of a high voltage, say 15,000 volts, delivering a relatively small current; when every-

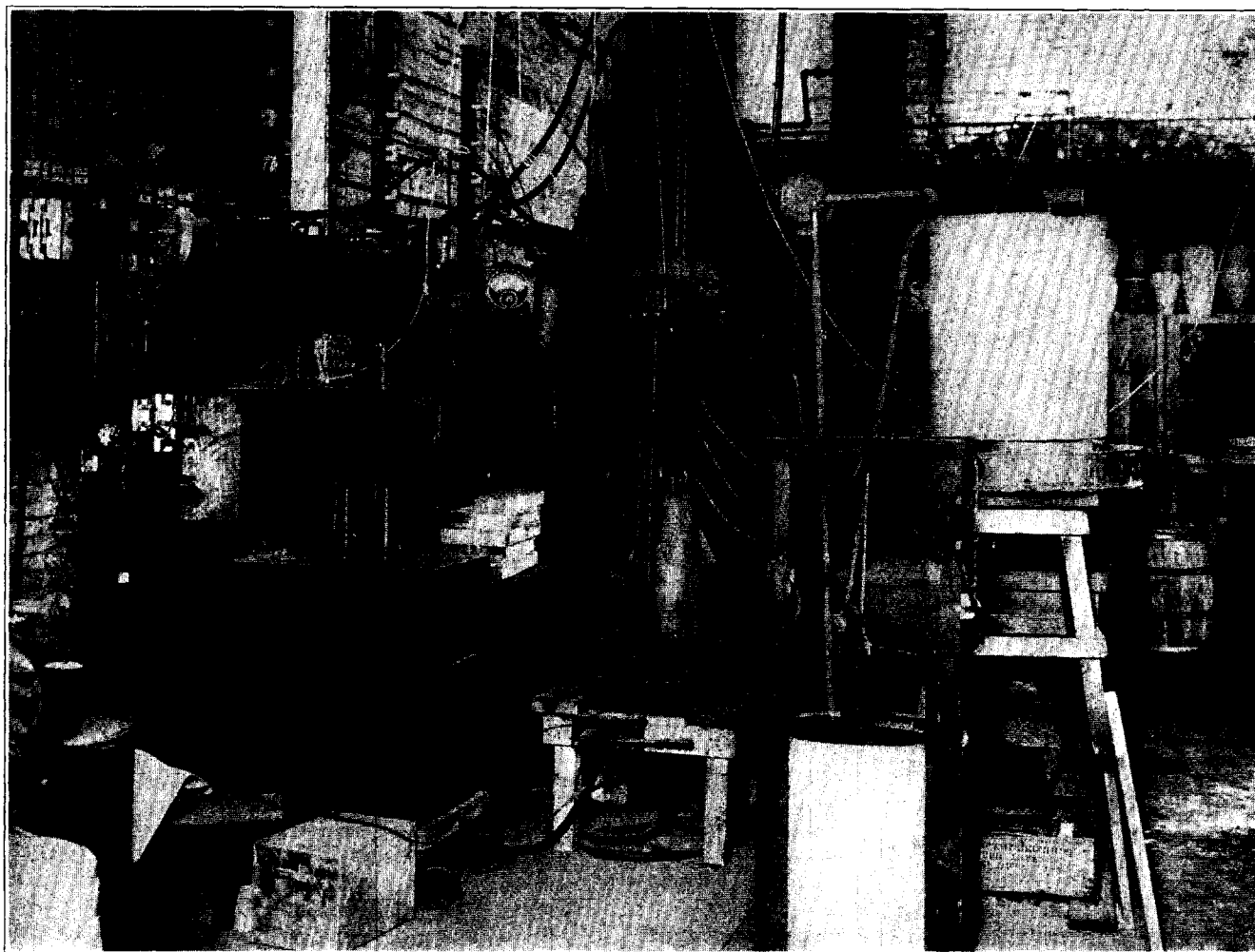


FIG. 9

tent and once developed in case of boron have shown themselves capable of applications in other fields.

The starting point in the preparation of technically pure boron is the reaction which has been studied before, the reaction between boric anhydride and magnesium or alkali metals. This reaction gives different products according to the relative weights of the substances employed, the temperature, and so forth. One interesting product obtained under certain conditions is boron sub-oxide of the approximate formula B_2O_3 . Under other conditions a product consisting of boron and magnesium boride is obtained. Detailed description of this reaction can be found in the article of 1909, in the *Transactions of the American*

thing is heated up the voltage is changed to about 2200 volts, the current to 50 amperes. After a while it is possible to reduce the voltage to 500 volts D. C. and increase the current to the desired value; by using 200-300 amperes it is possible to melt down a few pounds of boron at once.

Fig. 9 shows the small furnace used for some time but now replaced by a much bigger one. In this size of furnace only about a pound of boron can be melted in one operation. The iron hood can be moved up and down and through it hydrogen is flowing. Inside is a water-cooled copper cup containing the boron sub-oxide powder. Through the top a water-cooled copper terminal passes and is insulated from the hood.

A window is provided through which the operation of the furnace can be watched. The high voltage transformer and busbars conveying 2000 volts—(50 amperes and 500–700 volts—300 amperes, respectively)—are shown to the left of the furnace.

In this way it was found at first impossible to make absolutely pure boron. As long as the impurities are boiling out of the boron there is no danger of contamination with the material of the upper electrode; but when the state of purity of boron is approached the danger becomes considerable, and one has to stop a little short of absolute purity. With great care a material can be obtained containing about 99 per cent.

purposes they are apt to contaminate the material to be treated. They do not give a clear heat, that is, high temperature and nothing else. The new furnace is based on the use of a mercury electrode. It is a mercury *arc* furnace in an indifferent atmosphere.

Another method for making boron was developed before this one, and is capable of giving absolutely pure boron but is not as well adapted for producing large quantities. This method takes for its starting point boron chloride. If boron chloride is brought together with hydrogen at a high enough temperature it is reduced to boron and hydrochloric acid. All the materials taking part in the reaction are gaseous

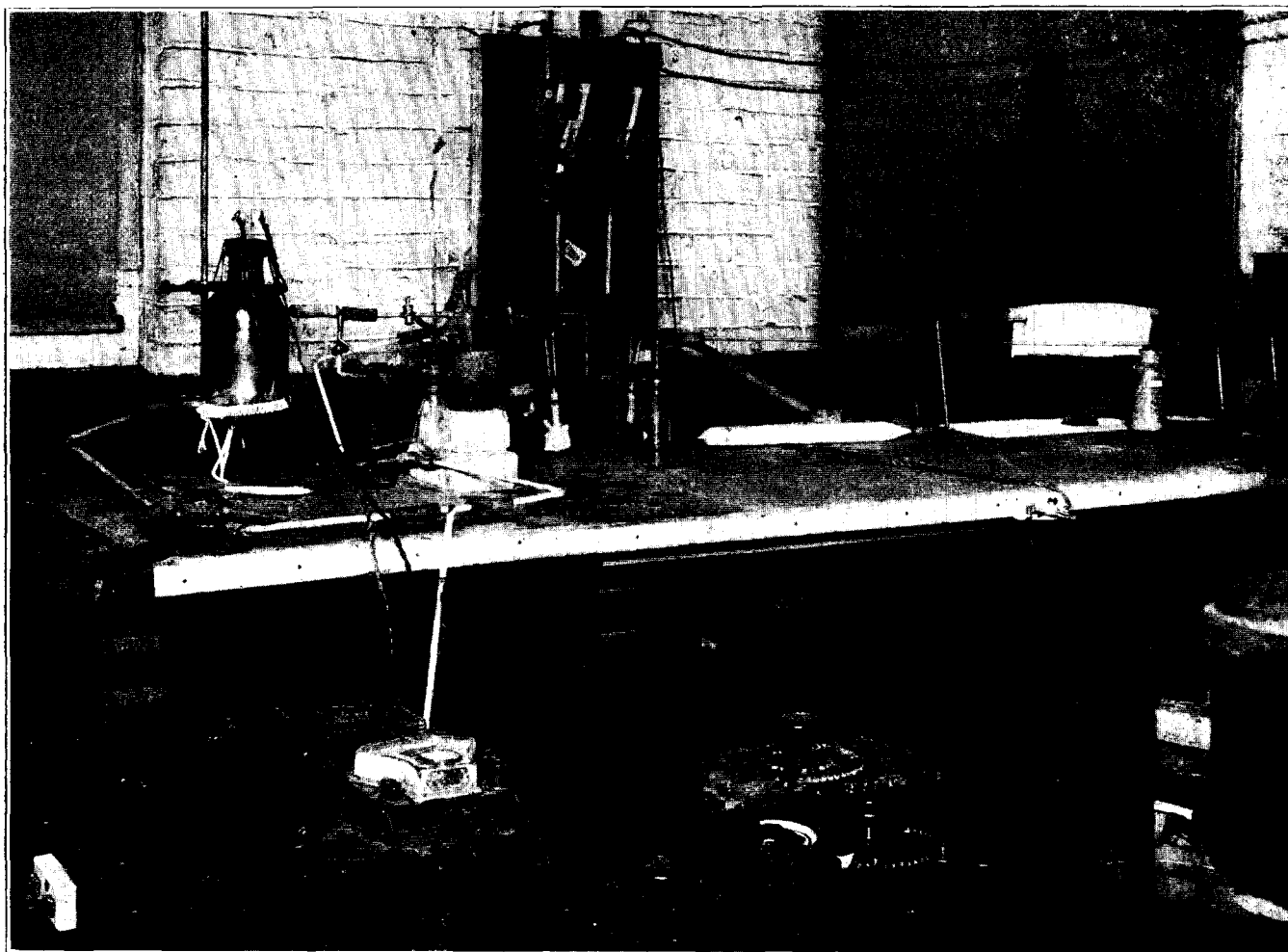


FIG. 10

boron. The average product will perhaps be not more than 97 to 98 per cent. boron. This product is good for some applications, but not for others.

It was necessary to develop an arc furnace in which no impurities at all would be introduced, either by chemical combination or mechanical admixture of the product of disintegration of the cooperating electrode. This has been accomplished and a furnace has been constructed in which practically any material can be melted down without introducing the slightest impurities into it. Most of the known arc furnaces use carbon electrodes or water-cooled metallic electrodes, and while they are excellent for some

except boron itself. As boron chloride can be made very pure and the slight impurities that may be in it are not reduced by hydrogen, the product is chemically pure boron in the most accurate sense of the word. If boron in the form of powder or lumps is wanted the reaction is carried out in an arc discharge between copper, or better, boron terminals. For practical applications (for making regulators, cut-outs, meter jewels) where a deposit of boron is wanted the gases are passed into a vessel in which a filament or plate is brought to incandescence by current.

The apparatus used is shown in Fig. 10. The hydrogen, freed from oxygen and dried in the usual way,

passes through liquid boron chloride which is kept at a definite temperature and the mixture passes then through the vessel on the left, where it is caused to react by the incandescent body. The procedure is modified in case it is desired to deposit a definite amount of boron. A vessel of definite dimensions is then filled with boron chloride and hydrogen of definite pressure.

SHAPING BORON

The arc furnace delivers irregular lumps of boron. Starting with these, the problem is to make a rod of boron. Casting in a mold is excluded because the

material is to be used for the envelope? The temperature must be high around 2300° . At that temperature the material must not have an appreciable conductivity or else it would take current itself, but above all it must not attack boron. Boron is very active at high temperatures and attacks practically everything known, all oxides included. The solution of the difficulty was found in the use of a boron compound—boron nitride, a substance described in all books of chemistry, but whose physical properties have not been well known. It is infusible, even at the temperature at which tungsten melts. More than



FIG. 11

melting point of boron is about 2300° C. It was found impossible to agglomerate the very hard boron by mere compression. The use of binding material would introduce impurities; but even if the material were agglomerated, the subsequent sintering by current would be a difficult matter on account of the large negative temperature coefficient resistance of boron. The current has a tendency to concentrate in lines which happen to have a higher conductivity; streaks develop, causing melting through in points before the whole mass is sintered. It was necessary to surround the boron with an envelope which would equalize the temperature and preserve the shape. But what ma-

terial is to be used for the envelope? The temperature at high temperatures, much poorer than any other known insulator. For instance, a small disc of magnesium oxide $\frac{1}{8}$ inch thick passed in some of the tests an appreciable current at 800° C. with 110 volts applied. A disc of boron nitride of the same dimensions heated at 1200° C. and put across 500 volts gave no measurable current. These two properties, infusibility and high insulating power at high temperatures, make it the ideal substance for an envelope in sintering boron. One more thing is necessary in order to make the sintering furnace complete; it is necessary to insure good contact between

the boron powder and the electrodes which conduct the current to it. This was done by applying pressure, and so a new type of furnace was developed, the "boron nitride sintering furnace." With this furnace not only boron, but also boron carbide, tungsten, and a large number of other materials have been formed into rods. On account of the inertness of boron nitride almost any refractory material can be sintered in this furnace, and it is difficult at present to foresee all the possibilities of this furnace in the direction of sintering materials or combining materials at elevated temperatures under high pressure.

Of course, in order to use boron nitride in this furnace, methods had to be developed for making boron nitride relatively cheaply and in large quantities. Fig. 11 shows a small size boron nitride furnace.

Due credit is given to the men of the West Lynn Research laboratory of the General Electric Co. who ably assisted in this work—G. Weintraub (work on the arc furnace, on the boron nitride furnace, etc.), H. Rush (designing of boron nitride furnace), E. Kraus (boron chloride reaction), F. W. Lyle (development of applications of boron to electrical engineering) and F. Kroner (copper casting). To them the success of this investigation is largely due.

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ACETYLENE SOLVENTS¹

By JOSEPH H. JAMES

The rapidly advancing price of acetone makes it desirable to have commercially available a solvent that can be obtained in any quantity and which shall not advance in price abnormally.

To determine the relative industrial value of various acetylene solvents, a number of laboratory tests and large scale experiments were made. The purpose of this paper is to report the results of this work.

The acetylene used in the laboratory tests was made from commercial carbide in an ordinary "Carbide to water" laboratory generator. It was purified to remove ammonia, sulfur compounds, and phosphorus compounds, by passing through a purifying train consisting of the following: (1) a ten per cent. sulphuric acid solution, (2) a fifteen per cent. hydrochloric acid solution saturated with mercuric chloride, (3) two towers, each containing approximately equal parts of a mixture of bleaching powder and slaked lime, (4) slaked lime, (5) two towers of fused calcium chloride.

In order to get as closely as possible at the true figure for the absorption of acetylene in any of its solvents, it is necessary that the gas be free from impurities and that the solvents be of the highest purity attainable. It has been demonstrated that the solubility drops off rapidly when impurities are present, either in gas or solvent. To get the highest commercial efficiency it will pay to purify the gas and select solvents of highest purity. Care with reference

to the purity of gas and solvent is at present not given the attention in this industry that it deserves.

The detailed method of carrying out this absorption test was as follows:

A carefully measured volume of the solvent (usually 1.5 cc.) was placed in an ordinary five inch side neck test tube, fitted through a two-hole rubber stopper with a glass tube gas inlet, and a thermometer, the bulb of which was immersed in the solvent.

This tube with solvent was immersed in a freezing mixture (ice and salt) and cooled to -18° or -19° C. before starting. The purified gas circulated through a four foot coil immersed in the freezing mixture, thus bringing the gas to the temperature of the solvent.

The acetylene was bubbled through the absorption tube at the rate of about one bubble per second. Since volatile solvents are appreciably vaporized during this process of saturation, the exit gas and vapor in each case was passed through an ordinary potash bulb containing 95 per cent. alcohol; thus the amount of vaporized solvent was determined and proper correction made on the volume of solvent actually used. In twelve minutes the amount of solvent usually taken was completely saturated with the gas at atmospheric pressure; pressure and temperature were always noted. The exit of the absorption tube was then connected to a similar tube two-thirds full of saturated calcium chloride solution which in turn had been saturated with acetylene. The calcium chloride was connected to an ordinary Hempel measuring burette (the liquid in the latter also being a saturated solution of calcium chloride subsequently saturated with acetylene).

The purpose of the calcium chloride was to absorb any solvent vapor that might be carried out in the evolution of the gas, and which would otherwise be measured with the gas giving too high a result. The saturated calcium chloride solution has a very low absorptive capacity for acetylene and it has been proved that it condenses and absorbs completely the vapors of each of the solvents tested. The efficiency of the saturated calcium chloride as an absorbent for the vapor of the various organic liquids used for acetylene solvents was demonstrated by boiling the solvents, and passing the vapor into such an absorbent tube, when the absorption was found to be complete. In several of the experiments noted below, evolved gas from the measuring burette was bubbled back through a "potash" bulb containing 95 per cent. alcohol, but no trace of solvent was found.

Gas evolution begins soon after the absorption tube is removed from the freezing mixture. While the solvent was saturated at -18° C. usually, to guard against the possibility of the solvent not being saturated at the place taken as the starting point, the readings were not noted until the temperature of the solvent had risen to -10° C. The gas evolved from a known volume of the solvent, saturated at -10° C. (since if gas is evolved between -18° and -10° it must be saturated at -10° C.) up to 30° C. is then measured, the figure obtained being recorded in each of the de-

¹ Paper presented at the Fifth Annual Meeting of the American Institute of Chemical Engineers, Detroit, December, 1912.