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NO. 1

## THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY

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## EDITORIALS.

### THE NEW YORK CHEMISTS' BUILDING.

A most significant event in the annals of American chemical development is the incorporation of the Chemists' Building Company, of New York City. Enterprising New York chemists have had the matter under consideration for some time and the plans, as now put forward, indicate a wealth of detail and carefully thought-out plans, worthy of highest respect and general support. *The Percolator*, the bulletin of the New York Chemists' Club, for November 15th makes the following statement:

"The incorporation of the Chemists' Building Company is now complete, the incorporators being Messrs. Baekeland, Bogert, Coblenz, Elliott, Hasslacher, McMurtrie, Plaut, Baskerville, Chandler, Doremus, Falk, Loeb, Nichols, Jr., I. F. Stone, Toch.

"At a meeting held on Friday, November 12th, the following directors were chosen:

"Morris Loeb, *President*; Charles F. Chandler, *Vice-President*; Albert Plaut, *Treasurer*; W. H. Nichols, Jr., *Secretary*; Leo H. Baekeland office.

"The architects are busy with the plans of the building, and it is hoped that estimates can be obtained

shortly. The Finance Committee, on the other hand, is still lacking about \$30,000 in subscriptions before the directors will be warranted in declaring the plan operative, and it is the duty of all those interested in the project to lighten the arduous labors of this committee.

"While the Chemists' Club will profit largely by the erection of this building, the fact cannot be emphasized too strongly that all the chemists of the United States will be benefited to an equal degree, not merely those residing in and about New York. Non-residents will find it a very suitable stopping place during their occasional visits to the city, and particularly convenient by reason of the library and other professional conveniences which this building will contain. This library in itself will be made so complete that it will be the natural bureau of information for all members of our various chemical societies, and the museum, if the present plans are carried out, will perform for chemistry the services which the Smithsonian Institution renders to natural history. The individual laboratories will frequently enable the temporary resident of New York to perform the experiments essential to the progress of his work, as well as affording commercial and research chemists, resident in the city, but not attached to any particular institutions or corporations, an opportunity to carry out their work economically and expeditiously.

"Our members have, therefore, every incentive to subscribe themselves, and to urge such companies or individuals to contribute to its funds, who owe their own business success, in a large measure, to the advance of chemical science.

"To eliminate all possible misunderstanding, we again repeat that the Chemists' Building Company is independent of any management from the Chemists' Club. Subscribers to the stock will have a permanent equity in New York real estate, and while their dividends are limited to 3 per cent., so long as the Chemists' Club is in prosperous existence, they are to be shareholders in a well-constructed office building, in the best possible business location, should the Club, for any reason, fail to fulfil its present prospects.

"This is a very different plan from the ordinary scheme, whereby a Club erects its own building on a very slender margin, and obtains additional funds by selling bonds to its own membership, which can only be liquidated through expensive foreclosure proceedings.

"We have, therefore, no hesitation in asking persons who do not themselves expect to profit by the

social advantages of the Club, to subscribe liberally for the erection of the building, which is to be devoted to the advancement of Chemical Science throughout the United States.

"Subscriptions now amount to \$210,000 received from the following subscribers:

"Jerome Alexander, Edward G. Acheson, John Anderson, L. H. Baekeland, Wilder D. Bancroft, John T. Barry, A. A. Breneman, Percy S. Brown, W. Bowman, Chas. A. Browne, E. A. Byrnes, Cassella Color Co., C. F. Chandler, W. B. Cogswell, B. F. Drakenfeld & Co., Dr. J. Douglas, Wm. H. Erhart, Arthur H. Elliott, Fritsche Bros., General Chemical Co., Wm. S. Gray, Grasselli Chem. Co., Theo. Geisenheimer, Edward Gudeman, Mrs. Esther Harrmann, Jacob Hasslacher, Heyden Chemical Works, Dr. C. F. Hirschland, A. von Isakovics, H. J. Krebs, A. Kuttroff, Lerger Aniline & Extract Co., Morris Loeb, H. Lieber, A. Lichtenstein, A. R. Ledoux, Adolph Lewisohn, A. C. Langmuir, Parker C. McIlhiney, H. A. Metz, John McKennon, Jr., Dr. McKenna, Wm. H. Nichols, Niagara Electrochem. Co., Nichols Copper Works, Nat'l Aniline & Chem. Co., Emil Pfizer, Chas. Pfizer, Jr., Herbert Phillip, A. L. Plaut, Powers-Weightman-Rosengarten Co., Franz Roessler, Roessler & Hasslacher Chem. Co., Clifford Richardson, W. E. Rowley, I. F. Stone, Nathan Sulzberger, C. C. Speiden, C. F. Stiefel, Schoellkopf, Hartford & Hanna Co., R. Seldner, Henry M. Toch, Maximilian Toch, J. Takamine, C. P. Townsend, A. P. Van Gelder, Dr. Edward Weston, H. W. Wiley, David Wesson, Dr. Whitney, W. Hull Wickham.

It is noticeable that in practically all of the cities the engineers' clubs are better housed than are the chemists', or, indeed, other scientific organizations representing but a single branch. It is true, that at various points academies of science, scientific museums, and similar institutions are equipped with excellent buildings and apparatus, chiefly, however, for museum purposes. Nevertheless, outside of colleges and universities the scientific branches have altogether inadequate meeting-places. This condition will not obtain for any great length of time; the New York chemists have shown the way and set the pace.

With the increase in the number of chemists working in the pure science and in industrial lines, it may be expected that in the near future we shall see buildings erected in all the principal cities to be used as meeting-places for chemical organizations, for club rooms and for laboratories, following to a large extent the plants developed by the New York members of the profession.

#### THE LENGTH OF A TECHNICAL PAPER.

WE Americans are a busy people and as such are in the habit of using and encouraging the use of time-saving devices in our business. Time is money, especially in business hours, and has a distinct value even in the hours of relaxation, when serious matters are to be considered or read.

The short sermon, terse and meaty and right to the point, is the kind the modern church-goer demands, instead of the hour-long variety of our ancestors.

The presidential message that covers a whole newspaper page is read by comparatively few. If it were boiled down to two or three columns, nearly all would read it entire. As it is, for lack of time most people read abstracts of it, or editorial comments.

Even the advertisement must be short and pithy to be read by many.

Only for purposes of entertainment or recreation, as in the case of lecture, novel or play, does the average busy man tolerate with indifference the long-drawn-out or padded feature of the work, and even then it must be extremely clever, witty or entertaining to merit his approval.

Under every-day business or professional conditions, and applying with special force to technical papers, whether for delivery before scientific bodies or for publication in the journals, a good motto to adopt is "make them short" if the author desires them to be widely appreciated and read.

How often have we sat through conventions, listening politely to long-winded papers that would seem to never end and from very weariness deprived of the mental effort to comprehend. It reminds us of the old Scotch clergyman, who when asked why he made his sermon so long, replied that he didn't have time to make it shorter.

An author should remember that no one is quite so intensely interested in his particular paper as he is himself, and, perhaps, to a diminishing degree a few other kindred souls pursuing the very same line of work. At the same time there are many others who would like to read it if it were really readable. Many a busy reader in glancing through the columns of a journal sees an article on a subject that casually interests him, which, if short, he would read at once. As it is, the mere length deters him and he passes it by for the time, fully meaning to read it in the future, but probably never doing so.

Having gotten together material for a paper, would it not be well in all cases before submitting it for publication to see if it cannot be pruned down and deprived of useless material, crystallizing out the main features and presenting them tersely, and, if possible, in an entertaining form. The ability to write a technical paper in such a manner that to read it is a pleasure is indeed a rare gift, though by no means an impossible one. To make the paper so concise that those interested will want to read it throughout, and not merely glance it through sufficiently to gather the main points or conclusions is a long step forward.

It is, of course, understood that in long or important investigations it is not always possible to avoid papers of considerable length, but too often, even in these cases, much condensation might with advantage be applied before sending them to the editor.

ALBERT E. LEACH.

## ORIGINAL PAPERS.

### ELECTRIC VACUUM FURNACE INSTALLATIONS IN THE RESEARCH LABORATORY OF THE GENERAL ELECTRIC COMPANY.

By W. C. ARSEM.

Received November 15, 1909.

In 1906 I described an electric vacuum furnace<sup>1</sup> at the Ithaca meeting of the American Electrochemical Society. Since that time other types of vacuum furnace have been developed and installed as a part of the equipment for high tem-

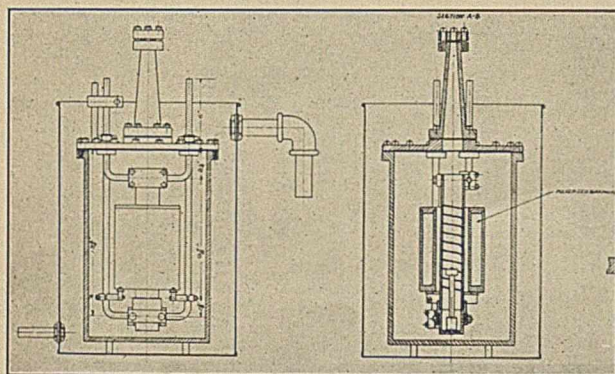


Fig. 1.—Small vertical vacuum furnace.

perature investigations in the General Electric Company's Research Laboratory at Schenectady, N. Y.

The general design of a vacuum furnace comprises a heater enclosed in a vacuum chamber, the heater being of such shape that it almost en-

<sup>1</sup> *Trans. Am. Electrochem. Soc.*, 9, 153, 171 (1906); *J. Am. Chem. Soc.*, 28, 921, 935 (1906).

tirely encloses the object to be heated. The heaters in the various types of furnace differ somewhat, according to the size of the furnace and the use to which it is to be put. In one case the heater is a graphite helix, in an upright position; in another case, it is a horizontal tube; and in another it is composed of 4 grids, made by sawing graphite slabs.

The vacuum chamber is a casting of bronze or gun-metal, the surfaces being machined and tinned to close the pores. All joints are made tight by lead gaskets. The electrode joints have

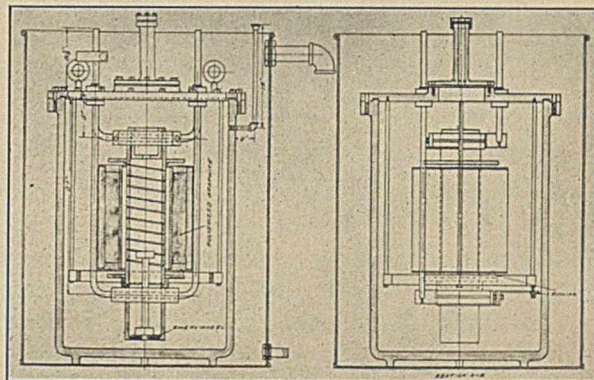


Fig. 2.—Large vertical vacuum furnace.

to be made so that they are air-tight, electrically insulated, and not liable to deterioration by heat. The same type of joint is used on all the furnaces and is illustrated in Fig. 5.

The "radiation screen" is a device for diminishing the amount of heat lost by direct radiation from the heater, and thus increasing the efficiency of the furnace. This is shown by the fact that the temperature *vs.* energy curve is approximately a semi-cubical parabola, whereas without the radiation screen, the curve would follow the Stefan-Boltzmann fourth-power law, like an incandescent lamp.

In the small vertical type furnace, the radiation screen is an annular graphite box, filled with graphite powder which is a poor conductor of heat.

Temperature calibration curves for the vertical furnaces are plotted by means of the equation  $(y-20)^n = ax$ , in which  $y$  is the centigrade temperature of a crucible in the furnace and  $x$  is the corresponding energy in kilowatts. The constants  $n$  and  $a$  are obtained by determining the energy necessary to maintain temperature equilibrium at the melting points of copper and platinum.

In the box type furnace I used Seger cones for calibration, because of their convenience.

Fig. 6 gives examples of calibration curves for the different furnaces.

The laboratory equipment consists of two installations, each containing four vacuum furnaces

with the necessary transformers, pumps, etc. I shall first describe the different types of furnace:

*Small Vertical Type (Fig. 1).*

This is the type described in the article above referred to. The heater is a vertical graphite helix, in the centre of which may be supported a crucible

using a maximum of 15 kw., but most experiments do not require a higher temperature than 2500° C., which can be attained with 10 kw.

This type of furnace is especially useful for small scale experiments that can be performed in crucibles 1½" diameter and 4" high. Of the various

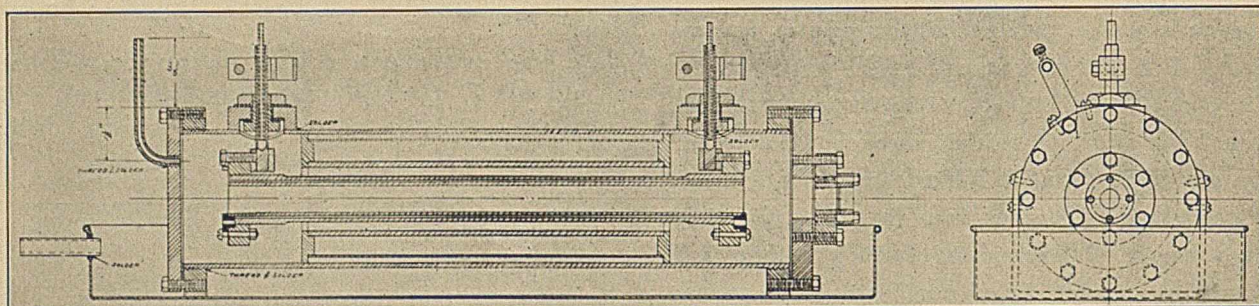


Fig. 3.—Horizontal tube furnace.

of carbon, graphite, alumina, tungsten or other material, according to the nature of the experiment.

The temperature can be quickly brought to any desired point, as determined by the calibration curve, and maintained constant for long periods,

uses which naturally suggest themselves, I mention the following:

Preparation of metals, alloys, carbides, silicides and other compounds.

Determination of melting points of metals, alloys, glazes, slags, refractories, etc., by an optical pyrometer, or by reference to the furnace calibration curve.

Calibration of optical pyrometers.

Distillation of refractory substances for separation or purification.

Study of equilibrium in reactions depending upon the pressure of the gaseous phase.<sup>1</sup>

Many reactions can be studied quantitatively with accurately weighed quantities.

*Large Vertical Type (Fig. 2).*

This is similar to the small vertical furnace, but its construction is somewhat modified because of the size and weight of the parts.

The heater is a vertical helix 5½" outside diameter, 4½" inside diameter, and 21" long. The radiation screen is an annular box, octagonal outside and cylindrical inside, and was built up in sections (see Fig. 11).

A crucible 10" high and 4" in diameter can be heated to 2500° or higher. This is the largest vertical vacuum furnace yet constructed for laboratory use.

It can be used for the same purposes as the

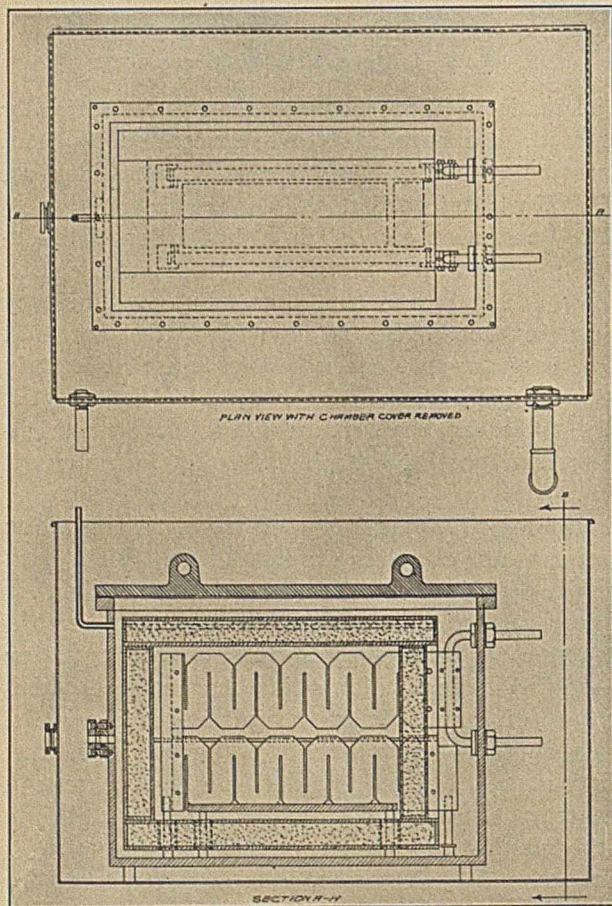


Fig. 4.—Box type vacuum furnace.

while the behavior of the article being heated may be observed through the mica window at the top. The range of temperature extends to 3100° C.,

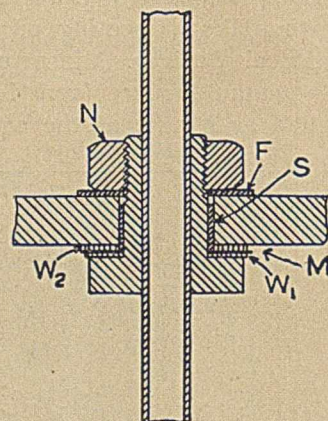


Fig. 5.—Electrode joint: W<sub>1</sub> and W<sub>2</sub>, lead washers; M, mica washer; F, fiber washer; S, fiber sleeve. N, brass nut.

<sup>1</sup> C. A. Hansen, *Electrochem. Ind.*, 7, 427-29 (1909). M. deKay Thompson, *Trans. Am. Electrochem. Soc.*, 1909.

smaller furnace of the same type, with the special advantage of greater capacity. Some of the rarer metals, for instance, can be made in commercially important quantities.

*Horizontal Tube Type (Fig. 3).*

The heater is a thin-walled graphite tube, held in graphite clamps with a tight sliding fit, to permit

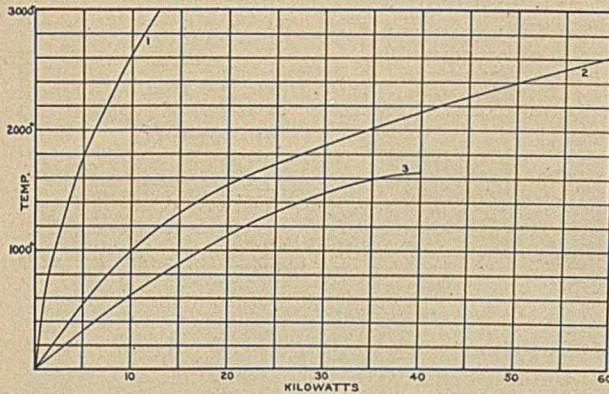


Fig. 6.—Calibration curves: 1, small vertical type; 2, large vertical type; 3, box type.

expansion and contraction. The radiation screen is of the same form as that used in the small vertical furnace, except that its outer wall is made of

brass. The electrode tubes are of somewhat different design from those in the other furnaces, as is seen in the sectional view.

To protect the rather frail heater there is an inner tube of graphite, insulated from the heater at the ends. This inner tube has an internal diameter of  $1\frac{1}{8}$ " and a length of 21", of which 10" is quite uniformly heated. There is a window at one end, for observation purposes.

The furnace is especially well adapted to the annealing or heat treatment of metal bars or rods.

It takes 4 kw. at  $1700^{\circ}$ , or about 400 amperes at 10 volts.

*Box Type (Figs. 4 and 7).*

The chamber of this furnace is a rectangular gun-metal box, with a removable cover. The heater is made up of four graphite grids, connected in series. These grids are made by sawing slots in graphite slabs  $5" \times 18" \times \frac{3}{4}"$ .

The heated space enclosed by the grids is large enough to take a box-shaped crucible 4" wide, 12" long and 8" high, or 384 cubic inches.

The radiation screen is made up of 8 box-like sections, each hollow and fitted with graphite

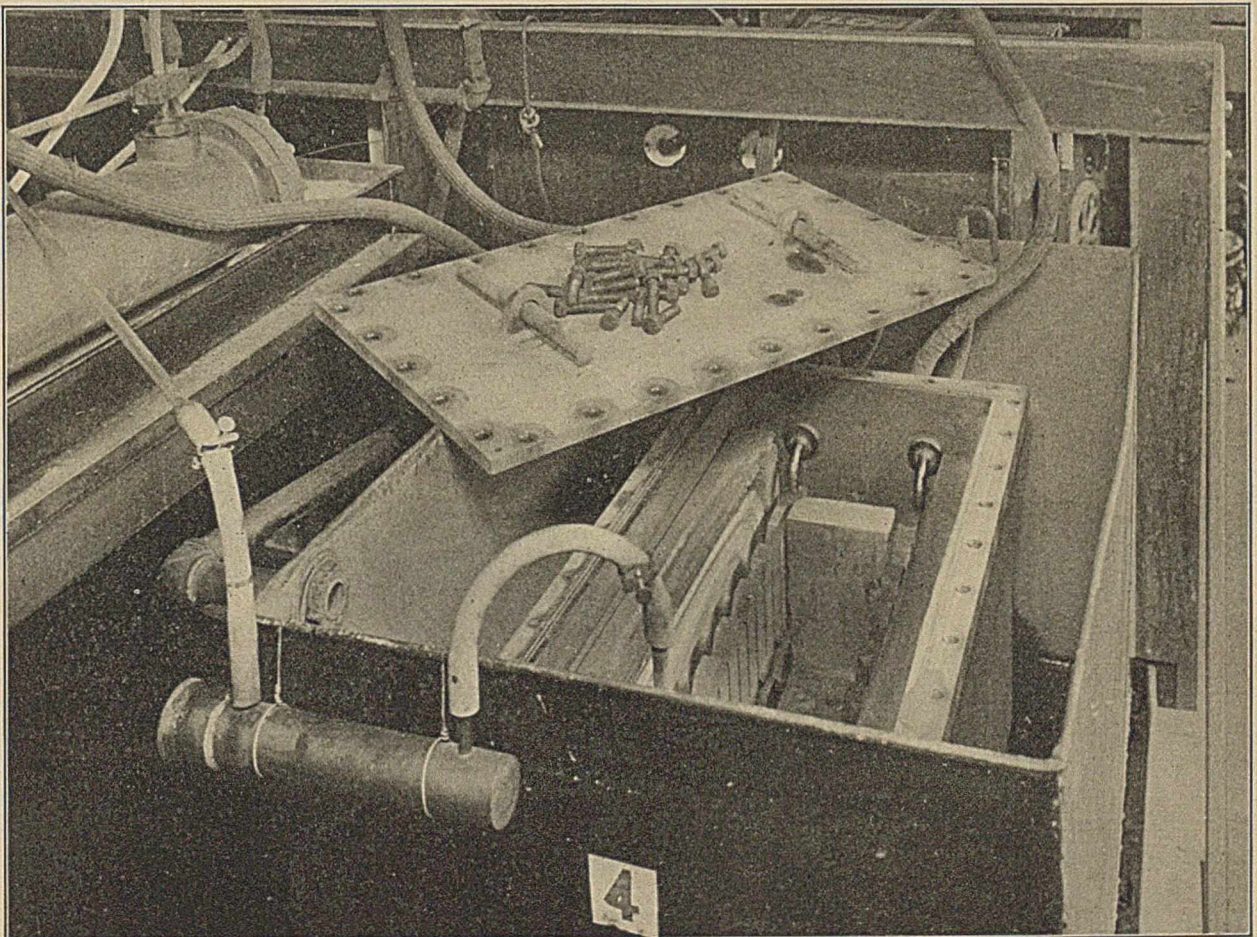


Fig. 7.—Box type vacuum furnace.

powder, the top section being removable when charging the furnace.

There is a window at one end of the furnace which is in line with a window in the water-jacket and with a hole in the end section of the radiation screen, thus giving a good view into the hot space. Fig. 7 shows the interior of the furnace, looking from above, the top section of the screen having been removed. The heater grids are plainly visible.

This furnace may be used for making fairly large amounts of materials which are otherwise

1700° C., while that of the large vertical furnace is over 2500° C.

TABLE OF FURNACE DATA.

	Small vertical.	Large vertical.	Horizontal tube.	Box type.
Maximum kilowatts.....	15	60	4	40
Maximum current, amperes.	250	800	400	570
Maximum voltage.....	60	75	10	70
Maximum temperature.....	3100°	2600°	1700°	1700°
Crucible volume.....	5 cu. in.	125 cu. in.	7.8 cu. in.	344 cu. in.
Crucible height.....	4 in.	10 in.	10 in.	8 in.
Crucible section.....	1 1/4" diam.	4" diam.	1" diam.	4" by 12"
Height of furnace.....	21 in.	48 in.	15 in.	24 in.
Base of furnace.....	15" diam.	36" diam.	12" by 36"	24" by 36"

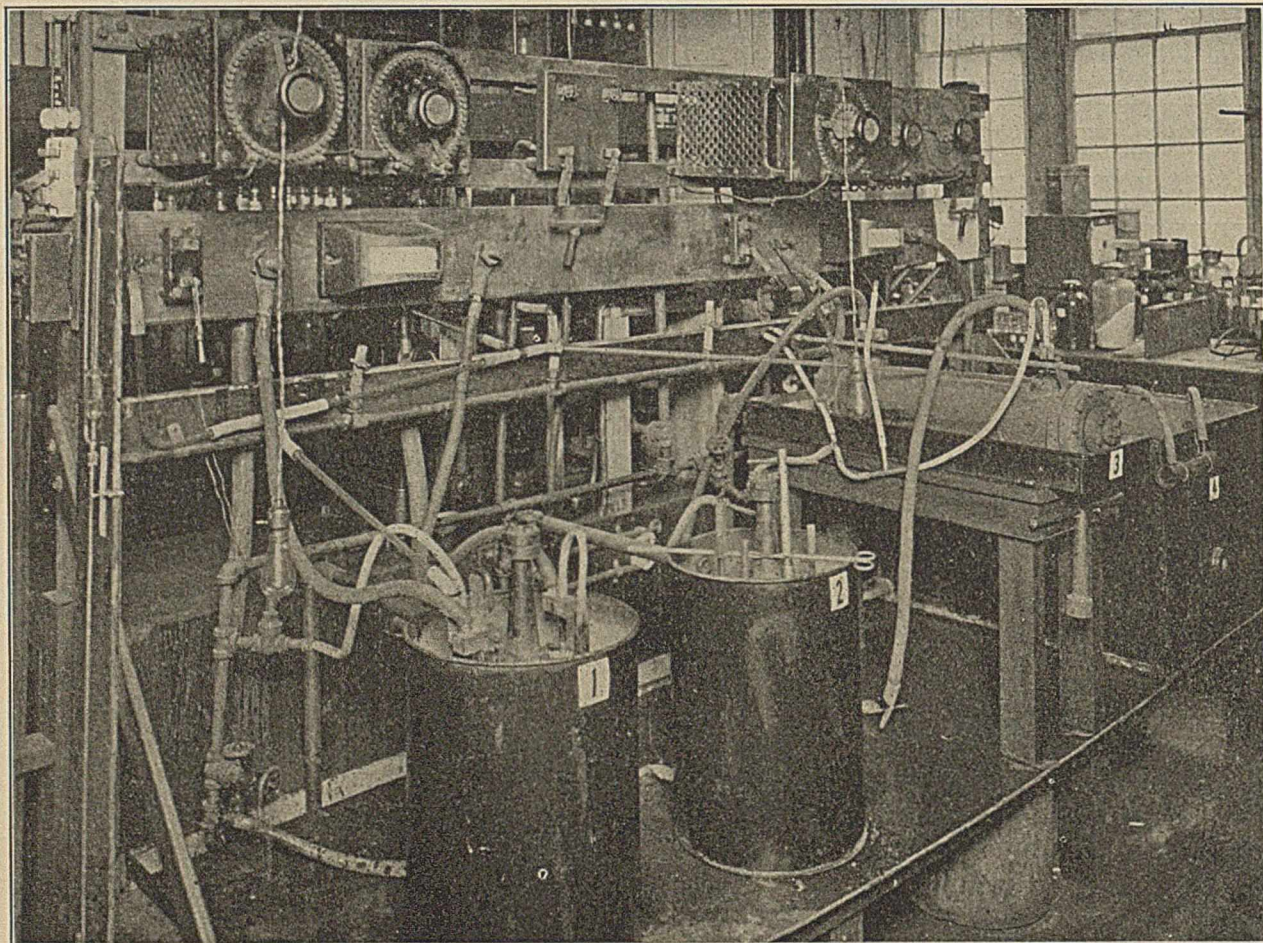


Fig. 8.—Front of Installation A.

difficult to produce, as, for example, certain rare metals which can be made in quantities of several pounds by reducing their oxides by carbon.

To perform such an experiment it is the work of only a few moments to put the charge in, fasten the cover down, exhaust the furnace, and start the run. As the temperature of the reaction is under perfect control and the material not exposed to oxidizing influences, the conditions are ideal.

The capacity of this furnace is considerably greater than that of the large vertical type, but its temperature limit in its present form is about

#### Installation A.

This installation consists of four furnaces. No. 1 and No. 2 are of the small vertical type, No. 3 horizontal tube type, No. 4 of the box type. These furnaces are set up in a row upon a low bench, having a galvanized iron top made in the form of a shallow pan, which drains into a deep sink behind the furnaces. This arrangement takes care of the outflow from the water jackets of the furnaces. Behind the furnaces are arranged the pump and the transformers.

Furnaces Nos. 1 and 2 are operated by a 25 kw.

transformer, and Nos. 3 and 4 are run on a "flux shunt" transformer, such as is used for thawing water-pipes. This is rated at 30 kw., but can be overloaded to 40 kw. for short periods. The energy supply to the furnace is usually controlled by rheostats in the field circuit of the generator supplying the current. The "flux-shunt" transformer, however, may be connected to any 1100 or 2200 volt circuit, and the voltage on the secondary side, which is connected to the furnace, may be regulated by simply turning a hand-wheel to raise or lower the "flux-shunt."

0.03 mm. of mercury in a cold furnace of the small vertical type.

The pressure during a run depends, of course, upon the nature of the charge, but an excellent vacuum can be maintained at the highest temperatures if the charge does not liberate gases.

Between the furnaces and transformers stands a switchboard upon which are mounted the switches, field rheostats, wattmeters, and transformer terminals. Water for cooling purposes is supplied by a tap near each furnace.

At the end of the switchboard stands a McLeod

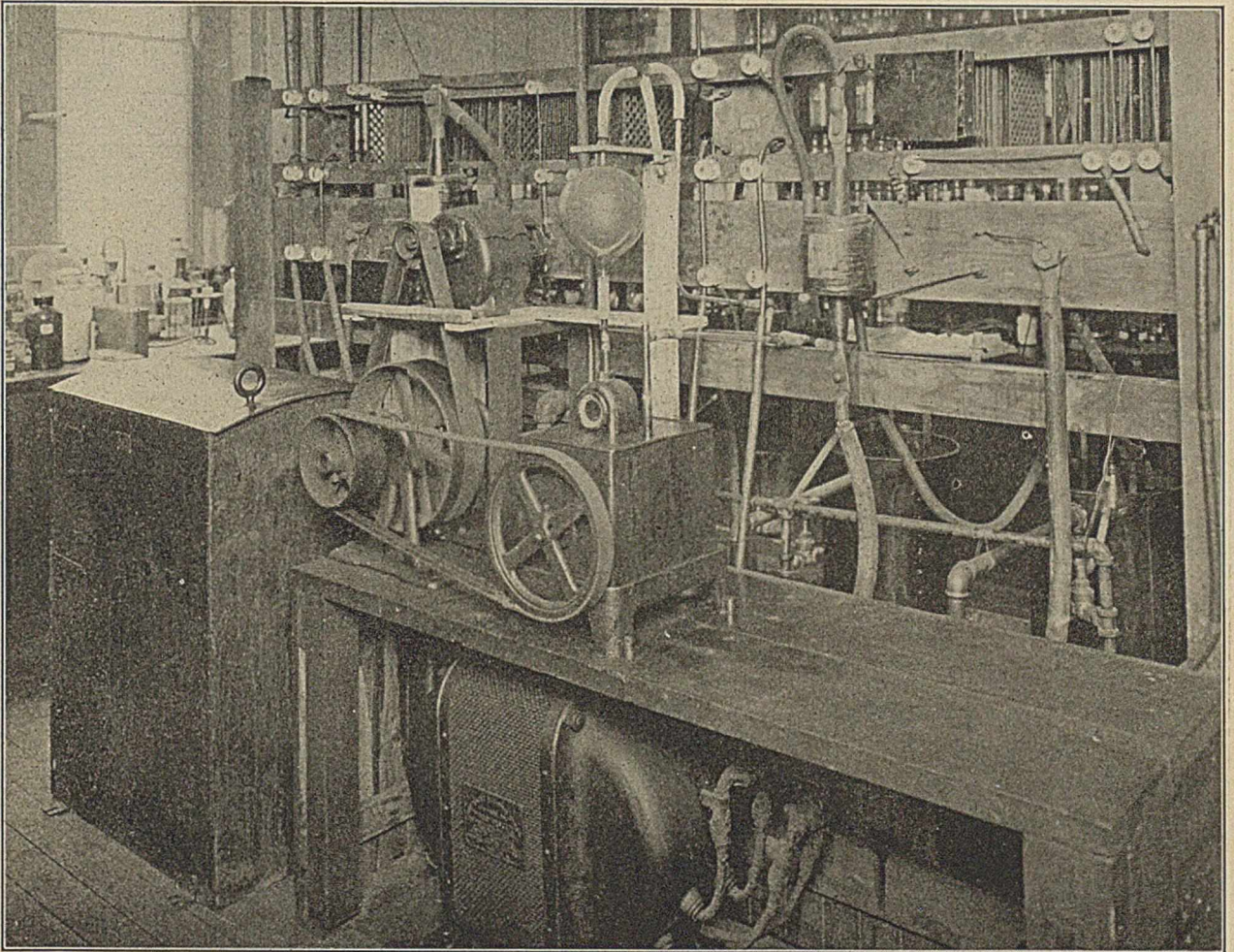


Fig. 9.—Back of Installation A.

The Siemens-Schuckert rotary oil-pump used for evacuating the furnaces is mounted on a bench over the 25 kw. transformer, the discharge side of the oil-pump being connected to the laboratory "rough-pump." In exhausting a furnace it is customary to first reduce the pressure to 4 or 5 mm. by means of the rough pump, and then to use the rotary pump to get a vacuum good enough for operating the furnace. With the rotary pump there is no difficulty in reducing the pressure to

gauge for measuring pressure in the furnaces more accurately than can be done with an ordinary manometer.

Figs. 8 and 9 show the front and back of installation A.

#### *Installation B.*

This installation consists also of four furnaces. Nos. 5 and 6 are small vertical furnaces, No. 7 is a box type, and No. 8 is a large vertical furnace.

Two double-cylinder Geryck pumps and a three-

cylinder Packard pump are used for evacuation, in connection with the laboratory "rough pump."

Regular General Electric Type H transformers are used to supply current, regulation being effected by field control. The usual form of mercury manometer is used here.

These furnaces are in charge of a skilled furnace operator, who performs any experiments desired by the members of the laboratory force.

A general view of this installation is shown in Fig. 10.

The heaters are inexpensive, and the replacement of one is a very simple matter.

Over 500 runs have been made in one of the small vertical furnaces, and over 200 runs in the box-type furnace No. 4. The total number of runs made in all these furnaces to date is about 2000.

A number of institutions of learning have installed small vertical vacuum furnaces in their electrochemical departments. Among these are Cornell, Mass. Institute of Technology, New Hampshire College and Rensselaer Polytech. Inst. The

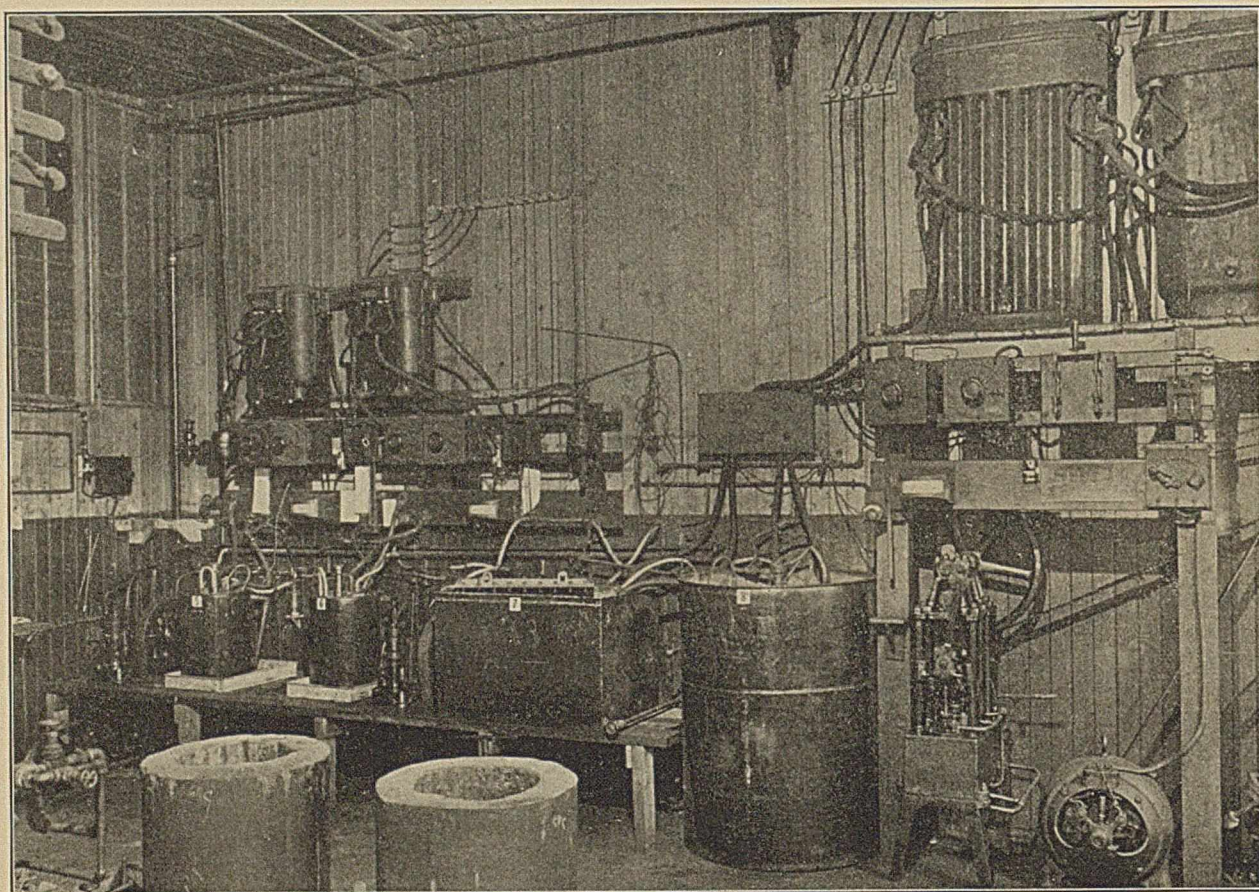


Fig. 10.—Installation B.

As regards the durability of these furnaces, the following may be of interest.

Since the furnaces are evacuated, there is, of course, no wasting of the heater and other graphite parts by oxidation.

Volatilization or disintegration of graphite begins to be apparent at  $2500^{\circ}\text{C}$ ., so that at this temperature a heater will waste away at its middle point in about 9 hours, while at  $3100^{\circ}$  it will last less than one hour. Up to  $2000^{\circ}$ , however, graphite does not volatilize appreciably, so that a heater which is never run above this temperature ought to last indefinitely.

Bureau of Standards at Washington also has a furnace.

In designing installations where field control as a means of regulating the voltage on the furnace is not permissible, several other means of securing variable voltage are available:

1. A transformer with a series of taps giving different voltages.
2. An ordinary transformer used in connection with an induction regulator.
3. A "flux-shunt," or pipe-thawing transformer.

A very convenient installation for small scale experimental work would consist of a small vertical



type vacuum furnace, a 10 kw. "flux-shunt transformer, a Geryck pump, and a 15 kw. wattmeter.

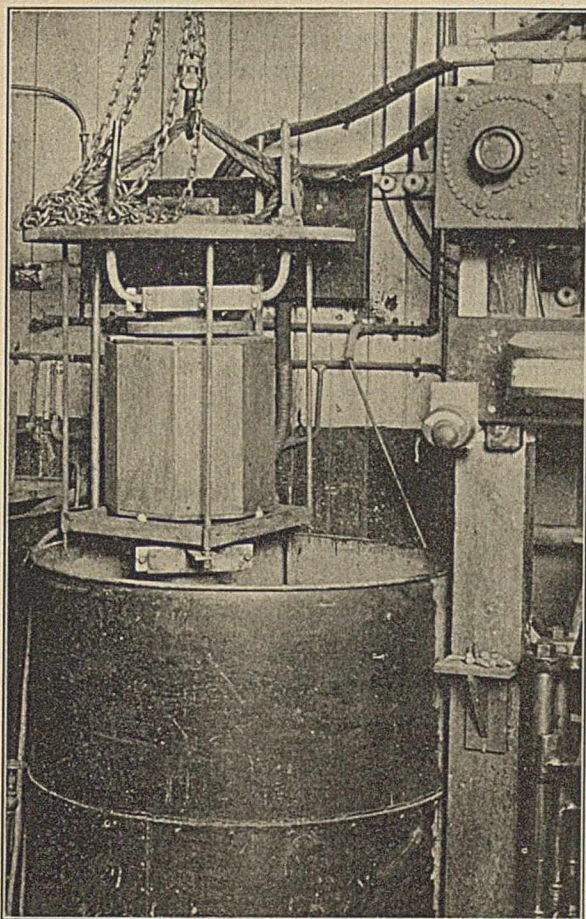
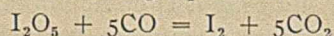


Fig. 11.—Large vertical type vacuum furnace, showing radiation screen.

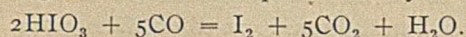
### THE DETERMINATION OF CARBONIC OXIDE IN ILLUMINATING GAS BY IODINE PENTOXIDE.

By AUGUSTUS H. GILL AND EDWARD H. BARTLETT.

The reaction made use of in this method for the quantitative determination of carbon monoxide is that first indicated by Ditte.<sup>1</sup> He showed in 1870, that if carbon monoxide was passed over iodine pentoxide at temperatures between 150° and 200° C., a reaction ensued by means of which the carbon monoxide was oxidized to carbon dioxide, this action being accompanied by a simultaneous reduction of the I<sub>2</sub>O<sub>5</sub> to free iodine, expressed by the following equation:



An analogous reaction is produced by iodic acid,



Apparently the first to actually make use of this reaction were de la Harpe and Reverdin,<sup>2</sup> who used it in 1888 for the detection of carbon

monoxide in the atmosphere. They passed filtered air over dry iodic acid heated to 150° C. and then into a solution of starch paste. If the air contained carbon monoxide this fact was made apparent of the blue color in the starch solution.

Phillips,<sup>1</sup> in 1894, mentions this reaction, and says that before it can be used for analytical work it is necessary to remove the higher olefines, acetylene, and vapors of benzene and alcohol. He says further that the lower paraffins are without action on the iodine pentoxide up to temperatures at which the iodic acid dissociates.

In 1898 both Nicloux and Gautier report having used this reaction quantitatively. Gautier did not publish his method until after that of Nicloux had appeared. He claims, however, to have been using his method for several years previously. Both confined their attention to the determination of carbonic oxide in the small quantities in which it is met with in the air.

Nicloux<sup>2</sup> first removed carbon dioxide, sulphuretted hydrogen, sulphurous acid and water vapor from the air, and passed it over iodic anhydride at a temperature of 150°. The iodine set free was carried along by the stream of air through a tube filled with a solution of potassium hydrate—where it was absorbed. The amount of iodine was then determined by the method of Rabourdin, which consists in acidifying the potassium hydrate solution with sulphuric acid, adding a few centigrams of nitrite of soda and 5 cc. of chloroform or carbon bisulphide, and then shaking the whole vigorously. This sets free the iodine which is then taken up by the chloroform or carbon bisulphide; the color of the solution obtained is compared with the color produced from a solution of potassium iodide, the strength of which is determined by titration. Nicloux tested this method by using air, the contents of which in carbonic oxide varied from 1 part in 1000 to 1 part in 50,000. He reports his results to have shown the method to be very accurate. He also says that neither hydrogen nor methane give an analogous reaction at this temperature.

Gautier<sup>3</sup> devised two quantitative methods using this reaction. In his first method, he passed the air first through a tube containing I<sub>2</sub>O<sub>5</sub>, and then through a small tube filled with pulverulent copper. The iodine set free by the reaction was taken up from the air by copper, after which its weight was obtained by taking the difference between the weights of the tube of copper before and after the experiment. Gautier soon abandoned this method, however, for another one in which he determined

<sup>1</sup> *Am. Chem. Jour.*, 16 (1894).

<sup>2</sup> *Compt. rend.*, 126, 746.

<sup>3</sup> *Ibid.*, 126, 793 and 931.

<sup>1</sup> *Bull. Soc. Chim.*, 13, 318.

<sup>2</sup> *Chem. Z.*, 12, 1726.

the amount of carbon dioxide formed instead of the iodine set free. He now passed the air after it had been in contact with the iodine pentoxide into a tube filled with glass beads, which contained a known volume of potassium hydrate solution free from carbonate. The carbon dioxide thus absorbed was again reobtained in the gaseous state by neutralizing the potassium hydrate solution with sulphuric acid. The volume of carbon dioxide thus obtained equaled the volume of carbonic oxide in the volume of air taken for analysis. Gautier investigated the accuracy of this method, using dilutions of carbonic oxide varying from 1 part in 500 to 1 part in 30,000 and keeping the tube of iodine pentoxide at temperatures ranging from 65° to 80°. He states that the method is fully accurate enough for practical purposes.

Gautier says that he has observed that the oxidation of the carbonic oxide begins at temperatures of less than 30° C., that it is active between 40° and 45°, and complete at 60°-65°. He says that this is true whatever be the dilution of the carbonic oxide in the air. With the exception of acetylene he says further that most of the hydrocarbons are not acted upon by iodine pentoxide at these temperatures. Certain gases like ethylene, however, although themselves being oxidized very difficultly at these temperatures, nevertheless have the property of hindering greatly the oxidation of carbonic oxide. He says further that contrary to the general rule this reaction goes completely, the carbonic oxide being oxidized to the very last trace. For every cubic centimeter of carbonic oxide at 0° and 760 mm. there is set free 0.00227 gram of iodine. Finally, he says that at these temperatures, 65°-80°, methane and hydrogen are not oxidized.

The problem of adopting this reaction for use in the determination of the carbonic oxide in the atmosphere was next taken up by Kinnicutt and Sanford<sup>1</sup> in 1899. By way of premise they say that all the methods which have been used for this purpose may be divided into two classes, to wit: those employing the reaction between carbon monoxide and the haemoglobin of blood by means of which carbon monoxid haemoglobin is formed, and those in which carbonic oxide is oxidized to carbon dioxide. The processes of the first class are of but little value from a quantitative standpoint. The most suitable oxidizing agent for use in the processes of the second class has been found to be iodine pentoxide. They report that they experimented with the methods of Nicloux and Gautier, and failed in both instances to obtain accurate results. They then proceeded to devise a method of their own. Their method is a modi-

fication of that of Nicloux. They passed the air first through a tube containing pieces of stick potash and a tube containing sulphuric acid, in order to remove from it sulphuretted hydrogen, sulphurous acid and similar reducing gases and all unsaturated hydrocarbons. They then passed the air through a tube containing iodine pentoxide heated to a temperature of 150°, after which it was passed into a solution of potassium iodide which absorbed the iodine. Finally they titrated this solution with a N/1000 sodium thiosulphite solution to determine the iodine. By measuring the carbonic oxide used in making up their samples in capillary tubes, they were enabled to read the volume to 0.001 cc. The content of carbonic oxide varied from 1 part in 2440 parts of air to 1 part in 43,479 parts of air. They report the results of fourteen analyses which show the method to have been very accurate. They found that they could determine as little as 2.5 vols. of CO in 100,000 volumes of air.

They next made experiments to determine whether or not the presence of illuminating gas in the air would vitiate the accuracy of these results. They found that the presence of the gas had no disturbing effect whatever, as is shown by the following results:

	cc.	cc.
Carbonic oxide calculated.....	0.0191	0.02789
Carbonic oxide found.....	0.019	0.027

This indicated that hydrogen and methane were not acted upon at this temperature. They had previously made experiments which had shown the same result. They also made experiments which showed that the reaction was not quantitative at temperatures lower than 150°. This is in direct contradiction to Gautier who says that the reaction is complete at temperatures as low as 60°-65°. These results of Kinnicutt and Sanford show that this method gives very accurate results when applied to the determination of carbonic oxide in the small quantities which are found in the atmosphere.

In 1900 Smits, Raken, and Terwogt,<sup>1</sup> of the University of Amsterdam, proposed utilizing this oxidation of carbon monoxide for its quantitative determination in the analysis of illuminating gas. After reviewing briefly the work of Nicloux and Gautier, they say that it is obvious that this "very sensitive" reaction, heretofore only used in the analysis of air, is also applicable to the determination of carbon monoxide in illuminating gas. They say, however, that it is out of the question to think of placing the carbonic oxide determination in line in a complete gas analysis, if the exact method of either Nicloux or Gautier is used. They

<sup>1</sup> *Jour. Am. Chem. Soc.*, 22, 14.

<sup>1</sup> *Zeit. für angew. Chem.*, 1900, 1002.

therefore modified Gautier's method and proceeded as follows: The gas from which the carbon dioxide, the illuminants, and oxygen had been removed in the ordinary Hempel scheme of analysis, was passed from a Hempel burette, through a U-tube containing iodine pentoxide heated to 150° by an oil bath into a Hempel pipette containing a solution of caustic potash. The caustic potash absorbed the carbon dioxide formed by the oxidation of the carbonic oxide so that the contraction in volume after the absorption was complete gave the volume of the latter present. The correction for the vapor tension of iodine was found to be negligible. It does not appear from their article that they ever actually tried their method in an actual analysis of illuminating gas. They did, however, undertake to find out whether or not hydrogen or methane would undergo oxidation on being passed through the iodine pentoxide tube. To this end they made by their method an analysis of a mixture of hydrogen and carbon monoxide, and another analysis of a mixture of methane and carbonic oxide. They report the following results:

	Found, cc.	Mixed, cc.
Hydrogen.....	72.7	72.6
Carbon oxide.....	8.7	8.8
Methane.....	42.7	42.8
Carbon oxide.....	8.1	8.0

It will be noticed that they give results for two experiments only, and that neither of these properly represents the conditions of practice, as in neither were both hydrogen and methane associated with the carbon monoxide. They do not hesitate, however, to say that their method is very exact and much superior to that in ordinary use.

The method of determination of carbonic oxide by cuprous chloride is so unsatisfactory that it was thought this promised well, and their work was repeated with the following results:

TABLE I.—SHOWING ABSORPTION OF CARBONIC OXIDE BY IODINE PENTOXIDE.

CO used.	CO found.	Loss.	Per cent. lost.	Bath temp.	Time, min.
21.8	20.8	1.0	3.6	150	90
23.2	22.2	1.0	4.3	150	150
25.9	25.5	0.4	1.0	152	32
25.6	25.6	0.0	0.0	154	29
26.4	26.0	0.4	1.0	161	32

In the last three results the carbonic oxide was mixed with air. It will be noticed that after two and a half hours' passage of the gas through the tube more than 4 per cent. of the gas remained unabsorbed. This would seem to show the impracticability of the method.

To still further test the method a mixture of carbonic oxide, methane, hydrogen and nitrogen was made of about the composition of that left after carbon dioxide, "illuminants" and oxygen had been absorbed from an illuminating gas and

passed through the iodine pentoxide. The results are shown in Table II.

TABLE II.—ABSORPTION OF CARBONIC OXIDE BY IODINE PENTOXIDE FROM MIXTURE OF GASES.

Hydrogen.	Meth- ane.	Carb. oxide added.	Carb. oxide found.	Gain.	Per cent. gain.	Bath temp.	Time, min.
24.1	44.8	25.6	28.5	2.9	11.3	157	24
24.4	42.3	26.4	31.3	4.9	18.6	152	29
23.6	46.1	25.6	27.1	1.5	5.9	152	28
26.1	46.2	24.1	27.1	3.0	12.4	152	51
24.6	53.5	25.7	28.1	2.4	9.3	152	75
20.4	43.7	27.8	31.5	3.7	13.3	148	30
24.0	46.0	26.1	26.9	0.8	3.1	150	26

It will be noticed from this table that the amount of carbonic oxide found considerably exceeds the amount originally added: The errors of the analysis are not sufficient to account for this and they show no regularity. Moreover, another observer a year previously had obtained almost identical results, the average error found by him being 7.8 per cent., whereas the average error of these results is 9.1. This error, as was shown by two different observers working independently, is not due to the action of the iodine pentoxide upon either the methane or the hydrogen nor the number of times the tube had been used nor the length of time taken by the gas in passing through the tube, but may be due to the breaking up of the iodine pentoxide.

In conclusion it may be said that the method of absorption of considerable quantities of carbonic oxide by iodine pentoxide is inaccurate, irregular in action and tedious and troublesome to use.

Finally our acknowledgments are due to Messrs. J. B. Finnegan and R. W. Lindsay by whom some of the experiments here detailed were performed.

### THE COEFFICIENT OF EXPANSION OF GLYCERINE.

By ARTHUR M. COMEY AND CECIL F. BACKUS.

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Upon examination of the literature on the coefficient of expansion of glycerine, it was found there was some discrepancy between various authorities. Hehner<sup>1</sup> states that it amounts to 0.00058 for each degree centigrade in the neighborhood of 15.5° C. It can be calculated from the results of Gerlach's observations<sup>2</sup> that the value of this factor varies with the temperature according to the following table:

Tempera- ture.	Coefficient of expansion.
5.0°	0.00057
12.5°	0.000587
20.0°	0.00060
30.0°	0.000619

The necessity of using an accurate coefficient in figuring the results obtained by determining

<sup>1</sup> J. S. C. I., 8, 8 (1889).

<sup>2</sup> Zeit. anal. Chem., 24, 111.

the specific gravity of glycerine at temperatures varying considerably from 15.5° led to the following investigation:

*Weights and Balances Used.*—The weights and balances used in the following determinations were carefully checked just before beginning the work. The weights that showed slight errors were adjusted so that each was a multiple of the other. The balance beam was found to give accurate weighings when the load and weights were shifted to opposite pans.

*Water Bath.*—The water, or constant temperature bath, employed to keep the pycnometers at 15.5°, 20°, 25° and 30° C. for the standardizations and determinations consists of two wooden boxes with an air space of one inch between the sides and bottom. The inner box is twenty inches long, fifteen inches deep and fifteen inches wide, lined with tin, having a shelf 10 × 8 inches, eight inches from the bottom. The bath was filled with water so that it covered the shelf to a depth of four and one-half inches. When the pycnometers are set upon the shelf with the water at this depth, the capillaries are entirely submerged, the rubber tubing, described later, serving to protect the contents of the pycnometer. By entirely submerging the body and capillary of the pycnometers, the contents are brought to the same temperature throughout. After the proper temperature was attained, the water was bailed out or drawn off until about one-half inch of the capillary projected out of the water. The temperature of the bath can be regulated by the addition of small pieces of ice, cold water, or hot water to within very narrow limits—less than 0.05° C.

Several small streams of air served to keep the water agitated, the temperature of the bath being the same at all points at a given time.

*Pycnometer.*—Geissler specific gravity bottles of 50 cc. capacity with centigrade thermometer graduated to 0.2° ground into neck of bottle, and with side capillary tube with ground glass cap were used in these determinations.

*Detailed Description of the Method Employed for the Standardization of Pycnometers.*—Freshly boiled, distilled water was rapidly cooled to about 15° C. in a flask which was covered with a watch-glass to keep out the air. The capillary of the pycnometer was inserted into a piece of clean and dry rubber tubing (2.5 mm. in diameter), which was fitted snugly over the capillary and which was 63 mm. in length. The pycnometer was then carefully filled with the air-freed, distilled water by pouring down the side and the thermometer set firmly in place. It was then placed in the water bath and after the pycnometer-thermometer and the standard thermometer reached 15.5° C.,

the temperature was kept constant for fifteen minutes more.

The pycnometers were submerged about four and one-half inches in the water bath during the time required for the water in them to attain the desired temperature. The water in the bath was then drawn off until about one-half inch of the capillary projected out of it. The rubber tubing was then removed and the top of the capillary wiped with a dry finger before removing from the water bath. The pycnometer was taken from the bath immediately after the excess water was removed from the top of the capillary and the ground portion of the capillary wiped with a piece of filter paper and the cap placed on tight. The whole pycnometer was then quickly dried by wiping with a dry cloth and weighed as rapidly as possible, without swinging the balance (which is sensitive to 0.2 mg. without swinging) to the fourth decimal place. The pycnometers were all standardized at the same time, but each one was weighed before the next was taken out of the water bath, due to the fact that there is a gradual loss of weight if the pycnometers stand for even a few minutes when they contain water. The loss of water is probably due to evaporation through the ground glass joint between the pycnometer and the thermometer.

The water that is forced through the capillary into the rubber tube when the thermometer is set in place is enough to replace any contraction due to lowering of the temperature, provided the difference in temperature is not too great. The rubber tube also allows the capillary to be entirely immersed and yet keeps out the water of the bath.

*Standardization of Pycnometer-Thermometers and Pycnometers.*—The pycnometer-thermometers were compared with a standard thermometer, which has been checked against several good thermometers at four points, *viz.*: 15.5°, 20°, 25° and 30°, and corrections were applied whenever needed.

The capacities of the pycnometers were determined in duplicate in a water bath at 15.5° C., while the temperature of the room registered 18° to 20° C. The capacities at 15.5° C. for two of the pycnometers checked results obtained some time previous at room temperatures and figured to grams of water at 15.5° C. From the weight of water obtained at *t* degrees the capacity at 15.5° is obtained from the following formula:

$$C = W \frac{D}{d} \frac{1}{1 + a(t - 15.5)}$$

in which,

*t* = Observed temperature.

W = Capacity in grams of water at *t*° C.

D = Density of water at 15.5° C.

*d* = Density of water at *t*° C.

$a$  = Coefficient of cubic expansion of glass, which is taken as 0.000025 per 1° C. between 15.5° and 30° C.

$C$  = Capacity in grams of water at 15.5° C.

Table I gives the capacities found and the corrections applied to the thermometers.

TABLE I.—CAPACITIES AND THERMOMETER CORRECTIONS FOR PYCNOMETERS.

Pycnometer marked.	Duplicate determinations at 15.5°.	Average capacity used at 15.5°.	Average determinations at t° C.	Thermometer corrections.			
				15.5°.	20.0°.	25.0°.	30.0°.
A.....	{ 49.7422 49.7422	49.7422	49.7421	0	0	0	-0.20
B.....	{ 50.2178 50.2182	50.2180	50.2179	0	0	0	0
C.....	{ 50.1063 50.1063	50.1063	.....	-0.10	-0.20	-0.20	-0.30
G.....	{ 49.6595 49.6596	49.6596	.....	-0.10	-0.05	-0.10	-0.10
H.....	{ 49.9742 49.9736	49.9739	.....	-0.10	-0.10	-0.10	-0.15
I.....	{ 49.9535 49.9537	49.9536	.....	-0.05	-0.10	-0.20	-0.25
J.....	{ 50.7740 50.7743	50.7742	.....	-0.10	-0.15	-0.20	-0.15
K.....	{ 50.2140 50.2140	50.2140	.....	0	0	-0.10	-0.10
L.....	{ 50.1220 50.1217	50.1219	.....	0	0	-0.10	-0.15
M.....	{ 50.0172 50.0168	50.0170	.....	-0.10	0	-0.05	-0.05
N.....	{ 49.5540 49.5541	49.5541	.....	0	0	+0.05	0

*Detailed Description of the Specific Gravity Determinations; Glycerine Used and Method of Transferring it from the Large Sample Bottles to the Pycnometers.*—Several samples of dynamite glycerine were used in these determinations, which had been obtained from different manufacturers. These were tested as received and also diluted to cover specific gravities ranging from 1.254 to 1.264. The glycerine sample in each case was thoroughly shaken at times on three successive days and then allowed to stand four days to free itself of air bubbles before making the determinations. The glycerine was transferred directly from the centre of the large sample bottles to the pycnometers, through a siphon arrangement. With a slight air pressure, the glycerine would flow as rapidly as desired without air bubbles, thus eliminating the danger of any diluted glycerine from the neck of the sample bottle or from the surface of the sample getting into the pycnometer.

*Specific Gravity Determinations.*—The pycnometer was first dried with alcohol and ether and then subjected to a current of dry air to remove all traces of ether, weighed and filled by allowing the glycerine to flow from the bottle through the siphon arrangement down the side as rapidly as possible. When filled, the thermometer was inserted into the neck of the pycnometer until the glycerine stood in a globule on top of the capillary. A small quantity of glycerine was then drawn from the large sample into a small, dry beaker

and a dry rubber tube 2.5 mm. in diameter and 63 mm. in length, supplied with a glass tube drawn out to a fine capillary, was then filled from it by drawing up some of the glycerine. The rubber tube full of glycerine was then quickly placed on the capillary of the pycnometer and the thermometer set firmly in place. This forced the excess of the glycerine through the capillary into the rubber tube and out through the small capillary. A neater way of manipulating the rubber tube attachment would be to first put it on the pycnometer, fill the pycnometer with glycerine to overflowing and then draw the tube full of glycerine before inserting the thermometer. The pycnometer was then submerged to a depth of 4<sup>1</sup>/<sub>2</sub> inches in the water bath, which was kept first at 15.5°, then at 20°, 25° and 30° C., and allowed to remain for ten or fifteen minutes longer after its thermometer and the standard thermometer in the bath registered the desired temperature. The extra time allowed for the glycerine in the pycnometer to attain a uniform temperature throughout may not have been necessary but was allowed as a precaution.

Just before the pycnometer was ready to be taken from the bath, the water was drawn off until its capillary projected about one-half inch above the water. The rubber tube was then carefully removed, the excess glycerine wiped from the top of the capillary with a dry finger, the pycnometer quickly removed from the bath and the ground portion of its capillary wiped dry with a piece of filter paper and the cap placed on tight. The pycnometer was then wiped first with a wet and then with a dry cloth until dry and allowed to stand until it had attained the temperature of the room before weighing. Moisture collects very rapidly in damp weather upon the surface of the pycnometer when its temperature is below room temperature. There is no loss in weight upon standing when the pycnometer contains glycerine, as is the case when it contains water, for glycerine is more viscous and less volatile than water. The specific gravity was first determined at 15.5° C. and successively at 20°, 25°, and 30° C., using the same pycnometer containing the same glycerine for all four determinations, for as the temperature increased the glycerine expanded and was forced out into the rubber tube, which was filled with glycerine each time as described above.

The rubber tube attachment permits the glycerine to be transferred from the sample bottle at room temperature to the pycnometer without first changing its temperature if the specific gravity is to be determined in the water bath at any temperature between 15.5° and 30° C. A tube of the size mentioned above with a glass capillary

will hold enough glycerine to supply the deficiency caused by contraction due to a reduction in temperature of 20° C. with a liberal excess. It also allows the pycnometer to be submerged to a depth of four and one-half to five inches in the bath whereby all of its contents attain the same temperature throughout, while the capillary admits air and keeps out the water. Even though water were to get into the rubber tube, the viscosity of the glycerine would not allow it to get into the pycnometer. This was proven by three of the pycnometers being overturned in the water just after

C = Capacity of pycnometer in grams of water at 15.5°.

a = Thermal coefficient of expansion of glass = 0.000025 per 1° C.

B = Thermal coefficient of expansion of glycerine.

t = Observed temperature.

Explanation of Tables:

Table I shows the pycnometers used, their capacities and thermometer corrections.

Table II shows the glycerine used, the specific gravity determinations made at 15.5° C., with the

TABLE II.—SPECIFIC GRAVITY AT 15.5° AND AT 20°, 25° AND 30° C.  
CALCULATED FROM THE FORMULA  $\frac{W}{C} \frac{1}{1 + a(t - 15.5)}$  WITHOUT CORRECTION FOR EXPANSION OF GLYCERINE.

Glycerine. (1).	Sp. Gr. at 15.5° 15.5° (2).	Av. sp. gr. at 15.5°. (3).	Specific gravity at 20° without glycerin expansion correction.		Specific gravity at 25° without glycerin expansion correction.		Specific gravity at 30° without glycerin expansion correction.		Pyc- nometer used. (10).
			Results. (4).	Average. (5).	Results. (6).	Average. (7).	Results. (8).	Average. (9).	
A.....	1.25465	1.25465	1.25190	1.25190	1.24878	1.24878	1.24564	1.24564	A
	1.25465		1.25190		1.24877		1.24564		C
B.....	1.25736	1.25736	1.25461	1.25461	1.25149	1.25149	1.24836	1.24836	G
	1.25736		1.25461		1.25148		1.24833		H
C.....	1.26010	1.26012	1.25735	1.25736	1.25422	1.25423	1.25108	1.25108	I
	1.26013		1.25737		1.25423		1.25108		J
D.....	1.26023	1.26026	1.25746	1.25749	1.25435	1.25438	1.25123	1.25126	M
	1.26029		1.25751		1.25440		1.25129		N
E.....	1.26078	1.26077	1.25801	1.25800	1.25492	1.25491	1.25177	1.25176	A
	1.26075		1.25798		1.25489		1.25174		C
F.....	1.26135	1.26135	1.25858	1.25858	1.25548	1.25548	1.25233	1.25233	G
	1.26135		1.25858		1.25547		1.25232		H
G.....	1.26175	1.26177	1.25897	1.25899	1.25587	1.25589	1.25272	1.25273	I
	1.26178		1.25901		1.25591		1.25273		J
H.....	1.26239	1.26238		1.25965		1.25659		1.25348	G
	1.26240				1.25659		1.25348		H
	1.26232								I
	1.26238		1.25965		1.25659		1.25348		J
	1.26241		1.25965		1.25659		1.25348		K
	1.26238		1.25965		1.25659		1.25349		M
	1.26314		1.26037						H
I.....	1.26313	1.26312	1.26037	1.26037	1.25725	1.25728	1.25414	1.25416	I
	1.26309		1.26037		1.25730		1.25417		H
J.....	1.26339	1.26340	1.26064	1.26067		1.25758		1.25448	A
	1.26342		1.26066				1.25446		C
	1.26347		1.26072				1.25447		G
	1.26336		1.26065				1.25447		A
	1.26338		1.26066				1.25447		C
K.....	1.26341	1.26368	1.26072	1.26092	1.25761	1.25783	1.25451	1.25469	G
	1.26365		1.26087		1.25780		1.25466		M
L.....	1.26370	1.26418	1.26094	1.26138	1.25786	1.25829	1.25472	1.25515	N
	1.26417		1.26138				1.25513		J
	1.26414		1.26136				1.25513		M
	1.26420		1.26142				1.25518		N
	1.26419		1.26136		1.25827		1.25513		J
	1.26417		1.26135		1.25828		1.25513		M
1.26421	1.26139	1.25833	1.25518	N					

being put into the bath, the determinations being completed and afterward redetermined to make sure that they were correct.

Formula for Calculating the Specific Gravity of Glycerine at 15.5° C. when Determined at t Degrees Centigrade.—

Specific Gravity at

$$\frac{15.5^\circ}{15.5^\circ} = \frac{W}{C} \frac{1}{1 + a(t - 15.5)} + B(t - 15.5)$$

in which

W = Weight of glycerine at t°.

average, the specific gravity without the coefficient of expansion correction at 20°, 25° and 30° C., with the averages and the pycnometer used in each determination.

Table III:

Column 1.—Glycerine used.

Column 2.—Specific gravity at 15.5° C.; Table II, column 5.

Column 3.—Specific gravity at 20° C. without glycerine expansion correction; Table II, column 5.

Column 4.—Difference between specific gravity

at 15.5° and 20° C. without glycerine expansion correction.

Column 5.—Expansion correction per degree at 20° C. with an average correction at 20° at the end of the column.

Column 1.—Glycerine used.

Column 2.—Specific gravity at 15.5° C.; Table II, column 3.

Column 3.—Specific gravity at 20° C. without glycerine expansion correction; Table II, column 4.

TABLE III.—DETERMINATION OF THE VALUE OF "B" IN THE FORMULA  $\frac{W}{C} \frac{1}{1 + a(t - 15.5)} + B(t - 15.5)$ .

$$"B" = \frac{(\text{Specific gravity at 15.5}) - \left( \frac{W}{C} \frac{1}{1 + a(t - 15.5)} \right)}{(t - 15.5)}$$

Glycerine.	Specific gravity at 15.5° C.	Specific gravity at 20° without glycerine expansion correction taken from Table II, Column 5.	Column 2 minus Column 3.	Value of "B" at 20° C.	Specific gravity at 25° without glycerine expansion correction taken from Table II, Column 7.	Column 2 minus Column 6.	Value of "B" at 25° C.	Specific gravity at 30° without glycerine expansion correction taken from Table II, Column 9.	Column 2 minus Column 9.	Value of "B" at 30° C.
(1).	(2).	(3).	(4).	(5).	(6).	(7).	(8).	(9).	(10).	(11).
A.....	1.25465	1.25190	0.00275	0.000610	1.24878	0.00587	0.000618	1.24564	0.00901	0.000621
B.....	1.25736	1.25461	0.00275	0.000610	1.25149	0.00587	0.000618	1.24835	0.00901	0.000621
C.....	1.26012	1.25736	0.00276	0.000613	1.25423	0.00589	0.000620	1.25108	0.00904	0.000623
D.....	1.26026	1.25749	0.00277	0.000616	1.25438	0.00588	0.000619	1.25126	0.00900	0.000621
E.....	1.26077	1.25800	0.00277	0.000616	1.25491	0.00586	0.000617	1.25176	0.00901	0.000621
F.....	1.26135	1.25858	0.00277	0.000616	1.25548	0.00587	0.000618	1.25233	0.00902	0.000622
G.....	1.26177	1.25899	0.00278	0.000618	1.25589	0.00588	0.000619	1.25273	0.00904	0.000623
H.....	1.26238	1.25965	0.00273	0.000601	1.25659	0.00578	0.000610	1.25348	0.00890	0.000614
I.....	1.26312	1.26037	0.00275	0.000610	1.25728	0.00584	0.000615	1.25416	0.00896	0.000618
J.....	1.26340	1.26067	0.00273	0.000601	1.25759	0.00581	0.000612	1.25448	0.00892	0.000615
K.....	1.26368	1.26092	0.00276	0.000613	1.25783	0.00585	0.000616	1.25469	0.00899	0.000620
L.....	1.26418	1.26138	0.00280	0.000622	1.25829	0.00589	0.000620	1.25515	0.00903	0.000623
Average.....	.....	.....	.....	0.000612	.....	.....	0.000617	.....	.....	0.000622

Columns 6, 7 and 8.—Specific gravity at 25° without glycerine expansion correction and so on as described for columns 3, 4, and 5 with an average correction per degree at 25° C. at the end of column 8.

Column 4.—Coefficient of expansion of glycerine taken as 0.00061 at 20° C.

Column 5.—Specific gravity determined at 20° and calculated to specific gravity at 15.5°, using 0.00061 as the coefficient of expansion at

TABLE IV.—SPECIFIC GRAVITY OF GLYCERINE DETERMINED AT 20°, 25° AND 30° AND CALCULATED TO 15.5° ACCORDING TO THE FORMULA:

$$\text{Specific gravity at } 15.5^\circ = \frac{W}{C} \frac{1}{1 + a(t - 15.5)} + B(t - 15.5)$$

Glycerine.	Specific gravity at 20° without glycerine expansion correction taken from Table II, 15.5° C.	Average expansion specific correction at 20°. B = 0.00061	Specific gravity at 20° without glycerine expansion correction taken from Table III, 15.5° C.	Glycerine expansion determined at 20° and calculated to sp. gr. at 15.5°	Specific gravity at 25° without glycerine expansion correction taken from Table II, 15.5° C.	Average expansion specific correction at 25°. B = 0.000615	Specific gravity at 25° without glycerine expansion correction taken from Table III, 15.5° C.	Glycerine expansion determined at 25° and calculated to sp. gr. at 15.5°	Specific gravity at 30° without glycerine expansion correction taken from Table II, 15.5° C.	Average expansion specific correction at 30°. B = 0.00062	Specific gravity at 30° without glycerine expansion correction taken from Table III, 15.5° C.	Specific gravity determined at 30° and calculated to sp. gr. at 15.5°
(1).	(2).	(3).	(4).	(5).	(6).	(7).	(8).	(9).	(10).	(11).	(12).	
A.....	1.25465	1.25190	0.00061 × 4.5	1.25465	1.24878	0.000615 × 9.5	1.25462	1.24564	0.00062 × 14.5	1.25463		
B.....	1.25736	1.25461	0.00061 × 4.5	1.25736	1.25149	0.000615 × 9.5	1.25733	1.24835	0.00062 × 14.5	1.25734		
C.....	1.26012	1.25736	0.00061 × 4.5	1.26011	1.25423	0.000615 × 9.5	1.26007	1.25108	0.00062 × 14.5	1.26007		
D.....	1.26026	1.25749	0.00061 × 4.5	1.26024	1.25438	0.000615 × 9.5	1.26022	1.25126	0.00062 × 14.5	1.26025		
E.....	1.26077	1.25800	0.00061 × 4.5	1.26075	1.25491	0.000615 × 9.5	1.26075	1.25176	0.00062 × 14.5	1.26075		
F.....	1.26135	1.25858	0.00061 × 4.5	1.26133	1.25548	0.000615 × 9.5	1.26132	1.25233	0.00062 × 14.5	1.26132		
G.....	1.26177	1.25899	0.00061 × 4.5	1.26174	1.25589	0.000615 × 9.5	1.26173	1.25273	0.00062 × 14.5	1.26172		
H.....	1.26238	1.25965	0.00061 × 4.5	1.26240	1.25659	0.000615 × 9.5	1.26243	1.25348	0.00062 × 14.5	1.26247		
I.....	1.26312	1.26037	0.00061 × 4.5	1.26313	1.25728	0.000615 × 9.5	1.26312	1.25416	0.00062 × 14.5	1.26315		
J.....	1.26340	1.26067	0.00061 × 4.5	1.26342	1.25759	0.000615 × 9.5	1.26343	1.25448	0.00062 × 14.5	1.26347		
K.....	1.26368	1.26092	0.00061 × 4.5	1.26367	1.25783	0.000615 × 9.5	1.26367	1.25469	0.00062 × 14.5	1.26368		
L.....	1.26418	1.26138	0.00061 × 4.5	1.26415	1.25829	0.000615 × 9.5	1.26413	1.25515	0.00062 × 14.5	1.26414		

Columns 9, 10 and 11.—Specific gravity at 30° without glycerine expansion correction and so on as described for columns 3, 4 and 5, with an average correction per degree at 30° C. at the end of column 11.

that temperature for comparison with the specific gravity when determined at 15.5° C.

Columns 6, 7 and 8.—Specific gravity at 25° without glycerine expansion correction and so on as described for columns 3, 4 and 5.

Table IV:

Columns 9, 10 and 11.—Specific gravity at 30° C. without glycerine expansion correction and so on as described for columns 3, 4 and 5.

#### SUMMARY.

In consideration of the importance connected with obtaining a true value for the thermal coefficient of expansion of glycerine, the greatest care was observed in every detail of the foregoing work. It may appear superfluous to report the determinations of the specific gravity to the fifth decimal place, but as they agreed so closely at that point when different pycnometers were used in doing duplicate determinations, it seemed better to show the actual results obtained, which also shows the degree of accuracy that entered into the work.

From the foregoing results, it is apparent that the published values for the thermal coefficient of expansion of dynamite glycerine are not true between 15.5° and 30° C.

Taking the average coefficients of expansion from Table II as 0.000612 at 20° C., 0.000617 at 25° C. and 0.000622 at 30° C., which are in round numbers 0.00061 at 20°, 0.000615 at 25° and 0.00062 at 30°, and applying them as shown in Table IV, the determinations show very close agreement with the determinations made at 15.5° C.

The idea in using the round numbers 0.00061, 0.000615 and 0.00062 as the coefficients of expansion for glycerine is that they are easily remembered and are accurate enough for all practical purposes.

EASTERN LABORATORY,

E. I. DUPONT DE NEMOURS POWDER CO.,  
September 1, 1909.

### FLASH, FIRE AND EXPLOSION TESTS ON MIXTURES OF CARBON TETRACHLORIDE AND NAPHTHA.

By EDW. A. BARRIER.

Received September 2, 1909.

The frequent occurrence of fires, explosions, or both, resulting from the wide-spread use of naphtha in removing oil from textile materials or metal goods in factories, makes it a matter of serious concern that no suitable, non-inflammable substitute has as yet been found. Of late years carbon tetrachloride has received some attention in this connection, but its comparatively high cost has barred its use except in a very limited way. Recent reductions in the price of this material, however, and the fact that a certain percentage of naphtha can be added without rendering the mixture inflammable have brought the cost of such a mixed solvent down to a point where it can be used in many cases, especially, if the spent solvent is reclaimed by distillation.

The importance of this matter led to the follow-

ing series of experiments which were undertaken with the object of determining how great a percentage of naphtha, of various gravities, could be added to carbon tetrachloride, and still leave the mixture free from fire and explosion hazard.

Four samples of commercial naphtha, labeled as below, were obtained from a petroleum refiner for the tests. The specific gravities of these samples were found to be as follows:

	Labeled.	Sp. gr. found.
I.....	54° Bé.	0.7556-55½° Bé.
II.....	62° Bé.	0.7251-63° Bé.
III.....	Motor gasoline, 68-72° Bé.	0.6982-70½° Bé.
IV.....	73-76° Bé.	0.6811-75½° Bé.

Distillation tests of these naphthas were made with the following results:

Gravity.	80-100°.	100-110°.	110-120°.	120-130°.	130-140°.
55¼°	.....	.....	.....	17.0 cc. <sup>1</sup>	20.5 cc.
63°	12.5 cc. <sup>2</sup>	23.5 cc.	22.0 cc.	18.0 cc.	8.0 cc.
Gravity.	140-150°.	150-160°.	160-170°.	170-180°.	Above 180°.
55¼°	17.0 cc.	11.0 cc.	9.5 cc.	7.0 cc.	17.0 cc.
63°	16.0 cc. above 140°				
Gravity.	50-60°.	60-70°.	70-80°.	80-90°.	
70½°	2.0 cc. <sup>3</sup>	5.0 cc.	19.0 cc.	24.5 cc.	
75½°	3.5 cc.	23.0 cc.	24.0 cc.	18.0 cc.	
Gravity.	90-100°.	100-110°.	110-115°.	Above 115°.	
70½°	21.0 cc.	11.0 cc.	4.0 cc.	14.0 cc.	
75½°	13.0 cc.	5.5 cc.	12.0 cc. above 110°		

#### FLASH AND FIRE POINT TESTS.

The flash and fire point tests were made in the open cup of the New York State Board of Health tester, and also in some cases, in a six-inch evaporating dish. No difficulty was experienced in determining the flash points of the various mixtures, but in no case could a definite fire point be found. On raising the temperature from the flash point, the liquid would usually flash and burn for one or two seconds, and then go out of itself. A further raising of the temperature resulted in some cases in lengthening the time of burning, and, in other cases, in decreasing the time of burning. Repeated tests of the same mixture, however, gave reasonably concordant results.

The mixtures containing the limiting amounts of naphtha, which are later stated to be reasonably safe, were tested in an evaporating dish, in addition to the open cup, as it was thought that this form of container with a large evaporating area was more comparable to the ordinary open bath frequently found in works.

On account of the absence of a true fire point the temperature was gradually raised from the flash point to the boiling point, and the observations recorded below were taken.

The results are given in the following table:

<sup>1</sup> Distillation began at 123°.

<sup>2</sup> Distillation began at 85°.

<sup>3</sup> Distillation began at 55°.



Kind of naphtha and flash-point.	Mixture tested.	Flash-point of mixture.	Observations.		
			Temp.	Time of burning, sec.	Remarks.
55½° Bé. 8° C.	50 per cent. naphtha and 50 per cent. tetrachloride.....	.....	.....	..	Did not flash at any temperature.
	60 per cent. naphtha and 40 per cent. tetrachloride.....	35° C.	.....	..	Did not flash at temperatures above the flash point.
	70 per cent. naphtha and 30 per cent. tetrachloride.....	28° C.	45° C. 55° C.	4 6	Above 55° C. the mixture would not burn.
	80 per cent. naphtha and 20 per cent. tetrachloride.....	24° C.	35° C. 45° C.	5 8	Increasing the temperature appeared to have no appreciable effect.
63° Bé. -12° C.	40 per cent. naphtha and 60 per cent. tetrachloride.....	38° C.	.....	..	On raising temperature mixture would not flash.
	50 per cent. naphtha and 50 per cent. tetrachloride.....	18° C.	35° C. 45° C.	3 5	Further increase of temperature decreased the time of burning.
	55 per cent. naphtha and 45 per cent. tetrachloride.....	10° C.	25° C. 35° C.	1 5	Further increase of temperature appeared to have no appreciable effect.
	60 per cent. naphtha and 40 per cent. tetrachloride.....	0° C.	25° C. 35° C. 45° C. 70° C.	3 10 20 ..	Burned continuously until extinguished.
70½° Bé. Below -18° C.	30 per cent. naphtha and 70 per cent. tetrachloride.....	-5° C.	25° C. 35° C.	1 2	At higher temperature would not burn.
	40 per cent. naphtha and 60 per cent. tetrachloride.....	-11° C.	25° C. 35° C.	10 15	Further increase of temperature appeared to decrease time of burning.
	50 per cent. naphtha and 50 per cent. tetrachloride.....	-18° C.	0° C. 25° C. 35° C.	2 10 ..	Burned continuously until extinguished.
75½° Bé. Below -18° C.	30 per cent. naphtha and 70 per cent. tetrachloride.....	-11° C.	10° C. 25° C. 35° C. 45° C.	1 2 5 8	Further increase of temperature appeared to decrease time of burning.
	40 per cent. naphtha and 60 per cent. tetrachloride.....	-18° C.	0° C. 25° C. 35° C. 45° C.	3 12 15 ..	Burned continuously until extinguished.

## EXPLOSION TESTS.

The explosion tests were carried out in a specially constructed box, 10" X 11" X 15" (see Fig. 1), heavily built, and made air-tight. The cover was provided with a tongue on the under side, which fitted into a channel placed on the upper, inside edges of the box. The channel was partly filled with water, thus making an air-tight seal. The box was also provided with an inlet pipe at the top of one end, and an outlet pipe at the bottom of the other end, both of 1/4" bore. Two copper bars passing through

the outlet end of the box served as terminals for making connections with an electric circuit.

The vapor was introduced into the box by connecting the outlet pipe with a suction pump, and the inlet pipe with an Erlenmeyer flask, fitted with a rubber stopper and two glass tubes, one of which dipped into the mixture to be tested. The amount of vapor sucked into the box was regulated by using a constant flow of water through the suction pump, and varying the time suction was applied. When the desired amount of vapor



had been drawn into the box, the suction pump and flask were disconnected, and the terminals were wired to an electric circuit. The charge was then fired by "short-circuiting" a one ampere fuse placed on the inside of the box.

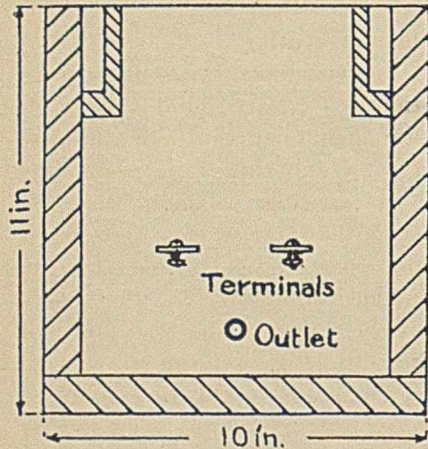


FIG. 1.

M.M.W. Jr.

mine within what range the maximum explosive

Before the final series of tests were run a number of preliminary experiments were made with pure naphtha, in which the time of application of the suction was varied, in

order to determine

effect could be obtained. This was found to be from 30 seconds to four minutes, and this range, except in certain cases where no explosion could be obtained, was accordingly used in the series of tests which appear in the following tables:

TABLE I. 55 $\frac{1}{4}$ ° BÉ. NAPHTHA. TEMPERATURE 20°.

Mixture.	Time suction was applied.	Height cover was lifted.	Remarks.
Pure naphtha.....	1 min.	Did not move.	No explosion.
" .....	2 min.	"	"
" .....	4 min.	"	"
" .....	8 min.	"	"

TABLE II. 63° BÉ. NAPHTHA. TEMPERATURE 20°.

Mixture.	Time suction was applied.	Height cover was lifted.	Remarks.
Pure naphtha.....	1 min.	Did not move.	No explosion.
" .....	2 min.	"	"
" .....	4 min.	"	"
" .....	8 min.	"	"

All of the above tests with the 70 $\frac{1}{2}$ ° naphtha were repeated using a temperature of 38°, with the idea of simulating conditions which occur in summer. Practically the same results were obtained as with the lower temperature, with the exception that the same explosive effect was obtained in a shorter time.

From an examination of the tables, it will be

III. 70 $\frac{1}{2}$ ° BÉ. NAPHTHA TEMPERATURE 21°.

Mixture.	Time suction was applied.	Height cover was lifted.	Remarks.
Pure naphtha.....	1 min.	7 ft.	No flame visible.
" .....	2 min.	2 $\frac{1}{2}$ ft.	Considerable flame, evident excess of vapor.
50 per cent. CCl <sub>4</sub> and 50 per cent. naphtha.....	1 min.	Did not move.	No explosion.
" .....	2 min.	"	"
" .....	4 min.	"	"
40 per cent. CCl <sub>4</sub> and 60 per cent. naphtha.....	1 min.	"	"
" .....	2 min.	"	"
" .....	4 min.	Very slight movement.	"
30 per cent. CCl <sub>4</sub> and 70 per cent. naphtha.....	1 min.	"	"
" .....	2 min.	$\frac{1}{2}$ ft.	"
" .....	4 min.	$\frac{1}{4}$ ft.	Green flame, evident excess of vapor.

IV. 75 $\frac{1}{2}$ ° BÉ. NAPHTHA TEMPERATURE 21°.

Mixture.	Time suction was applied;	Height cover was lifted.	Remarks.
Pure naphtha .....	30 sec.	Did not move.	
" .....	1 min.	6 ft.	No flame visible.
" .....	2 min.	2 ft.	Some flame visible
" .....	4 min.	1 ft.	Considerable flame, evident excess of vapor.
60 per cent. CCl <sub>4</sub> and 40 per cent. naphtha.....	1 min.	"	"
" .....	2 min.	Did not move.	"
" .....	4 min.	"	"
50 per cent. CCl <sub>4</sub> and 50 per cent. naphtha...	1 min.	"	"
" .....	2 min.	Moved slightly.	"
" .....	4 min.	2 inches.	"
40 per cent. CCl <sub>4</sub> and 60 per cent. naphtha.....	1 min.	3 inches.	"
" .....	2 min.	9 inches	"
" .....	4 min.	1 $\frac{1}{2}$ ft.	"
30 per cent. CCl <sub>4</sub> and 70 per cent. naphtha.....	1 min.	4 inches	"
" .....	2 min.	2 $\frac{1}{2}$ ft	"
" .....	4 min.	2 ft.	Flame visible, evident excess of vapor.

seen that no explosion could be obtained with the  $55\frac{1}{4}^{\circ}$  and the  $63^{\circ}$  naphthas. This is in line with what experience has generally shown, namely, that there is but slight explosion hazard connected with the ordinary use, at the usual room temperatures, of naphthas having gravities around  $60^{\circ}$  or below.

#### CONCLUSIONS.

As a result of these tests the following conclusions seem warranted:

(1) That a certain percentage of naphtha can be added to carbon tetrachloride and still leave the mixture free from fire and explosion hazard.

(2) That the percentage which can be safely added varies with the gravity of the naphtha.

(3) That  $55^{\circ}$  naphtha at ordinary room temperatures is practically free from explosion hazard, but in order to be reasonably safe from fire hazard, it should contain at least 30 per cent. of carbon tetrachloride.

(4) That  $63^{\circ}$  naphtha at ordinary room temperatures presents but slight explosion hazard, but in order to be reasonably safe from fire hazard, it should contain at least 45 per cent. of carbon tetrachloride.

(5) That  $70^{\circ}$  naphtha in order to be safe from explosion hazard should contain at least 50 per cent. of carbon tetrachloride, and to be reasonably safe from fire hazard should contain 60 per cent.

(6) That  $76^{\circ}$  naphtha to be safe from explosion hazard should contain at least 60 per cent. of carbon tetrachloride, and to be reasonably safe from fire hazard should contain 70%.

The above statements apply, naturally, only to naphthas which show approximately the same results on distillation, especially with respect to the lower boiling fractions, as those tested. The expression "reasonably safe from fire hazard" is used advisedly, since the above mixtures, while possessing but little fire hazard in open containers, will burn if spread out over a considerable area, on any other readily combustible material such as cotton waste. The percentages of naphtha, stated in each case, are limiting ones, and good practice would call for an additional 5 per cent. of carbon tetrachloride in order to allow some factor of safety.

### THE MOISTURE DETERMINATION AS APPLIED TO CEREALS.

By SHERMAN LEAVITT.

Received October 5, 1909.

The object of this investigation was to find out if a variation of one or two degrees from the standard temperature of one hundred degrees centigrade in

the determination of moisture content of cereals would have an appreciable effect on the percentage of moisture obtained under otherwise similar conditions. These conditions were that all the determinations be made in a partial vacuum with a gentle current of air passing through and that the drying be continued until the sample ceased to lose and commenced to increase in weight, it being generally assumed that cereals are dry when they begin to increase in weight on further heating.

In the fall of 1907, the writer had occasion to make moisture determinations on a large number of samples of wheat. Quite a number of these samples, unknown to the writer at the time, had been accidentally duplicated by another chemist. The results obtained by him on the same wheats were found to be in general about two per cent. higher on the basis of the fresh substance than ours. As this difference in results was fairly constant and was material in amount, the present investigation was started to determine, if possible, the cause for this divergence.

A cylindrical, heavy, brass, vacuum oven which was constructed with a water jacket was used. The oven was provided with a vacuum gauge and a thermometer, standardized by the Bureau of Standards, for which the correction was known. During the drying process, a slow current of air, dried by passing through strong sulphuric acid, was drawn over the samples, maintaining meanwhile a vacuum of about twenty-four inches of mercury. The samples were different varieties of wheat, ground to pass a one millimeter sieve, being well mixed and kept in tightly stoppered bottles. The experiment was devised and carried out so as to have as nearly as possible only one variable, *i. e.*, the temperature of the interior of the oven. As stated before, the end point of the drying operation was when the samples ceased to lose weight and showed a tendency to increase in weight. This was generally accomplished in five hours, the time being taken as soon as the oven had reached the desired temperature in the partial vacuum and current of air. The temperature was changed by the following simple conditions: When the oven was unprotected by asbestos covering and heated by an ordinary bunsen burner, with the oven jacket partially filled with tap water, a very even temperature of  $97^{\circ}$  Centigrade was maintained. By using a larger gas burner of the gridiron type and asbestos covering for the oven and flame, temperatures of  $98^{\circ}$  and  $99^{\circ}$  could be obtained under the standard vacuum conditions of twenty-four inches of mercury and two or three bubbles of air to the second passing through the oven.

It is interesting to note at this point that the temperature of the interior of the oven, as indicated by the standard thermometer, never reached  $100^{\circ}$  by

a whole degree.<sup>1</sup> The thermometer was always kept at the same depth of immersion in the bath, which was about at the point on the scale corresponding to 97°, so that practically the whole mercury column was immersed. Any correction on the thermometer was made according to the printed rules of the Bureau of Standards certificate accompanying the thermometer.

By using a mixture containing 20 per cent. of glycerine by volume (the percentage being determined experimentally) in the water jacket, an even temperature of 100° C. was obtained. Temperatures of 101° and 102° C. were obtained in a similar oven which was without the water jacket compartment and was heated directly by the gas flame, the temperature being regulated by a thermostat. This type of oven had been used in obtaining the high results referred to in the first part of this paper.

Circular, flat, aluminum dishes, with tightly fitting covers, were used to hold the samples (2 gram samples) so that the dry and very hygroscopic grain could not absorb an appreciable amount of moisture while on the pan of the balance during weighing. In Table I the results have been arranged so as to show the divergence between duplicates and the corresponding results for each sample at different temperatures:

TABLE I.

No. of sample.	(1) 97°.		(2) 98°.		(3) 99°.		(4) 100°.		(5) 101°.	
	Per cent. H <sub>2</sub> O.	Aver- age.	Per cent. H <sub>2</sub> O.	Aver- age.	Per cent. H <sub>2</sub> O.	Aver- age.	Per cent. H <sub>2</sub> O.	Aver- age.	Per cent. H <sub>2</sub> O.	Aver- age.
1993	{ 8.15 8.19	8.17	{ 8.11 8.32	8.22	{ 8.69 8.82	8.75	{ 9.37 9.43	9.40	{ 9.72 9.78	9.75
1994	{ 8.07 8.05	8.06	{ 8.77 8.73	8.75	{ 9.04 8.98	9.01	{ 9.61 9.62	9.62	{ 9.78 9.84	9.81
1995	{ 9.12 9.00	9.06	{ 8.85 9.08	8.97	{ 9.44 9.40	9.42	{ 9.83 9.82	9.83	{ 10.45 10.54	10.50
2151	{ 7.09 8.69	*	{ 8.23 8.41	8.32	{ 8.37 8.53	8.45	{ 9.02 9.12	9.07	{ 9.34 9.41	9.37
2152	{ 8.29 8.28	8.29	{ 7.90 7.62	7.76	{ 8.66 8.58	8.62	{ 9.31 9.41	9.36	{ 9.47 9.36	9.41
2154	{ 8.29 8.42	8.35	{ 8.09 8.12	8.11	{ 8.59 8.75	8.67	{ 9.23 9.33	9.28	{ 9.59 9.59	9.59
2155	{ 8.36 8.44	8.40	{ 8.50 8.61	8.55	{ 8.77 8.73	8.75	{ 9.46 9.28	9.36	{ 9.65 9.61	9.63
2156	{ 8.38 7.07	*	{ 8.09 8.25	8.17	{ 8.40 8.56	8.48	{ 9.04 9.14	9.09	{ 9.60 9.59	9.60
2176	{ 8.18 7.85	*	{ 8.22 8.38	8.30	{ 8.51 8.40	8.45	{ 9.16 9.16	9.16	{ 9.51 9.53	9.52

\* Duplicates not sufficiently close to average.

## EXPLANATION OF TABLE I.

All results were taken at point where samples commenced to increase in weight.

Columns (1), (2) and (3), corresponding to temperatures 97°, 98° and 99° C., were obtained in the oven with water in the jacket.

Column (4), corresponding to 100° C., was obtained

<sup>1</sup> It has been suggested that the expansion of the bulb of the thermometer in the vacuum was responsible for this one degree low reading of the thermometer. The writer, however, is of the opinion that the loss of heat, due to radiation because of an air leak in the oven, which was afterwards discovered, was responsible for it, because the oven failed to reach the boiling point of water when there was very little vacuum on.

in the same oven with a 20 per cent. by volume solution of glycerine and tap water.

Column (5), corresponding to 101° C., was obtained in the hot air vacuum oven regulated by a thermostat.

Column (6), not given because exact figures were mislaid or lost, corresponding to 102° C. Gave results all above 11 per cent. and approximating the high results previously referred to.

Table II has been arranged so as to show the effect of heating at 100° C. for different lengths of time:

TABLE II.

All temperatures 100° C.

No. of sam- ple.	(a) 3 1/2 hours.		(b) 4 1/2 hours.		(c) 5 hours.		(d) Over 5 hours.
	Per cent. H <sub>2</sub> O.	Aver- age.	Per cent. H <sub>2</sub> O.	Aver- age.	Per cent. H <sub>2</sub> O.	Aver- age.	
1993	{ 9.24 9.32	9.28	{ 9.37 9.43	9.40	{ 9.37 9.43	9.40	Sample increased in weight.
1994	{ 9.42 9.45	9.44	{ 9.61 9.62	9.62	{ 9.61 9.62	9.62	Sample increased in weight.
1995	{ 9.09 9.10	9.09	{ 9.83 9.82	9.83	{ 9.96 9.91	9.94	Sample increased in weight.
2151	{ 9.00 8.87	8.93	{ 9.02 9.12	9.07	{ 9.30 9.17	9.24	Sample increased in weight.
2152	{ 9.19 9.24	9.21	{ 9.31 9.41	9.36	{ 9.31 9.41	9.36	Sample increased in weight.
2154	{ 9.16 9.08	9.12	{ 9.23 9.33	9.28	{ 9.23 9.33	9.28	Sample increased in weight.
2155	{ 9.05 8.93	9.00	{ 9.46 9.28	9.36	{ 9.50 9.37	9.42	Sample increased in weight.
2156	{ 8.72 8.71	8.72	{ 9.04 9.14	9.09	{ 9.16 9.13	9.15	Sample increased in weight.
2176	{ 8.77 8.88	8.82	{ 9.16 9.16	9.16	{ 9.29 9.32	9.31	Sample increased in weight.

## EXPLANATION OF TABLE II.

Temperature constant at 100° C., weighings taken at different periods of time, giving some comparisons of length of time of heating on fresh samples in oven jacketed with 20 per cent. by volume solution of glycerine and tap water. We notice that the results in column (c), Table II, agree very well with the results in column (4), Table I, showing that practically all of the moisture had been driven off in from four and one-half to five hours' heating at 100° C.

From these results we conclude that a variation of one degree either below or above 100° C. will make a marked and appreciable difference in the moisture results on wheat, and probably would with other cereals as well. It is usually customary to calculate all analytical results on cereals to the *water-free basis* for comparison; hence, it is evident that the moisture determination is of the highest importance, as an error in this determination will change all of the other analytical results.

No attempt is made in this article to explain why it is that a long slow drying, at a lower temperature than 100° C., did not give the same results as at 100° C. Theoretically, we should expect the same results, but in the hands of the writer they barely gave within two per cent. of the same results and further heating showed quite a rapid increase in weight of

all samples examined, especially at the lower temperatures.

This work was carried out in the fall and winter of 1907, in the laboratory of Vegetable Physiological Chemistry of the Bureau of Chemistry of the U. S. Department of Agriculture, at Washington, D. C. The writer wishes to make special acknowledgment to Dr. J. Arthur LeClerc, chief of the above laboratory, for his many kindnesses and courtesies and for many valuable suggestions in connection with this study. The writer is also indebted to Professor Charles A. Mooers, of this station, for reading and criticizing this paper.

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[CONTRIBUTION FROM THE DEPARTMENT OF AGRICULTURAL CHEMISTRY OF  
THE UNIVERSITY OF MISSOURI.]

## THE GLYCOGEN CONTENT OF BEEF FLESH.

(FIRST PAPER.)

By P. F. TROWBRIDGE AND C. K. FRANCIS.

Received July 6, 1909.

*Consideration of the Problem.*—In connection with the "Use of Food" experiment with beef animals now in progress at the Agricultural Experiment Station of Missouri, one of the points to be studied is the variation in the composition of the flesh as affected by feed, age or breed of the animal. It was thought quite probable that variation in quantity of feed would particularly influence the glycogen content. With this in view, an experiment was outlined to study the glycogen content of certain parts of the steers to be slaughtered. Since the animals were to be slaughtered under similar conditions as to length of time off feed, it was possible to secure the samples under quite uniform conditions. The liver was selected as an organ especially to be examined, and for a sample of the flesh it was decided to take a sample from the lean clod muscle of the shoulder, as this could be obtained without seriously mutilating the carcass.

*Method of Securing and Preparing the Samples.*—The following method was used in securing and preparing the samples for analysis: The animals were given their regular feed at 6 A.M. and were knocked in the head at about 8.30 A.M. A chain was wrapped around the hind legs, the carcass hoisted from the floor, and the animal bled thoroughly. As soon as the bleeding was complete, and before the animal was lowered to the floor, a piece of the hide was stripped from over the shoulder of the left side and about one kilo of lean clod muscle removed and taken at once to the laboratory. There, as rapidly as possible, it was put through an Enterprise meat grinder three times with thorough mixing between each grinding. A sample was taken immediately from this

ground meat for determination of all the analytical data desired, including the glycogen determination.

At the slaughter-house the work of dressing the carcass was carried on as rapidly as was possible, securing and weighing the necessary samples, taking notes, etc. As soon as the contents of the abdominal cavity were removed, the liver from which the gall bladder had been separated, was weighed and sent to the laboratory, where it was immediately ground and mixed in the same manner as the sample of flesh. The samples were weighed out for the analyses as rapidly as possible. This work has been carried on with fifteen animals slaughtered during the past two years, under conditions as nearly uniform as it has been possible to make them. At first several hours elapsed from the time the sample was removed from the carcass until the portions were weighed out and treated with alkali. Later it was found possible to reduce this time very materially. This will be noted in the tables and in the discussion of the data.

*Method of Analysis as Adopted.*—With the first animal different methods<sup>1</sup> were studied and compared with the adoption of the following method as the most satisfactory procedure.<sup>2</sup>

*Solution of the Sample.*—Weigh out by difference in triplicate, about 25 grams of the finely ground and thoroughly mixed sample. Place the sample in 400 cc. beakers, mixed with 50 cc. of strong potassium hydroxide<sup>3</sup> solution. Cover the beaker with a watch-glass and digest on the water bath for two hours, with occasional stirring. At the end of the two hours, dilute to approximately 200 cc. with cold water.

*Separation of the Glycogen.*—Add to the solution an equal volume of 95 per cent. alcohol, cover with the watch-glass and set aside for 10 or 12 hours, in order that precipitation of the glycogen may be complete. Decant the supernatant liquid through a folded filter (S. & S. No. 588, 18<sup>1</sup>/<sub>2</sub> cm.), allowing the glycogen to remain in the beaker and wash by decantation with 66 per cent. alcohol (2 vol. of 95 per cent. alcohol with 1 vol. of water) until the glycogen is white, or nearly so. Usually about four washings are required. Transfer the washed precipitate from the beaker to the filter and wash two or three times with 66 per cent. alcohol. The solution filters slowly and the funnel should be covered with a watch-glass to prevent excessive evaporation. The albuminous substance present retards the filtration if permitted to dry on the paper. If the washing by decantation

<sup>1</sup> Pflüger, *Arch. f. d. Gesamte Physiologie*, 95 and 96 (1903); Allen, "Organic Analysis," 1, 284; 2, 275 (1898).

<sup>2</sup> Mr. L. F. Shackell, now assistant in the physiological laboratory of St. Louis University School of Medicine, was placed in charge of the laboratory work during the first part of this work and credit is hereby given him for his assistance in securing the data here presented.

<sup>3</sup> 150 grams of pure KOH free from carbonate to every 100 cc. of water.

is not made nearly complete, it will be difficult to obtain the glycogen free from the coloring matter.

*Purification of the Glycogen.*—After the washing is completed, close the bottom of the funnel by a piece of rubber tubing and a pinchcock. Fill the funnel with warm water, cover with the watch-glass and let stand two or three hours, or overnight. Remove the pinchcock and allow all of the solution to pass through the filter into a beaker. Close the funnel with the pinchcock and fill with warm water as before. Allow this water to remain in the funnel for one hour and then filter as before. At first the glycogen solution appears quite turbid. This washing with warm water should be continued until the filtrate becomes perfectly clear. To the solution of glycogen in water, add a double volume of 95 per cent. alcohol and let stand overnight to complete the re-precipitation of the glycogen. Filter and wash as before with 66 per cent. alcohol.

*Quantitative Estimation of the Glycogen.*—If desired, the last filtration can be made through tared Gooch crucibles and the weight of glycogen determined after drying to constant weight. This gives results that are approximately correct. A more satisfactory method, however, is to hydrolyze the glycogen with hydrochloric acid and determine the amount of the resultant glucose by means of Fehling's solution. This method proceeds as follows: The well washed precipitate of glycogen is dissolved on the filter as described above, collecting the filtrate in a 250 cc. measuring flask. The volume of the solution should be kept within 225 cc. Add 12.5 cc. of hydrochloric acid, specific gravity 1.19, and place the flask in a boiling water bath for three hours. When the contents of the flask are cooled make up to the mark with water and mix thoroughly.

By means of preliminary tests determine the amount of the glucose solution that can be used with 60 cc. of the mixed Fehling's solution. With a burette or pipette measure out in triplicate the amount of the glucose solution which the preliminary test has shown can be used with the 60 cc. of Fehling's solution. Neutralize the solution with potassium hydroxide and if the volume be less than 75 cc. add water to make about this amount. To this solution add 60 cc. of the previously mixed Fehling's solution. Heat rapidly to the boiling point over the naked flame and boil for exactly two minutes. Remove and add at once about 100 cc. of cold water. Allow the precipitate to settle completely, decant through a quantitative filter paper, transfer the precipitate to the filter paper and wash with hot water until the filter is free from alkali. Ignite the precipitate to cupric oxide, weigh and calculate the results to weight of glucose by means of Allihn's<sup>1</sup> tables. Calculate the

weight of glucose in the aliquot to the total sample, determining the amount of glycogen from the glucose by multiplying by 0.9<sup>1</sup> and calculate this result to per cent. in the fresh sample.

That the triplicate determinations agree reasonably well and also that the gravimetric determination gives approximately the same results as the method by hydrolysis is shown by the following results with steer No. 505, a fat yearling grade Hereford. Each result is from a separately weighed sample:

Lab. No.	Sample.	Method of hydrolysis, glycogen per cent.	Glycogen by weight, per cent.
822x	Liver.....	0.908	0.889
822y	Liver.....	0.951	0.889
822z	Liver.....	0.923	0.879
823x	Lean muscle.....	0.387	0.439
823y	Lean muscle.....	0.411	0.401
823z	Lean muscle.....	0.328	0.388

During the progress of the work it was found to be practicable to slaughter more than one animal at a time, therefore some of the animals were killed after a longer time off feed than were others. In the table below we tabulate the results, taking the average of the triplicate determinations arranged according to time off feed and in order of the amounts of glycogen in the liver:

Slaughtered within two or three hours after regular morning feed:

Lab. No.	Glycogen in liver, lean muscle,		Description of animal.
	per cent.	per cent.	
91102-3	3.803	0.663	No. 48, very fat short-horn, 4½ years old.
92102-3	2.337	0.697	No. 63, lean Jersey cow, 6 years old.
71251-2	1.210	0.243	No. 121, fairly fat Shorthorn, 3½ years old.
822-3	0.927	0.375	No. 505, fat yearling Hereford.
8122-3	0.820	0.309	No. 504, fat Hereford, 20 months old.

Slaughtered five or six hours after regular morning feed:

9252-3	0.965	0.569	No. 595, thin Hereford, 2 years old.
9152-3	0.913	0.416	No. 592, very thin Hereford, 23 months old.
8255-6	0.625	0.158	No. 503, thin Hereford, 11 months old.

Slaughtered about nine hours after regular morning feed:

92151	1.160	not det.	Jerry, very fat prize Hereford, 3 years old.
89102-3	0.284	0.140	No. 593, fairly fat Hereford, 18 months old.

The data are insufficient to draw any conclusions but it is possible to point out the probable factors, that may influence the glycogen content.

(1) It is quite possible the older the animal the greater the tendency to store the glycogen. All

<sup>1</sup> Methods of Analysis A. O. A. C., published as U. S. Dept. of Agriculture, Bureau of Chem., Bull. 107 (revised), p. 50.

<sup>1</sup> C<sub>6</sub>H<sub>10</sub>O<sub>5</sub> + H<sub>2</sub>O = C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>.  
162 + 18 = 180.

of the animals 3 years old or over show more than one per cent. of glycogen in the liver.

(2) The condition (fatness) of the animal may favor the storing of glycogen. Our results are a little contradictory. No. 504 was very fat, No. 63 very thin, No. 503 not fat but in good thrifty condition, while No. 592 was extremely thin, approaching starvation. On the other hand, No. 48 was much fatter than No. 121, both being mature animals and being comparable in every respect except degree of fatness.

(3) The length of time that elapses after feeding before the animal is slaughtered seems to be a very important factor in determining the amount of glycogen that remains stored in the organs and muscles of the animal. If fatness is a favorable factor, Jerry should have shown fully as much glycogen as No. 48, but he does show less than one-third as much, having been slaughtered in the afternoon, eight hours off feed, while No. 48 was slaughtered in the morning, two hours off feed. On the other hand, Nos. 595 and 592 compare favorably with No. 505 and No. 504 as to glycogen content and age, but were much thinner and were slaughtered 3 or 4 hours later after feeding.

While we have just considered some of the factors that may influence the amount of glycogen that the animal stores up, it must not be overlooked that the time that elapses from the killing of the animal until the sample is digesting in the alkali may be the chief factor that influences the amount of glycogen found.

With our first animals slaughtered no exact record was taken of the time. The sample was always sent to the laboratory as soon as obtained and the chemist immediately went to work to prepare the sample for the analysis, thinking in this way to have all results comparable.

The failure of the results to establish any definite law and knowing how rapidly the acidity of flesh increases under similar conditions, led us to consider more carefully the element of time as measuring perhaps the amount of enzymatic reaction. With animals No. 63, No. 595 and Jerry, slaughtered February 22, 1909, the exact time was carefully noted.

No. 63, a thin Jersey cow six years old (not in milk), was killed at 8.55 A.M. The sample of lean muscle was received at the laboratory at 9.20. All weighings were completed and the alkali was added to the last sample as 10.10. The alkali was added to each sample as soon as it was weighed out, so that sample *x* had slightly less time for enzymatic hydrolysis than did *y*, and *y* had a corresponding less time than did *z*; sample *x* gave on analysis 0.714 per cent. glycogen and *y* gave 0.680 per cent. The beaker containing sample *z* was broken and at 3.26 P.M. the alkali was added to another sample *z*. In the meantime the ground meat had stood in a closed jar in the

laboratory; result of the analysis, 0.320 per cent. of glycogen, or less than one-half as much.

The liver from this same animal was received at 10.18 A.M. and the alkali was added to the last sample at 11.00 A.M. The glycogen found was *x*, 2.24 per cent.; *y*, 2.48 per cent.; and *z*, 2.29 per cent.

Steer No. 595 was killed at 11.15 A.M. The sample of lean muscle was received at the laboratory at 11.37 A.M. and the alkali was added to the last sample at 12.06 P.M. *x* gave 0.685 per cent. glycogen, *y* 0.526 per cent., and *z* 0.495 per cent. The liver was received at the laboratory at 12.06 P.M. and the alkali added to the last sample at 12.53 P.M. *x* gave 1.015 per cent., *y* 0.99 per cent., and *z* 0.89 per cent. glycogen. These results would seem to suggest that an appreciable amount of enzymatic hydrolysis may take place between the weighing out of the samples, although not confirmed by the results on No. 63, given just preceding or on the liver of Jerry, which follows.

Jerry was killed at 2.00 P.M. No sample of lean muscle was taken. The liver was received at the laboratory at 3.10 P.M. The grinding was delayed, as the mill was being used for another sample and had to be cleaned so that the alkali was not added to the last sample until 4.35 P.M. *x* gave 1.17 per cent., *y* 1.14 per cent. and *z* 1.17 per cent. glycogen.

If further work confirms the present results as to a rapid hydrolysis of the glycogen, it will show how utterly worthless the glycogen determination is for the detection of the presence of horse flesh.

*Low Temperature Checks Hydrolysis.*—A very fat sheep was killed at 2.45 P.M., March 21, 1908. The liver was removed at 3.20 P.M., and a portion was immediately sliced and frozen by covering with cracked ice and salt. When frozen, the ice and salt were brushed off and the liver was ground and thoroughly mixed. The temperature of the mixture remained below 0° C. The sample was placed in the ice-box and in the evening triplicate portions were weighed out and treated with the alkali, the temperature of the sample not exceeding 10° C.:

Alkali added to Sample 1 at 7.20 P. M., determination lost.

Alkali added to Sample 2 at 7.35 P. M., 1.84 per cent. glycogen found.

Alkali added to Sample 3 at 7.50 P. M., 1.81 per cent. glycogen found.

The main portion of the sample was kept in the ice-box until the next evening (3/22/'08), temperature of sample was 9° C. Triplicate samples were again taken. Alkali was added to sample 1 at 7.20 P.M. 1.70 per cent. glycogen found.

Alkali was added to Sample 2 at 7.35 P.M., 1.71 per cent. glycogen found.

Alkali was added to Sample 3 at 7.50 P.M., 1.59 per cent. glycogen found.

The remainder of the sample was kept in the ice-box until the morning of (3/24/'08), temperature of the sample did not exceed 10° C. Triplicate samples were taken:

Alkali was added to Sample 1 at 9.00 A.M., 1.73 per cent. glycogen found.  
Alkali was added to Sample 2 at 9.15 A.M., 1.76 per cent. glycogen found.  
Alkali was added to Sample 3 at 9.30 A.M., 1.71 per cent. glycogen found.

The following morning (3/25/'08) triplicate samples were taken, temperature did not exceed 6° C.

Alkali added to Sample 1 at 9.45 A.M., 1.65 per cent. glycogen found.  
Alkali added to Sample 2 at 10.00 A.M., 1.70 per cent. glycogen found.  
Alkali added to Sample 3 at 10.15 A.M., 1.73 per cent. glycogen found.

In the evening (3/25/'08) a sample was weighed out at a temperature of 8° C., which gave 1.67 per cent. glycogen. These results indicate that at 10° C. or lower no appreciable hydrolysis of the glycogen takes place. This problem is to be studied further during the ensuing year.

COLUMBIA, MISSOURI.

[CONTRIBUTION FROM THE HAVEMEYER LABORATORIES OF COLUMBIA UNIVERSITY, No. 175.]

### A SOURCE OF ERROR IN THE EXAMINATION OF FOODS FOR SALICYLIC ACID.

By H. C. SHERMAN.

Received September 20, 1909.

In testing foods for preservatives it is commonly assumed that a constituent volatile with steam, soluble in ether, capable of sublimation and crystallization, and giving a violet reaction with ferric chloride, must be salicylic acid. This assumption is, however, incorrect.

It has recently come to the attention of the writer that certain baked cereal products and prepared foods containing such products yield a constituent which shows all of these properties and which would therefore, upon ordinary examination, cause these foods to be reported as containing salicylic acid; but further study showed that the constituent in question did not come from the raw materials used but was developed during the baking process. Salicylic acid was in fact absent and the source of error was indicated by an examination of the literature of the ferric chloride test for salicylic acid and was fully demonstrated by experiment.

#### LITERATURE.

Brand found, in 1893,<sup>1</sup> that an extract of caramel malt gave a reaction with ferric chloride like that given by salicylic acid, and in a paper published the following year<sup>2</sup> cites Erich<sup>3</sup> and Munsche<sup>4</sup> as having made the same observation. In his paper of 1894,<sup>2</sup> Brand recorded the actual separation from such caramel malt of a crystalline substance, soluble in ether or water (much less soluble in cold water than hot), volatile with steam, capable of sublimation and reacting like salicylic acid with ferric chloride, but which did not give the red reaction of salicylic acid with Millon's reagent. By condensing the vapors given off during the roasting process in the manufacture

of a so-called malt coffee, extracting with ether, purifying and recrystallizing in different ways, Brand obtained the reacting substance in crystalline form both as leaflets and as needles. Brand named the substance "maltol," and showed that it had phenolic character and agreed with the formula  $C_6H_6O_3$ .

Kiliani and Bazlen<sup>1</sup> also prepared maltol in crystalline form, showed that it has an acid reaction, and studied its chemical behavior.

Will<sup>2</sup> studied its effect upon yeast.

Abraham<sup>3</sup> pointed out that dark-colored beers may readily contain maltol which, in the examination of the beer for preservatives, will behave like salicylic acid, both in steam distillation and extraction with ether, and give a marked violet coloration with ferric chloride. The ferric chloride reaction being thus inapplicable and the Millon reaction not suitable, Abraham recommended the Jorissen reaction.

This reaction for salicylic acid, proposed by Jorissen in 1882,<sup>4</sup> is as follows:

*Jorissen's Reaction.*—To the solution to be tested add 4 or 5 drops of a ten per cent. solution of potassium (or sodium) nitrite, 4 or 5 drops of acetic acid, 1 drop of a ten per cent. solution of copper sulphate and heat to boiling. In the presence of salicylic acid the solution turns reddish and with more than a very minute amount becomes blood-red. According to Jorissen phenol behaves in the same way but benzoic acid does not. Abraham found that maltol does not give this reaction and recommended it as the most reliable test for salicylic acid.

da Silva<sup>5</sup> used Jorissen's reaction among others.

Klett<sup>6</sup> recommends Jorissen's reaction for the detection of salicylic acid in substances containing citric acid.

Jorissen's reaction was one of those used by Portes and Desmoulières<sup>7</sup> as demonstrating the presence of small amounts of salicylic acid in strawberries.

Windsch,<sup>8</sup> investigating the natural occurrence of salicylic acid in fruits, and having obtained violet reactions with ferric chloride from raspberries and strawberries, adopted the Jorissen reaction to decide the question whether the reacting substance was actually salicylic acid.

Thus it is evident that the Jorissen reaction for the detection of salicylic acid is fairly well established in Europe.

<sup>1</sup> Ber., 27, 3115.

<sup>2</sup> Ztschr. ges. Brauw., 21, 307; abs. Ztschr. Unters. Nahr. Genussm., 1, 720.

<sup>3</sup> Journ. de Pharmacie, de Liege, 1898, 5, 173. Abstracted in *Repert. Pharm.* [3], 10, 347, and *Ztschr. Unters. Nahr. Genussm.*, 1, 857.

<sup>4</sup> *Bulletins de l'Académie Royal des Sciences, des Lettres, et des Beaux-arts de Belgique*, 3rd series, 3, 259.

<sup>5</sup> *Compt. rend.*, 131, 423 (1900); *Bull. soc. chim.* [3], 23, 795 (1900).

<sup>6</sup> *Pharm. Centr.*, 41, 452; *Ztschr. Unters. Nahr. Genussm.*, 4, 469.

<sup>7</sup> *Annal. chim. analyt.*, 6, 401 (1901); *Ztschr. Unters. Nahr. Genussm.*, 5, 468.

<sup>8</sup> *Ztschr. Unters. Nahr. Genussm.*, 6, 447.

<sup>1</sup> *Ztschr. f. d. ges. Brauw.*, 15, 303.

<sup>2</sup> Ber., 27, 806.

<sup>3</sup> *Der Bierbrauer*, 24, 465.

<sup>4</sup> *Woch. f. Brauerei*, 10, 739.



## EXPERIMENTAL.

Fifty grams of a prepared food consisting in part of a baked cereal product were mixed with water, acidulated with phosphoric acid and distilled with steam in an Erlenmeyer flask surrounded by oil heated to 120°-130°. Portions of distillate tested from time to time gave distinct violet reaction with ferric chloride, but no reddish color in Jorissen's test. The distillation was continued until the ferric chloride reaction was no longer obtained, when the total distillate measured 800 cc. When this was mixed and one-hundredth of it (8 cc.) tested with ferric chloride a distinct reaction like that of salicylic acid was obtained. That the reacting substance was not salicylic acid was shown by extracting thoroughly the other ninety-nine-hundredths of the distillate with ether, treating it as recommended by the Association of Official Agricultural Chemists<sup>1</sup> for the complete recovery of salicylic acid, subliming and testing the product obtained. Although this represented practically one hundred times as much of the substance as had last been tested and found to react with ferric chloride, no reaction whatever was obtained with the Jorissen test.

Comparative tests with known solutions indicated that in the writer's hands about twice as much salicylic acid is required for the Jorissen reaction as for the reaction with ferric chloride, whereas in the above case a distinct coloration was obtained with ferric chloride in a test representing 0.5 gram of the sample and no coloration when the Jorissen reaction was applied to a solution of about equal volume, which represented about 50 grams of the same sample, or about one hundred times as much. Hence if the reaction with ferric chloride in the first case had been due to salicylic acid there would have been present in the second case about 50 times as much as is required to give a distinct coloration with the Jorissen reaction. It is therefore abundantly evident that the substance which gave the violet reaction with ferric chloride was not salicylic acid. It was probably Brand's maltol or some very similar substance. The production of this substance is evidently due to the baking process or caramelization rather than to the presence of malt, though it may be produced more abundantly in the baking of malt than in the baking of flour.

When 25 grams of the brown outer crust of ordinary wheat bread (partly from "patent" and partly from "whole wheat" flour) were treated in the same manner as the prepared food described above, the distillate gave a distinct violet reaction with ferric chloride, though it was, of course, free from salicylic acid and did not give the Jorissen reaction.

It may also be noted that Abraham<sup>2</sup> obtained the

violet (maltol) reaction with ferric chloride from roasted coffee as well as from caramel malt.

## SUMMARY.

Maltol, an acid substance volatile with steam, soluble in ether, capable of sublimation, crystallizing sometimes in leaflets and sometimes in needles, and giving with ferric chloride the same violet color as salicylic acid has been shown to be formed in the baking or roasting of malt and the same or some similar substance is produced in the roasting of coffee and the baking of wheat bread.

Such a substance would be reported as salicylic acid when the present ferric chloride test is used, no matter how carefully the test be made and even if the precaution be taken to sublime the acid and a crystalline sublimate be obtained.

In order to avoid this source of error some reaction other than that with ferric chloride must be used for the detection of salicylic acid in foods. Jorissen's reaction, already well established in European literature, is not subject to the same source of error. In carrying out this reaction about 10 cc. of the liquid to be tested were used and to this were added 4 or 5 drops of ten per cent. potassium nitrite, 4 or 5 drops of fifty per cent. acetic acid, 1 drop of ten per cent. copper sulphate; the liquid, after shaking, was then heated to boiling and, if necessary, boiled for half a minute and allowed to stand one or two minutes for the red color to develop. Under these conditions 0.00005 gram of salicylic acid in 10 cc. water (1 : 200,000) gives a distinct reddish color, easily recognizable after a very little practice, especially on comparison with a blank test carefully made with the same amounts as the reagents. With larger amounts of salicylic acid an unmistakable blood-red color develops quickly on heating.

September 14, 1909.

## ADDRESSES.

A NEW STEP IN INDUSTRIAL EDUCATION.<sup>1</sup>

By ALLEN ROGERS, PH.D.

At the present time when there is so much being said in the daily papers and magazines in regard to industrial education, it may be of interest to give an outline of what we are doing at Pratt Institute, and what we hope to do in the future.

Before undertaking, however, to describe what we are doing, I should like to point out two things that we do not attempt. In the first place we are not a trade school; that is, we do not claim to graduate men who are fitted to do only one kind of work. On the other hand we are far removed from the ordinary technical school or university. Although we may teach the same or similar subjects as given in trade or technical schools, we approach them in an entirely different manner, while the attitude of our students toward their work is also quite different. Our field, therefore, may be said to lie practically half way between the two.

In describing the aims of this course in Applied Chemistry,

<sup>1</sup> Read at New Haven Meeting, American Chemical Society, July 1, 1908.

<sup>1</sup> Bull. 107 (Revised), Bur. Chem., U. S. Dept. Agriculture.

<sup>2</sup> Loc. cit.

I do not wish to give the impression that it is the only course given at Pratt Institute, for as a matter of fact, there are more than sixty other courses, many of them day courses, and others evening courses, in all of which there is an enrolment of over four thousand students, drawn from nearly every state in the Union, and from a number of foreign countries. It requires nine different buildings to house these classes, other buildings also being in process of construction.

Some idea of the importance of this type of education can be gathered from the fact that the institute, which Mr. Pratt founded only twenty years ago, started with twelve students, and has grown to the proportions mentioned above, and during these twenty years has given instruction to more than sixty thousand individuals.

It is a significant fact that Mr. Pratt did not, in his will, lay down any hard and fast rules for the Institute. The Trustees have, therefore, made changes in its courses and methods whenever they seemed necessary, but have never changed its purpose, though this is also in their power.

It is owing to this elastic condition of affairs and to the increased demand for young men with technical training that it was deemed advisable to introduce a course in Applied Chemistry; consequently in September, 1905, the new work was undertaken, at which time the writer accepted the position as Instructor in Industrial Chemistry, and was told to organize his course and equip his laboratory for practical instruction. This may seem like a very simple proposition, but rest assured it was no small task.

Being an entirely new departure in this method of teaching the subject, there was no school which could be visited in order to gain any information. It was therefore a question of originality. And now after three years of experience it is with a great deal of satisfaction and pride that I wish to correct a statement which appeared in a recent issue of one of our prominent trade journals.

The article referred to in describing a certain English technical training school says:

"We ought to have had an institution of this kind in America, where we are rather vain of our practical way of handling things; but we have not, and it remains for an Englishman to establish in the heart of England the only real American idea school in the world. Therefore if the bright sons of far-sighted American manufacturers want a real education in their father's business, they must have it made in England."

That we have had, and continue to have, such a school in America, if not already known, let us trust will be made evident by the following remarks.

There is much truth in the analogy drawn by our director when he says:

"When boys are preparing to play a game of football they do not go into a lecture room and listen to a discussion of the best methods of playing the game, but rather get out on the field and try to produce the same conditions as nearly as possible to those they will meet when thrown into contest with their adversaries."

Consequently if we are to teach young men the methods employed in the manufacture of commercial products, we must bring them in as close relationship as possible to the actual working conditions, by which means they will learn as much of the how and the why in one week as they would by months of class-room instruction. Therefore, in arranging for this course in Industrial Chemistry the above belief has been put into practice, five miniature factories having already been installed. These factories are thoroughly equipped for manufacture on a fairly large scale, each plant being a unit in itself is worked independently of the others and occupies a floor space of fifteen by five feet.

They are as follows: Chemical works, soap factory, tannery,

dry color, paint and varnish works, bleaching, dyeing and print works.

In order to suit the needs of a large number of young men who cannot afford either the time or money for a four-year course with college requirements, the course has been made but two years in length. It is designed especially to give the necessary technical knowledge and practical training to those who desire to become foremen, superintendents or heads of departments in the important chemical industries.

As a preliminary to the applied side of his subject the student is given a training in general inorganic chemistry, organic chemistry, qualitative and quantitative analysis together with the necessary amount of physics, mathematics and mechanical training. The last two subjects extend over both years and include mechanics, strength of materials, steam and power transmission, carpentry, forge, foundry, machine work and design.

As it is not within the scope of this paper to dwell upon the details of the above-mentioned subjects, therefore, it will be best to leave them out of the discussion and pass on to the industrial training which begins in the second year.

In order to acquaint the student with commercial processes one hour each day is devoted to the study of general industrial chemistry, by which means he becomes familiar with the details involved in the various branches of industry, thus materially broadening his field of view, one of the principal objects being to show the relation which exists between them, and where one depends upon the other, at the same time bringing into prominence the value of chemical control and the stages in the numerous processes wherein chemical knowledge is not only beneficial, but absolutely essential. On completing each industry one or more trips are made to such factories as best illustrate the points which have been studied, thus more firmly fixing the knowledge gained by class-room instruction.

Furthermore, each student is required to write a complete description of the trip, which becomes a part of his record and is returned to him upon graduation.

The laboratory instruction connected with this course differs very greatly from that given in most schools, inasmuch as it is entirely practical, consisting of the analysis of such substances as gas, water, coal, lubricating oils, vegetable oils, animal oils, soaps, baking powder, foods, milk, sizes, pulp colors, dry colors, paint, varnish, steel, copper, brass and other alloys, boiler compounds, chrome liquor, tan barks, extracts, etc., etc.

Whether this method has any advantage over the one usually employed is an open question with strong arguments on both sides.

It is on our industrial work proper, however, that we place special emphasis, as it is through this method of training that we hope to prepare young men to fill the positions that have been mentioned above.

To illustrate the methods employed in giving this training it will be necessary to give at the same time an account of the equipment which has been provided for this novel line of work.

As the term opens at the beginning of the school year one man out of the class is assigned as foreman of the chemical works and is given three or four of his classmates as assistants. In his assignment he is instructed to make an estimate for the manufacture of a certain chemical; to see that his machinery is put in perfect condition; to inspect his shafting, belts and motors; and to see that everything is in order, and that his factory is kept clean.

This being his first experience at handling men, he is watched very closely. The first chemical which is manufactured is usually barium chloride or a similar compound. Hence while he is having the plant put in working condition he details one of his crew to make an analysis of the witherite in order to determine the content of barium. This being ascertained he

makes his estimate as to the quantity of material necessary to produce any desired amount. He may then be told to estimate on four thousand pounds, it being understood that the weighings are to be made in grams (4000 grams). He includes in his estimate the market prices of raw material, freight f. o. b. New York, wear and tear of machinery, cost of labor and containers. Having decided that he can manufacture this product at a profit, he next puts his men to crushing and grinding the mineral, which is accomplished by means of a power-driven crusher, and grinder. The powdered mineral is then introduced into a porcelain-lined ten-gallon steam jacketed kettle where it is treated with the necessary amount of water and acid. In using the kettle in place of the evaporating dish he becomes acquainted with the method of heating by steam, and learns to regulate his pressure to secure the best results. Having precipitated the iron and other foreign matter, he passes the hot solution through a suction filter, which operation takes about five minutes for the contents of his kettle. This you will note is somewhat shorter and more satisfactory than the old time filter paper. The filtrate is immediately returned to the kettle or vacuum pan and run down to a gravity of about 65° Bé. As this density is reached the hot solution is transferred to crystallizing tanks and allowed to cool, the tanks used for this purpose being old alcohol barrels which have been sawed in the middle. When all of the salt has crystallized the mother liquor is returned to the kettle for further concentration while the crystals are dried in the centrifugal machine. Should the compound prove to be impure, it is again recrystallized, as only a C. P. product is accepted.

The remainder of the equipment in the chemical works consists of a vacuum still, vacuum pump, iron steam jacketed kettle, a porcelain-lined kettle with arbor, two filter presses, copper retort, condenser and large drying-oven. During the time which each student occupies in the factory he is given preparations which necessitate the use of all of the above machinery. As one gang finishes its assignment, another takes its place, continuing the work of the previous week and making new products as required. When all of the class have served a sufficient time in the chemical works, this factory is closed and the machinery cleaned and oiled.

Following the above line of work comes instructing in soap-making which is accomplished by means of a miniature soap factory, consisting of a lye tank, kettle, crutcher, frames, slabber, cutting machine, chipper, mill, plodder and press. The foremanship system is, of course, employed here, the one in charge being held responsible for the quality and quantity of the output. Here the student becomes familiar with the preparation of boiled, half-boiled and cold-made soaps, as well as the methods of slabbing, cutting, chipping, milling, perfuming, plodding and pressing. The size of our crutcher is sufficient for a one hundred pound charge, which when worked up into market form produces about eight hundred cakes.

Outside of the knowledge gained in the manufacture of soap this training gives self-confidence and the satisfaction of making something which is of practical use. During the past year we have made about twenty-five hundred pounds of toilet soap, which I am pleased to say has found a ready market.

The installation for the manufacture of pigments, paint, and varnish consists of three steam-heated precipitating tanks, three settling tanks, filter press, ball-mill, change can mixer, cone mill, kneading machine and varnish kettle. The object of this plant is to give instruction in the preparation of lakes and pigments, the grinding in oil, the manufacture of ready-mixed paints, the manufacture of oil stains, the boiling of oils and the preparation of varnish.

For instruction in the manufacture of leather the equipment consists of three pits 2 × 4 × 4 feet made of cypress and used for soaking, lining and for vegetable tannage. To demon-

strate the process of leaching a set of four vats 18 inches by 3½ feet are arranged in battery and steam-heated. Adjoining the tan-yard is a power-driven pin-mill, 3 feet in diameter by 13 inches wide, making 18 revolutions per minute. Next to the pin-mill is a paddle box 3½ feet long, 2½ feet wide by 2 feet deep, being driven at the rate of 40 revolutions per minute. A glass top slating table, beams, staking stand, slickers, knives, etc., are also provided, while in the roof of the building we have our drying-room, dust box and coloring table.

Thus it will be seen that the equipment lacks nothing for hand work. Splitting machines, shaving machines and such accessories are yet beyond our reach, but we have hopes that some day in the not far distant future these may also be added to our list. The skins as we receive them are in the dry salted or pickled condition and are put through all of the operations of soaking, lining, puering, tanning, scouring, shaving, re-tanning, dyeing and finishing, both mineral and vegetable tannage being employed.

As we have numerous excellent textile schools in this country we do not try to devote much time to the mechanical side of the industry. On the other hand we do give quite a bit of attention to the methods employed in the manufacture and use of dye-stuffs. Thus our organic preparation work is devoted almost exclusively to the manufacture of various coal tar product. For the practical application of these colors there is provided a reel, three two-gallon steam jacketed copper kettles, a steam-heated calcium chloride bath with opening for ten beakers, four dye vats, one jigger for piece goods and a printing machine. A kior is also installed for bleaching purposes.

To give an idea of the extent to which this practical work is conducted I will give a list of the materials manufactured by the students during the past year.

Chemicals (inorganic).....	500 lbs.
"    (organiC).....	80 "
Dye stuffs.....	20 "
Synthetic oils.....	5 "
Toilet soap.....	2500 "
Ready-mixed paint.....	50 gals.
Spirit varnish.....	10 "
Oil varnish.....	10 "
Oil stains.....	10 "
Worsted yarn (dyed).....	20 lbs.
Cotton yarn    ".....	20 "
Silk            ".....	8 "
Piece goods    ".....	600 yards
Leather (tanned).....	240 skins

Whether or not this method of instruction will meet the requirements of the manufacturer remains yet to be seen. Undoubtedly changes will have to be made as we progress in the work. We feel confident, however, that it is a move in the right direction, and we hope for ultimate success.

PRATT INSTITUTE,  
BROOKLYN, N. Y.

## NOTES AND CORRESPONDENCE.

### THE DETERMINATION OF IRON AND ALUMINA IN INORGANIC PLANT CONSTITUENTS.

That the estimation of iron and alumina in the presence of phosphoric acid, manganese, calcium and magnesium is a somewhat troublesome operation is evidenced by the fact that the Association of Official Agricultural Chemists has adopted no method for the determination of these two elements in inorganic plant constituents.

The method which has been in use for some time in this laboratory has been found quite satisfactory; and, as it contains some modifications that we have not seen suggested as applicable to the analysis of ash, we have briefly outlined it below.

*Separation of the Iron and Alumina.*—To an aliquot of a solution of the ash corresponding to one gram add pure ferric chloride of known strength more than sufficient to combine with all phosphoric acid present. To the cold solution add concentrated solution of sodium carbonate until permanent precipitate just begins to form. Clear with 1 cc. of 80 per cent. acetic acid, add one gram of sodium acetate and boil for three or four minutes. When the precipitate has settled sufficiently to determine that the solution is colorless, filter at once before the precipitate becomes slimy. To insure the removal of all the lime from this precipitate it should be dissolved with hydrochloric acid into the same beaker, thereby preventing the necessity of washing beaker and precipitate. The iron and alumina are then reprecipitated with ammonia, filtered, dried, ignited, and weighed. The weight obtained, minus the  $\text{Fe}_2\text{O}_3$  and  $\text{P}_2\text{O}_5$  found by other methods and the known amount of  $\text{Fe}_2\text{O}_3$  added, will be the weight of the  $\text{Al}_2\text{O}_3$  in the ash.

In this method a great deal depends upon the proper adjustment of the sodium carbonate and the acetic acid. If too much sodium carbonate is used lime and magnesia may precipitate. Too much acetic acid may prevent the proper precipitation of iron and alumina.

The solution of ferric chloride which is added produces a red precipitate that is easier to filter and wash, makes a clearer filtrate and insures a complete precipitation of the phosphoric acid.

*Determination of Iron.*—To an aliquot of a solution of the ash determine iron by the Zimmermann-Reinhardt modification of the potassium permanganate method. Methods for determining iron in inorganic plant constituents by means of potassium permanganate have been offered before. The referee of the A. O. C.<sup>1</sup> recommended one modification of this as an official method of that association last year. All the methods, however, call for reduction of the iron by means of zinc, which necessitates the removal of the hydrochloric acid before the reduction can be proceeded with, and failures to remove all the hydrochloric acid, or to completely reduce the iron to the ferrous state are frequent sources of error when iron is determined in this manner. The danger of converting a part of the iron into an insoluble form when removing the hydrochloric acid by evaporation, and of not completely removing all zinc before proceeding with the titration are also frequent causes of error when this method is used. The Zimmermann-Reinhardt modification of the permanganate method in which the ferric iron is instantly reduced with stannous chloride, then titrated with the permanganate in the presence of the hydrochloric acid, after the addition of a "titrating" solution of manganese sulphate, phosphoric and sulphuric acid, is a decided improvement both in rapidity and accuracy over the use of zinc for reduction when iron is to be determined with potassium permanganate solution. R. F. HARE.

NEW MEXICO AGRICULTURAL COLLEGE.

#### THE ADAMKIEWICZ-SENEX TEST FOR CASEIN IN PAPER.

The Adamkiewicz test for casein sizing in paper consists in extracting the paper with borax solution, precipitating the dissolved casein with acid, drying and warming gently with a mixture of two parts glacial acetic acid and one part concentrated sulphuric acid. The gradual development of a purple color indicates the presence of casein. This test has been simplified by Senex,<sup>2</sup> who moistens the paper with the reagent and warms slightly.

The simplicity of this test induced us to try it in this laboratory, and it was found that a casein-sized paper readily gave

<sup>1</sup> U. S. Dept. Agr., Bur. Chem., *Bull.* 122, 93.

<sup>2</sup> *Papierfabrikant*, 1908, 3065; *Mitth. kgl. Materialprüfungsamt*, 27, 153 (1909).

the reaction; but unfortunately a check sample of paper sized with rosin also gave the purple color. This is not surprising in view of the similarity between the above mixture of acids and the acetic anhydride-dilute sulphuric acid (sp. gr. 1.530) mixture used in the Liebermann-Storch test for rosin. Further tests showed that powdered rosin, wood pulp and pine shavings, as well as casein, responded to this test. It is evident that before testing a sample of paper for casein it must be first thoroughly extracted with alcohol acidified with acetic acid in order to remove all rosin sizing that may be present.

C. E. WATERS AND J. C. BENEKER.

BUREAU OF STANDARDS.

#### THE SEPARATION OF BISMUTH FROM ALLOYS CONTAINING ALSO LEAD AND TIN.

The separation of bismuth from the associated metals in fusible alloys presents some difficulties when working according to the more obvious methods.

An expedient which renders the estimation simple, has not come to the attention of the writer, and he therefore risks the following:

The acid filtrate from the mixed nitrates filtered from the tin oxide, is treated with a moderate excess of potassium or sodium hydroxide solution. This precipitates the bismuth and cadmium if present, and redissolves the lead hydroxide.

The oxides are filtered off, washed, and dissolved in hydrochloric acid. The bismuth is precipitated in the well-known manner as basic chloride, by being poured into a large quantity of water.

By getting rid of the lead in the manner indicated there is no complication resulting from the precipitation of basic salts of bismuth where they are not wanted. CHAS. E. SWETT.

#### ON THE COMPOSITION OF LINSEED OIL FOOTS.

A few years ago G. W. Thompson published a very interesting article on the composition of the "break" from linseed oil.<sup>1</sup> He found that by heating a sample of linseed oil to about 400° F. he obtained 0.277 per cent. of a non-oleaginous substance which yielded 47.79 per cent. of ash consisting mainly of calcium and magnesium phosphates and less than 1 per cent. of nitrogen.

The authors had occasion to examine a sample of linseed oil "breaking-foots" which were obtained on a large scale from several thousand gals. of oil, the latter having been obtained by the naphtha extraction process. The oil had been heated to 500° F. through the direct introduction of superheated steam, and the foots were collected in a centrifugal machine. In this state the foots represented a brownish black dry mass of a consistency resembling that of soft cheese, and appeared free from oily matter. Extraction with carbon bisulphide showed, however, that 75.8 per cent. of the substance was still soluble in carbon bisulphide. The analysis of the soluble part proved it to be linseed oil, as shown by the following figures:

Specific gravity.....	0.9410 at 60° F.
Saponification value.....	191.3
Iodine value.....	160.6 per cent.
Hexabromides.....	8.2 per cent.
Acid value.....	5.3

The discrepancies between these values and those known for raw linseed oil are evidently due to the heating process to which the oil has been subjected.

The insoluble residue resembled in its appearance ground cocoonut shells and yielded the following analytical data:

<sup>1</sup> *J. Am. Chem. Soc.*, 1903, page 1005.

	Per cent.
Silica.....	0.23
Manganese oxide.....	1.00
Calcium oxide.....	23.53
Magnesium oxide.....	14.51
Phosphorus pentoxide.....	60.30
Sulphuric acid.....	none
Total.....	99.57

So far the results are a very close check to Thompson's work.

Another sample of foots which we subjected to an analysis represented settlings from the bottom of a tank containing raw linseed oil made by the extraction process. It was much more difficult to remove the oleaginous matter from these foots, but the appearance of the residue that proved insoluble in carbon bisulphide did not differ materially from the residue in the former case. The analysis of the extracted oil was as follows:

Saponification value.....	202.6
Acid value.....	14.5
Hexabromides.....	12.5 per cent.
Iodine value.....	145.3

Although these figures differ quite materially from those of raw oil, they point clearly to the fact that no other oleaginous matter but linseed oil was present in the foots. It will be remembered that the removal of the solvent at a comparative high temperature and the small quantities on hand cannot fail but exercise a strongly oxidizing influence.

The insoluble part of these foots contained 0.57 per cent. nitrogen, and the ash analyzed as follows:

	Per cent.
Silica.....	12.53
Manganese oxide.....	0.51
Calcium oxide.....	3.17
Magnesium oxide.....	6.25
Phosphorus pentoxide.....	72.75
Total.....	85.21

The final filtrate showed a strong qualitative test for potassium.

The presence of silica in these foots is quite interesting, and was not observed in the foots obtained by the "breaking process."

Another sample of settling foots from raw oil obtained by the "naphtha process" showed a yet higher percentage of silica in its ash:

	Per cent.
Silica.....	34.38
Calcium oxide.....	7.98
Magnesium oxide.....	8.39
Phosphorus pentoxide.....	46.50
Potassium oxide.....	present

It was feared that the silica in this case might be due to mechanically suspended impurities of a sandy nature. In order to settle this question, the foots were dissolved in petrol ether and filtered through properly selected paper, whereupon the solvent was evaporated. The subsequent analysis was a very close check to that reported above which seems to prove that the silicious matter was a genuine constituent of the foots in question.

Contrary to this, a sample of settling foots from oil obtained by the hydraulic press system showed a total absence of silica:

	Per cent.
Silica.....	none
Calcium oxide.....	3.26
Magnesium oxide.....	4.99
Potassium oxide.....	10.27
Phosphorus pentoxide.....	81.08

All these samples show a tremendous preponderance of acid radicals over basic radicals.

OTTO EISENSCHIME,  
H. N. COPHORNE.

AMERICAN LINSEED Co., So. CHICAGO, ILL.

## BOOK REVIEWS AND NOTICES.

**Food Inspection and Analysis.** BY ALBERT E. LEACH. Second Edition, Revised and Enlarged. Large 8vo., xviii, 954 pp., 120 figures, 40 full page plates. Cloth, \$7.50 net. New York: John Wiley & Sons.

When the first edition of Dr. Leach's book appeared five years ago it was eagerly seized and used by food chemists and inspectors and at once took a preeminent position among the works on the subject. It is seldom indeed that any book in its first edition shows such evidence of painstaking care in the preparation and in the selection and rejection of material. The first edition has now been thoroughly tried and in no sense has been found wanting. It is an excellent example of good authorship and good bookmaking.

The second edition is of the same high quality as the first. The pages have been increased from 787 to 954. New methods of analysis have been introduced, covering the following subjects: meat extracts, flour, noodles, paprika, prepared mustard, tea, coffee, cocoa products, ice-cream, maple products, honey, oils, distilled liquors and preservatives. There is a separate chapter on the various forms and applications of the refractometer and one on flavoring extracts, including those of almond, peppermint, wintergreen, rose, cassia and cloves.

The second edition of Dr. Leach's book will be welcomed by all chemists in the field of food inspection and analysis and will continue to occupy its important place for years to come.

W. D. RICHARDSON.

**Untersuchung der Mineralöle und Fette.** By DR. D. HOLDE. Third Edition, 12 mo. Pp. xii + 459. Berlin: Julius Springer. 1909. Price, 12 Marks.

The third edition of Dr. Holde's useful book contains several important modifications and additions. The chapters relating particularly to mechanical tests have been condensed, as have also other outdated matters and the analytical character of the book has been emphasized by utilizing the space thus obtained for extending the chapters: Specific Heat; Heat of Vaporization; Vaseline Lubricating Oil; Tar By-products; Turpentine, etc.

For a compact, concise book on mineral oils and fats, covering a large amount of ground, this work is unexcelled.

**Chemiker Kalendar, 1910.** By DR. RUDOLF BIEDERMANN. Two Parts. Part 1, xx + 50 + 385 pages. Part 2, 580 pages. Thirty-first year. Berlin: Julius Springer. 1910. Price, Cloth, 4 Marks; Leather, 4.50 Marks.

The Chemiker Kalendar, known to all, the chemist's *vade mecum*, now appears for the thirty-first consecutive year. It is probably true that Dr. Biedermann's work is possessed by more chemists and is more used from day to day than any other individual chemical book. It fully deserves its reputations because it is accurate, compact, comprehensive, well made and very moderate in price. For the current year nineteen pages have been added. As a suggestion to the author, the sulphuric acid tables of Lunge and Isler might well be supplemented by those of Ferguson and Talbot. Also the two parts might be combined and the whole bound in cloth or leather. Even so the book would be less bulky than many of the engineering handbooks.

**Iron Ores, Building Stones and Other Minerals.** WEST VIRGINIA GEOLOGICAL SURVEY. pp. 603, 24 page plates, 16 figures and maps. Post-paid, \$2.00; with maps, \$2.25.

This volume gives descriptions and analyses of all the principal iron ore deposits of the state, together with a history of the old charcoal furnace industry. The principal building stones of the state are also described and elaborate tests of their

strength and crushing limit made by the War Department of the Watertown Arsenal and also by the U. S. Geological Survey Testing Laboratory at St. Louis, together with chemical analyses, petrographic determinations, etc. The glass sands and salt industry are fully described and analyses given.

C. N. WILEY.

"The Utilization of Fuel in Locomotive Practice," is the subject of a bulletin just issued by the United States Geological Survey. The author, Professor W. F. M. Goss, of the Technologic Branch of the Survey, makes the statement that locomotives in service on the railroads of this country consume more than one-fifth of the total coal production of the United States:

Summary of results obtained from fuel burned in locomotives:

	Tons.
1. Consumed in starting fires, in moving the locomotive to its train, in backing trains into or out of sidings, in making good safety valve and leakage losses, and in keeping the locomotive hot while standing (estimated).....	18,000,000
2. Utilized, that is, represented by heat transmitted to water to be vaporized.....	41,040,000
3. Required to evaporate moisture contained by the coal.....	3,600,000
4. Lost through incomplete combustion of gases	720,000
5. Lost through heat of gases discharged from stack.....	10,080,000
6. Lost through cinders and sparks.....	8,640,000
7. Lost through unconsumed fuel in the ash..	2,880,000
8. Lost through radiation, leakage of steam and water, etc.....	5,040,000
	90,000,000

## SCIENTIFIC AND INDUSTRIAL SOCIETIES.

### AMERICAN INSTITUTE OF CHEMICAL ENGINEERS.

Second annual meeting, December 8 to 10, 1909. Sessions held at Hotel Walton, Philadelphia, Pa.

#### PROGRAM.

WEDNESDAY, DECEMBER 8, 1909.

10.00 A.M.—Meeting at Hotel Walton.

Address of Welcome, Mayor John E. Reyburn.

Business Session.

Reports of Officers and Council.

Reports of Committees.

11.30 A.M.—Reading of Papers.

(1) "Natural Draft Gas Producers and Gas Furnaces," Ernest Schmatolla. Discussion by O. K. Zwingenberger and G. E. Barton.

(2) "The Commercial Extraction of Grease and Oils," W. M. Booth. Discussion by Frederick Dannerth, Louis Olney and Samuel P. Sadtler.

12.30 P.M.—Luncheon (Alternative). (Table d'Hote 75 cents.)

Luncheon was served at Hotel Walton for members who attended the excursion to the University and Commercial Museum.

1.30 P.M.—Excursions.

Visit to Chemical, Engineering and Physical Laboratories of the University of Pennsylvania.

Visit to Commercial Museum of Philadelphia, Dr. W. P. Wilson.

1.00 P.M.—Luncheon (Alternative).

Luncheon was served by Harrison Bros. and Company, at their works for members holding admission cards for the excursion.

1.30 P.M.—Excursion.

Excursion through the Chemical Works of Harrison Brothers

and Company, Thirty-fourth Street and Gray's Ferry Road. (Admission by card only.)

7.30 P.M.—Meeting at Hotel Walton.

Address of Retiring President, Dr. Samuel P. Sadtler.

Address, "The Chemical Industries of America," Prof. Chas. E. Munroe.

THURSDAY, DECEMBER 9TH.

9.30 A.M.—Excursions.

Members of Institute and their Guests will assemble at Race Street Wharf on the Delaware and be conveyed by the City Boat *S. H. Ashbridge* to the Torresdale Filtration Plant—returning stop at Tacony to visit the Wool De-greasing Plant of Erben, Harding and Company.

12.30 P.M.—Luncheon.

Luncheon at the works of Welsbach Light Company.

1.30 P.M.—Excursion.

Excursion through the works of the Welsbach Light Company.

3.00 P.M.—Excursion.

Return by boat to Kaighn's Point, Camden, and excursion through By-Product Coke Oven Plant of the Camden Coke Company.

5.00 P.M.—Return.

Return to Chestnut Street Wharf, Philadelphia.

7.00 P.M.—Dinner.

Subscription Dinner at the Hotel Walton (\$3.00).

FRIDAY, DECEMBER 10TH.

9.30 A.M.—Meeting at Hotel Walton.

Installation of Officers and Business Meeting.

10.30 A.M.—Reading of Papers.

(3) "Multiple Effect Distillation," F. J. Wood (Mechanical Engineer of Marx and Rawolle's Glycerine Refinery).

(4) "The Advantages of the Multiple Effect Distillation of Glycerine and Other Products," A. C. Langmuir. Discussion by P. B. Sadtler and Chas. L. Reese.

(5) "Reclaiming of Waste India Rubber," S. P. Sharples. Discussion by Wm. M. Grosvenor.

(6) "The Permanence and Acidity of Hydrogen Peroxide Solutions," Launcelot W. Andrews.

12.00—Luncheon at Hotel Walton (Table d'Hote 75 cents).

1.00 P.M.—Excursion to Trenton.

Leave Broad Street Station for Trenton, N. J., at 1.00 P.M.

2.00 P.M.—Excursions.

Trenton Potteries.

The Hamilton Rubber Company.

Linoleum Works.

7.30 P.M.—Reading of Papers.

(7) "Materials for Textile Chemical Machines," Frederick Dannerth. Discussion by Louis A. Olney and J. M. Matthews.

(8) "A Method for Smelting Iron Ore in the Electric Furnace," Edward R. Taylor.

(9) "Chemical Composition of Illinois Coal," A. Bement.

(10) "Heat Efficiency of Smokeless Combustion, and Heat Absorbing Capacity of Boilers," A. Bement.

9.00 P.M.—Final Business Meeting.

SATURDAY, DECEMBER 11TH.

Visit to Cement Plant at Allentown, Pa.

The attendance at the meetings and excursions of the Institute at Philadelphia was very satisfactory. The dinner on Thursday evening was well attended and great enthusiasm shown both by members and guests in the importance of the work undertaken by the Institute, especially in maintaining a high standard for its membership.

The following officers were elected for the ensuing year:

*President*, Dr. Chas. F. McKenna, 50 Church St., New York; *First Vice-President*, Dr. F. W. Frerichs, 3828 Westminster Pl.,

St. Louis, Mo.; *Second Vice-President*, Dr. Edward G. Acheson, Niagara Falls, N. Y.; *Third Vice-President*, Dr. Eugene Harnel, Ottawa, Ont., Can.; *Secretary*, Dr. John C. Olsen, Polytechnic Inst., Brooklyn, N. Y.; *Treasurer*, William M. Booth, Dillaye Bldg., Syracuse, N. Y.; *Auditor*, Henry S. Renaud, 159 Front St., New York; *Directors for one year*, Geo. B. Adamson, Easton, Pa.; David Watson, 111 South Mountain Ave., Montclair, N. J.; Dr. Edward Gudeman, Postal Telegraph Bldg., Chicago, Ill. *Directors for two years*, Ludwig Heuter, Berkeley, Cal.; Thorn Smith, Portland, Mich.; H. F. Brown, DuPont Bldg., Wilmington, Del.; *Directors for three years*, Dr. William M. Grosvenor, 1123 Broadway, New York; Richard K. Meade, Nazareth, Pa.; Dr. S. P. Sadtler, 29 South 10th St., Philadelphia, Pa.

#### PHILADELPHIA MEETING.

##### Abstracts of Papers.

#### THE UTILIZATION OF WASTE INDIA RUBBER.

Abstract of paper by S. P. SHARPLES, S.B.

This paper gives a résumé of the various methods which have been employed in recovering the waste of rubber of commerce.

It calls particular attention to the processes which were employed by the late Nathaniel Chapman Mitchell in the Philadelphia Rubber Works. Mr. Mitchell was the first to make a commercial success in this country of methods for utilizing worn-out rubber articles, such as boots and shoes. This was largely due to the fact that he discovered that sulphuric acid of 15 to 25° B. could be employed to removed the fiber from these goods without injuring the rubber. The process was at once stolen and utilized all over the country until at the present time it is used in many works.

#### OIL AND GREASE EXTRACTION.

By W. M. BOOTH.

The properties of the solvents which can be used for the extraction of oils are given. The solvent power and the cost of each is given. On this basis the cheapest solvent is found to be naphtha. The construction of building tanks and extraction apparatus is discussed. Methods of operation are also given. The methods in use for the recovery of grease from gabbage tankage and other waste products is given. Drawings of extraction apparatus are given and also a number of tables and curves giving the properties of various fats and oils.

#### ELECTRIC FURNACE FOR THE SMELTING OF IRON ORE.

Abstract of paper by EDWARD R. TAYLOR, Penn Yan, N. Y.

Having found the electric furnace for the manufacture of carbon bisulphide one of the easiest of operation and control, an attempt has been made to apply these principles to other manufactures. The saving of the use of movable electrodes by the use of broken conductive carbons which in an important sense fulfil the same office. This system is especially adapted to large operations.

Arrangements are made to reduce the natural conductivity by feeding charcoal in a practically unbroken column down the center of the furnace and between the electrodes. Such column is also directed outwards in the four quarters of the furnace around which and between which the ore with fluxes is fed, maintaining four columns of coarse ore—outside of which are maintained mantles of fine ore directed by means of the lining of the furnace to form a movable lining and absorbent for the radiant heat—which is returned to the furnace with such heated ore. This operation is facilitated by screws working in the outer and cooler part of the fine ore forcing the same

into the heated interior of the furnace above the electrodes where the reduction and fusion is completed, as fast as melted the reduced iron falling between the electrodes into the hearth of the furnace from which it is tapped out.

Arrangements are also made for the circulation of CO to extend the zone of ore reduction.

#### HEAT EFFICIENCY OF SMOKELESS COMBUSTION AND HEAT ABSORBING CAPACITY OF BOILERS.

By A. BEMENT, Chicago, Ill.

This paper gives the result of tests carried out to ascertain the loss of heat due to incomplete combustion. The results are given in a table showing comparative heat balance of an ordinary water tube boiler in which the combustion was incomplete and an improved boiler arranged for the reception of a fire-brick tile furnace roof which made it necessary for the gases to travel to the rear of the boiler before coming in contact with the water tubes. By this arrangement, complete combustion was secured as shown by the absence of smoke. The loss of heat due to incomplete combustion in the ordinary boiler was found to be 9.3 per cent., while in the improved boiler there was no loss. The method of making the determinations and calculations is given in full.

In the second part of the paper, the causes governing the efficiency of a boiler are discussed. The results of the efficiency of three boilers. The first is a well-known water tube boiler in which the gases enter among the tubes at the rear and pass diagonally across the tubes to the exit. In the second boiler, a baffle was inserted among the tubes so that gases flowed twice the length of the tubes. In the third boiler, two baffles were inserted so that the gases flowed three times the length of the tubes. The fuel saving in the single baffle over the plain boiler was 8.68 per cent., while the saving in the double baffle was 20.68 per cent. Complete data with reference to the coal, temperature of gases, steam, draft, power developed, etc., are given.

#### THE ADVANTAGES OF MULTIPLE EFFECT DISTILLATION AND ITS APPLICATION IN OTHER LINES.

By A. C. LANGMUIR.

The Wood Process of glycerine distillation marks one of the most important advances made in recent years in this line.

The patent rights have been acquired by Marx and Rawolle, New York, in whose plant the process is now in successful use on a large scale.

The fuel economy is marked, a considerable increase in output per still being obtained at a fuel consumption of less than half of that formerly used.

A set of six stills in series is successfully at work with no diminution of efficiency in the last still as compared with the first.

The chief advantages of the process, aside from the fuel economy, consist in the ease of working, uniformity of product with varying crudes and the scientific control possible at all times. The process is largely automatic, less labor and less skill in handling being required.

Only a small body of glycerine is present in the heaters, stills and connections, and the prolonged heating of large bodies of glycerine with resulting loss in the average still is avoided.

The spraying of the glycerine through the steam avoids the shooting and foaming often met with when distilling certain crudes in the still generally used.

The process is applicable in any distillation where steam is used and the product obtained has a boiling point higher than that of water such as the distillation of the heavier products

of coal tar and petroleum of aniline, essential oils and the fatty acids of the candle industry.

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#### MULTIPLE EFFECT DISTILLATION.

By F. J. WOOD.

The various steps leading up to the present development of the art of glycerine distillation are described with particular reference to the fuel economy. The consumption of coal per pound of glycerine output is greater now than with the earlier system, as it has been found more economical to save glycerine from decomposition by the use of a flow of steam in a vacuum which greatly lowers the boiling point.

In the multiple effect apparatus, a superior economy of fuel is obtained by using but one flow of steam to a number of stills and condensers, the stills being arranged in series in regard to the steam flow, that is the steam from the steam jet of the first still flows through a series of stills alternating with condensers, the condensers being maintained at a temperature that will condense glycerine out of the steam, but not the steam itself.

The glycerine is forced through heaters by means of centrifugal pumps. The heaters are connected with the boilers and supply the heat necessary for the vaporization of the glycerine.

From the heaters the hot glycerine is sprayed down through the steam.

The combined vapors of steam, and glycerine pass into boiling water condensers whose temperature is maintained at such a point by means of a pressure regulator, that the glycerine, but not the steam, is condensed. The glycerine in condensing gives up its latent heat of evaporation to the boiling water. The steam passes on to the next still in the series to take up its quota of glycerine. Finally, after the last of the series of stills is passed, the steam together with the volatile impurities are condensed as sweet water in a cold water condenser.

In order to maintain a uniform vacuum throughout the system, there must be a free flow of steam and glycerine vapor without obstructions. In this system instead of forcing the steam down through the body of glycerine, the glycerine is pumped up and sprayed down through the steam and no back pressure to the flow of steam results.

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#### CHEMICAL COMPOSITION OF ILLINOIS COAL.

By A. BEMENT, Chicago, Ill.

This paper gives the chemical composition and heating power of the coal obtained from seams throughout the state. It is a summary of a large number of analyses.



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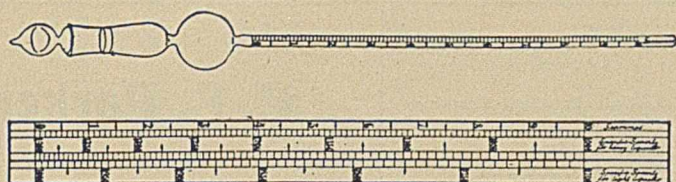


Fig. 2

¶ The Instrument is provided with three scales on the one stem, as shown in Fig. 2, representing the paper scale laid out flatly before being placed in the stem of the hydrometer.

¶ The first scale is graduated from 0.700 to 1.000 for light liquids in bulk.

¶ The second scale is graduated from 1.000 to 1.400 for heavy liquids in bulk.

¶ The third scale *serves as a balance*, being graduated 0 to 12 grammes in subdivisions, for determining Sp. Gr. of light or heavy liquids in small bulk (5 cc's) and SOLIDS, which are placed in the small perfectly stoppered cylindrical bulb graduated to contain 5 cc's of distilled water at 60° Fah.

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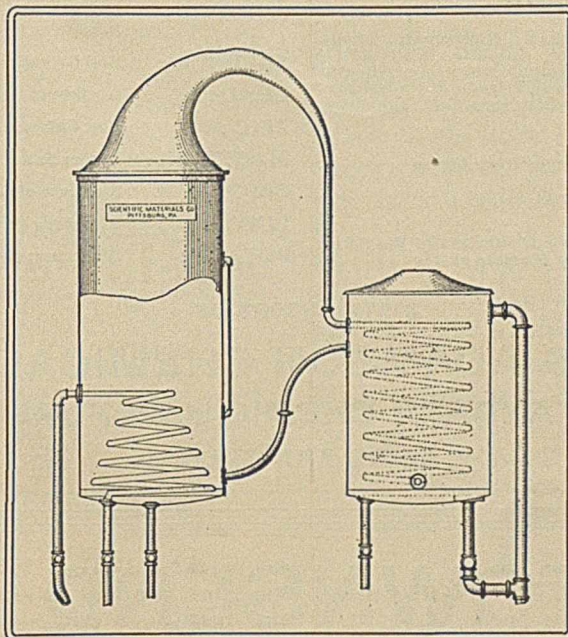
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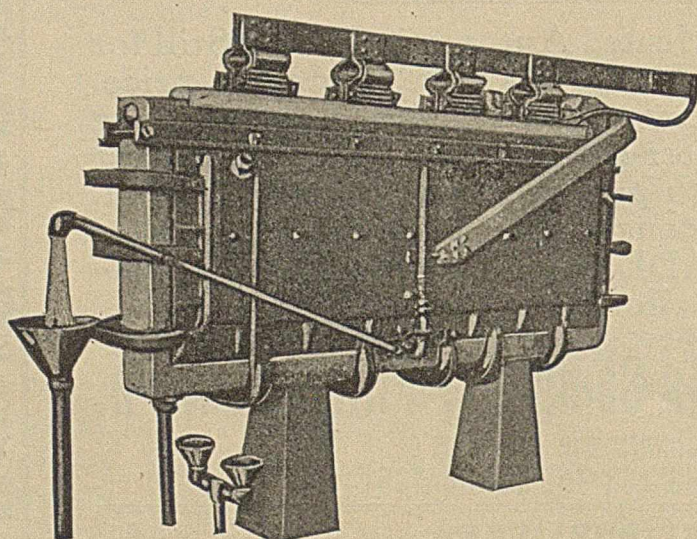
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