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## EDITORIALS

### RELIEF FOR THE PATENT OFFICE

On March 5, 1920, the Nolan Bill, H. R. 11984, which will give to the Patent Office resources for the enlargement and improvement of its personnel and general office operations, was passed by the House of Representatives.

This relief legislation embraces the general features which were urged by the Patent Committee of the AMERICAN CHEMICAL SOCIETY, whose chairman, Mr. E. J. Prindle, has worked indefatigably. For a while there seemed to be difficulty in getting the attention of Congress, but the hearings held before the Rules Committee of the House proved to be an eye opener. The bill went through the House without a roll call, and is now with the Senate Committee on Patents, Senator George W. Norris, chairman. It is very important that Senator Norris and senators in general should know how strongly chemists favor this bill and the pressing importance of its speedy passage. Letters from individual chemists to their senators and to the chairman would go far toward arousing interest in the bill. This seems to be the only thing needed to assure its passage. It will be a fortunate day for the American chemical industry when the Patent Office is enabled to accomplish more adequately the purpose for which it was founded.

### THE CHEMICAL WARFARE SERVICE

The Army Reorganization Bill passed the House of Representatives on March 18, 1920. It contains a section providing for the Chemical Warfare Service as a separate unit of the Army, with a chief holding the rank of brigadier general, and with 90 officers, ranging from colonel to second lieutenant, and 1500 enlisted men. It was in the House that friends of the Chemical Warfare Service had most to fear, for certain members had been outspoken in their opposition to the Service being made a separate unit. This opposition, however, gave way, and now the bill goes to the Senate, where it seems there is absolute unanimity of sentiment that the Chemical Warfare Service shall not only be a separate unit, but that full provision shall be made for its complete development and thorough efficiency. It is a safe prediction that any changes made in the section pertaining to the Chemical Warfare Service from now on will be only such as tend to strengthen and improve the Service.

The unexpected announcement of the transfer of Major General William L. Sibert from the head of the Chemical Warfare Service to the command of Camp Gordon, Atlanta, Ga., was a distinct shock to all who had been connected with this new branch of our military system. Whatever may have been the reasons which led to this transfer, one thing is certain: As he leaves that Service which he so aggressively developed, General Sibert carries with him not only the high personal esteem but also the genuine affection of all who now

are or who were during the strenuous days previous to the armistice members of the Chemical Warfare Service. Moreover, while this transfer removes him from the Washington atmosphere, nevertheless the clear, logical, and forceful testimony which he gave before committees of both the House and Senate made such an impression as to ensure the success of his ideas with the Congress, and this we take it is perhaps the greatest service General Sibert has performed, for he has done that which will safeguard in large measure the future peace of this country.

It is indeed fortunate that as a successor to General Sibert Lt. Col. Amos A. Fries was available. His fine service in the A. E. F. and the work he has done for the Chemical Warfare Service since his return give assurance that the active leadership of the Service in the future could not have been placed in stronger or wiser hands. Colonel Fries, whom we hope soon to hail as Brigadier General Fries, can count upon the whole-hearted and enthusiastic support of the civilian chemists of the country.

### "NOTHING TO EAT BUT FOOD"

We have just received Bulletin No. 1 of the Free Trade League, among whose members are noticed the names of prominent citizens. The bulletin is directed against the bills designed to protect the tungsten, magnesite, zinc, pearl buttons, scientific instruments, and coal-tar products industries.

The chief wail of the Free Trade League is that great secrecy has shrouded the introduction and progress of these bills. Secrecy, indeed! When we think of the journeyings to Washington of all parties affected by these bills, when we think of the groaning of the Government Printing Office under the mass of testimony presented at public hearings on the bills, the charge of lack of openness reminds us of the plaint of the man who sang:

Nothing to eat but food,  
Nothing to breathe but air,  
Nothing to wear but clothes and shoes  
And a hat on the top of my hair.

We don't imagine Bulletin No. 1 will have much influence on public thought or on congressional action, but we wonder how the members of the League square the term "secret movement," etc., with legislation which has on two separate occasions been urged by the President in his messages to Congress, which passed the House of Representatives after a full week of debate, and which now has enthusiastic non-partisan support in the Senate.

The Free Trade League is a little late in getting into the game, but it is very amusing to note the combined opposition of the American Protective Tariff League and the Free Trade League to the bill to make permanent the coal-tar chemical industry. The former describes the bill as a "grab;" the latter says it was "concocted by interests." A strange pair of bed-fellows that!



## AIR-CASTLES

The complexities of present day rates of foreign exchange lead to rather unexpected developments. For instance, the editor of a well-known German techno-chemical journal turns a deaf ear to the opinions frequently expressed in this country that this is no time for foreign loans. With that sublime confidence which frequently during the past few years has been so utterly out of place, he writes to an American chemist, known to him only through his subscription list and through a contributed article some seven or eight years ago. Without the slightest suggestion of security other than that of Teutonic good faith, he baldly requests a loan of five hundred dollars, not because of necessity, but avowedly because of the opportunity which the present rate on the mark as compared with the dollar offers, confident of course that at the end of the three-year period for which he requests the loan there will be a considerable increase in the value of the mark.

This five hundred dollars would represent at present about fifty thousand marks. If the exchange went back to normal, he could return two thousand marks, leaving him a profit of some forty-eight thousand marks, or twelve thousand dollars. It is not difficult to picture the air-castles that can be constructed on the basis of such rosy visions; the only difficulty, we learn from our correspondent, is that the loan will not be made, nor the letter even answered. We mention it here in the thought that possibly this may not be the only letter of its kind floating around the country. The wily doctor may have used a mimeograph. What next?

## NOTES

It's at St. Louis and the date is April 12th.

We had the good fortune recently to visit one of the large research laboratories connected with the new dye industry. An interesting sidelight of that tour of inspection was the great number of American-made optical instruments and thermometers in use, to the unexcelled character of which the chemists freely testified. The Bacharach bill makes sure the permanency of the American scientific instruments industry.

The letter of retiring Secretary Lane to the President proved one of the striking events of the month. Mr. Lane paid a generous compliment to the chemists of America in the following words:

It is not generally known that the largest society of scientists in the world is the AMERICAN CHEMICAL SOCIETY. If as appears now to be the promise, the immediate future is to be largely in the hands of the chemists, America need not look forward too sparingly, for we have a stock of men of this training which no other nation can excel.

The Sixth National Exposition of Chemical Industries will be held at the Grand Central Palace, New York City, in the week of Sept. 20, 1920. The management has issued a printed list of those exhibitors who have already engaged space. There are 308 names on the list, and the date is six months distant. Clearly the Sixth Exposition is to be a record breaker.

The way to begin is to begin. The matter of recovering the lost ground referred to in our last issue has already begun. President Mark W. Potter of the C. C. & O. Railway writes us that this road proposes to continue the utilization of the chemist and that steps are already under way to reopen their chemical survey laboratories. He writes:

It is our thought to be more progressive in the future. \* \* \* The function of the chemical engineer is of increasing importance. In the future more than in the past, successful achievement will mean the discovery of new opportunities, the increase of efficiency and elimination of waste, and in all the work that is ahead of us the chemical engineer must be of increasing importance.

Senator Moses (New Hampshire) seeks to amend the coal-tar chemical bill by removing the embargo feature and making it a straight tariff measure. In the *Daily News Record* of March 17, 1920, the Senator is quoted thus: "There can be no question as to who is behind my bill." Sure, that's an easy guess, the earmarks are perfectly plain. And by that same token there can be no question that the Senator's amendment is in no wise to be considered as an effort to safeguard or develop the American industry, but is simply an expression of the willingness of "who is behind my bill" to pay 20 per cent extra for the privilege of getting all the German dyes they want. Of course there are all sorts of ways of recouping that 20 per cent. It's a pretty safe guess, too, that every dye importer is loud in his praises of the Senator's amendment. Sure, they know the game. Maybe they are circularizing the mills again in order to assist Congress in its deliberations.

A correspondent suggests a useful form of coöperation between universities and industries: the establishment of prizes, in the chemistry departments of universities having adequate library facilities, for the best bibliographies compiled by students on subjects assigned by the industry. It is believed that the industry would be a two-fold gainer from such an investment, directly, in the value of the bibliographies compiled, and indirectly, through the better training in handling chemical literature which the competing students would receive.

Dr. William H. Walker, Director of the Division of Industrial Coöperation and Research at the Massachusetts Institute of Technology, contributes, page 394, this issue, an interesting discussion and explanation of the "Technology Plan" which was utilized so effectively in raising the \$8,000,000 endowment fund. The matter has aroused widespread interest and it is felt that the same plan in more or less modified form can and doubtless will be used by many other institutions. Cornell University has already adopted a very similar form of plan and contract. It will be interesting to note modifications and possible improvements as the thought of the educational and industrial world is brought to bear upon this plan, and equally interesting to note the outworking of the plan at Technology under the vigorous administration which it will without doubt receive from Dr. Walker.



## ORIGINAL PAPERS

PREPARATION OF NITROGEN AND HYDROGEN MIXTURE BY DECOMPOSITION OF AMMONIA<sup>1</sup>

By R. O. E. Davis and L. B. Olmstead

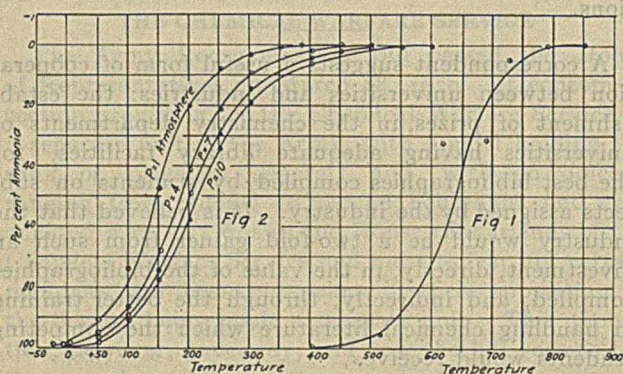
BUREAU OF SOILS, DEPARTMENT OF AGRICULTURE, WASHINGTON, D. C.

Received October 2, 1919

In work in the Bureau of Soils laboratory on the synthesis of ammonia done in coöperation with the Nitrate Division of the War Department, it was necessary to obtain a mixture of hydrogen and nitrogen in the ratio of  $3\text{H}_2$  to  $\text{N}_2$  in amounts of several hundred cubic feet per day. This gas should be free from impurities, especially carbon monoxide and oxygen. Anhydrous ammonia being easily obtained, it was decided to build an electric furnace to decompose it and use the decomposed gases as the required mixture of nitrogen and hydrogen.

## HISTORICAL

Investigations on the decomposition of ammonia were made in 1884 by Ramsay and Young<sup>2</sup> at temperatures of  $500^\circ\text{C}$ . and  $830^\circ\text{C}$ . They found that decomposition began under most favorable circumstances slightly below  $500^\circ\text{C}$ . Though decomposition occurred below  $500^\circ\text{C}$ . the rate of reaction was too slow to observe in the time of the test. The tests were made in tubes of porcelain and iron with pieces of broken porcelain as catalyst. Decomposition was found to be one and a half to two times greater in an iron tube than in a porcelain one.



The curve in Fig. 1 shows the quantities of ammonia found in the iron tube starting with pure ammonia. Decomposition was complete only at about  $780^\circ\text{C}$ . Haber<sup>3</sup> found that the velocity of reaction when  $t = 700^\circ\text{C}$ . was still so small that equilibrium could not be established with certainty. Plank<sup>4</sup> discusses the theoretical values at one to 10 atmosphere pressures. These values are shown in the curves of Fig. 2. These theoretical equilibria would exist only after months or years as the velocity of reaction is very slow.

Perman and Atkinson<sup>5</sup> made tests between  $t = 677^\circ\text{C}$ . and  $1111^\circ\text{C}$ . with a reaction vessel of porcelain.

<sup>1</sup> Published by permission of Chief of Ordnance.

<sup>2</sup> *J. Chem. Soc.*, 45 (1884), 88.

<sup>3</sup> *Z. Electrochem.*, 40, 2144.

<sup>4</sup> *Z. ges. Kalt-Industrie*, March and May, 1915, p. 8; reviewed in *Am. Soc. Refrig. Eng. J.*, 2 (1915), 34.

<sup>5</sup> *Proc. Roy. Soc. London*, 74 (1905), 110.

Mercury, platinum, and iron as catalysts were all found to accelerate the reaction.

Bodenstein and Kranendiech<sup>1</sup> operated between  $790^\circ\text{C}$ . and  $880^\circ\text{C}$ . using quartz glass for catalyzer and reaction vessel. Their results verify those of other investigations. From their experiments it is shown clearly that iron acts as a catalyzer toward this reaction in both directions.

## DESCRIPTION OF FURNACE

The furnace devised by us for carrying out this reaction is shown in Fig. 3.

A diagrammatic sketch of the furnace shows the outer tube *a* to be a piece of 5-in. standard wrought iron pipe 4 ft. long closed at the ends with flanges *c*, and the upper 34 in. wound with nichrome ribbon, *k*. Inside this pipe is a piece, *b*, of 2.5-in. standard iron pipe, 35 in. long, wound with nichrome wire, *k*. A pyrometer tube, *n*, extends 14 in. into the upper end of this inner pipe and the vacant space *f* (about  $\frac{1}{4}$  cu. ft.) is filled with the catalyst (in this case iron turnings or steel wool). Automobile spark plugs were adapted to carry the leads through the flanges. The lower portion is the heat interchanger *g* which actually consists of three coils of  $\frac{1}{8}$  in. pipe connected in parallel. The anhydrous ammonia enters the interchanger through the lower flange, passes upward over the interchanger coils, through the catalyst between the heaters, then passes down through the catalyst inside the inner pipe, through the interchanger coils, and emerges as a mixture of  $3\text{H}_2$ ,  $\text{N}_2$ , and  $\text{NH}_3$ , the percentage of  $\text{NH}_3$  depending upon the temperature and rate of flow. The gases are passed through a water scrubbing tower to remove the remaining ammonia and then through a meter to the gas holder. The ammonia remaining in the scrubbed gases rarely exceeded 0.2 per cent and quite often is nil.

It will be seen that the inner heater is very favorably situated since it operates in a reducing atmosphere. It consumes about 65 per cent and the outer coil about 35 per cent of the total energy. The source of current is a 25 cycle, 40 volt transformer. It was originally intended to use copper turnings as a catalyst and the furnace was wound inductively. With iron as catalyst the power factor is reduced to 92 per cent. The coils are connected so that their magnetic fluxes are opposed. The maximum power consumption at 40 volts is 2 k. v. a. which is inadequate to maintain the furnace at the proper temperature for a flow of ammonia as great as the catalyst can decompose. In a new furnace of similar design, the length is increased one foot and the diameter one inch. The inner coil is wound non-inductively on 3.5-in. cold drawn boiler tube and has a carrying capacity of 4 k. v. a. at 40 volts. The outer coil is also wound non-inductively with a maximum capacity of 3 k. v. a. The voltage is adjusted so that the inner coil will dissipate about 80 per cent of the energy. The capacity of the heat interchanger

<sup>1</sup> *Festschrift, W. Nernst* (1912), pub. by Knapp, Halle; *Chem. Abs.*, 7 (1913), 1647.



is considerably increased, and the inlet and outlet gases travel through about 10 ft. of double pipe in counter-current flow. Fine steel wool is used for catalyst.

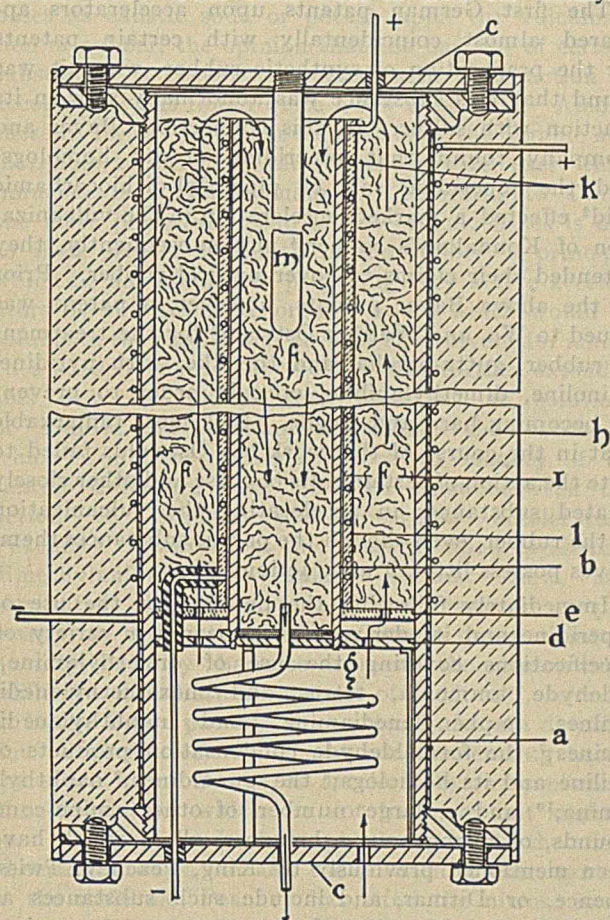


FIG. 3—FURNACE FOR THE DECOMPOSITION OF AMMONIA

- a—5-in. iron pipe  
 b—2.5-in. iron pipe  
 c—flange ends  
 d—flange end to inner pipe  
 e—perforated asbestos board  
 f—catalyst space  
 g—heat interchange coil  
 h—insulating material  
 i—alundum sheath  
 k—resistance coil of chromel wire  
 l—alundum cement  
 m—pyrometer well

The weight of the furnace is partially supported from above to prevent deformation and the sides and top are covered with 7 in. of infusorial earth.

#### RESULTS

Table I shows some results obtained in the furnace using iron shavings as catalyst. The flow is measured by the decomposed gas free from  $\text{NH}_3$  which passes through the meter.

TABLE I—DECOMPOSITION OF AMMONIA  
 (Catalyst: Iron. Space: 0.22 Cu. Ft.)

Temp. ° C.	Flow Cu. Ft. per Min.	Decomposition Per cent
370	0.01	73.0
470	0.15	84.0
540	0.01	94.0
600	0.20	97.0
600	2.00	94.5
610	0.50	93.5
650	0.62	99.6
650	1.50	97.4
675	0.50	99.7
675	1.00	99.5
700	1.50	99.7
710	2.00	99.6
735	0.82	99.7

For ordinary operation a temperature of 675° C. is maintained which enables one to obtain about 100 cu. ft. per hr. of the hydrogen-nitrogen gas mixture with a power consumption of 2 k. v. a. with loss

of 0.3 to 0.4 per cent of ammonia. The equilibrium of a system of  $\text{H}_2\text{-N}_2\text{-NH}_3$  at that temperature and atmospheric pressure theoretically should be about 0.02 per cent  $\text{NH}_3$ . The average time of contact in the catalyst space is about 5 sec. This time could probably be reduced considerably.

A small preliminary furnace in which copper chips and turnings were used for catalyst was employed before this one was constructed. Table II shows results obtained with this.

TABLE II—DECOMPOSITION OF AMMONIA  
 (Catalyst: Copper. Space: 0.11 Cu. Ft.)

Temp. ° C.	Flow Cu. Ft. per Min.	Decomposition Per cent
750	0.01	96.0
750	0.25	75.0
800	0.05	97.3
850	0.01	99.7
850	0.22	99.4
850	0.95	90.5
900	0.85	96.2
925	0.50	99.3
925	1.00	97.5

It will be seen that copper requires a temperature about 200° C. higher than iron, and a longer contact. The figures are not strictly comparable for the iron surface was probably of the order of ten times that of the copper. Beilby and Henderson<sup>1</sup> emphasize the fact that ammonia decomposes less rapidly in contact with copper than with iron.

Because of the lower operating temperature and cheapness, iron was chosen in preference to copper as a catalyst.

The furnace with steel shavings was in operation for about 6 mo. when the outer coil burned out. On taking apart the iron, shavings were found to have sintered into a compact mass and to be attached firmly to the sides of the iron pipe. The pipe itself, which had operated continuously in an atmosphere of hydrogen and nitrogen, showed attack from within to such a degree that it was granular and cracked about two-thirds the distance through the wall (0.25 in. thick) and crystallized the other third. The metal was very brittle and the pipe was readily broken up with a hammer. Beilby and Henderson<sup>2</sup> and Henderson and Galletly<sup>3</sup> found a similar deterioration of metals exposed to ammonia at high temperatures. Probably this was due to the formation of iron nitride or hydride, or both. The nichrome wire used as resistance winding seemed to be in as good condition as when first wound. The maximum temperature to which the furnace had been subjected was not over 900° C., and this temperature was reached inadvertently and maintained for a very short time.

#### THE ACTION OF CERTAIN ORGANIC ACCELERATORS IN THE VULCANIZATION OF RUBBER<sup>4</sup>

By G. D. Kratz, A. H. Flower and Cole Coolidge

RESEARCH LABORATORIES, THE FALLS RUBBER COMPANY, CUYAHOGA  
 FALLS, OHIO

Since the introduction of the use of organic substances to assist in, or to accelerate, the vulcanization

<sup>1</sup> *J. Chem. Soc.*, 79 (1901), 1245.

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

<sup>3</sup> *J. Soc. Chem. Ind.*, 27 (1908), 387.

<sup>4</sup> Presented before the Rubber Division at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 2 to 6, 1919.



of rubber, there has been remarkably little added to the literature upon the subject. Furthermore, what has been written has been confined largely to reviews of a general nature. Although King,<sup>1</sup> Peachey,<sup>2</sup> Twiss,<sup>3</sup> Spence,<sup>4</sup> and others<sup>5</sup> have contributed to this subject, the majority of investigators have remained reticent, despite the fact that the patent files are replete with information.

The present paper records a portion of the results procured in an attempt to obtain further information in regard to the action of certain organic substances in accelerating the vulcanization reaction. Throughout this work we have chosen to employ only substances which have previously been cited by others and whose definite chemical composition permits comparison with homologs or other closely related compounds. The activities of the substances from a practical standpoint were considered of secondary importance. Further, while we most assuredly recognize that the vulcanization reaction is, essentially, a catalytic one, we have chosen to employ the term "accelerator" rather than "catalyst," when referring to the organic substances in question. This distinction is deemed necessary to avoid misunderstanding, and to discriminate between catalysis as it is generally understood and as it must be applied to the vulcanization of rubber. In this regard, it is necessary to take into consideration the fact that a large number of the organic substances used to accelerate the vulcanization of rubber decompose below the temperatures at which vulcanization is ordinarily effected, and that many of them show a tendency to combine with the sulfur under vulcanizing conditions. Again, as most of the rubber mixtures in actual use contain mineral substances, the possibility of a reaction of these substances with the organic accelerators involving the alteration or decomposition of the latter, is far from remote. In fact, even before the general use of synthetic organic substances as accelerators, Esch<sup>6</sup> found it necessary to employ calcium and magnesium oxides with certain albuminous bodies in order to obtain the desired results. Likewise, Peachey<sup>7</sup> has noted a marked increase in the activity of *p*-nitrosodimethylaniline when used in conjunction with magnesia. Litharge was found to diminish the effectiveness of this accelerator.

Although we are confident that the use of organic substances as accelerators was practiced in the factories of this country long before the first patent on the subject was taken out abroad, the predominance of the early patents was undoubtedly German. In fact, the latter people, with their thoroughness and the realization of the possibilities in the subject, attempted to restrict the use of all nitrogenous substances by the use of such generalities as are found in one of Bayer

and Company's patents.<sup>1</sup> That few, if any, applications for license to use these patents were made during the war is an indication of our priority in this field.

The first German patents upon accelerators appeared almost coincidentally with certain patents for the preparation of synthetic rubber, when it was found that this substance was abnormally slow in its reaction with sulfur. In this connection, Bayer and Company found that piperidine or its homologs<sup>2</sup> and the piperidine salt of piperidyl-dithiocarbamic acid<sup>3</sup> effected a marked acceleration in the vulcanization of Kondakow's rubber,<sup>4</sup> and, subsequently, they extended their claims to cover natural rubber. Prior to the above Bayer patents, however, a patent was issued to Wo. and Walther Ostwald<sup>5</sup> for the treatment of rubber, gutta-percha, and the like, with pyridine, quinoline, dimethylaniline, etc., in order to prevent its becoming hard and fragile. It is most remarkable that in the course of this work the Ostwalds failed to note the action of certain salts of these and other closely related substances in accelerating the vulcanization of the rubber, even though the parent substances themselves possess little, if any, action.

Immediately following the patents for the use of piperidine and its derivatives appeared a variety of specifications covering the use of dimethylamine,<sup>6</sup> aldehyde ammonia, tetra- and hexamethylenediamines, *p*-phenylenediamine, and naphthylenediamines;<sup>7</sup> the formaldehyde condensation products of aniline and its homologs;<sup>8</sup> the guanidines;<sup>9</sup> naphthylamine;<sup>10</sup> and a large number of other amino-compounds, or nitrogenous substances, all of which have been mentioned previously by King, Peachey, Twiss, Spence, or Ditmar, and include such substances as quinoline and its salts, the quaternary ammonium bases, urea and thiourea derivatives, and a host of others. In their patents, Bayer and Company cover the urea and thiourea derivatives of saturated aliphatic amines, both open and closed carbon chains, which are saturated methylene open-carbon chain bases. The term "methylene base" is held to be generic to those bases which are saturated bases as distinguished from unsaturated bases as aniline. Suffice to say, that both aniline and diphenylthiourea were employed in this country long before the appearance of any of the above specifications.<sup>11</sup>

Although not specified in some of the foregoing patents, many of these were drawn upon the assumption that the activities of the various substances depended

<sup>1</sup> D. R. P. 280,198 (1914).

<sup>2</sup> D. R. P. 255,680, 266,618 (1912); 268,387, 265,221 (1912).

<sup>3</sup> D. R. P. 266,619 (1912); U. S. Patent 1,126,469 (1915).

<sup>4</sup> *J. prakt. Chem.*, **64** (1901), 109.

<sup>5</sup> D. R. P. 221,310 (1908).

<sup>6</sup> D. R. P. 269,512; French Patent 464,533.

<sup>7</sup> D. R. P. 280,198 (1914); U. S. Patent 1,149,580 (1915).

<sup>8</sup> British Patent 7,370 (1914).

<sup>9</sup> British Patent 17,760 (1916).

<sup>10</sup> U. S. Patent 1,296,469 (1919).

<sup>11</sup> In this connection it is interesting to note that in this country attention was directed to the use of urea as an accelerator before the appearance of these or any other patents upon urea or the thioureas. This is said to have been the result of observations made upon certain African rubbers, reputed to have been coagulated with human urine, and which were found to vulcanize with more than average rapidity.

<sup>1</sup> *Met. & Chem. Eng.*, **15** (1916), 231; **18** (1918), 246.

<sup>2</sup> *India Rubber J.*, **52** (1916), 603; *J. Soc. Chem. Ind.*, **36** (1917), 321; **36** (1917), 424, 950.

<sup>3</sup> *J. Soc. Chem. Ind.*, **36** (1917), 782.

<sup>4</sup> *THIS JOURNAL*, **10** (1918), 115.

<sup>5</sup> *Gummi Ztg.*, **29** (1915), 425; **30** (1916), 303; *India Rubber World*, **55** (1917), 190; *Caoutchouc et gutta-percha*, **15** (1918), 9637; *Rubber Age*, **3** (1918), 78.

<sup>6</sup> D. R. P. 273,482 (1912).

<sup>7</sup> *J. Soc. Chem. Ind.*, **36** (1917), 424.



largely upon their nitrogenous nature and the basicity of the substances in question.

In view of the well-known effect of such inorganic bases as caustic soda or potash upon the vulcanization of rubber, the opinion that the basicity of certain substances is closely connected with their action as accelerators has found quite a few adherents. Among these is Twiss,<sup>1</sup> who believes that both the organic and inorganic accelerators, all of basic nature, exert their influence in a similar manner. According to Twiss, the more powerful action of the organic bases is due to their greater solubility in the rubber, a principle which he has applied in preparing very active accelerators by the use of an organic liquid which would be soluble in the rubber and also act as a solvent for alkali. In a subsequent patent<sup>2</sup> he has still further extended this view to include such materials as solutions of sodium and potassium in aniline, the action of which would be difficult indeed to classify if essential differences are held to exist between the action of organic and inorganic accelerators. Peachey,<sup>3</sup> however, in his patent upon *p*-nitrosodimethylaniline, has taken another view, and attributes the activity of this substance, primarily, to the presence of the nitroso group. Subsequently, Peachey<sup>4</sup> has also expressed the opinion that, while the dissociation constant of  $1 \times 10^{-8}$ , referred to in the Bayer patents, does actually form a dividing line between accelerating and non-accelerating bases, there are other classes of accelerators (as *p*-nitrosodimethylaniline) which are not basic, or but slightly so. Further, in order to emphasize not only the effect of certain groups, but also the position of these groups in the molecule, Peachey has cited the nitroso bases as powerful accelerators, and pointed out that the isomeric nitrosamines, in which the nitroso group is not in direct combination with the nucleus, are inert.

Likewise, Spence has mentioned that the activity of certain substances which contain groups in the para position are more active than the isomeric substances, in which the groups are in the ortho, or meta position, and has cited the relative activities of *p*-phenetidine and *o*-phenetidine as an example. He has also called attention to the increase effected in the activity of substances as accelerators by the introduction of other groups into the nucleus. As a case in point, he has made reference to the increase in the activity of diphenylthiourea when it is converted into tetramethyldiaminodiphenylthiourea. While there is no doubt that these differences exist as expressed by Peachey and Spence, it is to be regretted that no figures were given by either of them by which the relative activities of the various substances might be recorded.

Our own experience, a portion of which is embodied in the experimental part of this paper, in the main confirms the results of Peachey and Spence; and in fact, in many ways, permits of their amplifications.

<sup>1</sup> *J. Soc. Chem. Ind.*, **36** (1917), 782; British Patent 110,059 (1916).

<sup>2</sup> British Patent 124,276 (1919).

<sup>3</sup> British Patent 4,263 (1914).

<sup>4</sup> *J. Soc. Chem. Ind.*, **36** (1917), 950.

Not only have we found certain groups, and the position of these groups in the primary nucleus, to have a marked effect upon the activity of the substance under investigation, but we have found that if the effect produced by one or more groups is to be considered on a strictly comparable basis, the size (molecular weight) of the parent substance must also be taken into account. On the other hand, we are equally well convinced that with many substances it is possible to place too great stress upon the presence of certain groups and their position in the nucleus. It would appear that the effect produced by the different groups is largely determined by their behavior and ultimate condition as the original substance undergoes decomposition or chemical change, and that the activity of substances not originally basic is due to their conversion into basic substances during vulcanization.

For example, in our preliminary experiments we found that urea was slightly more active than thiourea. Thiourea, in turn, was more active than monophenylthiourea, and the latter more active than diphenylthiourea. The same was true in the case of the guanidines, both monophenylguanidine and diphenylguanidine being found more active than triphenylguanidine, although the conditions of our experiment did not permit of a differentiation between the two substances first mentioned. In the case of the thiourea, the natural inferences would be that the activity of the parent substance depended largely upon the presence of the NH<sub>2</sub> groups, with a consequent diminution in the effect produced as phenyl replaces hydrogen. In the case of the guanidines, the great difference in activities of mono- and diphenylguanidines, when compared with that of triphenylguanidine, may be attributed to the fact that in the latter substance the phenyl group has replaced imido rather than amido hydrogen.

Again, when we made a comparison between a cyclic monamine and diamine, we found the latter, in accordance with the now general understanding, to be much more active than the former, if the groups are in the para position. On the formation of the anhydro-formaldehyde condensation products of these substances, however, although the activities of each were increased by the substitution of methylene groups for the hydrogen of the amino groups in the original substances, this increase in the case of the monamine was much less than that produced by the introduction of a second amino group into the original nucleus. Although the two types of compounds are not isomeric, this is in line with Peachey's statement as to the effect produced when groups are introduced into a side-chain rather than the original nucleus.

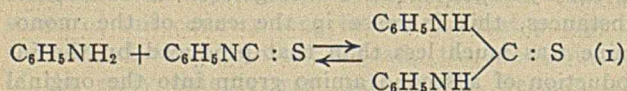
In our subsequent and more extensive experiments, wherein we employed aniline as a basis of comparison, we found that the replacement of the amino group in aniline by hydroxyl actually decreased the rate of vulcanization to less than that of the control mixture which contained no accelerator. The introduction of a second hydroxyl group in the para position was found to cause a still greater retardation in the rate of re-



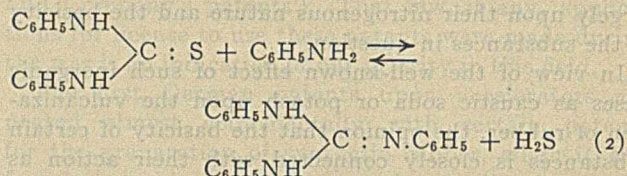
action. In fact, when allowance was made for the difference in the molecular weights of the two substances, the anti-accelerating effect of hydroquinone was found to be just twice that of phenol. The difference between the effect of one amino group and two amino groups in the para position has already been mentioned, and here again, after having corrected for the molecular weights of the two substances, *p*-phenylenediamine was found to be almost exactly twice as active as aniline. When we employed *p*-amidophenol, however, in order to obtain an amino group and an hydroxyl group in the para position to each other, our results were not strictly in accordance with those of Peachey and Spence, who found that this substance was without action or but mildly active. On the contrary, we found *p*-amidophenol to be more active than aniline. The inference from our results would be that although the effect of the (OH) group is that of a retardant, its position in the nucleus, and its subsequent influence upon the decomposition of the nucleus during vulcanization, are of less importance than the effect produced by the stronger and positive (NH<sub>2</sub>) group.

It is not our purpose, however, to attribute the action of certain substances as accelerators entirely to the inclusion and position of certain groups in the primary nucleus. Rather, as has been said, we have been led to the conclusion that the great majority of organic accelerators owe their activity to their decomposition products, when this decomposition takes place in the presence of sulfur and under vulcanizing conditions. This corresponds very closely with Spence's third class of accelerators, which he believes may be inactive in themselves, but which, at the temperature of vulcanization, either alone or in the presence of other substances, may break down into a substance, or substances, having the properties of powerful catalysts. He has mentioned diphenylthiourea as an example of this type.

In the final experiment recorded in this paper, we have worked along these lines with aniline as a standard and have compared its activity with that of other substances into which it can be converted, or from which it can be derived by decomposition under vulcanizing conditions. The following reaction suffices to explain our method:



That such a reaction, from right to left, actually takes place was indicated when we found that a mixture vulcanized with the assistance of a certain definite amount of diphenylthiourea had the same sulfur coefficient as that of a similar mixture, vulcanized under the same conditions, to which had been added aniline and phenyl mustard oil, in accordance with the amounts required by Equation 1. Similar sulfur coefficients also resulted for two mixtures, one of which was vulcanized with the assistance of triphenylguanidine and the other with quantities of diphenylthiourea and aniline, in accordance with Equation 2.



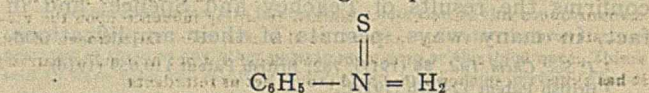
The above reaction, likewise, takes place from right to left. The diphenylthiourea and aniline formed, however, evidently undergo further decomposition with the formation of two molecules of aniline and one molecule of phenyl mustard oil.

Our interpretation of these results led us to the conclusion that both diphenylthiourea and triphenylguanidine owe their powers of acceleration to their tendency to decompose with the formation of aniline. On the other hand, and in view of our results as a whole and particularly those obtained with urea and thio-urea, we certainly do not feel warranted in ascribing to aniline alone those properties which Spence has termed the "active principle." Rather, we are inclined to regard aniline as one of the simpler substances that contains a group which, under vulcanizing conditions and temperatures, functions as a sulfur carrier.

To elaborate upon the action of accelerators beyond this point, however, is hardly warranted from the results obtained. Moreover, further discussion must, necessarily, involve the theory of vulcanization in its entirety, which is not our intention in this communication.

We have shown that under vulcanizing conditions and temperatures, certain accelerators decompose or suffer degradation to simpler substances which are responsible for the acceleration effected. The decomposition of diphenylthiourea to aniline is an example. Such being the case, similar decompositions would appear possible with other substances. This decomposition having been effected, the action of the decomposition or alteration products is most conveniently interpreted on the basis of a change in the valency of the nitrogen in active groups from 3 to 5.

It is not to be understood, however, that we believe aniline as such is, necessarily and quantitatively, formed by the decomposition of diphenylthiourea under vulcanizing conditions. Further, the various complexes which we have postulated for aniline, *p*-phenylenediamine, etc., serve to account only for the relationship noted between the activity of the substance in question and its nitrogen content. Theoretical deductions of a general nature are not at present possible, as results obtained by Erdmann<sup>1</sup> and Ostromyslenskii<sup>2</sup> indicate that the active sulfur may take the form of a polysulfide and may even not be attached to nitrogen. However, for the purpose of interpretation, rather than deduction, and with aniline as an example, under the conditions and temperatures at which vulcanization takes place, we postulate the existence of the following complex:



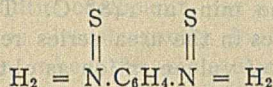
<sup>1</sup> *Ann.*, **362** (1908), 133.

<sup>2</sup> *A. H. King, Met. & Chem. Eng.*, **18** (1918), 245.

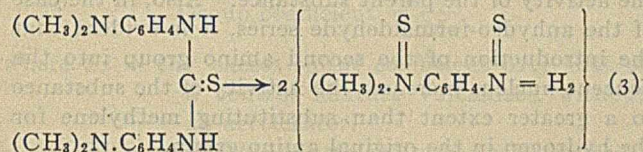


As this complex would not be stable, except under the conditions and temperatures at which vulcanization is effected, it would function as a catalytic sulfur carrier, releasing its sulfur in an active form, which would combine with the rubber more readily than the sulfur originally included in the mixture. Its catalytic action would depend upon its ability to maintain an equilibrium between the active and inactive sulfur.

The above is in strict accord with our results for the relative activity of aniline and *p*-phenylenediamine. The hypothetical complex for the latter substance would include 2 pentavalent nitrogen atoms, or active groups, as below:



Thus, when the effect of molecularly equivalent quantities of aniline and *p*-phenylenediamine are compared, the latter should be just twice as active as the former. This was actually found to be the case. From this it is also possible to explain why Spence found tetramethyldiaminodiphenylthiourea so much more active than diphenylthiourea; the former substance would probably decompose into 2 molecules of dimethyl-*p*-phenylenediamine with just twice the number of active groups found in the latter. Further, we venture to predict that the activity of one molecular part of tetramethyldiaminodiphenylthiourea is exactly equal to that of 2 equimolecular parts of dimethyl-*p*-phenylenediamine,<sup>1</sup> and less than that of 2 equimolecular parts of *p*-phenylenediamine. The difference in the basicities of the last two substances accounts for their relative activities



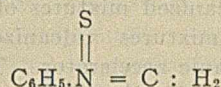
The latter observation is made in the light of our results with the ureas and guanidines. While our results with these substances are subject to the same general interpretation as the preceding, we found with them that the replacement of the hydrogen by phenyl in the active groups decreased the activity of the substance in question. It is possible that the substitution of larger groups for the hydrogen attached to the active nitrogen would cause the group to become inactive, due to steric hindrance precluding the formation of pentavalent nitrogen. Peachey's observation of the inactivity of the nitrosamine isomeric with *p*-nitrosodimethylaniline may be subject to this interpretation.

In view of this, our results with the anhydro-formaldehyde condensation products of aromatic

<sup>1</sup> It is interesting to note that Eaton, Grantham and Day (Dept. of Agr. Fed. Malay States, *Bulletin* 27 (1918), 319) found dimethyl-*p*-phenylenediamine hydrochloride to exert a marked retarding influence upon the vulcanization of a fast curing rubber. In this case, then, the nitrogen of the amino group is already pentavalent, and could not act as a sulfur carrier; it has already been shown that acid groups act as retardants.

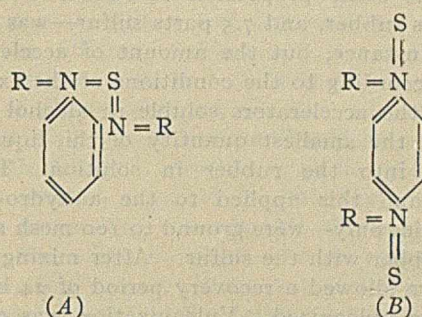
In this connection, it should also be pointed out that although dimethyl-*p*-phenylenediamine is more active than aniline, dimethylaniline is practically inert.

amines are more difficult to interpret, as the substitution of methylene for hydrogen increased the activity. In this instance, however, there will be two double bonds when nitrogen becomes pentavalent:



A complex of this type would be even more unstable, and more active, than that postulated for aniline.<sup>1</sup>

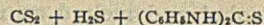
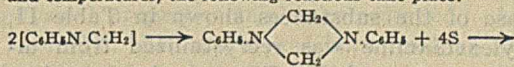
Lastly, in light of the readiness with which sulfur passes from the di- to the tetra- or hexavalent state, it is possible to adapt the former principles to account for the difference in the relative activities of the *o*- and *p*- products of the same substance. The proximity of two groups in the *o*-position might lead to a complex of type A, whereas, if the groups were in the *p*-position, the configuration would be that shown in type B.



In this case, then, the *p*- would be twice as active as the *o*-product, a condition with which we are not as yet familiar.

In the foregoing we have not taken into consideration the effect of the accelerators upon the physical properties of the mixtures investigated, and have confined our remarks to interpretations made on the basis of excess sulfur coefficients of vulcanization only. However, in our experiments with mixtures vulcanized with the assistance of aniline, diphenylthiourea and triphenylguanidine, we were able in no instance to recover the original amount of the accelerator in the acetone extract. While this does not permit us to state definitely that a portion of the accelerator or its decomposition products, as well as the sulfur, becomes closely bound to the rubber molecule, it is not impossible that such is the case. We agree with King, that the remarkable improvement imparted to mixtures vulcanized with the assistance of certain organic accelerators indicates that the binding between the

<sup>1</sup> In the paper by C. W. Bedford and W. Scott, read immediately after the above, the following explanation was offered for the behavior of this substance. According to the above authors, under vulcanizing conditions and temperatures, the following reactions take place:



We have already shown that diphenylthiourea probably decomposes into aniline and phenyl mustard oil. Further, we have shown that when compared on equal weights the activities of anhydro-formaldehyde aniline and diphenylthiourea are approximately the same. As the molecular weight of diphenylthiourea is over twice that of anhydro-formaldehyde aniline, and as two molecules of the latter and one molecule of the former would each decompose into one molecule of aniline, a comparison with equal weight of the two substances should show anhydro-formaldehyde aniline to be slightly more active than diphenylthiourea. This is in strict accordance with the above reactions and our findings.



"active principle," or active group, of certain of the organic accelerators and the rubber is a close one.

In the course of another investigation shortly to be published, we have shown that fundamental differences exist between vulcanized mixtures of rubber and sulfur, and similar mixtures vulcanized with the assistance of an organic accelerator. This is subject to still further modification if either type of mixture contains a mineral oxide.

#### EXPERIMENTAL PART

The same general method of procedure was pursued in this work as in that previously reported.

The rubber used was good quality, first latex, pale crêpe, and the same lot was employed in all mixtures. All mixtures were made under standard conditions; the average time of each batch on the mill was 17.5 min. The same proportion of rubber and sulfur—92.5 parts rubber, and 7.5 parts sulfur—was employed in each instance, but the amount of accelerator was varied, according to the conditions of the experiment.

All of the accelerators soluble in alcohol were dissolved in the smallest quantity of this liquid and introduced into the rubber in solution. Those not soluble—and this applied to the anhydro-formaldehyde bodies only—were ground to 100 mesh and added to the rubber with the sulfur. After mixing, the mixtures were allowed a recovery period of 24 hrs. before they were vulcanized. Vulcanization was carried on in a platen press of the usual type.

The vulcanized sample for analysis was then sheeted thin on the mill, subjected to 24 hrs. continuous extraction with acetone in the extractor recommended by the Joint Rubber Insulation Committee,<sup>1</sup> dried in air and *in vacuo* at 100° C. to constant weight. The free, or extractable, sulfur found in the acetone extract was estimated by the bromine oxidation method of the Bureau of Standards.<sup>2</sup> The combined sulfur in the extracted rubber was estimated by a method of our own, to be reported upon in the near future.<sup>3</sup> All figures given for per cent sulfur are the average of at least 2 determinations which checked to within 0.1 per cent.

**PRELIMINARY EXPERIMENTS**—With the exception of aniline, which was purified by distillation and boiled at 185° C., all of the accelerators shown in Table I were recrystallized from alcohol. The purified products were found to have the following melting points: urea, 132° C.; thiourea, 168° C.; monophenylthiourea, 153° C.; diphenylthiourea, 151° C.; monophenylguanidine, 193° C.; *sym*-diphenylguanidine, 147° C.; triphenylguanidine, 143° C.

In the case of the substances shown in Table II, the *p*-phenylenediamine was recrystallized from alcohol and melted at 146° C. The anhydro-formaldehyde bodies possessed the following melting points: anhydro-formaldehyde aniline, 140° C. (with decomposition); monoanhydro-formaldehyde *p*-phenylene-

diamine, 142° C.; dianhydro-formaldehyde *p*-phenylenediamine, 192° C.

TABLE I—RELATIVE ACTIVITIES—THIOUREA SERIES  
Parts Required to Equal One Part Aniline

Aniline.....	1.000
Urea.....	0.250
Thiourea.....	0.300
Monophenylthiourea.....	0.450
Diphenylthiourea.....	0.850
Monophenylguanidine ( $\alpha$ ).....	0.075
Diphenylguanidine ( <i>Sym</i> ).....	0.075
Triphenylguanidine.....	0.500

The activities of the various substances were compared in the mixture previously mentioned—92.5 parts of rubber and 7.5 parts of sulfur—taking as a standard<sup>1</sup> the effect obtained with one part of aniline, vulcanized for 90 min. at 148° C. The amounts of various substances in the urea series required to effect the same degree of vulcanization as obtained with one part of aniline are shown in Table I. The same is true for the anhydro-formaldehyde aniline series, the results of which are shown in Table II.

TABLE II—RELATIVE ACTIVITIES—ANHYDRO-FORMALDEHYDE SERIES  
Parts Required to Equal One Part Aniline

Aniline.....	1.00
Anhydro-formaldehyde aniline.....	0.75
<i>p</i> -Phenylenediamine.....	0.17
Monoanhydro-formaldehyde <i>p</i> -phenylenediamine.....	0.14
Dianhydro-formaldehyde <i>p</i> -phenylenediamine.....	0.14

As these preliminary tests were conducted solely to obtain information for guidance in subsequent experiments, and as no sulfur estimations were made, we do not feel warranted in drawing extensive conclusions from them. It would appear, however, that in the case of the urea series, the introduction of phenyl as a substituent for the hydrogen of the amido or imido group is responsible for a marked decrease in the activity of the parent substance. Also, in the case of the anhydro-formaldehyde series, it is evident that the introduction of the second amino group into the benzene nucleus increases the activity of the substance to a greater extent than substituting methylene for the hydrogen in the original amino group.

**FINAL EXPERIMENTS**—(I) This experiment was carried out in order to determine the relative accelerating effects produced by certain substances which contained hydroxyl or amino groups in their primary nucleus. Determinations were also made of the effect produced when the number, or kinds, of groups in the nucleus were altered or changed.

Every effort was made to obtain the various substances in the greatest degree of purity. Their physical constants were as follows: phenol, m. p. 41° C.; *p*-amidophenol, sublimed at about 180° C.; aniline, b. p. 185° C.; hydroquinone, m. p. 168° C.; *p*-phenylenediamine, m. p. 146° C.

The relative activity of these substances, as expressed by their sulfur coefficients, is shown in Table III.

<sup>1</sup> The comparisons were made at what was judged to be in each case the correct cure, as measured by the physical properties of the various mixtures only. Owing to this fact, the values assigned in Tables I and II were not found to be in strict agreement with their respective sulfur coefficients as determined in the subsequent experiments. Such comparisons, based on physical properties alone, are apt to be misleading, due to the toughening action imparted to the rubber when certain accelerators are employed; this effect is very noticeable in the case of *p*-phenylenediamine.

<sup>1</sup> THIS JOURNAL, 6 (1914), 514.

<sup>2</sup> Bureau of Standards, *Circular* 38 (1915), 66.

<sup>3</sup> This method was evolved and applied in the course of the present work, in order to overcome certain disadvantages in the Rosenstein-Davies method (*Chemist-Analyst*, 15 (1915), 4) previously employed.



TABLE III—RELATIVE ACCELERATION

First Latex Pale Crêpe..... 92.5 parts  
 Sulfur..... 7.5 parts  
 Accelerator..... 1 part  
 Vulcanized for 90 min. at 148° C.

ADDED SUBSTANCE	Combined Sulfur Per cent	Sulfur Coefficient	Excess Sulfur Coefficient	Group Coefficient
Control.....	1.792	1.937	.....	.....
Phenol.....	1.629	1.779	-0.158	-0.00168
Hydroquinone.....	1.430	1.561	-0.376	-0.00170
<i>p</i> -Amidophenol.....	4.772	5.210	+3.273	.....
Aniline.....	4.226	4.614	+2.677	+0.02870
<i>p</i> -Phenylenediamine <sup>1</sup> ..	7.286	7.955	+6.018	+0.02780

<sup>1</sup> As the combined sulfur obtained in this instance is so close to the total amount originally added to the mixture, this comparison was repeated with 0.25 per cent aniline and *p*-phenylenediamine. When employed in this amount, aniline gave an excess sulfur coefficient of 1.421 and *p*-phenylenediamine, 3.552. On calculation, their group coefficients were found to be 0.01530 and 0.01640, respectively.

From this table, it is seen that phenol, when expressed in terms of its sulfur coefficient, has a slight retarding influence which is almost exactly doubled in the case of hydroquinone, when the molecular weight of the latter is taken into consideration. Aniline and *p*-phenylenediamine are seen to exert a marked accelerating effect, as would be expected, and here, again, the effect produced by two amino groups is almost exactly twice that of a single group when the molecular weights of the two substances (aniline and *p*-phenylenediamine) are taken into consideration.<sup>1</sup> Thus, when equal weights of certain accelerators which contain active groups in the same primary nucleus are employed, it is possible to compare the activity of these groups *per se* by means of a group coefficient. Or, conversely, molecular quantities of substances which contain the same active group (or groups, if in equal number) in the primary nucleus should produce the same accelerating activity.

In the case of equal quantities by weight, this may be expressed as follows:

$$\text{Group coefficient} = \frac{Sx}{Mn}$$

where  $Sx$  = the excess sulfur coefficient over that of a control mixture vulcanized without an accelerator.

$M$  = the molecular weight of the accelerator.

$n$  = the number of the groups in the primary nucleus.<sup>2</sup>

When the results recorded in Table III are substituted in the above expression, the coefficient for the (OH) group is seen to be in good agreement for both phenol and hydroquinone; the same is true for the coefficients obtained for the (NH<sub>2</sub>) group in both aniline and *p*-phenylenediamine. In other words, the introduction of a second group in the para position just doubles the activity. In light of our remarks in the introductory part of this paper, in regard to the function of these groups in accelerating the reaction between rubber and sulfur, we regard this point of

<sup>1</sup> The alkaloidal substances mentioned by Spence as decidedly active are undoubtedly of a high molecular weight.

<sup>2</sup> The term "nucleus" is used as distinct from "molecule," in order to preclude the inclusion of similar groups in the side chains which, as Peachey has already noted, are not comparable in the effect produced. Likewise, as our work has been confined to two groups in the para position, further extension of this principle would require the introduction of another factor to compensate for the appearance of groups in the meta and ortho positions. In the most cases, the ortho, meta and para products are increasingly active in the order named. Such differentiations, however, may be carried on indefinitely to include naphthylamines, etc. We have found  $\beta$ -naphthylamine to be almost twice as active as  $\alpha$ -naphthylamine.

much more importance than the numerical coefficient of the group itself, although a properly arranged table of group coefficients would undoubtedly be of value in anticipating the activities of untried substances.

II—In the previous experiment we employed substances of comparatively simple composition, and have attributed their action as accelerators to the presence of certain active groups. In the introductory part of this paper, however, we have pointed out that the action of certain other accelerators depends upon their ability to decompose, under vulcanizing conditions and temperatures, into degradation products containing an active group which is responsible for the acceleration effected. The present experiment was designed to further emphasize this point.

For this purpose, diphenylthiourea was chosen as the standard accelerator, on the assumption that Equation 1 is reversible and proceeds from right to left under the conditions and temperatures at which vulcanization ordinarily takes place.

Such being the case, and in view of the probable inactivity of phenyl mustard oil, the activity exerted by one part of diphenylthiourea should be no greater than that effected by a quantity of aniline corresponding to the amount required for the above reaction, when it is proceeding towards the left. Likewise, if the accelerating effect obtained with diphenylthiourea is dependent solely upon the amount of aniline formed by the decomposition of the former substance during vulcanization, the same should be true for triphenylguanidine, in accordance with Equation 2, which should also proceed from right to left during the vulcanization.

As mentioned in the introductory part of this paper, the diphenylthiourea and aniline formed undoubtedly undergo a subsequent decomposition with the formation of 2 molecules of aniline, and 1 molecule of phenyl mustard oil.

In order to test the above, our standard mixture was vulcanized for 90 min. at 148° C., with the assistance of one part of diphenylthiourea, and compared with similar mixtures which contained molecularly equivalent amounts of either one or both of the various decomposition products indicated in the above equations. The actual amounts of the different substances employed are shown in the second column of Table IV; they showed the following physical constants: phenyl mustard oil, b. p. 220° C.; aniline, 185° C.; diphenylthiourea, m. p. 151° C.; triphenylguanidine, m. p., 143° C.

Before any sulfur determinations were made, the different slabs were submitted to thumb and tooth tests in order that a rough estimation of their relative physical properties might be recorded. As shown in Table IV, the control has been used as the standard for comparison and given the number 1. The value 2, given to diphenylthiourea, simply indicates that it was faster than 1; number 3 was considered faster than 2, and so on for the rest of the series. The differences existing between the physical properties of the various slabs, however, were in no sense proportional to the numbers given them. The odor of the cured slabs was recorded in each instance and found to substantiate



TABLE IV

First Latex Pale Crêpe..... 92.5 parts  
Sulfur..... 7.5 parts  
Accelerator..... X parts  
Vulcanized for 90 min. at 148° C.

	X = Parts Accelerator Added to the Mixture	Physical Prop- erties	ODOR OF CURED SLAB	—Acetone	Extract—	Free Sulfur Per cent	Com- bined Sulfur Per cent	Total Sulfur Per cent (By Ad- dition)
				Per cent (Uncor- rected)	Per cent (Cor- rected)			
Control.....	0.00	1	Standard	8.630	2.894	5.736	1.792	7.528
Phenyl Mustard Oil.....	0.59	—1	Phenyl mustard oil	8.443	2.729	5.714	1.726	7.440
Aniline.....	0.41	4	Decomposition products of aniline	7.065	3.137	3.928	3.625	7.553
Phenyl Mustard Oil and Aniline	$C_6H_4NCS = 0.59$ $C_6H_5NH_2 = 0.41$	3	Phenyl mustard oil and decomposition prod- ucts of aniline, the latter predominating	7.966	3.595	4.371	3.100	7.471
Diphenylthiourea.....	1.0	2	Phenyl mustard oil and decomposition prod- ucts of aniline, the latter predominating	7.429	3.119	4.310	3.078	7.388
Diphenylthiourea and Aniline....	$(C_6H_5NH)_2CS = 1.0$ $C_6H_5NH_2 = 0.41$	6	Decomposition products of aniline predom- inating	7.301	3.963	3.338	3.959	7.297
Triphenylguanidine.....	1.25	5	Strong, but indistinguishable. Phenyl must- ard oil could be detected	7.523	4.063	3.460	3.872	7.332

the preceding equations. The results obtained for the acetone extracts of the different mixtures, together with all the sulfur estimations made, are also recorded in detail in Table IV. To facilitate comparison, the combined sulfurs, expressed as sulfur coefficients, are tabulated in Table V in the order of their relative magnitude.

TABLE V

First Latex Pale Crêpe..... 92.5 parts  
Sulfur..... 7.5 parts  
Accelerator..... X parts  
Vulcanized for 90 min. at 148° C.

ACCELERATOR	Com- bined Sulfur Per cent	Sulfur Coef- ficient	Excess (+) Sulfur Coef- ficient
Phenyl Mustard Oil.....	1.726	1.877	—0.060
Control (No Accelerator).....	1.792	1.937	.....
Diphenylthiourea.....	3.078	3.361	1.424
Phenyl Mustard Oil and Aniline.....	3.100	3.385	1.448
Aniline.....	3.625	3.935	1.998
Triphenylguanidine.....	3.872	4.240	2.303
Diphenylthiourea and Aniline.....	3.959	4.340	2.403

From Table V it is seen that the sulfur coefficients obtained confirmed very closely the estimates made upon the physical properties. Further, it is shown that the activity of diphenylthiourea is almost exactly equal to that obtained with an amount of aniline corresponding to the quantity formed in its decomposition products as expressed in Equation 1. Likewise, triphenylguanidine was found to have the same activity as obtained with an equivalent amount of diphenylthiourea and aniline, as required by Equation 2, when the reaction proceeds toward the left.

It is evident that the action of both diphenylthiourea and triphenylguanidine as accelerators is due to their tendency to decompose, under vulcanizing conditions and temperatures, with aniline as one of the most probable degradation products. Moreover, it would appear that the aniline so formed is responsible for the acceleration effected, and owes its activity to the presence of an active group containing nitrogen, which functions as a sulfur carrier. That in no case were we able to recover the original amount of accelerator in the acetone extract, is an indication—and an indication only—that a portion of the active principle may also remain bound to the rubber as well as the sulfur.

Final emphasis is laid upon the fact that all of our results have been obtained with a mixture composed of rubber, sulfur, and accelerator only. The presence in the mixture of inorganic oxides has been found to have a marked influence on both the chemical and physical results obtained after vulcanization.

## CONCLUSIONS

In view of the preceding results, we have been led to the following conclusions:

1—Comparisons of organic substances as accelerators should be made with molecularly equivalent amounts of the substances in question, and should be based on the values obtained for their excess sulfur coefficients over that of a control which contains no accelerator.

2—The action of certain substances, such as diphenylthiourea, is due to their tendency to decompose under vulcanizing conditions and temperatures into simpler substances which contain an active nitrogen group which is responsible for the acceleration effected.

3—Molecularly equivalent quantities of substances which contain the same active nitrogen group in their primary nucleus effect the same accelerating activity.

4—The replacement of the hydrogen in the active nitrogen group by other and larger groups, or radicals, decreases the activity of the parent substance.

5—The activity of the nitrogen in certain groups is most readily interpreted as due to a change in valency from three to five, with the temporary addition of sulfur; the active nitrogen group would thus function as a sulfur carrier.

CARBON BLACK—ITS PROPERTIES AND USES<sup>1,2</sup>

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An investigation of the carbon black industry has been undertaken by the United States Bureau of Mines as a result of economic issues brought up during the war. In the present process of manufacture carbon black is made by burning natural gas with a supply of air insufficient for complete combustion and collecting the liberated carbon on a metal surface by actual contact of the flame on the surface. This process produces from 0.5 lb. to 1.5 lbs. of carbon black from 1000 cu. ft. of gas or 1.5 per cent to 3.5 per cent of the total carbon in the gas. The process at first sight seems most wasteful but examination of the problem shows that it is at any rate the only process in practical operation which produces carbon black

<sup>1</sup> Published by permission of the Director, U. S. Bureau of Mines.

<sup>2</sup> Presented at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 2 to 6, 1919.



suitable for the ink trade and rubber industry consumers whose combined use is over 30,000,000 lbs. annually. The whole situation appears to necessitate thorough investigation before any opinion can be given in regard to the wastefulness of the present process.

Accordingly the industry has been carefully studied as to methods of manufacture, properties, and uses of the finished product. Plants in Louisiana, Oklahoma, and West Virginia have been studied by Bureau engineers. Other processes for making carbon black have been investigated. The uses of carbon black have been studied with the idea of determining the properties of the product which users of carbon black demand and with an attempt at designating in which of these uses carbon black is essential and in which a substitute material might be employed. Microscopic investigation of a large number of blacks has been made and test methods studied with a view to finding the reason for the very different behavior exhibited by different blacks.

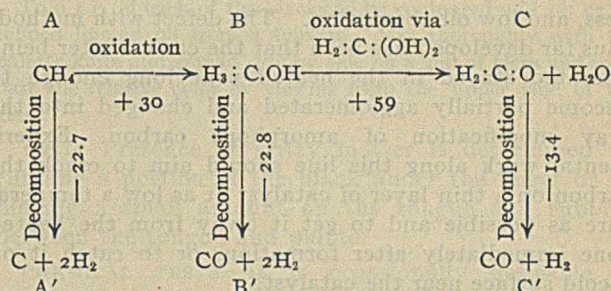
#### METHOD OF MANUFACTURE

Carbon black, as known to the American trade, is the fluffy, velvety black pigment produced by burning natural gas with a smoky flame against a metal surface. It is entirely different in physical characteristics from lampblack, which is made by burning oil or other carbonaceous material with insufficient air for complete combustion and collecting the smoke in settling chambers. Lampblack is gray in contrast to the deep black of carbon black, often contains considerable quantities of empyreumatic matter, and when used in printing ink gives a product with very different properties from an ink of similar composition made from carbon black. The process of manufacture used to the greatest extent at the present time is the so-called channel system, in which the black is deposited on the smooth under-surface of steel channels by lava-tip burners set at a distance of 3 or 4 in. below the channel. The channel irons are usually built up in tables of eight, sometimes 100 ft. long, and are given a slow reciprocating motion which scrapes the black deposited on them into hoppers from which it is carried by screw conveyors to the packing house, where it is bolted and sacked. The mechanism is enclosed in sheet-iron buildings in order that the amount of air may be regulated. Varying the amount of air, speed of scraping, and pressure of the gas controls the quality of the product. The shape of the burner and distance from the collecting surface also affects the quality of the black, but these are constant for any one plant. Other similar processes differ only in the nature of the collecting surface and burners. Godfrey L. Cabot, a pioneer in the carbon black industry, has described in a very able manner the history of the development of the manufacture of carbon black.<sup>1</sup> Mr. R. O. Neal, of the Bartlesville Station of the Bureau of Mines, is preparing a technical paper describing in detail the present method of manufacture and discussing the economic side of the industry.

<sup>1</sup> 8th Intern. Cong. Applied Chem., 12, 13.

#### THEORY OF FORMATION OF CARBON BLACK

When natural gas burns in an incomplete supply of air, the carbon is liberated, not as a result of preferential combustion of hydrogen, but as a direct decomposition of unburned gas in the heat of the flame. According to Bone<sup>1</sup> and co-workers, combustion takes place in steps as a result of hydroxylation:



It is evident that the tendency is always to run from A to C. When the proportion of methane to oxygen is  $\text{CH}_4 : \text{O}_2$  the reaction goes from A to B to C to C'. If the ratio is  $2 \text{CH}_4 : \text{O}_2$  or higher, only a part of the methane can be oxidized through the reaction A to C and part is decomposed at A by the heat evolved in the A to C reaction. The lowest per cent of oxygen in which a methane flame will burn is 15.6 per cent. Carbon will be evolved only in the inner part of the flame, where the oxygen supply is low but where there is sufficient heat to break up the methane, and the per cent of carbon to be expected by the incomplete combustion of methane is low. Gases rich in ethane and the higher homologs produce greater yields by this process.

The function of the cold surface is to cool the liberated carbon in the flame sufficiently to prevent its combustion. This permits the use of a sufficiently hot flame to give carbon uncontaminated with hydrocarbons or their partial oxidation products and produces a finely divided material which has been prevented from agglomerating by the sudden cooling. It is evident that there must be an optimum temperature and an optimum position for the surface in the flame. Too cold a surface may prevent the maximum separation of carbon; too hot a surface will allow too much carbon to be burned and may change the properties of the carbon which remains unburned. The temperature of the channels in the present processes is about  $300^\circ \text{C}$ .

Numerous methods for producing a larger yield of carbon black from natural gas have been proposed and patented. In most of these processes the gas is broken up into carbon and hydrogen by passing through a retort filled with refractory material at a temperature of  $1200^\circ \text{C}$ . or over. A much higher yield of carbon, sometimes as high as 40 per cent of the theoretical, is produced in this way, but it has not yet been made of a grade commercially salable. The carbon, while often finely divided, is usually gray in color and contains particles of grit which may be agglomerates of carbon or particles of the refractory material. It seems highly possible, however, that with properly

<sup>1</sup> Trans. Roy. Soc. London, 215 (1915), 275.



controlled conditions a carbon suitable for use in rubber might be made by this method. The hydrogen produced can be sold at a nice profit and even if the hydrogen were not used, the black could be produced for less than 5 cts. a lb. For use in ink, carbon black made by the closed retort method up to the present time has been of no value, due to inferior color, grittiness, and low oil absorption. The defect with methods thus far developed has been that the carbon after being liberated stayed in the heated zone long enough to become partially agglomerated and changed into the gray modification of amorphous carbon. Experimental work along this line should aim to crack the carbon on a thin layer of catalyst at as low a temperature as possible and to get it away from the heated zone immediately after formation, or to catch it on a cold surface near the catalyst.

Other methods have been patented, *e. g.*, chlorination of natural gas to hydrochloric acid and carbon black, explosion of acetylene, rupture of hydrocarbon vapors with electric spark, exploding hydrocarbon vapors with CO, CO<sub>2</sub> and O<sub>2</sub>, but to the authors' knowledge none of them have ever produced any quantity of a salable product in this country.

#### USES OF CARBON BLACK

In order of importance the uses of carbon black are as follows:

- 1—Printers' ink.
- 2—Automobile tires and other rubber goods.
- 3—Black and gray paint and enamel.
- 4—Stove polish.
- 5—Other products—such as phonograph records, carbon paper, crayons, typewriter ribbons, black and gray paper, glazed paper, tarpaulins, black leather, bookbinders' board, marking and stenciling inks, rubber sheeting and clothing, hard rubber, artificial stone and black tile, Chinese and India inks, fireworks, insulating materials, crucible steel, case hardening.

The amounts used by these industries in 1918 were approximately as follows:

	POUNDS
Printers' ink.....	10,000,000-12,000,000
Rubber goods.....	20,000,000
Stove polish.....	4,000,000-5,000,000
All others.....	1,000,000

Besides this, in normal times probably 15,000,000 lbs. are exported.

**PRINTERS' INK**—Lampblack has been used as a pigment for printers' ink ever since the invention of the printing press, and was used almost exclusively up to 1864. For certain sorts of printing where an extremely fine-grained ink was required, great trouble was taken to purify the black, but after the advent of carbon black in 1864, and its increased production and diminished cost after 1880, lampblack became less and less used in the ink trade, and at the present time only a small amount is used, and then only to impart certain qualities to an ink already containing carbon black.

Carbon black has certain properties which make it peculiarly suitable to the needs of the modern printing art. It is well adapted to the modern fast running presses and to the fine half-tone screen work which enters into most of our illustrations. Certain carbon blacks

give what is known as a short ink, that is, an ink of buttery consistency which does not flow rapidly. This is especially desirable in lithographic and offset work, and in slow speed presses and most half-tone work. Lampblack does not give the right consistency for such work and is too gray in color. For fast running presses, carbon black made by other processes is also good in that it makes a very fluid long ink which at the same time has sufficient opacity to give a black letter obscuring the paper well. Ink manufacturers and users believe that carbon black is absolutely essential to their business.

**RUBBER TIRES**—Prior to 1914 little carbon black was used by the rubber industry and then only in small amounts as a coloring material. Little distinction was made between carbon black and lampblack, the two compounds being used indiscriminately. At this time, due partly to the stimulus afforded by the rising price of zinc oxide, it was found that carbon black could be used in very large amounts as a filler for rubber, with a correspondingly smaller amount of zinc oxide. Carbon black is used in rubber in quantities of 3 per cent to 20 per cent by weight. Many manufacturers claim very unusual properties for rubber so compounded. It is said to increase the tensile strength very greatly and to give increased toughness and resistance to abrasion. It is believed by some authorities that the life of the rubber is increased. Other rubber chemists are more conservative in their praises of carbon black and do not admit that it possesses any properties which make it irreplaceable.

From the point of view of cost, carbon black is much cheaper than zinc oxide. Carbon black, absolute specific gravity 1.8, suitable for compounding in rubber, can be procured as low as 10 cts. a lb. at the present time. Zinc oxide costs at least as much and has a specific gravity of 5.8. Carbon black evidently costs one-third as much as zinc oxide on a volume basis, supposing that equal volumes were compounded with the rubber, but in practice a greater volume of carbon black is used than of zinc oxide so that the resulting mix with carbon black contains less rubber per unit volume than the corresponding zinc oxide mix.

From a theoretical consideration, carbon black should be an ideal filler for rubber on account of its extremely fine state of division and the correspondingly large surface energy developed when intimately mixed with the gum rubber. It also serves to protect the rubber substance from the effects of light and may possibly retard oxidation.

Whatever may be the true state of affairs as to the irreplaceability of carbon black in rubber, an enormous amount is now consumed by the rubber companies, probably 20,000,000 lbs. annually, and 10,000,000 lbs. exported for foreign trade in normal times.

**PAINT**—Carbon black is coming into extensive use in the paint trade. It has a higher tinting strength than any other black and a given weight will obscure a greater area of surface. Carbon black is acknowledged superior in varnishes and enamels and evidently has many followers in making black and gray paints for general purposes. The U. S. War Department re-



quires the use of carbon black in black enamels and various black and gray paints for general purposes. Some authorities consider lampblack superior to carbon black and it is probably true that in certain gray tints lampblack is superior, owing to its bluish gray tones.

**OTHER USES**—For other purposes carbon black is employed chiefly because of its cheapness and high coloring power. It produces a carbon paper of very smooth surface which will give a large number of copies before the letters lose their opacity. For phonograph records it gives a smooth surface for recording the vibrations, although lampblack is often used for this latter purpose.

#### TESTING METHODS

The final test of the suitability of a black for a given purpose is the actual trying out of the working qualities. In rubber making a sample mix is made and the finished piece tested for tensile strength, per cent elongation, toughness, and resistance to abrasion. In ink making a sample batch of ink is made up and the suitability of the black determined by an actual run on the press for which the ink is intended, in which the working qualities of the ink and the amount used for a given number of impressions are noted.<sup>1</sup>

There are, however, a number of laboratory tests which are of use in matching a standard sample. The tests most commonly employed are for tinting strength, color, and grit. It is also desirable to determine moisture, ash, and acetone extract.

**TINTING STRENGTH**—According to the American Society for Testing Materials, tinting strength is "the power of coloring a given quantity of paint or pigment selected as a medium or standard for estimating such power." Tinting strength, then, as applied to carbon blacks is the measure of the ability of the black to impart a color to a definite weight of standard white. It depends on the size of the particles and the specific gravity of the black. In making the test, the black is always compared with a standard black.

Weigh out accurately on a sensitive balance, 0.100 g. of the black to be tested and 10.0 g. of a standard zinc white kept especially for the purpose. Transfer to a glass or marble slab and add from a burette exactly 3.5 cc. refined linseed oil. Mix with a palette knife and rub out thoroughly with the palette knife (or, better, a glass muller) until no streakiness or difference of color is observed, when successive small portions are spread out on a clean piece of window glass and viewed from the upper side. It is important that the rubbing out be thorough; 10 min. are usually sufficient. Follow the same procedure with the standard black. Then spread a small amount of each mix side by side on a clean glass (a microscope object glass serves the purpose very nicely). Examination of the samples from the other side of the glass, particularly at the line where they overlap, will show the difference in tinting strength.

To make a quantitative estimation of the tinting strength of the sample as compared to the standard, more white is added to the stronger mix until the colors match. A new sample of the stronger black is then weighed out, using the calculated

<sup>1</sup> For a detailed discussion of printing inks see Norman Underwood and John V. Sullivan, "The Chemistry and Technology of Printing Inks," Van Nostrand Co., 1915; "The Composition, Properties, and Testing of Printing Inks," Bureau of Standards, *Circular 53*, Government Printing Office, Washington, 1915.

amount of zinc white, and the process repeated until mixes of the same color are obtained. If, for example, it was necessary to mix 15 g. of zinc white with 0.1 g. of the standard to match a mixture of 10 g. zinc white and 0.1 g. of the sample, the latter has two-thirds the strength of the standard.

**COLOR**—By this term is meant the relative blackness of the material when mixed in oil.

To 0.3 g. of each of the blacks to be compared add 1.3 cc. of refined linseed oil from a burette. Mix thoroughly with the palette knife and spread side by side on a slip of glass and compare the relative color by viewing from the upper side of the glass.

**GRIT**—Presence of gritty matter is determined by rubbing a portion of the black under the finger or by placing a small amount on the tongue and rubbing between the tongue and palate.

**CHEMICAL TESTS**—It is occasionally desirable to make a few quantitative chemical tests of carbon black. A black containing more than 0.2 per cent ash is probably adulterated with mineral black or charcoal. An acetone extract over 0.1 per cent indicates adulteration with a poorly calcined lampblack. Too great a percentage of moisture is undesirable from the point of view of working qualities. Certain blacks will absorb as much as 15 per cent of their weight of moisture, making a total moisture content of 20 per cent or more. Most blacks for ink making contain from 2 to 4 per cent of moisture, although certain blacks may contain as high as 7 per cent.

**Moisture**<sup>1</sup>—A 1-g. sample of the black is placed in a weighed porcelain crucible and heated for one hour at 105° C. in a constant temperature oven in circulating dry air. The crucible is then removed from the oven, covered, and cooled in a desiccator over sulfuric acid. The loss in weight multiplied by 100 is recorded as the percentage of moisture.

**Ash**<sup>1</sup>—The crucible containing the residue from the moisture determination is heated gradually with a Meeker burner (or better, in a muffle furnace) to cherry-red (about 750° C.). Ignition is continued until all the particles of carbon have disappeared. The crucible is then cooled in a desiccator and weighed, after which it is heated again for 15 min., cooled in a desiccator, and reweighed. If the change in weight is more than 0.0002 g., the process is repeated until successive weighings are constant to this figure. The weight of the crucible and ash minus the weight of the crucible is taken as the weight of the ash.

**Acetone Extract**—A 2-g. sample is weighed into an alundum or paper extraction thimble of 20 cc. capacity and the extraction carried out for one hour, using any standard apparatus of the Soxhlet type. The weight of the residue after evaporation of the acetone is taken as the acetone extract. The extract for a pure carbon black is usually zero.

#### SPECIFICATIONS

The Bureau has received a great many inquiries in regard to tests which a carbon black must meet to be suitable for use in printing ink or rubber.

The following specifications represent an attempt to gather together the requirements adopted by the trade. It should be realized that there are no hard and fast specifications for carbon black, and that the test on which a black stands or falls is the practical test.

<sup>1</sup> For details of method, see F. M. Stanton and A. C. Fieldner, "Methods for Analyzing Coal and Coke," Bureau of Mines, *Technical Paper 8*.



## I—PRINTING INK

## Chemical Tests

Moisture—Less than 5.0 per cent  
Ash—Less than 0.1 per cent  
Acetone Extract—Less than 0.1 per cent

## Physical Tests

Color—Must match standard  
Tinting Strength—Must equal standard  
Grit—None

## Practical Tests

The black when made into ink must have satisfactory working qualities as determined by an actual run on the press for which the ink is intended. The ink must have satisfactory transfer, tack, drying properties, color, and must print a sufficient number of pages per pound. The oil must not separate from the pigment and there must be no offset or smutting.

## II—RUBBER

## Chemical Tests

Moisture—Less than 4 per cent  
Acetone Extract—Less than 0.5 per cent  
Ash—Less than 0.25 per cent

## Physical Tests

Grit—None  
Tinting Strength—Not less than 90 per cent of the strength of standard

## Practical Tests

Rubber mixes are made up containing equal weight of the sample to be tested and of the standard. Mixes are cured under exactly the same conditions. The finished sheet is tested for tensile strength, per cent elongation, toughness, and resistance to abrasion.

## III—PAINT

## Chemical Tests

Moisture—Less than 5 per cent  
Ash—Less than 1.25 per cent

## Physical Tests

Grit—None  
Tinting Strength—Not less than 95 per cent of the strength of standard

## PRELIMINARY WORK ON OTHER LABORATORY TESTS

Some preliminary work has been done on the problem of devising tests which will predict in a quantitative way the performance of the black when made into ink.

Tests which suggest themselves are measurements of viscosity, cohesion, and adhesion of mixtures of black and oil. Determination of these three properties should throw light on the probable performance of the black in use.

**VISCOSITY**—Attempts to measure the viscosity of mixtures of black and linseed oil in a Saybolt viscosimeter were unsatisfactory because an extremely dilute mixture was necessary. Accordingly, it was decided to try out the MacMichael torsion viscosimeter<sup>1</sup> which is used in the Petroleum Laboratory at the Pittsburgh Station for determining the viscosity of heavy oils. In this instrument a brass disk connected to a graduated torsion head is suspended from a piano wire in a rotating cup containing the liquid to be tested. Deflection times the time of one revolution is proportional to the viscosity. By use of suitable wire, liquids of very high viscosity can be tested. The apparatus is calibrated with a liquid of known viscosity.

Mixtures of equal weights of various blacks with the same amount of raw linseed oil were made up and the viscosity determined by means of the MacMichael apparatus. It was found that carbon blacks prized by ink makers on account of their "length" gave a lower reading on the viscosimeter than other blacks.

<sup>1</sup> THIS JOURNAL, 7 (1915), 961.

At this time the author's attention was drawn to an illuminating paper by Bingham and Green<sup>1</sup> on measurements of the mobility and "yield value" of paint. Bingham distinguishes between the *viscosity* of true liquids and the *rigidity* of plastic solids. Measurements of the flow through a capillary when different pressures are applied to the liquid show that the curve of pressure against the volume flowing through in unit time is different for true liquids and for plastic solids. In a true liquid the curve passes through the origin; for a plastic solid the curve cuts the pressure axis at some distance on one side of the origin. This distance Bingham calls the yield value or force which must be applied to the plastic solid before any deformation takes place.

It appeared possible to obtain similar curves using the torsion viscosimeter by measuring the deflection at different speeds of rotation, the speed of rotation corresponding to the volume flowing through the capillary and the deflection corresponding to the pressure.

Mixes of several blacks were made up, using 10 g. of black to 100 cc. of raw linseed oil. They were run at different speeds in the torsion viscosimeter at a temperature of 80° F. Curves are shown in Fig. 1.

It will be noticed that at the higher speeds the points lie on a straight line and that the extrapolation of this line back to zero r. p. m. does not pass through zero deflection. These curves are similar to those obtained by Bingham with the capillary method. It is possible to obtain a relative figure for the rigidity or mobility and the yield value from these data from the formula

$$R_{rel.} = t(d_t - d'_{t\infty})$$

where  $t$  is the time of one revolution in seconds,  $d_t$  the deflection, and  $d'_{t\infty}$  the deflection at zero r. p. m.

Data and calculated relative rigidity and mobility are given in Table I.

TABLE I—VISCOSITY OF MIXTURES OF CARBON BLACK AND LAMPBLACK WITH LINSEED OIL

Rev. per Min.	Deflection	Time of One Revolution Seconds	Relative Rigidity	Relative Mobility
SHORT CARBON BLACK				
124	90	0.48	15.4	0.0650
80	79	0.75	15.7	0.0637
47	70	1.28	15.4	0.0650
21	59	2.86	..	..
11	54	5.45	..	..
Yield Value = 58				
LAMPBLACK				
124	78	0.48	20.6	0.0486
90	66	0.67	20.8	0.0481
63	57	0.95	20.9	0.0478
38	46	1.58	17.4	0.0575
Yield Value = 35				
LONG CARBON BLACK				
124	55	0.48	15.4	0.0650
102	49	0.59	15.3	0.0654
67	40	0.90	15.2	0.0658
33	31	1.82	14.6	0.0685
11	22	5.45	..	..
Yield Value = 23				
RAW LINSEED OIL				
124	7	0.48	3.4	0.0194
70	4	0.86	3.4	0.0194
Yield Value = 0				
SUMMARY				
		Relative Rigidity	Relative Mobility	Yield Value Deg. Deflection
Short carbon black plus linseed oil		15.0	0.067	59
Long carbon black plus linseed oil		15.3	0.065	23
Lampblack plus linseed oil		22.0	0.046	33
Raw linseed oil		3.4	0.019	0

<sup>1</sup> "Paint, a Plastic Material and Not a Viscous Liquid," Reprint, American Society for Testing Materials, 22nd Annual Meeting, June 24-27, 1919.



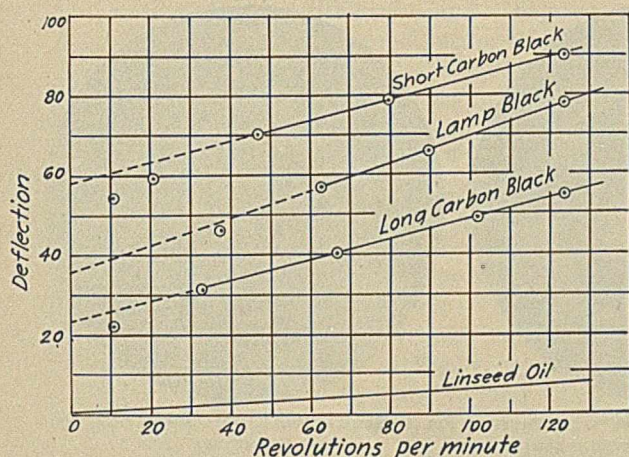


FIG. 1—"VISCOSITY" OF MIXTURES OF BLACK AND LINSEED OIL USING MACMICHAEL TORSION VISCOSIMETER

**DISCUSSION OF RESULTS**—It is at once seen both from the table and the curves that the difference between the long and short blacks tested is a difference principally in yield value, and that the mobilities of the two mixtures are nearly the same. The lampblack, with a steeper slope, has a lower mobility and a yield value intermediate between the long and short blacks.

The yield value is apparently the resistance, due probably to attractive forces between the particles of black, which the mixture offers to deformation when the load is applied above a certain rate. If the load is applied slowly, a different condition obtains, as instanced by the falling off of the curve from a straight line at the lower speeds of revolution. If the rotation of the cup is stopped while a mixture is being tested, the deflection drops back quickly to a point near the yield value, and then very slowly decreases nearly to zero over a period of several minutes.

It is hoped to continue work with the torsion viscosimeter and to compare results as obtained with it to the results obtained by the capillary method. Results are, of course, empirical, but might nevertheless be of practical value.

**COHESION**—Cohesion is defined as the attraction between the particles of the same substance. It is the resistance which a substance offers to deformation. An ink with low cohesion is a long ink, *i. e.*, it allows itself to be changed in shape and easily drawn out into strings. One with higher cohesion offers greater resistance to deformation and breaks off when an attempt is made to draw it out into a string with the palette knife.

Cohesion may be measured by determining the force necessary to draw a flat, circular plate away from the surface of the liquid on which it is resting. In order to get duplicable results with a mixture such as ink, the force must be applied quickly. If it is applied slowly, the same indefinite results are obtained as in the viscosity determinations at the lower speeds, due probably to slippage. Results of a few measurements of this character with long and short black mixtures show long blacks to have lower cohesion.

**ADHESION**—The molecular attraction between the surface of bodies in contact is called adhesion. The

adhesion of an ink for the type and paper must influence its working qualities. This is what the printer calls "tack."

Adhesion of a liquid may be measured by dipping a weighed metal plate in the liquid, allowing it to drain at a constant temperature, and reweighing. For a plastic material, such as ink or paint, the value for the adhesion so obtained would probably be modified by the yield value of the mixture.

In making any tests of mixtures of carbon or lampblack in oil, care must be taken that the black is thoroughly incorporated with the oil if duplicable results are to be obtained. The mixture should be thoroughly ground in a paint mill or three-roller ink mill. An inert mineral oil will probably be better than linseed oil for testing purposes.

**CHEMICAL ANALYSIS**—Carbon blacks consist of from 85 per cent to 95 per cent amorphous carbon, 1 per cent to 7 per cent water, 0.5 per cent to 0.8 per cent hydrogen, and from 2 per cent to 8 per cent oxygen, present partly as CO and CO<sub>2</sub>, and partly as fixed oxygen. Chemical analysis serves to show in a general way whether a carbon black will give a long or short ink. A black giving a long ink is usually low in carbon, and high in volatile matter and oxygen, while shorter blacks are correspondingly lower in the latter and higher in carbon. Typical analyses of long and short blacks are given in Table II.

TABLE II—ANALYSES OF CARBON BLACKS

CHARACTER	Long Black 1	Long Black 2	Long Black 3	Short Black 1	Short Black 2	Short Black 3
PROXIMATE ANALYSIS:						
Moisture.....	3.56	7.13	5.30	2.25	3.02	3.12
Vol. Matter.....	11.99	13.41	10.40	5.60	5.48	5.58
Fixed Carbon.....	84.40	79.44	84.16	92.13	91.47	91.22
Ash.....	0.05	0.02	0.14	0.02	0.03	0.08
ULTIMATE ANALYSIS:						
Hydrogen.....	1.19	1.32	1.11	0.74	0.88	1.05
Carbon.....	88.17	84.56	87.98	94.78	93.50	93.63
Nitrogen.....	0.04	0.04	0.08	0.09	0.04	0.05
Oxygen.....	10.54	14.00	10.68	4.37	5.25	5.19
Sulfur.....	0.01	0.06	0.01	0.00	0.30	0.00
Ash.....	0.05	0.02	0.14	0.02	0.03	0.08
ULTIMATE ANALYSIS (Moisture-free):						
Hydrogen.....	0.82	0.57	0.55	0.50	0.52	0.72
Carbon.....	91.42	91.05	92.91	96.96	96.41	96.64
Nitrogen.....	0.04	0.04	0.08	0.09	0.04	0.05
Oxygen.....	7.66	8.26	6.30	2.43	2.69	2.51
Sulfur.....	0.01	0.06	0.01	0.00	0.31	0.00
Ash.....	0.05	0.02	0.15	0.02	0.03	0.08

**ADSORBED GASES**—Carbon black contains considerable quantities of carbon monoxide, carbon dioxide, and oxygen. The oxygen is probably present as "fixed oxygen," that is, in some kind of combination with the carbon. Through the coöperation of Dr. G. A. Hulett and H. E. Cude, of Princeton University, the nature and amount of the adsorbed gases were determined. The apparatus used was that designed by Dr. Hulett to determine the nature of the adsorbed gases in gas-mask charcoal. The gases are pumped off at any desired temperature by means of a Töpler pump and analyzed, the water being caught on a bulb containing solid carbon dioxide.

At room temperature the composition of the gas which can be pumped off is practically that of air and the volume about that of the voids and capillary spaces. At 445° C. a larger volume usually comes off, consisting chiefly of CO<sub>2</sub>, CO, and N<sub>2</sub>. Carbon dioxide may be present in as large amount as one per cent of the



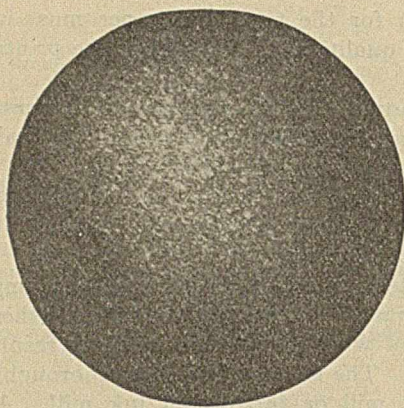


FIG. 2—SHORT BLACK, 18 MIN. AFTER PREPARATION ON SLIDE. MAGNIFICATION 500 DIAMETERS

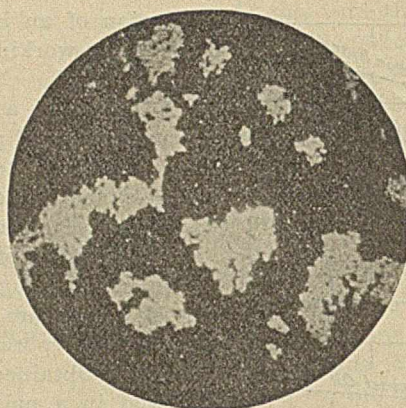


FIG. 3—SHORT BLACK, 2 HRS. AFTER PREPARATION ON SLIDE. MAGNIFICATION 500 DIAMETERS

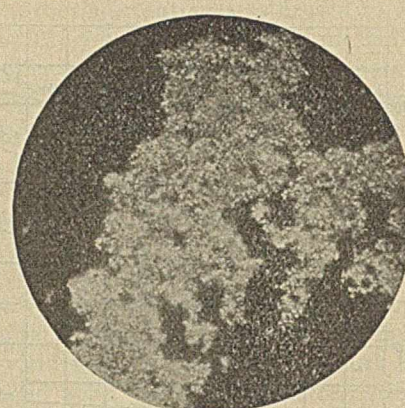


FIG. 4—SAME AS FIG. 2. MAGNIFICATION 2000 DIAMETERS

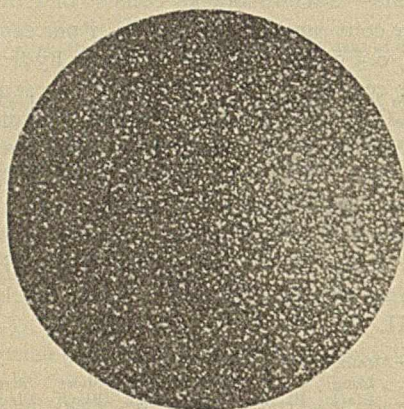


FIG. 5—LONG BLACK, 2 HRS. AFTER PREPARATION ON SLIDE. MAGNIFICATION 500 DIAMETERS

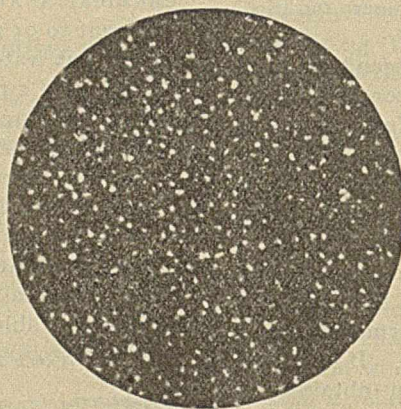


FIG. 6—LONG BLACK, 4 HRS. AFTER PREPARATION ON SLIDE. MAGNIFICATION 2000 DIAMETERS

MICROSCOPIC EXAMINATION—According to the ink makers, carbon blacks exhibit a wide range of properties,<sup>1</sup> depending on the method of manufacture.

Apparently a pigment which gives a long ink is one which affects the original properties of the vehicle to a less extent than an equal weight of a short pigment. At first sight this would seem to be merely a question of fineness of subdivision and it is probable that such is the case to a large extent. The tendency of the particles to form agglomerates may also play a part.

weight of the moisture- and gas-free black; oxygen about 0.01 per cent; CO, 0.1 to 0.2 per cent; methane and hydrogen, a trace; nitrogen, 0.05 to 0.2 per cent. An insufficient number of samples was analyzed to allow conclusions to be drawn in regard to the relation between adsorbed gases and physical and chemical properties. At the temperatures investigated, none of the hydrogen indicated by the ultimate analysis was pumped off. The carbon dioxide evolved would account for only part of the oxygen shown to be present by the ultimate analysis.

HYGROSCOPICITY—Carbon blacks absorb a considerable amount of moisture when exposed to damp air. Too large a moisture content is detrimental to the working qualities of the black in ink. Blacks containing a high percentage of volatile matter, *i. e.*, long blacks, are as a rule more hygroscopic than are those containing a lesser amount of volatile matter. Table III shows the results of exposing weighed amounts of differ-

TABLE III—HYGROSCOPICITY OF CARBON BLACK

SAMPLE	Percentage Loss in Weight over H <sub>2</sub> SO <sub>4</sub>		Percentage Gain in Weight over H <sub>2</sub> O		Moisture at 105° C.
	24 Hrs.	72 Hrs.	24 Hrs.	72 Hrs.	
Long Black 1.....	3.9	4.2	4.9	10.3	3.6
Long Black 2.....	6.2	6.9	7.0	14.1	7.1
Short Black 1.....	2.4	2.6	3.1	6.7	2.3
Short Black 2.....	2.2	2.5	3.6	7.7	3.0

ent blacks over water and over sulfuric acid at a temperature of about 25° C. The blacks when initially weighed were in equilibrium with a humidity of about 60 per cent at 25° C.

We have examined a large number of carbon blacks under the microscope at magnifications as high as 2000 diameters. The particles are very small, below the resolving power of the microscope. Indications are that the average size of the particles of carbon black is about 100 millimicrons. Microscopic examination indicates that the particles of long blacks are slightly larger than those of short blacks, although the apparent difference in size may be due to the effect of different surface conditions on the diffraction of light. Time has not been available to make exact measurements of the size.

Dark ground illumination with the ultra condenser has been found to be the most satisfactory method of examination for most purposes. Examination by transmitted light in the ordinary manner does not distinguish between single particles and small agglomerates of particles. Examination by transmitted light is of advantage in showing up large agglomerates or particles of grit and serves to show the relative opacity of the blacks. In general, the higher the percentage of carbon in a black, the blacker it looks under the microscope. Well calcined lampblacks are more opaque than carbon black or lampblacks containing much volatile matter.

Under the microscope freshly prepared mixtures of thin lithographic varnish and short or long carbon

<sup>1</sup> Underwood, "The Chemistry and Technology of Printing Inks."



black look at first precisely similar. They consist of ultraparticles or agglomerates of two or three particles. After a few minutes, however, a decided difference is apparent. The short black has begun to agglomerate into groups of 20 or 30 particles and in an hour over 100 may be grouped together. These agglomerates are loosely held together and may be dispersed by pressing down on the cover glass but they come together again in a few minutes. The long black, on the contrary, remains completely dispersed after several hours. Fig. 2 shows a short black 18 min. after preparation on the slide, Fig. 3 shows the same black after 2 hrs., Fig. 4 is a duplicate of Fig. 3, magnified 2000 diameters. Figs. 5 and 6 show a long black after several hours, magnifications 500 and 2000, respectively. The concentration of black in oil on the slide is about 1 part black to 1,000,000 parts oil.

Long blacks are usually made with cylindrical burners, a cool flame which tends to produce a black high in volatile matter. It seems probable that these adsorbed impurities prevent the carbon particles from agglomerating. In support of this, we have found that if a long black be treated with steam at 500° C. so that the occluded matter is burned off, the black forms a distinctly thicker mixture in oil and is seen to be agglomerated under the microscope. Conversely, if a definite quantity of a short black be treated with a dilute alcoholic solution of tannin and mixed with oil after the alcohol has evaporated, it makes a much more fluid mixture than an equal amount of untreated black. Under the microscope the tannin-treated mixture is found to be completely dispersed and to show no tendency to agglomerate.

To summarize, blacks which give long ink probably consist of slightly larger particles than those which make a shorter ink, hence exposing less surface per unit weight and taking less oil to form a mixture of given consistency. Furthermore, the difference in surface conditions due to the fact that long blacks contain a high percentage of adsorbed gases, probably influences the properties of the mixture of the black with oil. Microscopic examination of dilute mixtures of black and oil shows one effect of this difference in surface conditions, namely, the tendency of the blacks containing little adsorbed impurities to agglomerate, while the blacks containing large amounts of volatile matter remain dispersed.

#### SUMMARY

It has been pointed out that although the present process of making carbon black recovers only a few per cent of the carbon in the gas, no other process in practical operation produces a material with similar properties. Inability to secure carbon black would prove a serious blow to the printing industry and would probably inconvenience rubber manufacturers and others. In view of the diminishing supply of natural gas, developmental work on more efficient methods of production and production from other materials is needed. The problem is not an easy one. Developmental work should probably be directed toward the investigation of entirely new methods, such as the

thermal decomposition of gas or other hydrocarbons in absence of air, or explosion with CO, CO<sub>2</sub>, O<sub>2</sub>, or their mixtures.

The uses of carbon black have been discussed, test methods outlined, and a brief account given of preliminary work on microscopic and chemical differentiation of blacks giving long and short inks.

#### ACKNOWLEDGMENTS

We wish to express our appreciation of the valuable assistance and suggestions of Mr. A. C. Fieldner under whose supervision the work was carried out. We appreciate very much the assistance of Mr. J. O. Lewis, Chief Petroleum Technologist of the Bureau of Mines, and of Mr. R. O. Neal, Chemical Engineer in the Bureau of Mines, who made it possible for one of us to visit carbon black plants in Oklahoma and West Virginia.

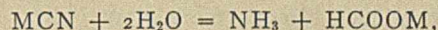
### THE PRODUCTION OF AMMONIA AND FORMATES FROM CYANIDES, FERROCYANIDES, AND CYANIZED BRIQUETS<sup>1</sup>

By G. W. Heise and H. E. Foote

U. S. CHEMICAL PLANT, SALTVILLE, VIRGINIA

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The hydrolysis of the cyanides of the alkali or alkaline earth metals by steam, whereby ammonia and formates are produced in accordance with the equation



has been known for a long time and was employed on a commercial scale at least as early as 1843.<sup>2</sup> In the past, however, the great demand for cyanides, and their value as compared with ammonia, have prevented this method from achieving any degree of commercial success. Three pounds of sodium cyanide are required for the production of one pound of ammonia, hence it is obvious that this method cannot compete successfully with the extraction of ammonia from gas liquor unless sodium cyanide can be procured very cheaply.

In recent times the great demand for nitrogen products, and their increased cost, has again directed attention to the hydrolysis of cyanides as a means of obtaining ammonia. Especially in the process of nitrogen fixation<sup>3</sup> with coke and soda ash in the presence of iron (the present success of which is in large measure due to Bucher,<sup>4</sup> who for the first time accurately analyzed the controlling factors), the subsequent hydrolysis of the cyanized briquetted product offers attractive possibilities, since the reaction products might be returned directly to the process, and the expense and difficulty of lixiviation and cyanide recovery avoided.

<sup>1</sup> The work described in this paper was done under the auspices of the Research Section, Nitrate Division, Ordnance Department, U. S. A., at Saltville, Va. Published by permission of Chief of Ordnance.

<sup>2</sup> By Newton, in England. T. H. Norton "Utilization of Atmospheric Nitrogen," Dept. of Commerce and Labor, Bur. of Manufacturers, Special Agents Series, *Bulletin* 52 (1912). See also Thorpe, "Dictionary of Applied Chemistry," 2, 599, in which reference to original literature and patents is made.

<sup>3</sup> Marguerite and Sourdeval, Eng. Patent 1,027 (1860).

<sup>4</sup> J. E. Bucher, *THIS JOURNAL*, 9 (1917), 233; *Met. & Chem. Eng.*, 16 (1917), 315; *Trans. Am. Inst. Chem. Eng.*, 9 (1916), 335.



Since the data available on the hydrolysis of cyanides are meager, especially as regards details of manipulation, most favorable conditions of temperature and pressure, and yields to be secured, a series of experiments was undertaken on the steaming of sodium cyanide, sodium ferrocyanide, and cyanized briquets. Work was limited to variations of pressure, partly because it was known that the effect of temperature was being studied elsewhere; partly because we believed that increased pressure would give improved yields. The equation for the reaction indicates that there is an actual shrinkage in volume during hydrolysis, two volumes of steam being absorbed for every volume of ammonia formed; hence pressure might be expected to exert favorable influence on the course of the reaction.

Fortunately, the results of the work on the effect of varying temperatures have been made available to us and are summarized and discussed in this report.

#### EXPERIMENTAL PART

**APPARATUS**—All hydrolysis experiments were conducted in autoclaves made of 4-in. wrought iron pipe, large enough to accommodate a charge of a kilogram of material and a liter of water. The form of apparatus finally adopted is shown in the accompanying diagram (Fig. 1). For a number of experimental runs at less than 150 lbs. pressure, no water was added, steam being introduced directly from plant boilers through a quarter-inch pipe entering near the bottom of the autoclave. In any case the autoclave was immersed in a bath of cylinder oil, kept above the reaction temperature, to obtain the desired pressure when water was used inside the autoclave, and to prevent condensation of water when steam was introduced.

Pressures were read on a Schaeffer and Budenberg ammonia gauge, reading directly to 300 lbs., and temperatures were recorded by means of ordinary laboratory thermometers immersed in the oil bath, and in some experiments by a mercury-in-iron thermometer (not shown in Fig. 1) inserted near the top of the autoclave.

**MANIPULATION**—To make an experimental run, the oil bath was brought to the temperature (determined by experiment) required to heat the autoclave to a point corresponding to the desired pressure. The autoclave was then immersed and connected through a quarter-inch union to the condenser. When the desired temperature and pressure had been reached, ammonia (together with any excess steam or other gases) was drawn off through an iron ammonia valve into a water-cooled iron condenser, thence into a barrel or large bottle of dilute sulfuric acid, and determined by titration of the sulfuric acid solution. Titrations were made at frequent intervals in order to follow the course of the reaction.

**ANALYTICAL METHODS**—All cyanogen compounds used, including the briquets, were analyzed for cyanide content by the method of Lundell and Bridgman<sup>1</sup> in which the cyanide solution or extract is titrated in a

solution made slightly alkaline with ammonia against standard nickel ammonium sulfate solution, using dimethylglyoxime as an indicator.

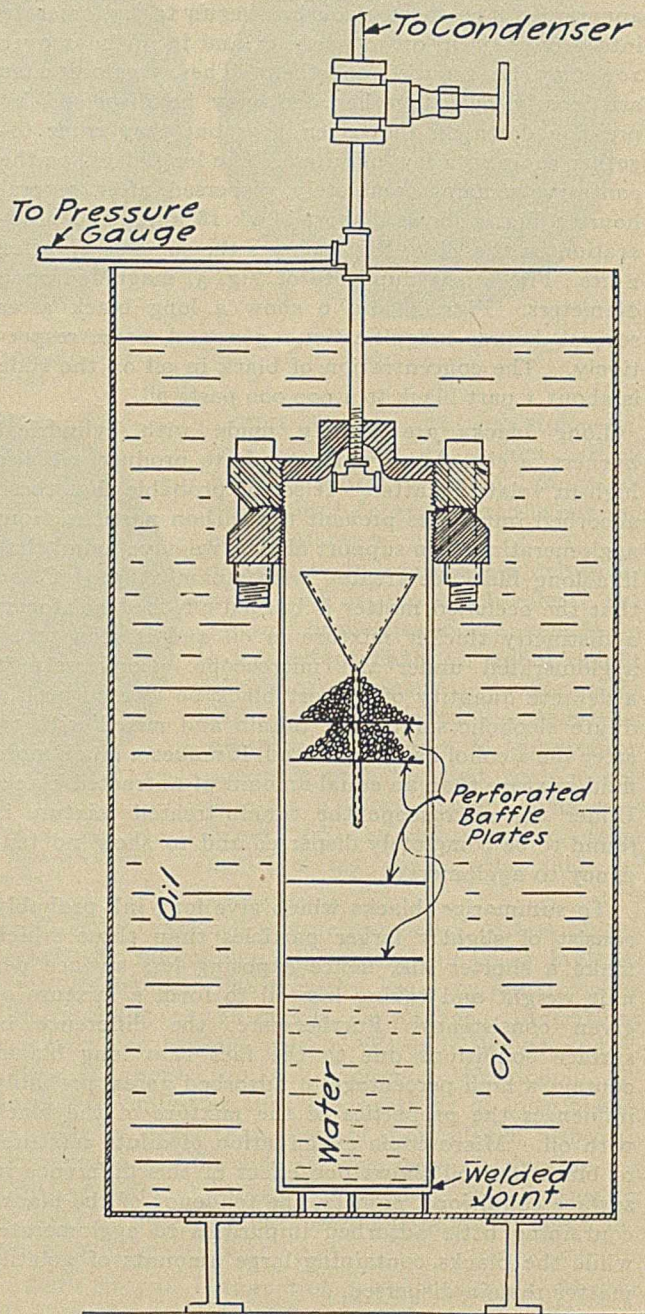


Fig. 1

Formates were determined in the usual manner by titration with permanganate in warm alkaline solution.<sup>1</sup>

#### EXPERIMENTAL DATA

##### A—Experiments with Sodium Cyanide

The following results were obtained by passing steam over sodium cyanide at pressures of 50–80 lbs.

Expts. 1 and 2 are summarized in Table VI.

Expt. 3—The analyses of the reaction mass before and after treatment are given in Table I.

<sup>1</sup> THIS JOURNAL, 6 (1914), 554.

<sup>1</sup> Lieben, *Monatsh.*, 14, 746; 16, 219.



TABLE I—ANALYSIS OF CYANIDE MASS

	Before Hydrolysis	After Hydrolysis
Sodium cyanide.....	80.77 per cent	0.07 per cent
Sodium cyanate.....	5.22 per cent	..
Sodium formate.....	..	91.96 per cent
Sodium oxalate.....	..	Nil
Weight.....	111 g.	130 g.

Expt. 4—The autoclave smelled strongly of ammonia at the end of the run, and there were other indications that the hydrolysis was proceeding as long as water was present, and that better results would have been achieved had more water been present. The analyses before and after hydrolysis are given in Table II.

TABLE II—ANALYSIS OF CYANIDE REACTION MASS

	Before Hydrolysis	After Hydrolysis
Sodium cyanide.....	83.9 per cent	8.58 per cent
Sodium cyanate.....	1.99 per cent	..
Sodium formate.....	..	90.6 per cent
Weight.....	115 g.	135 g.

### B—Experiments with Sodium Ferrocyanide

Because of the possibility of and tendency toward ferrocyanide formation in cyanized briquets, and the resulting decreased yield of ammonia on hydrolysis, Expts. 5 to 11, inclusive, were undertaken on the steaming of sodium ferrocyanide.

Expts. 5 and 6 are summarized in Table VI.

Expt. 7—The foregoing attempts to hydrolyze ferrocyanide having resulted in virtual failure on account of frothing, the effect of the addition of sodium hydroxide was studied with the result noted in Table VI.

Expt. 8—In spite of all precautions, frothing occurred after 2 hrs. when 27.1 per cent of the theoretical yield of ammonia had been produced.

Expt. 9—A pressure of 300 lbs. developed in 2 hrs. The hydrolysis was terminated before its completion because the water entirely distilled out of the autoclave while the reaction was still in progress.

Expt. 10—To continue experiments on the effect of alkalis, lime was used. A partial analysis of the reaction product is given in Table III.

TABLE III—ANALYSIS OF REACTION PRODUCT FROM FERROCYNANIDE HYDROLYSIS

	Per cent
Sodium ferrocyanide.....	28.8
Formate (as HCOONa).....	12.8
Lime (CaO).....	13.8

Expt. 11—In a similar run where sodium carbonate was employed, the reaction product gave the following analysis:

TABLE IV—ANALYSIS OF REACTION PRODUCT FROM FERROCYNANIDE HYDROLYSIS

	Per cent
Sodium ferrocyanide.....	44.3
Sodium carbonate.....	23.7
Formate (as HCOONa).....	28.5

The foregoing experiments show that cyanide can readily be hydrolyzed at comparatively low pressure, and that ferrocyanide is acted upon slowly and with difficulty. Alkalies, other than perhaps caustic soda, did not greatly affect ferrocyanide decomposition.

### C—Experiments with Cyanized Briquets

The briquets were obtained from U. S. Chemical Plant, Saltville, Virginia. Preliminary experiments showed that good yields of ammonia cannot be ob-

tained, from cyanized briquets at 50 lbs. steam pressure, the reaction products being essentially ferrocyanide and hydrogen.<sup>1</sup> It was found further that, though some hydrogen was also formed at 120 to 150 lbs. pressure, good yields of ammonia could be secured.

Expts. 12 and 13 are sufficiently well described in Table VI.

Expt. 14—In this experiment the ferrocyanide content rose from 8 per cent in the original mixture to 10 per cent in the residue, the increase corresponding to a formation of approximately 10 g. of sodium ferrocyanide during the reaction. No oxalate was detected in the residue. Sodium formate, 93 per cent pure and free from carbonate, was recovered from the residue by extraction.

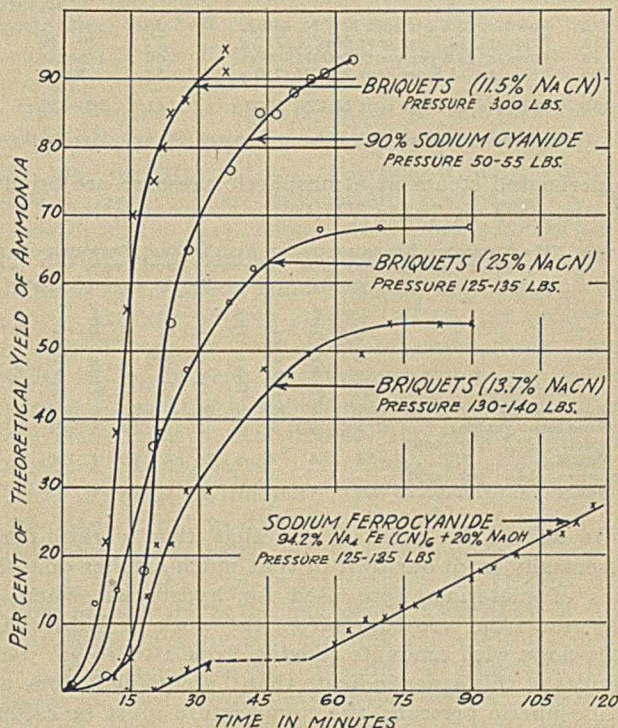


FIG. 2—HYDROLYSIS OF SODIUM CYANIDE, FERROCYNANIDE, AND CYANIZED BRIQUETS

Expt. 15 requires no special comment.

Expt. 16—The routine analytical results from the briquets before and after treatment are given in Table V.

TABLE V—ANALYSIS OF BRIQUETS

	Before Steaming Per cent	After Steaming Per cent
Sodium cyanide.....	11.2	0.0
Sodium cyanate.....	0.4	Trace
Sodium carbonate.....	23.87	29.16
Sodium ferrocyanide.....	1.38	0.16

The experimental results are summarized in Table VI, and typical runs are shown graphically in Fig. 2.

### DISCUSSION OF RESULTS

For comparative purposes the results obtained with

<sup>1</sup> C. F. Bierbauer and L. S. Finch, U. S. Patents 1,295,262 and 1,295,293, *Met. & Chem. Eng.*, 20 (1918), 431, attempt to obviate this difficulty by dissolving crude cyanide as rapidly as possible by blowing pulverized briquet material into a tank equipped with water spray and agitator, rapidly filtering the mixture, and heating the solution with steam under 125 lbs. pressure, the ammonia being allowed to escape through a relief valve set at 50 lbs.



TABLE VI—HYDROLYSIS OF SODIUM CYANIDE, SODIUM FERROCYANIDE AND CYANIZED BRIQUETS

EXPT. No.	CHARGE	Time Min.	Steam Pressure in Autoclave Lbs.	TEMPERATURES ° C.			Oil Bath	Ammonia Yield Per cent of Theoretical	REMARKS
				Corresponding to Steam Pressure Recorded					
SODIUM CYANIDE									
1A	90 per cent NaCN, 300 g.	80	70-80	158-161	140-155	165-175	65	Charge frothed into condenser, stopping run	
2A	90 per cent NaCN, 150 g.	65	50-55	148-150	145-148	210	93	Charge frothed into condenser, stopping run	
3B	83.2 per cent NaCN, 111 g.	60	200 (Av.)	185-210	...	215-220	100	500 cc. water in autoclave	
4B	85.5 per cent NaCN, 115 g.	120	55	150	...	210	82	1000 cc. water in autoclave. Reaction proceeded until autoclave was dry	
SODIUM FERROCYANIDE									
5A	Na <sub>4</sub> Fe(CN) <sub>6</sub> ·10H <sub>2</sub> O, 500 g.	30	120-135	170-181	180	160-200	Trace	Frothing put end to run	
6A	94.25 per cent Na <sub>4</sub> Fe(CN) <sub>6</sub> , anhyd., 310 g.	70	120-135	170-181	180	220-240	Little	Frothing, pronounced odor of NH <sub>3</sub> at end of run	
7A	94.25 per cent Na <sub>4</sub> Fe(CN) <sub>6</sub> , anhyd., 150 g. + 30 g. NaOH	80	125-145	173-184	180-185	200	17	Frothing, pronounced odor of NH <sub>3</sub> at end of run	
8A	94.25 per cent Na <sub>4</sub> Fe(CN) <sub>6</sub> , anhyd., 150 g. + 35 g. NaOH	120	125-135	178-181	178	225	27.1	Frothing, pronounced odor of NH <sub>3</sub> at end of run	
9B	96.5 per cent Na <sub>4</sub> Fe(CN) <sub>6</sub> , anhyd., 52 g.	285	300-330	220	...	250	46.6	800 cc. water in autoclave	
10B	90.5 per cent Na <sub>4</sub> Fe(CN) <sub>6</sub> , anhyd., 106 g. + 25 g. CaO	112	300-330	220	...	250	15.8	1000 cc. water	
11B	96 per cent Na <sub>4</sub> Fe(CN) <sub>6</sub> , anhyd., 35 g. + 15 g. Na <sub>2</sub> CO <sub>3</sub>	170	300-340	220	...	260	25.3	1000 cc. water	
CYANIZED BRIQUETS									
12A	Powder, 23.5 per cent NaCN, 440 g.	55	135	180	180	...	50	Charge frothed into condenser	
13A	Briquets, 13.7 per cent NaCN, 730 g.	72	130-140	180-183	175-178	220	54	Hydrogen formed	
14A	Powder, 23.5 per cent NaCN, 400 g.	60	125-135	178-181	174-177	220	68	Residue free from cyanide	
15B	Briquets, 13.8 per cent NaCN, 750 g.	38	300	220	...	230	90.7	500 cc. water in autoclave. Briquets recovered unbroken	
16B	Briquets, 11.5 per cent NaCN, 1000 g.	41	300-330	220	...	240	92.5	700 cc. water. Good briquets recovered	

In experiments marked "A" steam was passed into autoclave; in those marked "B" water was introduced directly.

superheated steam at atmospheric pressure are briefly summarized in Table VII.<sup>1</sup>

TABLE VII—STEAMING EXPERIMENTS AT ATMOSPHERIC PRESSURE—PERCENTAGE OF THEORETICAL AMMONIA PRODUCTION

MATERIAL	200° C.		300° C.		400° C.		500° C.		600° C.	
	Per cent	Time, Hrs.	Per cent	Time, Hrs.	Per cent	Time, Hrs.	Per cent	Time, Hrs.	Per cent	Time, Hrs.
Sodium cyanide.....	0.8(?)	4	56	6	61	6	73	6	74	6
Sodium ferrocyanide.....	0.5	4	0.5	6	5	6	88	4	91	4
Briquets, 20.5 per cent NaCN.....	54	4	57	4	84	6	99	4	100	4
Briquets, 14.7 per cent NaCN.....	60	4	47	4	79	6	84	4	85	4

It should be pointed out that this work differs essentially from our own in that much smaller quantities of materials were used for hydrolysis; whereas we used from 100 to 1000 g., the work in Table VII was done with amounts ranging from 2 to 10 g. The results of this series indicate that if steaming is done at atmospheric pressure, the temperature must be raised to at least 600° C. to obtain quantitative yields of ammonia. Although the experiments in question were continued for periods of 4 to 6 hrs., the greatest portion of the ammonia was obtained in from 30 min. to one hour.

Using steam under pressure with different degrees of superheating, results were obtained as shown in Table VIII.

TABLE VIII—EXPERIMENTS WITH STEAM UNDER PRESSURE

Gauge Pressure (pounds)...	0		25		50		75		100	
	Per cent	Time, Hrs.	Per cent	Time, Hrs.	Per cent	Time, Hrs.	Per cent	Time, Hrs.	Per cent	Time, Hrs.
Briquets, 20.5 per cent NaCN (saturated steam)	12	3.5	25	4	30	4	36	4	44	4
Briquets, 20.5 per cent NaCN, Reaction at 300° C.	56	4	61	4	70	4	80	4	86	4
Briquets, 20.5 per cent NaCN, Reaction at 400° C.	75	4	83	4	93	4	100	4	102	4

The above data indicate that with a steam pressure of 100 lbs., a temperature of at least 400° C. must be

<sup>1</sup> The data in Tables VII and VIII were obtained by Mr. J. A. Beattie of the Nitrate Division, working in the Geophysical Laboratory.

maintained to secure quantitative yields of ammonia. Our own work indicates that the exceedingly high temperatures noted above are not necessary for the hydrolysis of cyanides on a commercial basis. We have found that a pressure of 300 lbs. without superheat will insure satisfactory results.

The hydrolysis of cyanides may be accomplished without great difficulty at temperatures and pressures which can readily be secured in commercial practice. The reaction proceeds more rapidly at high steam pressure than at low, as might be expected. That pressure and not merely the higher temperature of compressed steam is a deciding factor is proved by the fact that much higher temperatures are required for hydrolysis at atmospheric pressure than those recorded in the present work.

A possible advantage of work at the comparatively low temperatures here recorded arises from the fact that hydrolysis proceeds without indications of side reactions, virtually quantitative yields of formate being secured. The available literature indicates that there is little danger of decomposition of formates below 200° C. Above this temperature oxalates might be formed, and at still higher temperatures oxalates decompose to form carbonates. (The temperature of saturated steam at 300 lbs. pressure is 217° C.)

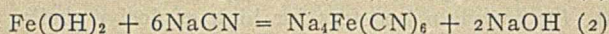
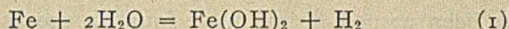
In runs made at the same pressure, better results were secured by passing steam into the autoclave than by introducing water direct. This is to be expected, since, in the former case, fresh steam comes in contact with the reaction mass.

The experiments with ferrocyanide showed that hydrolysis takes place very slowly, even at 300 lbs. steam pressure. Results were not materially benefited by the addition of alkalies, though at a pressure of 125 lbs. the addition of caustic soda gave indications of an increased yield.

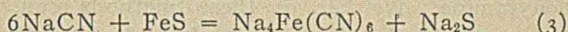
The hydrolysis of cyanized briquets to form ammonia does not proceed very far at low pressures, the best yield obtained at 125 lbs. being only 68 per cent.



This is due, in a large measure at least, to the formation of ferrocyanide, probably as follows:



The formation of ferrocyanide, which takes place readily enough, seemed further to be influenced appreciably by the sulfur content of the briquets, in accordance with the reaction:



At low pressures the formation of ferrocyanide in briquets seems to progress more rapidly than its decomposition, so that the ferrocyanide content materially increases, as shown in Expt. 13. It is therefore clear that to obtain a maximum yield, ammonia should be liberated from the cyanide as rapidly as possible, preferably at a temperature and pressure at which ferrocyanide is hydrolyzed with appreciable velocity. These conditions were obtained in Expt. 16, in which a maximum yield (92.5 per cent) of ammonia was obtained, and in which the ferrocyanide content was actually decreased. The good results obtained at 300 lbs. indicate the desirability of working at this pressure. It does not seem advisable to go much higher in commercial operation.

Bierbauer and Finch<sup>1</sup> describe a procedure whereby, in the presence of much ferrocyanide, all the cyanide in the pulverized briquet mass is converted into ferrocyanide by digestion with finely divided iron, whereupon the mass is filtered, and hydrolyzed at 80 to 150 lbs. steam pressure. In our own work we were unable to secure satisfactory yields of ammonia by steaming ferrocyanide at such low pressures.

If condensation in the autoclave is prevented, briquets may be steamed without physical disintegration, and can be returned to the cyanizing retorts. It has been shown from previous experiments that at the cyanizing temperature of 950° to 1000° C., carbon monoxide (formed in this case by the decomposition of formates to carbonates) does not interfere in any way with the nitrogen fixing reaction. It is believed that briquets could be put through the process three times without disintegration, so that a great saving in the cost of briquetting would be effected. It is clear also that unless sodium formate (or oxalate) is to be recovered there is no very apparent advantage to be derived from the use of soda ash as one of the raw materials for the cyanizing process. Barium compounds would naturally suggest themselves because of the relative ease with which they fix nitrogen,<sup>2</sup> and even calcium compounds might be applicable.

Although no appreciable temperature effect was noted in the present work, the fact that the reaction is strongly exothermic might prove advantageous both by reducing the amount of heat to be supplied from without and by tending to prevent condensation of moisture which causes the briquets to stick together or disintegrate. On the other hand, a very considerable rise in temperature might induce secondary reactions such as the conversion of formate to oxalate and of the

latter to carbonate. It is also possible that the ammonia might be partially decomposed by being heated in contact with finely divided iron, a reaction which begins at about 500° C., though the results of high temperature experiments quoted above do not corroborate this view.

It is believed that the actual cost of the process of making ammonia from cyanide is low. Whether it can be made profitably depends largely on the cost of cyanide in the iron-coke briquet mass. In any case, at least three times the cost per pound of cyanide in the briquets would have to be added to the cost of the process, since 3 lbs. of cyanide are required for the production of one pound of ammonia. For example, assuming that cyanide in the iron-coke mass costs 10 cts. per lb., and that the cost of ammonia production is 4 cts., the total cost of the ammonia would be 34 cts. if the formate formed were not recovered.

Two dispositions may be made of the formate: it may be left in the briquets and returned to the process, or it may be extracted and recovered if market conditions warrant. There are certain advantages to be gained from the first course. If the briquets are kept dry during the steaming, they are obtained at the end of the process in good condition and may be returned directly to the cyanizing process. They can probably be passed through the complete cycle three times before it is necessary to rebriquet them. As far as cyanide formation is concerned, formates would be equivalent to carbonate because the former could be converted into the latter on heating. Thus, the entire cost of the fresh sodium carbonate otherwise necessary, and one-half to two-thirds of the cost of kneading, briquetting, and drying, would be eliminated.

If the formate is to be returned to the process, the whole process might be cheapened somewhat by the substitution of barium or possibly calcium for sodium in the manufacture of cyanide. Margueritte and Sourdeval used barium carbonate, carbon, and iron, and state that barium compounds react with nitrogen much more readily than potassium compounds (and presumably more readily than sodium compounds). Thus not only might the process of cyanide formation be cheapened through the use of cheaper material, but it might also be made more efficient, either by lowering the temperature of the reaction or increasing the yield, or both. This suggests some interesting possibilities in the way of future experiments on nitrogen fixation along the line of comparison between barium or calcium and sodium compounds under the same conditions.

On the other hand, the formate may be recovered in fairly pure form by lixiviating the steamed reaction mass with alcohol. It may also be obtained at less cost, but in cruder form, by lixiviation with water. The formates might be used as a starting point for other compounds, such as esters, formic acid, oxalates, or oxalic acid.

#### SUMMARY

The formation of ammonia from sodium cyanide, sodium ferrocyanide, and cyanized briquets was

<sup>1</sup> *Loc. cit.*

<sup>2</sup> Margueritte and Sourdeval, *Loc. cit.*



studied. Good yields of ammonia were obtained from sodium cyanide by steaming at 50 lbs. pressure. A quantitative yield was obtained at 200 lbs.

The hydrolysis of ferrocyanides proceeded very slowly. A maximum yield of 46 per cent was obtained by steaming  $4\frac{3}{4}$  hrs. at a pressure of 300 to 330 lbs.

With cyanized briquets, yields averaging over 90 per cent were obtained in 30 to 45 min. by steaming at 300 to 330 lbs.

To obtain satisfactory results with steam at atmospheric pressure, a temperature of 600° C. was necessary.

High temperatures were necessary to obtain good results with steam at low pressure. A temperature of 600° C. with atmospheric pressure and 400° C. with 100 lbs. pressure gave satisfactory results.

At the temperatures involved in the experiments with saturated steam there were no indications of side reactions, only formates and ammonia being produced.

#### THE PREPARATION OF HEXANITRODIPHENYLAMINE AND ITS USE AS A BOOSTER FOR SHELL CHARGES<sup>1</sup>

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Early after the entry of the United States into the war it became evident that the country's capacity for the manufacture of secondary detonating materials or "booster" explosives for high explosive shells was insufficient to provide for the extensive program planned by the Ordnance Department.

The material which had been used to the best advantage for this purpose and the one apparently preferred above all others was tetryl, or tetranitromethylaniline. Closely approaching this compound in value was tetranitraniline, though the latter had some disadvantages connected with its use on account of its questionable stability in the presence of moisture.

It was evident therefore that in order to compete with these compounds and at the same time supplement the available supply of secondary detonating agents, it would be necessary to produce a material of similar properties, *i. e.*, high melting point, sensitiveness to detonation, and power as a detonator, and at the same time to produce it by some method whose simplicity would make it possible to compete with the relatively low prices of tetryl and TNA. Added to these requirements it was considered necessary to develop such a compound from sources other than toluene in order to conserve as far as possible the supply of this material.

A survey of the field indicated that the symmetrical hexanitrodiphenylamine would probably meet these requirements, though the literature was singularly vague on the properties of the material. A careful study was therefore made early in 1918 on the manufacture and properties of this compound. The results in a large measure justified the hopes which

had been felt, and it is probable that had the war continued, hexanitrodiphenylamine, or hexil, as it has been named, would have found a use as a substitute for tetryl in boosters for high explosive shells.

The first description of this compound was given in 1874 by P. T. Austen,<sup>1</sup> who prepared it by nitration of picryl-*p*-nitraniline, and during the same year it was prepared by Gnehm<sup>2</sup> by the nitration of diphenylamine. Neither of these methods can have any practical value on account of the high cost of the intermediates involved.

The synthesis described in a patent of the Chemische Fabrik Griesheim,<sup>3</sup> and elaborated in 1913 by T. Carter,<sup>4</sup> offered greater advantages, and it was this method which was settled on for development. This method as described by Carter depends on the reaction of 1,2,4-chlorodinitrobenzene with aniline to form dinitrodiphenylamine and aniline hydrochloride, with subsequent nitration by nitric acid in two stages to hexanitrodiphenylamine.

It should here be pointed out that a recent article by Messrs. Hoffman and Dame<sup>5</sup> indicates that the Bureau of Mines was also interested in this subject, their work in a measure duplicating the early phases of our own work.

#### PREPARATION OF DINITRODIPHENYLAMINE

The first preparation of dinitrodiphenylamine was made in accordance with Carter's procedure. This method consists of agitating two molecules of aniline with one molecule of chlorodinitrobenzene, the reaction beginning slowly at moderate temperature. There is at first a slow rise in temperature followed by a more energetic one, which carries the temperature to 125° C., at which point the charge is drowned in water and extracted with water and dilute hydrochloric acid to remove excess aniline and by-product aniline hydrochloride.

In the laboratory excellent results were obtained with theoretical yields of a product melting at 149° to 152° C. When the process was tried on a larger scale, however, it was found difficult to control the temperature properly without drowning the charge before the completion of reaction, since the charge rapidly became so viscous that efficient agitation was out of the question.

The following more satisfactory method was then devised.<sup>6</sup> Two molecules of aniline and one molecule of chlorodinitrobenzene are added to three times their combined weight of water heated to 60° C., and the mixture is agitated mechanically to form an emulsion. Steam is then introduced to raise the temperature to 80° C., the reaction beginning during this heating, and coming to completion in one hour. The dinitrodiphenylamine precipitates in the form of thick clusters of red needles. Agitation at 80° C. is continued one-half hour longer to insure complete solution of aniline hydrochloride, after which the charge

<sup>1</sup> Ber., 7, 1248.

<sup>2</sup> Ibid., 7, 1399.

<sup>3</sup> D. R. P. 86,295, July 1885.

<sup>4</sup> Z. ges. Schiess-Sprengstoffw., 1913, 205-251.

<sup>5</sup> J. Am. Chem. Soc., 41 (1919), 1013.

<sup>6</sup> U. S. Patent 1,309,580, July 8, 1919.

<sup>1</sup> Presented at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 2 to 6, 1919.



is thrown on a filter, washed with dilute hydrochloric acid and water, and finally dried. 95 to 100 per cent of theoretical yields are obtained of material melting at 148° to 152° C. The mother liquor from this process is available for recovery of aniline, and in several runs made in the laboratory recoveries of 85 per cent of the theory were made. This method gave perfect reaction control, and in addition yielded a product in such form that it could be used at once for nitration. Several runs were then made on the 100 lb. scale with equally satisfactory results.

#### PREPARATION OF TETRANITRODIPHENYLAMINE

In the nitration of dinitrodiphenylamine thus prepared to the tetra stage, as before stated, the method of Carter involves the addition of dry dinitrodiphenylamine to nitric acid of approximately 40 per cent concentration at temperatures up to 90° C. Any method of nitration involving the use of straight nitric acid is open to serious objections on account of excessive consumption of nitric acid and the necessity for using enameled equipment; and in addition to these objections it was found on trial of Carter's method that the nitration was liable at times to serious and almost uncontrollable foaming.

Carter's article makes the definite statement that a mixed nitric-sulfuric acid cannot be used for this nitration on account of the insolubility of dinitrodiphenylamine in sulfuric acid, but since the claim appeared unreasonable, a series of experiments was instituted on the use of mixed acid for nitration. After considerable investigation it was found that the nitration could be carried out satisfactorily by the addition of dry dinitrodiphenylamine at 70° C. to 3.5 to 4 parts of a mixed acid containing 30 to 45 per cent HNO<sub>3</sub> and 50 to 40 per cent H<sub>2</sub>SO<sub>4</sub>, followed by raising the temperature to 80° to 90° until evolution of NO<sub>2</sub> was at a minimum. The nitration charge was then cooled and filtered for removal of the spent acid. The product of this nitration was a brownish yellow amorphous material, containing small percentages of higher nitration products, and was found to be in such condition that it could be used directly for the final nitration.

#### NITRATION OF TETRANITRODIPHENYLAMINE TO HEXANITRODIPHENYLAMINE

Carter's procedure for the final nitration also involves the use of straight nitric acid, tetranitrodiphenylamine being added at 40° to 70° C. to a 90 per cent nitric acid, with final heating at 90° C., followed by cooling and filtration. The product from this method consisted of a finely divided, almost amorphous material which dusted badly, and consequently involved danger of poisoning in handling.

It was found on experimenting that mixed acid could be used with satisfaction at this stage also, and that the character of the product could be varied at will from the amorphous form to the crystalline form by varying the proportion of nitric to sulfuric acid. For example, by the use of a mixed acid containing 25 per cent HNO<sub>3</sub> and 70 to 75 per cent H<sub>2</sub>SO<sub>4</sub>, an amorphous product was obtained, as compared with

a distinctly crystalline product when an acid containing 60 per cent HNO<sub>3</sub> and 40 per cent H<sub>2</sub>SO<sub>4</sub> was used. As a crystalline product is the more desirable, an acid of the last noted composition was finally used.

The final procedure consisted of adding the acid tetranitrodiphenylamine to 3.75 parts mixed acid at 70° C., followed by one hour's heating at 90° C., cooling to room temperature and filtering. The product thus obtained was a distinctly crystalline, yellow, free running material, melting sharply with decomposition at a temperature between 238.5° and 239.5° C.

This procedure, for both the tetra- and the hexa-stage nitrations, was also studied thoroughly on the 100 lb. scale, with entirely satisfactory results. Yields of 86 per cent of the theory were obtained, this point duplicating the result of the nitration with straight nitric acid. Spent acid recoveries throughout were very good and the spent acid composition was such that it could be handled in iron. The following are specimen analyses of the spent acid from the two stages:

	TETRA-STAGE SPENT ACID Per cent	HEXA-STAGE SPENT ACID Per cent
HNO <sub>3</sub> .....	14.28	33.62
H <sub>2</sub> SO <sub>4</sub> .....	50.95	40.03
HNOSO <sub>3</sub> .....	7.45	11.30
H <sub>2</sub> O.....	26.13	14.16
Ether soluble.....	1.07	0.89

#### PREPARATION OF HEXANITRODIPHENYLAMINE BY COMPLETE NITRATION OF DINITRODIPHENYLAMINE WITH MIXED ACID

In the article by Hoffman and Dame<sup>1</sup> the possibility is suggested of preparing hexanitrodiphenylamine by a one-stage nitration with mixed acid. This procedure was found to be possible by adding dinitrodiphenylamine to 3.75 parts of a mixed acid containing 60 per cent HNO<sub>3</sub> and 30 per cent H<sub>2</sub>SO<sub>4</sub> at temperatures up to 70° to 80° C. This results in the formation of the tetranitro compound. Two parts of fuming acid, analyzing 107 to 108 per cent H<sub>2</sub>SO<sub>4</sub>, are then carefully added at 80° to 90° C. to bring about nitration to the hexa stage and the charge is cooled after one hour, and filtered. 85 per cent of theoretical yields were obtained of amorphous material melting at 238° C. Cost estimates indicated that the two-stage process was preferable to this method, and it was not pursued further.

In this connection it was found that the use of acids of higher than 90 per cent acidity produced charring of dinitrodiphenylamine.

#### NEUTRALIZATION OF FREE ACID IN HEXANITRODIPHENYLAMINE

In the work so far discussed no mention has been made of the methods used for freeing the hexanitrodiphenylamine from the free acid carried by the particles of the material.

No alkali can be used with hexanitrodiphenylamine for the reason that the material acts as an acid in the presence of alkalis, forming highly soluble salts, and it is therefore necessary to depend on the use of water for washing. It was found that there

<sup>1</sup> *Loc. cit.*



was great difficulty in completely purifying amorphous hexanitrodiphenylamine, but that the acid could be completely removed from the crystalline material by means of a drowing wash, followed by 2 or 3 one-hour washes with boiling water, after which the product can be centrifuged and dried on trays at temperatures up to 80° C.

#### EXPLOSIVE PROPERTIES OF HEXANITRODIPHENYL-AMINE

While the foregoing work was being carried out on the preparation of hexanitrodiphenylamine, a thorough study was also made of the properties of the material in comparison with tetryl and tetranitraniline, the compounds which it was desired to replace, and with TNT.

The most important properties of an explosive for any purpose are unquestionably stability, sensitiveness to detonation, and explosive power. For use in a booster charge these items are especially important. The stability must be high, because of the high temperatures liable to occur in field service. The material must be sufficiently sensitive to give complete detonation with its primer charge, and, at the same time, must be sufficiently insensitive to eliminate danger from premature explosion. It must also be powerful enough to insure complete detonation of the bursting charge of the shell with a minimum size of booster charge.

As a test for stability, the Abel heat test has for a long time been recognized by the authorities as perhaps the most reliable. During the period of the war, however, the value of this test has been questioned many times when used for nitro compounds, and it is therefore being supplanted by other tests depending on the actual determinations of the products of decomposition of the nitro compound on heating. The most easily applied of this class of tests is the Obermuller, or gas evolution test,<sup>1</sup> and this was developed in our laboratory to a fair degree of satisfaction.

It consists of heating a weighed portion of the material in question at 120° C. *in vacuo* in a calibrated flask attached to a calibrated manometer. Pressure readings are taken every hour on the manometer, the increase in pressure being calculated back to cc. gas evolved per hour at the desired pressure.

Tested in this manner, hexil showed the following results, in comparison with other explosives:

	M. P.	Stability cc. per hr. per g. at 100 mm. pressure
Hexil.....	140.0	4.4
TNT.....	77.3	3.6
	80.1	2.1
Tetryl.....	127.0	13.8
	128.2	2.6
	129.0	1.6

It appears therefore that hexil has a stability close to that of TNT and tetryl, and it may be added that later routine tests on semi-works product indicated a normal stability somewhat better than tetryl of service quality.

<sup>1</sup> *J. Am. Chem. Soc.*, 30 (1908), 271.

#### SENSITIVENESS OF HEXANITRODIPHENYLAMINE TO DETONATION

The subject of sensitiveness to detonation has fortunately been more thoroughly studied in the past than has the subject of stability, and a number of recognized methods are at hand for this test.

Probably the most reliable of these methods is that in which determination is made of the minimum weight of priming charge to insure complete detonation.<sup>1</sup> This test consists essentially of firing, with varying weights of fulminate-potassium chlorate priming mixture, a series of blasting caps loaded with equal weights of the material to be tested. The lead plate test described by Brunswig<sup>2</sup> was used as an index to completeness of detonation.

The minimum priming charges for complete detonation of the several explosives compared were:

	Gram
Hexil.....	0.1800
Tetryl.....	0.2000
TNA.....	0.2000
TNT.....	0.2500

These tests showed therefore that hexil is slightly more sensitive to detonation than tetryl and TNA, and in addition indicated that the efficiency of hexil is practically the same as for tetryl and TNA, inasmuch as the perforations in the lead plates from all three compounds were comparable.

#### SENSITIVENESS TO IMPACT

The foregoing test for sensitiveness to detonation was supplemented by test for sensitiveness to impact and friction in order to gain an idea as to the safety of handling the material. Sensitiveness to impact was determined by the so-called drop test, in which 0.2 g. of explosive, placed on an anvil between two plungers, is subjected to the fall of a 20-kg. weight through a measured distance, the sensitiveness being expressed in terms of maximum height of drop at which no detonation occurred.

In this test, hexil gave no detonation at 5 in., while at the same height tetryl gave one detonation out of 10 trials, TNA four detonations out of 10 trials, and TNT no detonations. Hexil showed detonation at 6-in. drop, but TNT gave none until 25 in. drop was reached. It appears from this that hexil is somewhat less sensitive to shock than tetryl and TNA, and is possibly safer to handle.

#### SENSITIVENESS TO FRICTION

The sensitiveness to friction was studied in a pendulum friction apparatus such as was developed by the Bureau of Mines.<sup>3</sup> In this test 7 g. of explosive are placed on a bed plate and subjected to the friction of a 20-kg. weight attached to a pendulum swinging from a measured vertical height. Using a steel bed plate and a steel shoe, the pendulum falling from 1.5 meters vertical height and swinging 19 times before coming to rest, hexil, TNT, and tetryl gave no evidence of either local or general detonation. TNA, however, gave some local detonations.

<sup>1</sup> Bureau of Mines, *Technical Papers* 125 and 145.

<sup>2</sup> Brunswig, Monroe & Kibler, "Explosives," p. 112.

<sup>3</sup> Bureau of Mines, *Bulletin* 66, 15.



## RIFLE BULLET TEST

As a measure of sensitiveness to impact and friction combined, the rifle bullet test is recognized as most severe. This test consists of packing about 1 lb. of the explosive to be tested into a 3.5-in. cube box of cardboard or tin, and firing into it from a distance of 30 yards with standard U. S. Army rifle (New Springfield). With hexil no detonations were obtained with cardboard boxes, but with tin boxes 7 detonations and one failure were obtained. With TNT no detonations were obtained with cardboard boxes. With tin boxes, the first and second hit in every case set fire to the TNT and the third hit gave a detonation. With tetryl and TNA detonations were obtained with both cardboard and tin boxes.

The preceding results on sensitiveness appeared to indicate that hexil is as sensitive as TNA and tetryl to the action of a primer, but with regard to mechanical shock or friction is more in a class with TNT and is therefore safer to handle.

## EXPLOSIVE POWER OF HEXANITRODIPHENYLAMINE

The first tests which are usually applied in a determination of the explosive power of any compound are the ballistic mortar test and the determination of velocity. The ballistic mortar test<sup>1</sup> is widely used and its details are well known. Briefly, the strength is determined in terms of some well-known explosive, for example, TNT, by firing such weights of the two explosives in the ballistic mortar as to give equal swings of the pendulum. Taking the strength of TNT as 10, the following results were obtained:

TNT.....	10.0
Hexil.....	11.1
TNA.....	12.1
Tetryl.....	12.1

The strength of hexil is therefore midway between that of TNT and tetryl.

A determination of the velocity of detonation of hexil, made by the method of Dautriche,<sup>2</sup> showed a velocity of 6898 m/sec. at 1.58 density, and 7150 m/sec. at 1.67 density.

Similar figures from Marshall<sup>3</sup> for tetryl are:

Density	Velocity
1.53	7075 m/sec.
1.57	7155 m/sec.
1.63	7215 m/sec.

and for TNT, density 1.59 and velocity 6649 m/sec.

It appears, therefore, that in explosive power and velocity, hexil is inferior to tetryl, but superior to TNT.

## EFFECT AS A BOOSTER

It having been demonstrated that hexil has a satisfactory sensitiveness and power, it only remains to make a final comparison of hexil and tetryl as secondary detonators in their effect on the bursting charge of a shell. An index was given to this in the lead plate

test previously described in which it was found that while somewhat more sensitive than tetryl, hexil did not quite come up to tetryl as a booster material.

More conclusive results were given by a series of tests of the ability of tetryl and hexil, respectively, to detonate insensitive mixtures of TNT and iron oxide. In this test blasting caps were made up of 6.5 grains secondary charge (tetryl or hexil) and 5.3 grains primer (90-10 fulminate-chlorate mixture) and these caps used in firing a series of dynamite cartridges in which different mixtures of TNT and iron oxide had been handpacked. It was found that tetryl would give satisfactory detonation of a mixture containing 14 per cent iron oxide, while the limiting mixture for detonation with hexil contained only 11 per cent iron oxide. While not equal to tetryl as a booster explosive, therefore, hexil shows to good advantage.

A final test of effect as a booster was made in comparison with other booster materials by fragmentation tests on a number of 3-in. base detonating shells.

In this test the bursting charges of the shells consisted of cast refined TNT. Five each of the boosters were loaded with tetryl and TNT and 10 each with hexil and TNA as secondary detonators. The fragmentations were made by dropping the shells from a height of 25 ft. into a steel bomb-proof chamber, the detonation being produced by the striking of the shell on a heavy iron anvil. The fragments were then collected, weighed to obtain percentage of recovery, sorted by sieving, and counted. The average results obtained were as follows:

BOOSTER MATERIAL	TETRYL	TNT	TNA	HEXIL
Wt. booster charge, grams.....	28	27.5	27.5	27.5
Wt. shell charge, grams.....	478	480	446	450
Wt. of metal in shell, grams.....	6275	6261	6126	6156
Wt. of material recovered, grams..	6108	6116	6096	6112
Per cent of material recovered,				
total.....	97.5	97.8	99.5	99.3
Number of fragments on 2 mesh..	178	161	188	179
2-4 mesh.....	1824	1679	1781	1651
4-6 mesh.....	2002	1840	1969	1830

From these results it appears that hexil is not so good as tetryl or TNA as a booster material, and is more nearly comparable with refined TNT for this purpose. It does, however, give a satisfactory order of detonation.

In connection with the use of hexil as a booster it is interesting to make note of the densities given by the compressed material. It was found impossible to make satisfactory blocks by the compression of crystalline hexil alone, but by the addition of 1 to 2 per cent of stearic acid excellent blocks resulted.

Using one per cent stearic acid as a binder, the results were as follows, the absolute density of crystalline hexil being 1.653:

PRESSURE Lbs. per sq. in.	DENSITY
5,000	1.43
10,000	1.56
15,000	1.59
20,000	1.60

<sup>1</sup> Comey and Holmes, *8th Intern. Congr. Applied Chem.*, 25, 209.

<sup>2</sup> *Compt. rend.*, 143 (1906), 641.

<sup>3</sup> "Explosives," 1915, 403.



The best blocks were given by 10,000 lbs. and this was adopted as the standard pressure for preparing pellets for boosters.

#### CONCLUSIONS

From the work which has been outlined it appears that the following conclusions were justified:

(1) Hexil as a booster material is superior to TNT, but somewhat inferior to tetryl and TNA.

(2) It is extremely stable and is safer to handle than either tetryl or TNA, and makes a satisfactory booster.

(3) It can be manufactured by a simple process from sources which would not conflict with TNT manufacture, and because of the excellent yields obtained, and the cheapness of intermediates for it, its material cost should be less than for either tetryl or TNA.

(4) On account of the simplicity of the process, the installation of a plant for its manufacture would be less expensive than for an extension of the manufacturing facilities for either tetryl or TNA.

(5) On account of the simplicity of operating methods, labor costs would be less than for either of the other materials.

### ACIDITY AND ACIDIMETRY OF SOILS.<sup>1</sup> I—STUDIES OF THE HOPKINS AND PETTIT METHOD FOR DETERMINING SOIL ACIDITY

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The Hopkins and Pettit method of determining soil acidity<sup>2</sup> proposed in 1902 is essentially as follows: 100 g. of soil are shaken in a bottle of 400 cc. capacity with 250 cc. of 5 per cent commercial common salt solution for 3 hrs. 125 cc. of the clear liquid are taken off, boiled to expel carbon dioxide, and titrated using phenolphthalein as an indicator. The results are multiplied by 3 as a factor to determine the total amount of base required. Later<sup>3</sup> a normal solution of potassium nitrate was substituted for the 5 per cent commercial common salt and the factor 2.5 recommended. The modified method is still the provisional method of the A. O. A. C. for determining the acidity of soils.

Veitch<sup>4</sup> criticizes the Hopkins method upon the grounds that it indicates only the apparent need for lime or the most urgent need, and claims further that the acidity shown by this method is largely due to aluminates. He also notes that there is a great discrepancy between the Hopkins method and that proposed by himself<sup>5</sup> upon soils high in organic matter.

<sup>1</sup> From a thesis submitted to the faculty of the University of Illinois in partial fulfillment of the requirements for the degree of Doctor of Philosophy. Acknowledgment is made of many helpful suggestions and criticisms from Prof. C. G. Hopkins and Prof. A. H. Noyes.

<sup>2</sup> Nineteenth Annual Proceedings, O. A. C., U. S. Dept. of Agr., Bureau of Chemistry, *Bulletin* 73 (1902), 114.

<sup>3</sup> U. S. Dept. of Agr., Bureau of Chemistry, *Bulletin* 107 (1908), 20; Hopkins, "Soil Fertility and Permanent Agriculture," 1910, 566.

<sup>4</sup> *J. Am. Chem. Soc.*, 26 (1914), 637.

<sup>5</sup> *Ibid.*, 24 (1902), 1120.

Harris<sup>1</sup> claims that the acidity shown by this method is due to selective ion absorption by the soil colloids, basing his views upon the fact that the acidity shown by the extract is dependent upon the character of the salts used. Freer<sup>2</sup> also holds this view. Truog<sup>3</sup> strenuously combats the theory of colloidal absorption and brings evidence to support the view of Hopkins that the reaction is one of double decomposition between the acids or acid salts in the soil and the neutral salt solution. Parker<sup>4</sup> concluded from analysis of extracts prepared by treating soils with potassium chloride and potassium acetate that the base was absorbed to a little greater extent than it was liberated by the soil and that the excess of the anion should be accounted for by the presence of the corresponding acid. Brogue<sup>5</sup> states that it has been repeatedly proven that the base liberated by the soil is usually not merely equivalent to the base absorbed from the solution. Sullivan,<sup>6</sup> Morse and Curry,<sup>7</sup> Abbott, Conn and Smalley,<sup>8</sup> Ruprecht,<sup>9</sup> and others have noted the presence of aluminum and iron in salt extracts from acid soils. Rice<sup>10</sup> concludes from hydrogen-ion concentration studies upon 31 soils using the indicator method of Sørensen<sup>11</sup> that when so-called acid soils are shaken with salt solutions part of the cation of the salt is absorbed and an equivalent quantity of the base from the soil is given up to the solution.

It was to test the above points that the following investigations were made.

#### EXPERIMENTAL

Harris obtained different lime requirements for soils by repeated shaking with different salt solutions. These experiments were repeated in this laboratory using yellow-gray silt loam, and similar differences were obtained as was reported by Harris for different salts. As Hopkins claims that the reaction between the neutral salt solution and the soil is one of equilibrium, the end reaction would be practically impossible to realize by such a treatment. To overcome the objections which would arise from the above method provisions were made for forcing the salt solutions through the soil, so that the soil particles would be continually bathed by fresh solutions.

Twenty grams of yellow-gray silt loam<sup>12</sup> were placed upon a dry filter paper and the salt solution was allowed to filter through. The filtrate was boiled and treated with 0.04 *N* potassium hydroxide at room temperature using phenolphthalein as an indicator, with the results shown in Table I.

<sup>1</sup> Michigan Agr. College and Station, *Bulletin* 19 (1914).

<sup>2</sup> Penn. Dept. Agr., *Bulletin* 261 (1915), 106.

<sup>3</sup> *J. Phys. Chem.*, 20 (1916), 157.

<sup>4</sup> THIS JOURNAL, 6 (1914), 831.

<sup>5</sup> *J. Phys. Chem.*, 19 (1915), 665.

<sup>6</sup> U. S. Geol. Survey, *Bulletin* 312 (1907).

<sup>7</sup> New Hampshire Agr. Station, *Report* 1906-08, 271.

<sup>8</sup> Indiana Agr. Expt. Station, *Bulletin* 170 (1913).

<sup>9</sup> Mass. Agr. Expt. Station, *Bulletin* 161 (1915).

<sup>10</sup> *J. Phys. Chem.*, 20 (1916), 214.

<sup>11</sup> *Biochem. J.*, 21 (1909), 131; Walpole, *Biochem.*, 5 (1911), 207; 8 (1914), 628.

<sup>12</sup> Sample No. 1. Subsoil from Southern Illinois. Lime requirement: Hopkins Method 4.2T., Veitch Method 5.6T., per acre of soil of 2,000,000 lbs.

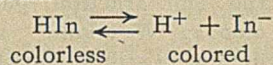


TABLE I—ACIDITY OF DIFFERENT FRACTIONS OF VARIOUS SALT SOLUTIONS FILTERED THROUGH AN ACID SOIL

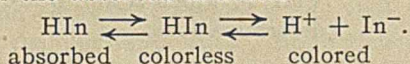
Salt Used	Cc. 0.04 N KOH Required for—				Total	Equivalent to T. CaCO <sub>3</sub>
	100 cc.	250 cc.	250 cc.	250 cc.		
N KNO <sub>3</sub> .....	36.40	2.8	1.2	0.5	40.5	4.05
N KCl.....	35.95	4.3	2.1	0.5	39.95	3.99
N NaNO <sub>2</sub> ....	26.50	9.2	1.8	1.4	38.9	3.89
N NaCl.....	31.20	6.4	1.4	..	39.0	3.90
N CaCl <sub>2</sub> .....	31.40	5.4	1.3	0.5	38.6	3.86

The greatest difference shown is 0.19 T., calculated as calcium carbonate, which may easily be accounted for by errors in reading the end-point. It will be noted that the acidity of the sodium nitrate extract was quite marked even after 600 cc. had filtered through, while the first 100 cc. showed the lowest acidity. That none of the extractions were carried to completion is evident, but all, with the possible exception of that with sodium nitrate, were carried to a point beyond which it was impossible to measure the acidity with any degree of accuracy by the ordinary indicator methods. The calcium salt extract would be expected to show a slower reaction after the first surface reaction because of the greater insolubility of calcium compounds which would be formed upon the surface of the soil grains.

INDICATOR EFFECTS—In the first two series of extracts, a precipitate which had a rather marked effect upon the indicator was always formed in considerable quantity. The pink color produced by the addition of a slight excess of base disappeared after a time even when the titrating flask was tightly stoppered. By the further addition of base the color could be brought back. The end-point is also markedly influenced by the amount of indicator present. It is quite apparent that the indicator is absorbed to a marked extent by the precipitate, and instead of the simple equilibrium



there must be taken into consideration the equilibrium with the absorbed indicator



Two equal quantities of a potassium salt extract of an acid soil gave readings, as shown in Table II, which clearly indicate the variation in results which may be obtained by using different amounts of indicator.

TABLE II—EFFECT OF DIFFERENT AMOUNTS OF INDICATOR UPON TITRATION END-POINT

Phenolphthalein Used Drops	0.04 N KOH Required Cc.
4	62.7
10	61.1

In several instances a point was reached which showed no visible color change with four or five drops of indicator, while upon the addition of larger quantities a marked color change was observed. To overcome as far as possible the variation due to the indicator the same quantity was used in each case unless otherwise stated.

EFFECT OF TEMPERATURE UPON TITRATION—The temperature at which the titration is carried out was found to produce an effect which is shown in Table III. Two equal quantities of potassium nitrate extract were titrated with 0.04 N potassium hydroxide

using the same quantity of phenolphthalein as an indicator. The only difference between the duplicates was that of temperature.

TABLE III—EFFECT OF TEMPERATURE UPON TITRATION END-POINT

Temp. ° C.	Cc. KOH
22	16.7
85	19.2

EFFECT OF TEMPERATURE UPON THE AMOUNT OF ACIDITY SHOWN—If the Hopkins method is a measure of the colloidal adsorption for the base by a soil as maintained by Harris<sup>1</sup> there should be a temperature effect which could be measured. Travers<sup>2</sup> has shown that the adsorption of carbon dioxide by charcoal decreases markedly with rise in temperature, and we may expect a similar change to be shown by soils in contact with neutral salt solutions.

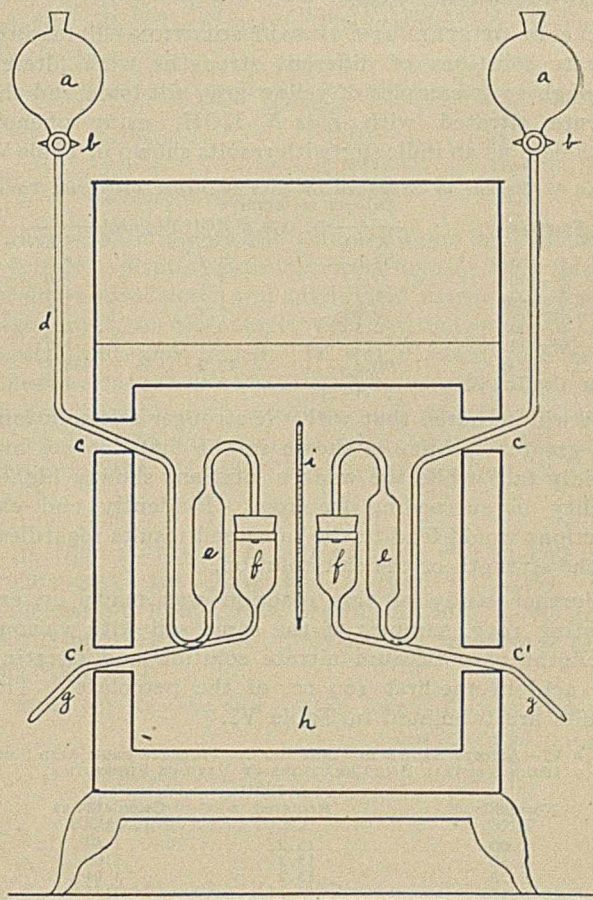


FIG. 1

To test this theory an apparatus, shown in Fig. 1, was arranged in a constant temperature electrical oven of the Freas type. The apparatus was arranged in duplicate. *a* is the receptacle for the neutral salt solution, *b* a stopcock for regulating the flow of the neutral salt through tube *d* which passes through the ventilating openings *c* provided by the manufacturers in the stock oven. The bulb *e* serves to bring the salt solution to the temperature of the oven before it runs into the receptacle *f* which contains the soil under investigation. The filtrate passes out of the apparatus through tube *g*, through ventilating openings in the oven at *c'*, and is caught in measuring flasks;

<sup>1</sup> *Loc. cit.*<sup>2</sup> *Proc. Roy. Soc. London*, 74 (1904), 126.



*i* is a thermometer placed in the center of the oven for noting the temperature.

Twenty grams of soil were placed in the receptacle, normal potassium nitrate solution allowed to percolate through, and titrated at room temperature with results as shown in Table IV.

TABLE IV—EFFECT OF TEMPERATURE UPON REACTION BETWEEN ACID SOIL AND NEUTRAL SALT SOLUTION

PERCOLATE Cc.	TEMPERATURE			
	90° ± 1°		25° ± 1°	
	0.04 N KOH Cc.	CaCO <sub>3</sub> T.	0.04 N KOH Cc.	CaCO <sub>3</sub> T.
100	37.85	3.785	36.4	3.64
400	3.15	0.315	3.0	0.30
TOTAL 500	41.00	4.1	39.4	3.94

The temperature effect is very slight, and certainly does not indicate colloidal adsorption, unless this may be an exceptional case.

EFFECT OF STRENGTH OF SALT SOLUTION—Potassium nitrate solutions of different strengths were filtered through 20 g. samples of yellow-gray silt loam and the filtrate titrated with 0.04 N KOH, using phenolphthalein as an indicator with results shown in Table V.

TABLE V—EFFECT OF STRENGTH OF SALT SOLUTION UPON THE TOTAL AMOUNT OF ACIDITY

Fractions Cc.	Cc. 0.04 N KOH Required—		
	N KNO <sub>3</sub>	0.5 N KNO <sub>3</sub>	0.1 N KNO <sub>3</sub>
1—100	35.0	33.3	18.3
2—100	3.5	4.7	7.6
3—100	1.2	1.5	4.3
4—100	0.8	0.9	3.7
5—100	0.5	0.6	3.3
6—100	..	..	1.6
TOTAL.....	40.8	41.0	38.8
as T. CaCO <sub>3</sub> .....	4.08	4.10	3.88

It will be noted that with the stronger salt solutions the greatest acidity is shown in the first 100 cc. but rapidly falls while the weaker solutions show a higher acidity in succeeding fractions. Evidently end extractions would lead to the same end results regardless of the strength of the salt solution.

Further study of this reaction was made by extracting 10 g. samples of the same soil with various strengths of potassium nitrate solution and titrating the acid in the first 100 cc. of the percolate. The results are tabulated in Table VI.

TABLE VI—ACIDITY SHOWN IN FIRST 100 CC. FILTRATE FROM ACID SOIL, USING NEUTRAL SALT SOLUTIONS OF VARIOUS STRENGTHS

Normality of KNO <sub>3</sub>	0.04 N KOH	
	Required Cc.	Calculated as T. CaCO <sub>3</sub>
1.00	15.2	3.40
0.50	15.3	3.06
0.25	15.2	3.04
0.125	13.25	2.65
0.0625	8.00	1.60
0.0312	4.10	0.82
0.0156	1.95	0.39
0.0078	1.17	0.23

The acidity of the first portion of the extract increases with increase in concentration of the neutral salt solution.

TABLE VII—AMOUNTS OF LIME ABSORBED BY ACID SOIL FROM SOLUTIONS OF VARIOUS STRENGTH

Lime Added Calculated as T. CaCO <sub>3</sub>	Absorbed by Soil T. CaCO <sub>3</sub>	Left in Solution T. CaCO <sub>3</sub>	Final Normality of Solution
12	11.71	0.29	0.000285
14	13.56	0.64	0.00064
16	14.59	1.41	0.0014
20	16.75	3.25	0.0032
30	20.93	9.07	0.009
40	24.96	15.04	0.015

#### EXAMPLES OF SO-CALLED COLLOIDAL ABSORPTION

As examples of what are usually considered colloidal phenomena the following experiments are submitted.

EXPERIMENT I—20 g. of yellow-gray silt loam were shaken with various amounts of lime contained in 200 cc. of solution for 12 hrs. and aliquot parts of the clear liquid were titrated with results as shown in Table VII.

A greater portion of the lime is absorbed from the dilute solutions than from the more concentrated, thus apparently following the colloidal absorption law.

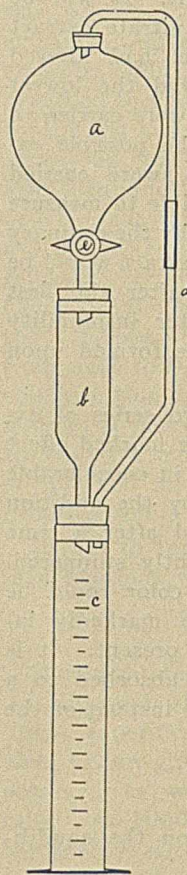


FIG. 2

EXPERIMENT II—20 g. of soil were placed in an extracting apparatus (*b*, Fig. 2), and a 0.04 N calcium hydroxide solution allowed to percolate through. In the diagram *a* is the receptacle for the base, *c* a stopcock for regulating the flow of the base into *b*, and *d* a graduated receiver connected with tube *d* which serves to equalize the pressure. The apparatus is a closed system and duplicate results were easily obtained. 237.2 cc. of filtrate<sup>1</sup> passed through before a pink color could be detected, with phenolphthalein as an indicator, representing a lime adsorption at this point of 23.7 T. as calcium carbonate. At the close of the experiment 537.6 cc. of filtrate had percolated through, the last 50 cc. being 0.0285 N base, while the soil had absorbed a total of 35.28 T. of lime as calcium carbonate. 15.82 T. of lime were washed out by the first 700 cc. of distilled water, the end fraction passing through 0.00242 N alkali. 19.46 tons of lime were still left in the soil.

The details are given in Table VIII, in which A and B are duplicate determinations.

TABLE VIII—AMOUNT OF LIME ABSORBED BY ACID SOIL FROM SOLUTION OF CONSTANT STRENGTH PERCOLATING THROUGH THE SOIL

Percolate Cc.	A		
	Calculated as T. CaCO <sub>3</sub> Absorbed	Per cent Absorbed	Calculated as T. CaCO <sub>3</sub> in Percolate
236.5	23.65	100.0	0.00
27.0	2.50	92.6	0.20
28.5	1.95	68.4	0.90
45.0	1.97	43.7	2.53
51.4	1.61	31.3	3.53
48.6	0.93	19.1	3.93
50.0	1.04	20.8	3.96
50.5	1.45	28.7	3.60
TOTAL 537.5	35.10		
		B	
238.0	23.80	100.0	0.00
43.2	4.17	96.6	0.15
58.0	3.04	52.2	2.76
50.5	1.24	24.5	3.81
50.0	1.08	20.2	3.92
50.0	1.15	23.0	3.85
50.0	0.99	19.8	4.01
TOTAL 539.7	35.47		

At the completion of the experiment water was added and the first 700 cc. carried through the equivalent of 16 tons of lime as CaCO<sub>3</sub>, or nearly half the amount absorbed.

<sup>1</sup> Figures given are average of results A and B below.



EXPERIMENT III—Twenty gram samples of yellow-gray silt loam were shaken for 3 hrs. with 200 cc. of 0.04 *N* calcium hydroxide and potassium hydroxide, respectively. Since the solution containing the potassium hydroxide would not settle, 25 cc. of normal potassium nitrate were added to both the calcium and potassium hydroxide solutions, and filtered. Titrations of 100 cc. of filtrate with 0.04 *N* hydrochloric acid gave the results included in Table IX.

TABLE IX—AMOUNT OF BASE NOT ABSORBED BY AN ACID SOIL FROM SOLUTIONS OF EQUIVALENT STRENGTH

BASE USED	0.04 <i>N</i> HCl Required to Neutralize Filtrate Cc.
Ca(OH) <sub>2</sub> .....	7.7
KOH.....	31.9

As the potassium hydroxide solution was neutralized, a copious precipitate of aluminum hydroxide was formed. No precipitate was noted upon neutralizing the calcium hydroxide solution.

It would appear from this experiment that the soil has a greater absorption power for calcium than for potassium, which is not indicated by other experiments. A chemical difference in the action of the two bases seems the more simple explanation. Potassium aluminate is soluble while calcium aluminate is not. Both are unstable except in the presence of a base. Since the potassium aluminate passes into solution it is titrated above while the calcium aluminate is precipitated around the soil particles.

This apparently throws doubt upon the magnitude of the colloidal adsorption effects which may be assumed from Expts. I and II above. The probable cause would seem to be precipitation effects. Hydrolysis will account for the rapid washing out of the lime when water is added in Expt. II.

#### EXCHANGE OF BASE

There is considerable doubt whether, when an acid soil is shaken up with a neutral salt, there is a complete exchange of base. Rice<sup>1</sup> comes to the conclusion that there is an equivalent exchange and that the acidity is due to the aluminum salts. He further claims that the ordinary methods of analysis are too crude to determine this accurately. Sharp and Hoagland<sup>2</sup> by use of the hydrogen electrode show that soil acidity is due to an excess of hydrogen ions, and that the acidity is increased by the presence of certain neutral salts.

Analysis was made of the potassium nitrate extract of yellow-gray silt loam with results shown in Table X.

TABLE X—ANALYSIS OF POTASSIUM NITRATE EXTRACT OF AN ACID SOIL  
627 Cc. of 0.04 *N* Acid =

	Gram
SiO <sub>2</sub> .....	0.04741
P <sub>2</sub> O <sub>5</sub> .....	0.00576
Al <sub>2</sub> O <sub>3</sub> .....	0.38822
Fe <sub>2</sub> O <sub>3</sub> .....	Trace
CaO.....	0.06025
MnO.....	0.04839
MgO.....	0.19901

The acid combined with the alumina would be equivalent to 570 cc. of 0.04 *N* acid leaving an excess of 57 cc. to be accounted for in other ways.

<sup>1</sup> *Loc. cit.*

<sup>2</sup> *J. Agr. Res.*, [3] 7 (1916), 124.

Since, as has been shown by Blum,<sup>1</sup> alumina is completely precipitated before the hydrogen-ion concentration drops to a value of 10<sup>-7</sup> and, conversely, alumina will not pass into solution until the hydrogen-ion concentration reaches a value higher than 10<sup>-7</sup>, it is reasonable to conclude that there must be some absorption of base before the alumina will pass into solution. If this is true we must assume that the dissolution of alumina is a secondary reaction, and the above analysis certainly points in this direction.

It was noted that strongly acid extracts of this soil were highly colored with iron. To test the question of why iron is not also taken out in larger quantities by a similar secondary reaction, 100 g. samples of yellow-gray silt loam were shaken with 250 cc. of 0.04 *N* acids and a partial analysis made of 125 cc. of the filtered extracts. The results are given in Table XI.

TABLE XI—PARTIAL ANALYSIS OF WEAKLY ACID EXTRACTS OF AN ACID SOIL

ACID USED	Al <sub>2</sub> O <sub>3</sub> Gram	Fe <sub>2</sub> O <sub>3</sub>
Acetic.....	0.00383	Trace
Nitric.....	0.06913	Trace
Hydrochloric.....	0.07049	Trace

The solutions were still acid and upon neutralization white precipitates of aluminum hydroxide were formed in the hydrochloric and nitric acid extracts but none was noted in the acetic acid extract until it was neutralized and boiled. It would seem that when dilute acids are allowed to act upon an acid soil alumina is first brought into solution, and that neutral salts, when brought into contact with an acid soil, show the properties of a weak acid in this respect.

#### DIALYSIS

Normal potassium nitrate solution was shaken with yellow-gray silt loam and allowed to settle. The supernatant liquid was drawn off into a collodion bottle and subjected to dialysis with the results given in Table XII.

TABLE XII—DIALYSIS OF A NEUTRAL SALT IN CONTACT WITH AN ACID SOIL

	0.04 <i>N</i> KOH to Neutralize Cc.
1st water fraction.....	35.1
2nd water fraction.....	12.06
3rd water fraction.....	24.9
Left in flask.....	24.9
TOTAL.....	96.96

74 per cent of the titratable acid had passed through the membrane. As the liquid left in the dialyzing flask was being titrated a heavy precipitate formed, while that which passed through remained clear upon neutralization. It was evident that the acid passed through while the aluminum hydroxide did not. This, however, was to be expected, as this is one of the recognized methods for the preparation of colloidal aluminum hydroxide.<sup>2</sup>

#### DISTILLATION OF SOIL EXTRACT

Attempts were made to remove acid from potassium nitrate and chloride extracts of soil by prolonged distillation with steam but without success. Better success followed the distillation of the potassium

<sup>1</sup> *J. Am. Chem. Soc.*, 7 (1916), 1282.

<sup>2</sup> *Graham, Ann.*, 121 (1866), 41.



acetate extract of the yellow-gray silt loam as shown in Table XIII.

TABLE XIII—DISTILLATION OF A POTASSIUM ACETATE EXTRACT OF AN ACID SOIL, AND OF STOCK SOLUTION OF POTASSIUM ACETATE (Acidity in Terms of 0.04 N Base)

Potassium Acetate Extract		Potassium Acetate Stock Solution	
Distillate	Residue	Distillate	Residue
72.9 Cc.	21.9 Cc.	8.6 Cc.	1.95 Cc.

Phenolphthalein was used as an indicator. This experiment shows the presence of appreciable quantities of acetic acid in the soil extract. About three-fourths of the acid shown by the extract was distilled over.

Walter Crum<sup>1</sup> prepared colloidal aluminum hydroxide by separating the acetic acid by heating, but as there were only traces of aluminum salts carried by the potassium acetate extract<sup>2</sup> it can hardly be conceived that the phenomena may be accounted for by the presence of these salts, but rather that there is an excess of acid.

#### COMPARISON OF CATION AND ANION ABSORPTION

To compare the cation and anion absorption of the yellow-gray silt loam from neutral salt solution a 0.0358 N solution of calcium chloride was allowed to percolate through 20 g. of the soil in the apparatus shown in Fig. 2. The extract was analyzed for calcium and chlorine. The calcium was determined in an aliquot portion of the extract which had been freed from iron and aluminum by first precipitating as the oxalate and titrating the precipitate with a standard potassium permanganate in the presence of dilute sulfuric acid. The chlorine was determined by the Volhard method<sup>3</sup> using 0.04 N solutions. The results are tabulated in Table XIV.

TABLE XIV—ANALYSIS OF FRACTIONS OF EXTRACT OF AN ACID SOIL BY A NEUTRAL SALT SOLUTION TO DETERMINE CATION AND ANION ABSORPTION

Extract Fractions Cc.	Calcium Normality	Chlorine Normality	Acid Normality
1—50	0.0180	0.0348	0.0046
2—50	0.0314	0.0358	0.0037
3—50	0.0332	0.0358	0.0028
4—50	0.0335	0.0358	0.0022

The cation is absorbed to a measurable extent, for which the change in acidity fails to account. There must, therefore, have been an exchange of base. This confirms the fact that there is a basic exchange regardless of the neutral salt used. (See analysis of potassium nitrate extract.) The anion is absorbed little or not at all. The slight absorption shown in the first 50 cc. of extract is probably due to the wetting of the particles and to a slight dilution of the extract by moisture in the air-dried soil. A small amount of precipitate was formed in each case upon neutralizing the extract.

#### SUMMARY

1—When normal solutions of potassium nitrate, potassium chloride, sodium nitrate, sodium chloride, and calcium chloride were percolated through an acid soil all gave the same end titrations, using phenolphthalein as an indicator. This corroborates Hopkins' statements.

<sup>1</sup> *Ann.*, 89 (1854), 168.

<sup>2</sup> Compare Conner, *This Journal*, 8 (1916), 35.

<sup>3</sup> *Ann.*, 190, 1.

2—The acidity of the salt extract of an acid soil is independent of the temperature within the range from 25° to 90° C.

3—The precipitate formed in titrating the soil extract obtained by the Hopkins method absorbs the indicator to a marked extent. The end result depends upon the temperature, time, and amount of indicator used.

4—The acidity of the first portions of the neutral salt extracts of an acid soil increases with increase in concentration of the neutral salts.

5—The difference in absorption of calcium and potassium from solutions of their bases by an acid soil may be accounted for by precipitation effects.

6—There is a marked basic exchange when a neutral salt solution is added to an acid soil, by which alumina is carried into solution. This, however, does not account for the total acidity of the solution.

7—When acid soil is extracted with potassium acetate solution, a portion of acetic acid may be distilled off from the extract, showing the presence of free acid.

8—Exchange of acid radicals when an acid soil is treated with a neutral salt solution was not noted.

#### THE USE OF CUPFERRON IN QUANTITATIVE ANALYSIS<sup>1</sup>

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BUREAU OF STANDARDS, WASHINGTON, D. C.

Received September 8, 1919

#### INTRODUCTION

The increasing use of cupferron (the ammonium salt of nitrosophenylhydroxylamine,  $C_6H_5.N.NO.ONH_4$ ) for the determination of zirconium in its ores and metallurgical products, as well as for minor purposes such as the separation of iron and titanium from manganese and aluminum in limestone analysis,<sup>2</sup> and other determinations for which it has been recommended, makes a review of the possibilities and limitations of this reagent highly desirable. This paper presents a review of the literature dealing with the use of cupferron as a quantitative precipitant, and gives the results of many tests which were performed at this Bureau in connection with an attempt to adapt the cupferron method to the determination of zirconium in its ores and metallurgical products.

#### GENERAL PRINCIPLES

Cupferron precipitates are salts in which the ammonium radical of the reagent has been replaced by metals. Precipitations are performed in cold solutions containing free mineral or organic acids. Cold solutions must be employed to prevent decomposition of the reagent into various organic substances, such as nitrobenzene, and the temperature of precipitation is usually specified as "cooled in ice water." A 6 per cent water solution of the reagent is used and complete precipitation is indicated by the formation of a temporary flash of a fine, white precipitate which redissolves, as contrasted with the flocculent insoluble cupferron

<sup>1</sup> Published by permission of the Director of the Bureau of Standards.

<sup>2</sup> Private communication from J. A. Holladay, Electro-Metallurgical Co., Niagara Falls, N. Y.



precipitates. Precipitation is usually immediate and filtration is carried on through paper and cone by means of gentle suction. The wash waters are always cold and range from 5 per cent (by volume) ammonium hydroxide for iron precipitates to 10 per cent (by volume) hydrochloric acid for titanium and zirconium precipitates. Ignitions of cupferron precipitates are carried on very carefully in the early stages owing to excessive liquefaction when wet precipitates are ignited, and to the heavy liberation of gaseous products from dried precipitates. The ordinary procedure calls for ignition to oxides, although some variations will be noted in certain methods reviewed below.

#### HISTORICAL

The use of cupferron as a quantitative reagent was recommended originally by Baudisch<sup>1</sup> who showed its application to the determination of iron in brown iron ore and of iron and copper in nickel ore. He also described the properties and behavior of the reagent, and enumerated instances where it could be used to advantage. Its uses have since been studied by many investigators and it has been proposed as a quantitative precipitant under varying conditions (often loosely defined in the earlier papers) for copper, iron, titanium, zirconium, thorium, and vanadium. For convenience, the work done on each of these determinations will be given below under separate headings.

Tables of results will not be given unless authors have given definite conditions for precipitation.

#### QUANTITATIVE DETERMINATIONS WITH CUPFERRON

##### COPPER

(1) HISTORICAL—Biltz and Hödtke<sup>2</sup> found that complete precipitation of copper takes place in weak solutions of hydrochloric acid and in solutions of acetic acid containing sodium acetate, but not in the presence of excessive amounts of mineral acids. They washed the precipitate first with water, then with 1 per cent sodium carbonate solution to remove excess reagent, finally with water, and then ignited to oxide. Their data demonstrated a very great accuracy for the method.

Hanus and Arn. Soukup<sup>3</sup> experienced considerable difficulty in precipitating copper from dilute sulfuric acid solutions but obtained good results when filtration on a gooch immediately followed precipitation. The precipitate was washed with cold water, dried, ignited to oxide, and reduced with methyl alcohol.

Baudisch<sup>4</sup> in a second paper dealt with the properties of cupferron and gave a method for its preparation.

Fresenius<sup>5</sup> demonstrated that copper can be quantitatively precipitated from acetic acid solution containing ammonium acetate. The precipitate was washed with 1 per cent sodium carbonate solution, dried, ashed, ignited with sulfur in a Rose crucible in an atmosphere of hydrogen, and weighed as cuprous sulfide.

<sup>1</sup> *Chem.-Ztg.*, **33** (1909), 1298.

<sup>2</sup> *Z. anorg. Chem.*, **66** (1910), 426.

<sup>3</sup> *Ibid.*, **68** (1910), 52.

<sup>4</sup> *Chem.-Ztg.*, **35** (1911), 913.

<sup>5</sup> *Z. anal. Chem.*, **50** (1911), 35.

Weber<sup>1</sup> dealt mainly with the technique of the use of cupferron and cited the work of Biltz and Hödtke and of Nissenson.<sup>2</sup>

Baudisch and King<sup>3</sup> reviewed the desirable features of the cupferron method, calling attention to the fact that copper can be quantitatively determined in mineral or acetic acids, and giving directions for the preparation of cupferron.

(2) FAVORABLE PROCEDURE FOR PRECIPITATION—For the determination of copper, high mineral acidity is objectionable, but weak solutions of hydrochloric or sulfuric acids are permissible, while acetic acid in sodium or ammonium acetate solutions gives the best results. Cold water is the most satisfactory washing medium. The ignition is usually carried to oxide, although some authors prefer to transform the oxide to cuprous sulfide or metallic copper.

(3) SEPARATIONS WHICH HAVE BEEN ATTEMPTED. (a) *Copper from Zinc*—Biltz and Hödtke demonstrated that a very satisfactory separation of copper from zinc could be accomplished in acetic acid solution. Hanus and Soukup tried the separation in weak sulfuric acid solution and obtained results which were not as satisfactory as those of Biltz and Hödtke.

(b) *Copper from Cadmium*—Biltz and Hödtke obtained good separation in weak hydrochloric acid but not in acetic acid. Hanus and Soukup performed the separation in weak sulfuric acid solution.

(c) *Copper from Iron*—Biltz and Hödtke found that a good separation could be secured if the copper-iron cupferron precipitate obtained in weak acid solutions was washed with ammonium hydroxide, which dissolved the copper precipitate and not the iron compound.

Fresenius obtained an excellent separation by precipitation in acetic acid solution containing ammonium acetate followed by washing with ammonium hydroxide.

(4) INTERFERING SUBSTANCES—Biltz and Hödtke pointed out that lead, silver, mercury, and tin interfere. Fresenius added bismuth to the list.

To the above list should be added iron, titanium, zirconium, cerium, thorium, tungsten, vanadium, and undoubtedly several other elements.

The interference of iron, titanium, zirconium, cerium, and thorium, and possibly lead, mercury, tin, and bismuth can be avoided by treating the cupferron precipitate with ammonia and reprecipitating the dissolved copper by the addition of weak acid.

(5) SUMMARY—The use of cupferron for the determination of copper is of classical interest and not of any practical importance.

#### IRON

(1) HISTORICAL—Baudisch<sup>4</sup> pointed out that iron was precipitated by cupferron as reddish brown flocks, soluble in ether and in acetone.

Biltz and Hödtke<sup>5</sup> showed that iron could be precip-

<sup>1</sup> *Z. anal. Chem.*, **50** (1911), 50.

<sup>2</sup> *Z. angew. Chem.*, **23** (1911), 969.

<sup>3</sup> *THIS JOURNAL*, **3** (1911), 629.

<sup>4</sup> *Chem.-Ztg.*, **33** (1909), 1298.

<sup>5</sup> *Loc. cit.*



itated with copper and quantitatively separated from it by the use of an ammonium hydroxide wash. Fresenius<sup>1</sup> used Biltz and Hödtke's method with good results in hydrochloric, sulfuric, and acetic acid solutions. He cited practical tests of the method with excellent results in the analysis of a manganese ore and the analysis of a ferromanganese.

Nissenson<sup>1</sup> used the reagent for the separation of iron from nickel and cobalt in speiss.

Ferrari<sup>2</sup> published a paper on the rapid determination of iron in the presence of soluble organic substances. He showed that quantitative determinations of iron in this class of material can be carried out in 1.5 hrs. by direct precipitation with cupferron of the chlorine-treated hydrochloric acid solutions of the organic material, followed by filtration of the precipitate and ignition to  $\text{Fe}_2\text{O}_3$ . He stated that such elements as copper, silver, lead, mercury, tin, bismuth, titanium, and zirconium interfere with the determination.

(2) FAVORABLE PROCEDURE FOR PRECIPITATION—Iron can be precipitated from dilute solutions of the mineral acids, hydrochloric, sulfuric, and nitric, and from acetic acid. Complete precipitation has been obtained at this Bureau in solutions containing 20 per cent by volume of sulfuric acid. The precipitate may be washed with cold 2 *N* hydrochloric acid, ammonium hydroxide, or water. Ignition is carried to the oxide.

(3) SEPARATIONS WHICH HAVE BEEN ATTEMPTED. (a) *Iron from Copper*—Biltz and Hödtke obtained perfect separations from copper. See COPPER 3c.

Fresenius demonstrated the exactness of this method. See COPPER 3c.

(b) *Iron from Manganese*—Fresenius carried out perfect separations of these elements in hydrochloric acid solution.

(c) *Iron from Zinc*—Fresenius showed that complete separation can be had in dilute sulfuric acid solution.

(d) *Iron from Aluminum and Chromium*—Biltz and Hödtke performed this separation in mineral acid solution, and washed the precipitate with 2 *N* hydrochloric acid, water, ammonium hydroxide, and finally water. Fresenius showed that the separation of iron from aluminum and chromium is complete in dilute hydrochloric acid solution.

(e) *Iron from Nickel and Cobalt*—Baudisch showed that this separation was satisfactory in hydrochloric acid solution. Biltz and Hödtke also obtained a good separation in mineral acids. Fresenius performed perfect separations of iron from nickel and cobalt in dilute sulfuric acid solution.

(f) *Iron from the Alkalies and Alkaline Earths*—Fresenius showed that no trouble was experienced in carrying out these separations in appropriate dilute mineral acids.

(g) *Iron from Phosphorus*—Fresenius also demonstrated the fact that iron can be completely separated from phosphorus in dilute hydrochloric acid solutions.

<sup>1</sup> *Loc. cit.*

<sup>2</sup> *Ann. chim. applicata*, 4 (1915), 341.

(4) INTERFERING SUBSTANCES—Copper, lead, silver, mercury, tin, and bismuth interfere. This, however, is not serious, since their prior separation by means of hydrogen sulfide is an easy matter.

Titanium, zirconium, cerium, thorium, tungsten, vanadium, and undoubtedly several other elements of this analytical group also interfere.

(5) SUMMARY—The determination of iron by the cupferron method does not offer any advantages over ordinary established procedures. This reaction may, however, be of value in certain group separations such as the separation of iron and titanium from aluminum and manganese in limestone analysis as proposed and used by Mr. Holladay.<sup>1</sup>

#### TITANIUM

(1) HISTORICAL—Schroeder<sup>2</sup> found that titanium could be quantitatively precipitated from acid solutions. Bellucci and Grassi<sup>3</sup> showed that cupferron quantitatively precipitated the yellow compound  $(\text{NO.NC}_6\text{H}_5\text{O})_4\text{Ti}$ , which they ignited to  $\text{TiO}_2$ .

Thornton<sup>4</sup> demonstrated that this method was one of high accuracy in a solution containing varying amounts of sulfuric acid and tartaric acid as is shown by Table I.

EXPT. No.	TiO <sub>2</sub> Used G.	TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> 1:1 Cc.	Tartaric Acid Used G.	Vol. of Solution Cc.
9	0.1428	0.1429	+0.0001	5	0.5	200
10	0.1066	0.1069	+0.0003	20	1.5	400
11	0.1064	0.1067	+0.0003	30	2.0	400
12	0.1064	0.1066	+0.0002	40	2.0	400

In Table II will be found the tabulation of results obtained in tests along the same line carried on at this Bureau. These show that complete precipitation can be made in solutions containing 40 per cent by volume of concentrated sulfuric acid.

TABLE II

EXPT. No.	TiO <sub>2</sub> Used G.	TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Vol.	Vol. of Solution Cc.
1	0.0999	0.0997	-0.0002	5	400
2	0.0999	0.1000	+0.0001	10	400
3	0.0999	0.0999	0.0000	15	400
4	0.0999	0.1001	+0.0002	20	400
5	0.0999	0.1002	+0.0003	40	400

Qualitative tests of the above filtrates showed no titanium.

Brown<sup>5</sup> applied Thornton and Hayden's method to the analysis of titanium and zirconium in baddeleyite and zircon with good results.

(2) FAVORABLE PROCEDURE FOR PRECIPITATION—Titanium can be quantitatively precipitated from solutions containing as much as 40 per cent sulfuric acid by volume. The presence of tartaric acid causes no trouble. Presumably titanium can also be precipitated from solutions containing considerable hydrochloric, acetic, or tartaric acids. Nitric acid in high concentrations is not permissible on account of its action on the reagent.

(3) SEPARATIONS WHICH HAVE BEEN ATTEMPTED. (a) *Titanium from Aluminum*—Bellucci and Grassi

<sup>1</sup> *Loc. cit.*

<sup>2</sup> *Z. anorg. Chem.*, 72 (1911), 89.

<sup>3</sup> *Atti accad. Lincei*, [V] 22 (1913), 30.

<sup>4</sup> *Am. J. Sci.*, 37 (1914), 173, 407.

<sup>5</sup> *J. Am. Chem. Soc.*, 39 (1917), 2358.



showed that the separation of titanium from aluminum is quantitative. This observation was confirmed by Thornton who found, however, as shown in Table III, that the separation is sharp only in highly acid solutions, and that addition of tartaric acid is desirable.

TABLE III

EXPT. No.	TiO <sub>2</sub> Used G.	Al <sub>2</sub> O <sub>3</sub> Used G.	TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> 1:1 Cc.	Tartaric Acid Used G.	Vol. of Solution Cc.
1	0.1066	0.1127	0.1179	+0.0113	5	...	200
2	0.1066	0.1127	0.1094	+0.0028	10	...	200
3	0.05715	0.1127	0.0590	+0.0018	5	1.2	200
4	0.0572	0.1127	0.0577	+0.0005	10	1.2	200
5	0.0575	0.1127	0.0579	+0.0004	15	1.2	200
6	0.0573	0.1127	0.0575	+0.0002	20	1.2	200
7	0.1067	0.1127	0.1072	+0.0005	20	1.5	400
8	0.1068	0.1127	0.1070	+0.0002	20	1.5	400

(b) *Titanium from Iron*—Thornton accomplished this separation by treating an acid tartaric acid solution of the two with hydrogen sulfide, adding ammonium hydroxide after the iron was reduced, continuing the addition of hydrogen sulfide and finally filtering off the iron sulfide. The titanium was then determined in the acidified filtrate. Data are given in Table IV.

TABLE IV

EXPT. No.	TiO <sub>2</sub> Used G.	Fe <sub>2</sub> O <sub>3</sub> Used G.	TiO <sub>2</sub> Found G.	Error Mg.
4	0.1428	0.2267	0.1424	-0.4
5	0.1428	0.2267	0.1430	+0.2
6	0.1066	0.2267	0.1068	+0.2
7	0.1063	0.2267	0.1061	-0.2

(c) *Titanium from Phosphorus*—Thornton demonstrated that phosphoric acid was without serious effect in strongly acidified solutions, as shown in Table V.

TABLE V

EXPT. No.	TiO <sub>2</sub> Used G.	P <sub>2</sub> O <sub>5</sub> Used G.	TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> 1:1 Cc.	Tartaric Acid Used G.	Vol. of Solution Cc.
13	0.1064	0.0711	0.1071	+0.0007	20	1.5	400
14	0.1066	0.0710	0.1067	+0.0001	25	1.5	400

Experiments carried on at this Bureau dealing with the effect of phosphoric acid in cupferron precipitations of titanium and zirconium are given in Table VI.

TABLE VI

ZrO <sub>2</sub> .TiO <sub>2</sub> (1:1) Taken G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Vol.	Tar-taric Acid G.	P <sub>2</sub> O <sub>5</sub> Used G.	Weight of Ignited Oxide, G.	Error G.	Vol. Solution Cc.
0.2119	10	1.0	0.02	0.2126	+0.0007	400
0.2119	10	1.0	0.05	0.2129	+0.0010	400
0.2119	10	1.0	None	0.2117	-0.0002	400
0.2119	10	1.0	None	0.2122	+0.0003	400

The results show a contamination greater than that obtained by Thornton and this is directly attributable to zirconium, which forms more insoluble phosphates.

(d) *Titanium from Iron, Aluminum, and Phosphorus*—Thornton separated iron, first, by precipitating the reduced iron by ammonium sulfide in ammonium tartrate solution as outlined in *b*, and then proceeding as indicated in Table III. The accuracy of the method is shown in Table VII.

TABLE VII

EXPT. No.	TiO <sub>2</sub> Used G.	Fe <sub>2</sub> O <sub>3</sub> Used G.	Al <sub>2</sub> O <sub>3</sub> Used G.	P <sub>2</sub> O <sub>5</sub> Used G.	TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> 1:1 Cc.	Tar-taric Acid G.	Vol. of Solution Cc.
15	0.1065	0.2036	0.1127	0.0154	0.1070	+0.0005	30	2	400
16	0.1066	0.2036	0.1127	0.0151	0.1068	+0.0002	30	2	400
17	0.1065	0.1018	0.1127	0.0153	0.1067	+0.0002	40	2	400
18	0.1067	0.1018	0.1127	0.0153	0.1069	+0.0002	40	2	400
19	0.1066	0.2267	0.2254	0.0153	0.1069	+0.0003	40	2.5	400
20	0.1066	0.2267	0.2254	0.0153	0.1073	+0.0007	40	2.5	400

(e) *Titanium from Platinum*—Since platinum is often introduced into titanium solutions by fusion

operations, Thornton analyzed some solutions to which chlorplatinic acid had been added. The data, given in Table VIII, demonstrate that platinum introduced into solutions by fusion operations is without effect.

TABLE VIII

EXPT. No.	TiO <sub>2</sub> Used G.	Pt Used (approx.) G.	TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> 1:1 Cc.	Vol. of Solution Cc.
21	0.1067	0.01	0.1071	+0.0004	40	400
22	0.1067	0.01	0.1066	-0.0001	40	400

(f) *Titanium from Boric Acid*—Since borax was used at this Bureau for the fusion of several ores, tests were carried out which showed that boric acid was without interfering action. The data obtained are given in Table IX.

TABLE IX

EXPT. No.	ZrO <sub>2</sub> .TiO <sub>2</sub> 1:1 G.	H <sub>3</sub> BO <sub>3</sub> Taken G.	Tar-taric Acid G.	ZrO <sub>2</sub> .TiO <sub>2</sub> Found G.	Error G.	H <sub>2</sub> SO <sub>4</sub> 1:1 Cc.	Vol. of Solution Cc.
1	0.2104	0.5	1	0.2106 <sup>1</sup>	+0.0002	40	400
2	0.2104	0.5	1	0.2103 <sup>1</sup>	-0.0001	40	400

<sup>1</sup> Cupferron filtrate contained neither zirconium nor titanium.

(g) *Titanium from Alkali Salts*—Tests made at this Bureau dealing with the effect of alkali salts confirmed the observation of Thornton and Hayden<sup>1</sup> concerning their effect in zirconium precipitations, namely, that the presence of excessive alkali causes high values and the obvious remedy when they are present is to carry out a preliminary precipitation with ammonium hydroxide, followed by solution of the precipitate in acid and precipitation by cupferron.

(h) *Titanium from Silica*—Experiments performed at this Bureau are listed in Table X, which shows that the separation of titanium (and zirconium) from silica is not complete. This is not a source of trouble, for silica in the final ignited precipitate can be volatilized with hydrofluoric acid with no loss of titanium (or zirconium) provided sufficient sulfuric acid is present.

TABLE X

EXPT. No.	Approx. Amount of Soluble Silica in Solution when Treated with Cupferron G.	Weight Ignited Cupferron Ppt. ZrO <sub>2</sub> -TiO <sub>2</sub> -SiO <sub>2</sub> G.	Weight of SiO <sub>2</sub> in Precipitate G.
1	0.05	0.1651	0.0036
2	0.05	0.1978	0.0013
3	0.05	0.2107	0.0006

(i) *Titanium from Vanadium*—Tests performed at this Bureau demonstrated that it is not possible to separate titanium (and zirconium) from vanadium in either the quadrivalent or quinquivalent state. It will be noted that the separation is more nearly complete when vanadium is in the quadrivalent state, and it may be safely stated that contamination varies inversely as the acidity. Table XI gives the data obtained.

TABLE XI

EXPT. No.	ZrO <sub>2</sub> .TiO Taken G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume G.	Tar-taric Acid Taken G.	V <sub>2</sub> O <sub>5</sub> Taken G.	V <sub>2</sub> O <sub>4</sub> Taken G.	Vol. of Solution Cc.	Weight of Cupferron Ppt. G.	Error G.
1	0.2120	10	1	None	None	400	0.2117	-0.0003
2	0.2120	10	1	None	None	400	0.2122	+0.0002
3	0.2120	10	1	None	0.02	400	0.2130	+0.0010
4	0.2120	10	1	None	0.05	400	0.2161	+0.0041
5	0.2120	10	1	0.02	None	400	0.2270	+0.0150
6	0.2120	10	1	0.05	None	400	0.2489	+0.0369



The presence of vanadium in both the cupferron precipitates and in the cupferron filtrates was qualitatively established in Expts. 3, 4, 5, and 6. The use of ammonium hydroxide wash solution does not lessen the contamination.

(j) *Titanium from Uranium*—Since it was considered likely that some zirconium steels might contain uranium, tests were made at this Bureau to establish the effect of uranium in the cupferron precipitation. The data given in Table XII demonstrate that uranium does not interfere when in the hexivalent state, but does interfere in the quadrivalent state.

TABLE XII

EXPT. No.	ZrO <sub>2</sub> .TiO <sub>2</sub> Taken G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume	Tar-taric Acid G.	UO <sub>3</sub> Taken G.	UO <sub>2</sub> Taken G.	Vol. of Solution Cc.	Weight of Cupferron Ppt. G.	Error G.
1	0.2114	10	1	None	None	400	0.2114	0.0000
2	0.2114	10	1	None	0.02	400	0.2122	+0.0008
3	0.2114	10	1	None	0.05	400	0.2179	+0.0065
4	0.2114	10	1	0.02	None	400	0.2118	+0.0004
5	0.2114	10	1	0.05	None	400	0.2116	+0.0002

Qualitative tests showed the presence of uranium in both precipitates and filtrates in Expts. 2 and 3 and no uranium in the precipitates in Expts. 4 and 5.

(k) *Titanium from Tungsten*—The data given in Table XIII show that tungsten interferes seriously in the precipitation of titanium (and zirconium) by cupferron.

TABLE XIII

EXPT. No.	ZrO <sub>2</sub> .TiO <sub>2</sub> Taken G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume	Tar-taric Acid G.	WO <sub>3</sub> Taken G.	Vol. of Solution Cc.	Weight of Cupferron Ppt. G.	Error G.
1	0.2114	10	None	None	400	0.2114	0.0000
2	0.2114	5	None	0.1266	400	0.2663	+0.0519
3	0.2114	20	None	0.1286	400	0.3043	+0.0929

The ignited oxides in Expts. 2 and 3 were pale blue in color and contained tungsten. It was found that the use of an ammonium hydroxide wash solution markedly lowered the contamination but did not completely eliminate it.

(l) *Titanium from Thorium*—The experiments performed at this Bureau and given in Table XIV demonstrate that it is not possible to separate titanium (and zirconium) from thorium by the cupferron method.

TABLE XIV

EXPT. No.	ZrO <sub>2</sub> .TiO <sub>2</sub> Taken G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume	Tar-taric Acid G.	ThO <sub>2</sub> Taken G.	Vol. of Solution Cc.	Weight of Cupferron Ppt. G.	Error G.
1	0.2114	10	1	None	400	0.2116	+0.0002
2	0.2114	10	1	0.02	400	0.2181	+0.0067
3	0.2114	10	1	0.05	400	0.2288	+0.0174
4	0.2114	10	1	0.20	400	0.2734	+0.0620

(m) *Titanium from Cerium*—Quadrivalent cerium interferes markedly in a cupferron precipitation of titanium (and zirconium) while trivalent cerium interferes but slightly. Data obtained in these separations are given in Table XV.

TABLE XV

EXPT. No.	ZrO <sub>2</sub> .TiO <sub>2</sub> Taken G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume	Tar-taric Acid G.	Ce <sub>2</sub> O <sub>3</sub> Taken G.	CeO <sub>2</sub> Taken G.	Vol. of Solution Cc.	Weight of Cupferron Ppt. G.	Error G.
1	0.2114	10	1	None	0.02	400	0.2140	+0.0026
2	0.2114	10	1	None	0.05	400	0.2139	+0.0025
3	0.2114	10	1	0.02	None	400	0.2121	+0.0007
4	0.2114	10	1	0.05	None	400	0.2130	+0.0016

(4) INTERFERING SUBSTANCES—It will be noted from the foregoing review that there is a formidable array of interfering elements. These may be divided under five heads, as follows:

(a) Interfering elements removable by a preliminary ammonium hydroxide precipitation. Alkalies, alkaline earths, and copper (incompletely) are in this class.

(b) Interfering elements removable by hydrogen sulfide in acid solution. These are copper, lead, silver, mercury, tin, bismuth, and other likely interfering elements of the hydrogen sulfide group.

(c) Interfering elements removable by ammonium sulfide in ammonium tartrate solution. Iron is the chief element, although this treatment also removes other elements, such as nickel, cobalt, and manganese.

(d) Interfering elements removable by the use of an ammonium hydroxide wash. Copper is the chief interfering element of this class. The interference of tungsten and vanadium is lessened by this treatment.

(e) Interfering elements not removable. This list includes zirconium, thorium, cerium in the quadrivalent state (less in the trivalent condition), uranium in the quadrivalent condition (but not in the hexivalent state), tungsten, vanadium in either quadrivalent or quinivalent condition, and silica. Phosphorus interferes but slightly in a 10 per cent sulfuric acid solution.

(5) SUMMARY—The determination of titanium in pure solutions by the cupferron method is most exact. The method is subject to so many interferences that it loses its value in technical analysis except for certain separations, such as, for example, titanium from chromium and aluminum which are indicated under (3).

## ZIRCONIUM

(1) HISTORICAL—Schroeder<sup>1</sup> stated that zirconium could be quantitatively precipitated by cupferron in acid solutions. Thornton and Hayden<sup>2</sup> demonstrated that the determination could be carried out in solutions containing from 5 to 7.5 per cent by volume of sulfuric acid. Ferrari<sup>3</sup> also showed that zirconium could be quantitatively determined in acid solution by means of cupferron. Brown<sup>4</sup> applied the method described by Thornton and Hayden to the analysis of zircon and baddeleyite with excellent results.

Tests, which are given in Table XVI, carried on at the Bureau of Standards, show that zirconium can be quantitatively precipitated in solutions containing 40 per cent by volume of sulfuric acid and that tartaric acid is without effect.

TABLE XVI

EXPT. No.	ZrO <sub>2</sub> Added G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume	Tar-taric Acid G.	Vol. of Solution Cc.	ZrO <sub>2</sub> Found G.	Error G.
1	0.1118	5	None	400	0.1118	0.0000
2	0.1118	10	None	400	0.1117	-0.0001
3	0.1118	15	None	400	0.1120	+0.0002
4	0.1118	20	None	400	0.1123 <sup>1</sup>	+0.0005
5	0.1118	40	None	400	0.1123 <sup>1</sup>	+0.0005
6	0.1118	5	1	400	0.1116	-0.0002
7	0.1118	10	5	400	0.1117	-0.0001

<sup>1</sup> Ignited precipitates contained traces of SO<sub>2</sub>.

<sup>1</sup> *Z. anorg. Chem.*, **72** (1911), 89.

<sup>2</sup> *Am. J. Sci.*, **38** (1914), 137.

<sup>3</sup> *Ann. chim. applicata*, **2** (1914), 276.

<sup>4</sup> *J. Am. Chem. Soc.*, **39** (1917), 2358.



(2) FAVORABLE PROCEDURE FOR PRECIPITATION—The conditions given under TITANIUM 2, governing the precipitation of titanium, apply directly to zirconium.

(3) SEPARATIONS WHICH HAVE BEEN STUDIED. (a) *Zirconium from Aluminum*—Data obtained by Thornton and Hayden are given in Table XVII and show a satisfactory separation.

TABLE XVII

EXPT. No.	ZrO <sub>2</sub> Taken G.	Al <sub>2</sub> O <sub>3</sub> Taken G.	ZrO <sub>2</sub> Found G.	Error Mg.	H <sub>2</sub> SO <sub>4</sub> 1 : 1 Cc.	Vol. of Solution Cc.
1	0.1091	0.1127	0.1090	-0.1	40	400
2	0.1088	0.1127	0.1090	+0.2	40	400
3	0.1086	0.1127	0.1090	+0.4	60	400
4	0.1091	0.1127	0.1094	+0.3	60	400

(b) *Zirconium from Iron*—The above authors applied the tartrate ammonium sulfide separation of iron, followed by cupferron precipitation of zirconium, and obtained excellent results as shown in Table XVIII.

TABLE XVIII

EXPT. No.	ZrO <sub>2</sub> Taken G.	Fe <sub>2</sub> O <sub>3</sub> Taken G.	ZrO <sub>2</sub> Found G.	Error Mg.	H <sub>2</sub> SO <sub>4</sub> 1 : 1 Cc.	Tartaric Acid G.
5	0.1088	0.1018	0.1091	+0.3	40	2
6	0.1090	0.1018	0.1093	+0.3	40	2

(c) *Zirconium from Phosphorus*—Thornton and Hayden claimed that this separation was not feasible.

Experiments carried on at this Bureau and also by Mr. Holladay<sup>1</sup> demonstrated that fair separations are possible if the acidity is high, 10 per cent sulfuric acid by volume, and the concentration of phosphorus pentoxide is not higher than 0.02 g. per 400 cc. of solution. Higher concentrations of phosphorus pentoxide require prior removal, or analysis and correction of the weighed precipitate. Table XIX gives data obtained at this Bureau.

TABLE XIX

EXPT. No.	ZrO <sub>2</sub> Taken G.	P <sub>2</sub> O <sub>5</sub> Taken G.	Tartaric Acid G.	Weight of Ppt. G.	Error G.	H <sub>2</sub> SO <sub>4</sub> Per cent by Volume	Vol. of Solution Cc.
1	0.1118	0.01	None	0.1151	+0.0033	5	400
2	0.1118	0.01	1	0.1135	+0.0017	5	400
3	0.1118	0.02	None	0.1123	+0.0005	10	400
4	0.1118	0.02	5	0.1121	+0.0003	10	400

Qualitative tests showed considerable phosphorus pentoxide in A and traces in B.

(d) *Zirconium from Iron and Aluminum*—Thornton and Hayden obtained very satisfactory separations of these elements, as shown in Table XX, by first separating the iron as described in *b* above and then the aluminum as in *a*.

TABLE XX

EXPT. No.	ZrO <sub>2</sub> Taken G.	Fe <sub>2</sub> O <sub>3</sub> Taken G.	Al <sub>2</sub> O <sub>3</sub> Taken G.	ZrO <sub>2</sub> Found G.	Error Mg.	Tartaric Acid G.	H <sub>2</sub> SO <sub>4</sub> 1 : 1 Cc.	Vol. of Solution Cc.
7	0.1087	0.1018	0.0564	0.1086	-0.1	2	40	400
8	0.1089	0.1018	0.0564	0.1088	-0.1	2	40	400
9	0.1101	0.2036	0.1127	0.1110	+0.9	2	40	400
10	0.1100	0.2036	0.1127	0.1103	+0.3	2	40	400

(e) *Zirconium from Platinum*—The observation of Thornton concerning the effect of chloroplatinic acid on a cupferron precipitation of titanium, which is given under TITANIUM 3e, will undoubtedly hold true for zirconium.

(f) *Zirconium from Boric Acid*—See TITANIUM 3f.

(g) *Zirconium from Alkali Salts*—Thornton and Hayden gave data, tabulated in Table XXI, which

<sup>1</sup> Private communication.

show that moderate excess of alkali causes no difficulties. See also TITANIUM 3g.

TABLE XXI

EXPT. No.	ZrO <sub>2</sub> Taken G.	K <sub>2</sub> O Taken G.	ZrO <sub>2</sub> Found G.	Error Mg.	H <sub>2</sub> SO <sub>4</sub> 1 : 1 Cc.	Vol. of Solution Cc.
11	0.1088	2.7027	0.1097	+0.9	40	400
12	0.1089	2.7027	0.1096	+0.7	40	400

(h) *Zirconium from Silica*—See TITANIUM 3h.

(i) *Zirconium from Vanadium*—See TITANIUM 3i.

(j) *Zirconium from Uranium*—See TITANIUM 3j.

(k) *Zirconium from Tungsten*—See TITANIUM 3k.

(l) *Zirconium from Thorium*—See TITANIUM 3l.

(m) *Zirconium from Cerium*—See TITANIUM 3m.

(4) INTERFERING SUBSTANCES—The elements which interfere with titanium also interfere with zirconium, except phosphorus which exercises a greater disturbing effect. The methods of removing the interfering elements also apply to zirconium.

(5) SUMMARY—The determination of zirconium in pure solutions by the cupferron method is most exact. The method is subject to so many interfering elements that it loses much of its value in technical analysis. The data given in this chapter demonstrate that the ignited and weighed cupferron precipitate must be most carefully tested in order to make certain of its true composition.

## THORIUM

(1) HISTORICAL—Thornton<sup>1</sup> found that precipitation of thorium by cupferron is incomplete in the presence of even a small excess of sulfuric acid. This observation was confirmed at this Bureau. The data given in Table XXII were obtained by Thornton by precipitation in acetic acid solution and by the use of a one per cent ammonium acetate wash.

TABLE XXII

EXPT. No.	ThO <sub>2</sub> Used G.	ThO <sub>2</sub> Found G.	Error G.	Ammonium Acetate G.	Vol. of Solution Cc.	H <sub>2</sub> SO <sub>4</sub> (approx.) 1 : 1 Cc.
1	0.0925	0.0924	-0.0001	15	500	1.25
2	0.0923	0.0917	-0.0006	15	500	1.25

(2) FAVORABLE PROCEDURE FOR PRECIPITATION—This is given in Table XXII.

(3) SEPARATIONS WHICH HAVE BEEN ATTEMPTED.

(a) *Thorium from Iron*—Thornton showed that a good separation of thorium from iron could be obtained by the procedure which he used in similar titanium and zirconium separations (see TITANIUM 3b and ZIRCONIUM 3b) and gave the data presented in Table XXIII.

TABLE XXIII

EXPT. No.	ThO <sub>2</sub> Used G.	Fe <sub>2</sub> O <sub>3</sub> Used G.	ThO <sub>2</sub> Found G.	Error G.	Ammonium Acetate G.	Acetic Acid	Vol. of Solution Cc.
3	0.0924	0.1018	0.0922	-0.0002	25	Concentration not stated	400
4	0.0924	0.1018	0.0916	-0.0008	25		500
5	0.1846	0.1018	0.1840	-0.0006	25		400

(4) INTERFERING SUBSTANCES—The elements listed under titanium (see TITANIUM 4) as interfering affect also the precipitation of thorium, and undoubtedly many more on account of the low permissible acidity.

(5) SUMMARY—The determination of thorium by the use of cupferron is of no practical importance since it is affected by as many interfering elements as an ammonium hydroxide precipitation of this element.

<sup>1</sup> *Am. J. Sci.*, 42 (1916), 151.



## VANADIUM

(1) HISTORICAL—Rodeja<sup>1</sup> showed that acid solutions of the alkali metavanadates give a red precipitate somewhat soluble in water. 0.000001 g. of vanadium per cc. gives a reddish coloration changing to green. The author claims that this is a more sensitive qualitative reaction than the potassium sulfocyanate or hydrogen peroxide reactions.

Rodeja<sup>2</sup> in another paper claims that cupferron does not quantitatively precipitate vanadic salts in dilute acid solutions but does precipitate vanadyl salts.

Turner<sup>3</sup> in an attempt to separate vanadium from uranium found that a metavanadate was completely precipitated by cupferron in 1 per cent solutions of hydrochloric or sulfuric acids. The precipitate was washed with one per cent sulfuric acid containing 1.5 g. cupferron per liter and then ignited to V<sub>2</sub>O<sub>5</sub>. His results are shown in Table XXIV.

TABLE XXIV

EXPT. No.	V <sub>2</sub> O <sub>5</sub> Taken G.	V <sub>2</sub> O <sub>5</sub> Found G.	Error G.
1.....	0.1655	0.1655	+0.0003
2.....	0.1655	0.1657	+0.0002
3.....	0.1655	0.1652	-0.0003
4.....	0.1655	0.1658	+0.0003

Table XXV gives data obtained by Turner which led him to conclude that Rodeja's statement concerning the incomplete precipitability of vanadic salts by cupferron was incorrect.

TABLE XXV

EXPT. No.	V <sub>2</sub> O <sub>5</sub> Taken G.	V <sub>2</sub> O <sub>5</sub> Found G.
15.....	0.0873 <sup>1</sup>	0.0873
16.....	0.0882 <sup>1</sup>	0.0882
17 <sup>2</sup> .....	0.2300 <sup>2</sup>	0.2300
18.....	0.1952 <sup>2</sup>	0.1954

<sup>1</sup> V<sub>2</sub>O<sub>5</sub> not reduced before precipitation.

<sup>2</sup> V<sub>2</sub>O<sub>5</sub> reduced before precipitation.

<sup>3</sup> Filtrate from this experiment showed vanadium.

(2) FAVORABLE CONDITIONS FOR PRECIPITATION—From the foregoing work it appears that quantitative determinations of vanadium by cupferron can be obtained in one per cent hydrochloric or sulfuric acid solutions of the element in either the quinquivalent or quadrivalent condition provided the precipitate is washed with one per cent acid containing cupferron.

(3) SEPARATIONS WHICH HAVE BEEN ATTEMPTED.

(a) Vanadium from Phosphorus—Rodeja<sup>4</sup> claimed that a satisfactory separation can be accomplished by acidulating with sulfuric acid, boiling with sulfur dioxide to reduce the vanadium, expelling the sulfur dioxide with carbon dioxide, precipitating with cupferron, washing with very dilute sulfuric acid, and igniting to vanadium pentoxide.

(4) INTERFERING SUBSTANCES—Since very dilute acidity only is permissible many elements will interfere in this determination. See THORIUM 4.

(5) SUMMARY—It is certain that the use of cupferron for the quantitative determination of vanadium will find application only in very rare cases.

<sup>1</sup> *Anales soc. españ. fis. quim.*, 12 (1914), 305; *Chem. Abs.*, 9 (1915), 2201.

<sup>2</sup> *Anales soc. españ. fis. quim.*, 12 (1914), 379; *Chem. Abs.*, 9 (1915), 2202.

<sup>3</sup> *Am. J. Sci.*, 41 (1916), 339; 42 (1916), 109.

<sup>4</sup> *Loc. cit.*

## SUMMARY

The present status of cupferron as a quantitative precipitant is as follows:

1—Cupferron has been successfully used for the quantitative determination of copper, iron, titanium, zirconium, thorium, and vanadium.

2—Many elements interfere with the determinations. In any given determination the partial or complete precipitation of copper, iron, titanium, zirconium, thorium, and vanadium must be considered in addition to the following known interfering elements: lead, silver, mercury, tin, bismuth, cerium, thorium, tungsten, uranium in the quadrivalent condition, silica, vanadium, and in certain cases when present in excessive amounts, phosphorus, alkali salts, and alkaline earths.

3—The cupferron method should not be employed unless the qualitative composition of the material to be analyzed is known, or a most careful quantitative examination of the ignited and weighed cupferron precipitate is made.

4—Cupferron can be used advantageously in certain separations, such as iron from manganese, and iron and titanium from aluminum and manganese.

BORIC ACID MODIFICATION OF THE KJELDAHL METHOD FOR CROP AND SOIL ANALYSIS<sup>1</sup>

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In 1913 Winkler<sup>2</sup> proposed the substitution of boric acid for sulfuric acid in the fixation of ammonia distilled over in the course of the Kjeldahl method for the determination of total nitrogen. He tried both methyl orange and congo red as indicators, the results with the latter being slightly better than the former as the following figures show:

## METHYL ORANGE INDICATOR

N HCl.....	8.28 cc.	0.1410 g. NH <sub>3</sub>
N HCl.....	8.27 cc.	0.1408 g. NH <sub>3</sub>
N HCl.....	8.25 cc.	0.1405 g. NH <sub>3</sub>

Av., 0.1407 g. NH<sub>3</sub>

## CONGO RED INDICATOR

N HCl.....	8.27 cc.	0.1408 g. NH <sub>3</sub>
N HCl.....	8.21 cc.	0.1398 g. NH <sub>3</sub>
N HCl.....	8.22 cc.	0.1400 g. NH <sub>3</sub>

Av., 0.1402 g. NH<sub>3</sub>

The ammonia present was equal to 0.1400 g. The fixing solution in this case consisted of 5 g. boric acid dissolved in 100 cc. distilled water.

Adler<sup>3</sup> in 1916 published a modification of the method adapted to the use of laboratories of the brewing industry, but as it has been impossible to obtain a copy of the original we have had to take our information regarding his work from abstracts.<sup>4</sup> No data regarding his results are given but only an outline of his recommendations for the modification of Winkler's method. He used 50 cc. of a 4 per cent solution of pure crystallized boric acid in the receiving flask. The condenser tube

<sup>1</sup> Published by permission of the Secretary of Agriculture.

<sup>2</sup> *Z. angew. Chem.*, 26 (1913), 231.

<sup>3</sup> *Z. ges. Brauw.*, 29 (1916), 161, 169.

<sup>4</sup> *J. Inst. Brewing*, 22 (1916), 50; *Chem. Abs.*, 11 (1917), 3371.



was allowed to remain below the surface of the absorbing solution for the first 15 min. but after that it was raised so that the subsequent distillate served to rinse the end of the tube. The distillate should reach the absorption flask at room temperature. Adler found that 20 min. of vigorous boiling sufficed to distil all the ammonia. In order to obtain maximum accuracy in titration it was necessary to employ a color standard; this was prepared by treating 250 cc. distilled water with 0.15 cc. of 0.1 *N* sulfuric acid and a few drops of methyl orange. The orange-yellow tint remained unchanged for a long time. The distillate, treated with the same amount of methyl orange, was titrated with 0.1 *N* sulfuric acid to the same tint, and the ammonia content calculated from the volume of acid required, no correction being necessary for the boric acid present. The maximum amount of ammonia which was absorbed without loss by the quantity of boric acid mentioned above (50 cc. of 4 per cent solution) was not determined, but it exceeded 70 mg. The results were as accurate as when sulfuric acid was used for absorbing the ammonia.

In laboratories where it is necessary to run a great number of nitrogen determinations, this modification is of special value because it requires only one standard solution, and since the absolute amount of boric acid in the receiving flask need not be accurately measured, an unskilled assistant may do this part of the work; this rough measurement of the acid saves from one-fourth to one-third of the time necessary to obtain equally accurate results by the old methods. Where the acid solution is made so that 1 cc. equals 1 mg. of nitrogen or some whole number multiple of this figure, the worker can read his results directly from the burette or else obtain them by the simplest mental arithmetic. For these reasons we have experimented with the method as a possible substitute for the one commonly used for the determination of total nitrogen in crops and soils.

#### SOLUTIONS

The standard sulfuric acid solution was made up so that 1 cc. equaled 1 mg. of nitrogen. The boric acid solution was made by weighing out 4 g. of the acid for each 100 cc. of distilled water used in preparing the 10-liter quantities. The boric acid was dissolved in water at room temperature.

Three indicators were employed: methyl orange, congo red, and bromophenol blue. With the first two it was necessary to make up solutions containing the regular quantity of indicator in 150 cc. of water and sufficient acid to produce the color to be used for a match in the regular titration. The methyl orange was prepared in the usual way by dissolving one gram in 1000 cc. water; congo red, 1 part in 100 parts of 30 per cent alcohol. One-tenth gram of the bromophenol blue was heated with 3 cc. of 0.05 *N* sodium hydroxide till dissolved and then diluted to 250 cc. with distilled water. The end-point of the bromophenol blue was observed by arranging an electric bulb within about 5 in. of a white surface near the burette stand in such a way that the flask during the titration was between the bulb and the reflecting surface; under these conditions the

end-point was the disappearance of the purple color. The change is sharp and distinct.

#### APPARATUS

The apparatus employed for these tests was an unmodified Kjeldahl digestion shelf and distilling stand with condensing tank.

#### EXPERIMENTS

**AMMONIUM SULFATE**—The method was first tested by distilling known quantities of nitrogen as ammonia into both sulfuric acid and boric acid. For this purpose a solution of ammonium sulfate containing approximately 0.5 mg. of nitrogen per cc. was used. Fifty cc. of this solution were pipetted into each of the Kjeldahl flasks and 200 cc. of distilled water and 50 cc. of saturated sodium hydroxide solution added. The resulting solution was boiled for 30 min. so that about 150 cc. of distillate passed over, and then 5 min. longer to allow for clearing the delivery tubes.

Two sets of 8 flasks each were titrated with congo red as an indicator, 3 drops being used in each determination; in one set 50 cc. sulfuric acid solution and in the other 50 cc. boric acid solution were used in the receiving flask. The excess sulfuric acid was titrated with ammonium hydroxide, 1 cc. of which equaled 1.011 cc. sulfuric acid.

In order to make the results with the two absorbing solutions comparable, we added 3 drops of congo red to 200 cc. of distilled water and then added 0.30 cc. of acid to produce the same color as was obtained with 150 cc. distilled water, 50 cc. 4 per cent boric acid and 0.35 cc. of standard acid. In the former case, as the end-point comes while the solution is still acid, the correction of 0.30 cc. was added to the burette reading for ammonia to reduce it to what would be obtained if the end-point were taken at the neutral point with this indicator. To correct for the presence of the boric acid it was necessary to subtract 0.35 from the burette reading for sulfuric acid. Two additional sets of distillates in boric acid were titrated, one with bromophenol blue, 3 drops, and the other with methyl orange, 3 drops; in each case the correction constant was 0.3 cc., which was subtracted from the figures obtained for each determination as shown.

TABLE I—NITROGEN RECOVERED FROM  $(\text{NH}_4)_2\text{SO}_4$  SOLUTION

SULFURIC ACID FIXING AGENT Congo Red	BORIC ACID FIXING AGENT		
	Congo Red	Bromophenol Blue	Methyl Orange
Mg.	Mg.	Mg.	Mg.
26.20	26.15	26.20	26.10
26.20	26.25	26.25	26.25
26.15	26.25	26.25	26.25
26.25	26.25	26.20	26.25
26.13	26.15	26.10	26.10
26.10	26.05	26.25	26.20
26.25	26.15	26.25	26.15
26.10	26.25	26.10	26.20
Av.	26.18	26.19	26.20

**PROCEDURE WITH CROPS AND SOILS**—The crops were weighed, placed in a Kjeldahl flask, and 30 cc. concentrated sulfuric acid containing 10 per cent phosphoric anhydride and 0.5 g. of anhydrous copper sulfate added before starting the digestion. The digested material in the acid was diluted with 175 cc. of distilled water and treated with 100 cc. concentrated sodium hydroxide and a few pieces of mossy zinc. The distillates from these solutions were received in approxi-



mately 50 cc. portions of 4 per cent boric acid and titrated with standard sulfuric acid, using bromophenol blue as an indicator. This indicator was selected in preference to any of the others because when used under the conditions described for observing the end-point, the disappearance of the purple color (which could also be observed through a screen as in hydrogen-determinations) is sharper and more distinct than either of the others and has the added advantage that it is not necessary to keep a color standard for comparison.

The soils were digested in 40 cc. of concentrated sulfuric acid containing 1 g. of salicylic acid in each 25 cc. The soil is allowed to digest half an hour at room temperature, then 0.5 g. of iron or zinc dust is added, and after standing half an hour longer 5 g. of Lipman's sulfate mixture<sup>1</sup> is added and the digestion completed over the flame. After digestion, when the solution has cooled sufficiently, distilled water is added and the liquid contents of the flask transferred to another Kjeldahl flask and the residue well washed with distilled water which is also transferred to the second flask. The water added should not be much over 175 cc. One hundred cc. of saturated caustic soda and some mossy zinc are then added and the solution distilled into boric acid and the distillate titrated the same as the distillate from crops.

Following the procedures outlined, first with the reagents employed for crops and for soils, and later with crops and soils, the figures shown in Table II were obtained.

TABLE II—NITROGEN FOUND IN CROPS AND SOILS

SUBSTANCE	Weight G.	Flask No.	Titration Corrected for Indicator Cc.	Less Check Cc.	Average Cc.
Reagents for Crops.....	.....	1	0.10	...	0.10
		2	0.10	...	
		3	0.10	...	
		4	0.10	...	
Reagents for Soils.....	.....	1	0.40	...	0.35
		2	0.40	...	
		3	0.30	...	
		4	0.30	...	
Corn Fodder.....	1	1	5.80	5.70	5.58
		2	5.60	5.50	
		3	5.65	5.55	
Corn Fodder.....	2	1	11.50	11.40	11.40
		2	11.50	11.40	
		3	11.50	11.40	
Wheat Grain.....	1	1	22.35	22.25	22.22
		2	22.30	22.20	
		3	22.30	22.20	
Wheat Grain.....	2	1	44.60	44.50	44.45
		2	44.60	44.50	
		3	44.45	44.35	
Sweet Clover.....	1	1	28.35	28.25	28.42
		2	28.60	28.50	
		3	28.60	28.50	
Sweet Clover.....	2	1	57.00	56.90	56.93
		2	57.00	56.90	
		3	57.10	57.00	
Soil No. 1.....	10	1	4.05	3.70	3.70
		2	4.00	3.65	
		3	4.10	3.75	
Soil No. 2.....	10	1	4.55	4.20	4.13
		2	4.40	4.05	
		3	4.50	4.15	

In tests with an ammonium sulfate solution it was found that when 50 cc. of 4 per cent boric acid solution were used 95 mg. of nitrogen as ammonia could be recovered with accuracy. This result was obtained without the use of glycerin which Adler recommends.

<sup>1</sup> K<sub>2</sub>SO<sub>4</sub>, 20 parts; FeSO<sub>4</sub>, 2 parts; CuSO<sub>4</sub>, 1 part.

## SUMMARY

Winkler's modification of the Kjeldahl method in which he proposed the use of boric instead of sulfuric acid in the receiving flask is found to be as accurate as when the latter reagent is employed. Ninety-five mg. of nitrogen as ammonia can be recovered in the distillate when 50 cc. of 4 per cent boric acid is used.

Bromophenol blue is a better indicator than those used by the other investigators.

The method has the following advantages:

1—It does away with the occasional errors that arise from slight mistakes in measuring the sulfuric acid into the receiving flask.

2—As the boric acid solution need be measured only approximately, much time can be saved, and an unskilled helper can measure it into the receiving flasks.

3—By proper adjustment of the strength of the standard acid and the weights of the samples taken the percentage of nitrogen can be read directly from the burette.

4—It is necessary to prepare accurately only one standard solution, *i. e.*, the sulfuric acid for titrating.

#### A RAPID AND ACCURATE METHOD FOR DETERMINING NITROGEN IN NITRATE OF SODA BY THE MODIFIED DEVARDA METHOD AND THE USE OF THE DAVISSON SCRUBBER BULB<sup>1</sup>

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There is among chemists with whom I have come in touch, and especially among those engaged in the fertilizer industry, a very strong conviction that the usual methods for the determination of nitric nitrogen, as in the analysis of nitrate of soda, are far from satisfactory for routine work and none too accurate in the hands of the average analyst. Procedures used in the average fertilizer works laboratory should of necessity be both rapid and accurate; moreover, it is very desirable that constant attention be not required. The modified Kjeldahl-Gunning method, which had been used in our laboratory for several years, produced fairly satisfactory results, provided considerable time were given to complete the solution and reduction of the nitrate in the salicylic acid mixture. In addition to this time-consuming procedure, agreeing duplicate determinations have been very difficult to obtain, unless the sample be dried and ground very fine. Naturally, therefore, our attention was turned to other direct methods.

The Devarda method, as refined in late years by W. S. Allen<sup>2</sup> and also E. R. Allen,<sup>3</sup> appeared the most promising, as both investigators obtained almost theoretical results. The method recommended by W. S. Allen, while very accurate, necessitated the use of the modified Knorre apparatus which was rather elaborate and more complicated as to operation than seemed desirable.

The procedure outlined by E. R. Allen for nitric nitrogen in soil extracts, which he designated as the Volmari-Mitscherlich-Devarda method, seemed more suitable in that the reduction and distillation were effected in a very dilute alkali solution which permits the reaction to proceed quietly, a distinct ad-

<sup>1</sup> Presented before the Fertilizer Division at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 4, 1919.

<sup>2</sup> 8th Intern. Congr. Appl. Chem., 1 (1912), 19.

<sup>3</sup> THIS JOURNAL, 7 (1915), 521.



vantage over other methods in which strong alkali solutions are used. E. R. Allen found that one gram of 60-mesh alloy was sufficient to reduce 25 mg. nitric nitrogen in 300 cc. of approximately 0.1 *N* NaOH, reduction and distillation being completed in 40 min., using the modified Mitscherlich apparatus in which the water cooling is dispensed with and the ammonia absorbed by passing the steam through a scrubber into the receiving flask.

By using the E. R. Allen method in this laboratory in connection with the regular Kjeldahl rack without a scrubber, favorable results were obtained, provided the reaction was allowed to proceed with gentle heat for several hours and the distillation then completed over a low flame. Even then at times we found that the alkali mist would be carried into the tube of the condenser, thereby rendering blank tests variable.

None of these procedures, therefore, seemed simple or rapid enough until the Davisson<sup>1</sup> type of scrubber came to our notice. This scrubber bulb is built similarly to and may be substituted for the Hopkins connecting bulb where the necessity arises for a scrubber for the ammonia vapors. Davisson found that the entrained alkali can be satisfactorily removed when the vapors are scrubbed through water previous to condensation. According to his statement, the first steam which passes into the scrubber condenses on the surface of the bulb and flows down about the small bulb and there acts as a scrubbing solution for the remaining vapors. This solution is neutral or slightly alkaline, and the long period of steam distillation removes all the ammonia.

When using this scrubber in connection with the modified Devarda method, it was found in this laboratory that since the alkali mist passed into the condenser tube before any condensation occurred in the bulb it was necessary to draw about 20 cc. of water by suction into the scrubber before beginning the distillation.

A large number of the solutions left in the scrubber at the end of the distillation period have been tested with Nessler's reagent. Out of a total of more than 30 solutions, only 2 have shown the reaction for ammonia and these showed only a very faint coloring. Likewise a large number have been tested by the phenolsulfonic acid method,<sup>2</sup> and in no case were nitrates found.

To determine whether or not any alkali passes the scrubber, ammonia-free water and sodium hydroxide were used in blank tests on three separate occasions, and the distillate titrated, one drop of 0.1 *N* sulfuric acid being sufficient in all cases to produce a distinct color change with methyl red indicator. This confirms Davisson's statement that thorough scrubbing of the vapors is accomplished.

The method used in this work is based on the findings of W. S. Allen, who recommended using 3 g. of Devarda's alloy to 1 g. of nitrate of soda, and the findings of E. R. Allen, who verified Volmari's conclusions that approximately 0.1 *N* sodium hydroxide solutions are sufficiently concentrated for the reduction and distillation, and that strongly alkaline solutions cause the reaction to be so violent that care must be continually exercised to prevent a loss of the determination.

The Devarda's alloy used in this work was obtained from J. T. Baker, and upon testing was found to be ammonia-free. A fineness test on the original sample showed: 100 per cent passing 10-mesh sieve, 76 per cent passing 20-mesh sieve, 19 per cent passing 60-mesh sieve.

To determine the effect of the degree of fineness on the results a few determinations were carried out using in one determination 3 g. of alloy remaining on 20 mesh and in the duplicate 3 g. of the alloy passing 20 mesh with the following results:

Test Number	Alloy Remaining on 20 Mesh Per cent Ammonia Found	Alloy Passing 20 Mesh Per cent Ammonia Found
1.....	19.97	19.96
2.....	19.96	19.98
3.....	18.85	18.82
4.....	18.81	18.81

These results indicate that an alloy passing a 10-mesh sieve is sufficiently fine under the conditions of the method to be outlined.

The time required for complete reduction and distillation was also studied. Tests were made, distilling 15, 30, 45, and 60 min. The results follow:

Minutes Distilling	15	30	45	60
Per cent ammonia found.....	16.21	18.49	18.99	19.03

To the contents of the flasks containing the condensate from an additional 15 min. distillation, one drop of 0.1 *N* sulfuric acid gave a distinct color change with methyl red indicator. This shows that one hour's reduction and distillation is sufficient for a practically complete recovery of the nitrogen.

#### METHOD OF ANALYSIS

The method in detail recommended for the analysis of nitrate of soda is as follows:

**PREPARATION OF SAMPLE**—The sample should be put through a 10-mesh sieve and mixed thoroughly. This we have found to be sufficiently fine to obtain good duplicate checks. Out of a total of 23 samples analyzed since adopting this procedure, duplicate determinations have shown the following agreements: 35 per cent within 0.05 per cent NH<sub>3</sub>, 78 per cent within 0.10 per cent, and all within 0.15 per cent.

**DETAILS OF MANIPULATION**—Weigh 17.034 g. and transfer to a 500 cc. volumetric flask (calibrated to correspond with the pipette to be used) and add 300 cc. of water. Warm to dissolve, cool, and make up to mark. After mixing thoroughly, pipette 25 cc., corresponding to 0.8517 g. of sample, into a Kjeldahl flask of 500 to 650 cc. capacity. Add 300 cc. of water, 3 g. Devarda's alloy (75 per cent of which will pass 20-mesh sieve), 3 to 5 cc. 45° Bé. sodium hydroxide, and connect at once with the Kjeldahl distilling apparatus fitted with the Davisson scrubber into which has been drawn 20 to 30 cc. of water. Conduct distillation of the ammonia synchronously with the reduction, regulating the boiling so that 175 to 200 cc. of distillate is collected in about one hour. When distillation is about half completed, it is recommended that part of the solution in the scrubber be sucked back into the distilling flask, allowing 10–20 cc. to remain in the bulb, this being accomplished by removing the flame or holding the flask out of place a moment while still connected. This removes the danger of the liquid splashing into the condenser tube, and by reducing the volume in the bulb facilitates the passing of the last traces of ammonia into the receiving flask.

<sup>1</sup> THIS JOURNAL, 11 (1919), 465.

<sup>2</sup> Scott, "Standard Methods of Chemical Analysis," 1917, p. 539.



Twenty-one cc. of 0.5 *N* sulfuric acid are used in which to absorb the ammonia and the titration is carried out as in the regular Kjeldahl method, using methyl red indicator.

Cc. 0.5 *N* acid required = per cent ammonia

ACCURACY OF METHOD—0.5 *N* sulfuric acid used in the following work was carefully standardized by the sodium carbonate method.<sup>1</sup> This solution in a series of tests averaged within 0.03 per cent of the theoretical ammonia equivalent of specially prepared and dried ammonium sulfate when ammonia in the salt was determined by the above method.

0.25 *N* sodium hydroxide was prepared by titration against the 0.5 *N* H<sub>2</sub>SO<sub>4</sub> using methyl red indicator.

A check sample of sodium nitrate was prepared from C. P. sodium carbonate and nitric acid, free from chlorides and sulfates, recrystallized and dried at 150° C. Upon analysis this salt showed the following:

H <sub>2</sub> O.....	0.08 per cent
NaCl.....	0.02 per cent
KNO <sub>3</sub> .....	0.62 per cent
Na <sub>2</sub> SO <sub>4</sub> .....	Trace
Fe <sub>2</sub> O <sub>3</sub> .....	Trace
Al <sub>2</sub> O <sub>3</sub> .....	Trace
CaO.....	Trace
MgO.....	Trace
SiO <sub>2</sub> .....	Trace

Correcting for the K<sub>2</sub>O found and deducting from 100 per cent all the impurities the result is 99.80 per cent sodium nitrate. The theoretical ammonia, therefore, equals 20.00 per cent.

Results which were obtained at various times on this check sample are as follows:

No.	Per cent Ammonia Found	No.	Per cent Ammonia Found	No.	Per cent Ammonia Found
1.....	19.99	5.....	20.03	9.....	19.95
2.....	20.00	6.....	19.95	10.....	19.96
3.....	19.99	7.....	19.96	11.....	19.99
4.....	20.04	8.....	19.98	12.....	19.99
Highest.....			20.04		
Lowest.....			19.95		
Average.....			19.99		

It will be noted the extremes show a variation of 0.09 per cent ammonia, equivalent to 0.45 per cent sodium nitrate. Blank determination showed an equivalent of 0.01 per cent ammonia, which was deducted from all results.

The effects of the presence of sodium chloride and also sodium carbonate were then determined with results as follows:

AMOUNTS ADDED TO ALIQUOT IN FLASK BEFORE DISTILLING

No.	NaCl		No.	Na <sub>2</sub> CO <sub>3</sub>	
	Added	Per cent Ammonia Found		Added	Per cent Ammonia Found
1.....	0.04 g.	20.03	6.....	0.04 g.	20.02
2.....	1 g.	19.94	7.....	1 g.	19.97
3.....	Added	19.99	8.....	Added	20.00
4.....	Per cent	20.03	9.....	Per cent	20.02
5.....	Ammonia	20.02	10.....	Ammonia	19.99
Av.....	Found	19.98	Av.....	Found	19.96

The foregoing figures indicate that neither of these compounds in quantities of 1 g. or less has an appreciable effect on the results.

In order to show whether or not the same degree of accuracy is attainable on commercial nitrate of soda, two samples were selected at random from a lot arriving at the laboratory, and each was run in duplicate by three analysts. Following are their results:

ANALYST	Sample 1	Sample 2
	Per cent Ammonia Found	Per cent Ammonia Found
1.....	18.79	18.97
	18.83	18.93
Average.....	18.81	18.95
2.....	18.82	18.97
	18.78	18.98
Average.....	18.80	18.98
3.....	18.83	19.00
	18.84	19.01
Average.....	18.84	19.01

Upon making analyses on these two samples for all impurities and using the average ammonia figures for Sample 1, 18.82 per cent, and Sample 2, 18.98 per cent, the following shows that the method had yielded exceptionally accurate results:

	Sample 1	Sample 2
H <sub>2</sub> O.....	3.08	2.74
Insoluble.....	0.21	0.26
Na <sub>2</sub> SO <sub>4</sub> .....	0.27	0.33
NaCl.....	0.51	0.56
NaClO <sub>3</sub> .....	0.05	0.05
NaClO <sub>4</sub> .....	0.94	0.51
Fe <sub>2</sub> O <sub>3</sub> }		
Al <sub>2</sub> O <sub>3</sub> }	0.10	0.09
CaO.....	0.09	0.07
MgO.....	0.18	0.10
NaIO <sub>3</sub> .....	0.05	0.04
P <sub>2</sub> O <sub>5</sub> .....	0.04	0.06
KNO <sub>3</sub> .....	3.48	2.55
NaNO <sub>3</sub> .....	90.98	92.57
TOTAL.....	99.98	99.93

	Sample 1	Sample 2
NaNO <sub>3</sub> by modified Devarda method....	93.92	94.72
NaNO <sub>3</sub> by deducting from 100 per cent all impurities and correcting for the K <sub>2</sub> O found.....	93.93	94.78

SUMMARY

The modified Devarda method as outlined, used in connection with the regular Kjeldahl rack, fitted with the Davison scrubber, in addition to being rapid, is capable of producing exceptionally accurate results and is suitable for routine work. Reduction and distillation may be completed in one hour.

The presence of 1 g. of sodium chloride or sodium carbonate in the sample is shown to have no appreciable effect on the determination.

Ten- to twenty-mesh alloy is shown to be sufficiently fine under the conditions of the method outlined.

In view of the excellent results obtained it is hoped that the fertilizer division will include this procedure or a modification of the same in any work that may be carried out toward the improvement in our methods for nitrate of soda analysis.

<sup>1</sup> Scott, *Loc. cit.*, p. 503.



## TESTING THE STRENGTH OF GLUE JELLEIS

By Wilson H. Low

THE CUDAHY PACKING CO., OMAHA, NEBRASKA

Received September 8, 1919

Testing of the strength of glue jellies has been carried on in this laboratory for a great many years, and under the direct supervision of the writer for the last 21 years. In that time all kinds of devices have been used for this purpose, but until the Smith glue tester (Fig. 1) came out, there was none on the market sensitive enough to show even fairly large differences or to duplicate the "finger test" so valuable in the hands of an expert.

This form of tester is far superior to any other we have seen, but in its original form the testing, although sensitive enough, was subject to some errors which affected the grading of the glues. Moreover, since it was filled simply with water colored with a dye to make it visible on the scale, glues were often met with which could not be measured on the scale. The only modification of the Smith glue tester that we have seen illustrated is that of E. C. Hulbert<sup>1</sup> (Fig. 3).

The apparatus used in this laboratory (Fig. 2) consists of a Smith glue testing apparatus modified to give more accurate results for grading glues correctly. Mercury is used in the U-tube, and above this colored water. The scale has slots at each end to allow of moving it and setting it by the screws that fit through the slots. All other parts of the instrument are as illustrated in Fig. 1.

It was first determined by calculation and experiment that the use of mercury covered by water in the scale tube gave correct results. It was found necessary to correct for the force required to deform the diaphragm itself, for this takes some force that should not be credited to the jelly, and this force is not a constant, for diaphragms vary much with different rubbers and vary from day to day through permanent stretch of the rubber. Another point is that it is necessary to find the force required to deform the rubber when the instrument is set in the position for a test, that is, when the water stands at point A in the diagrams.

Owing to changes in temperature of the water and

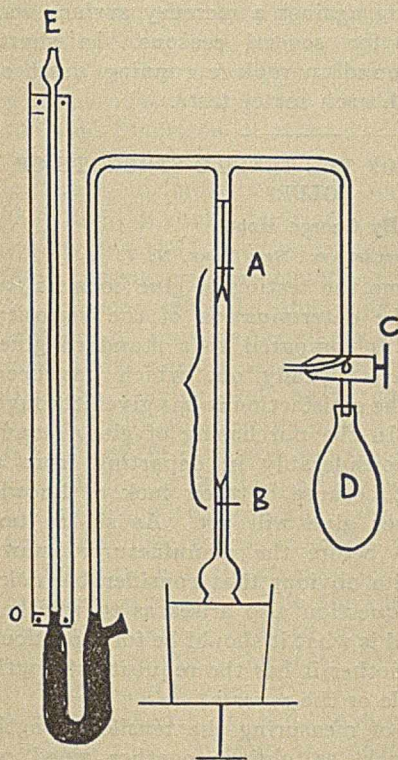


Fig. 1

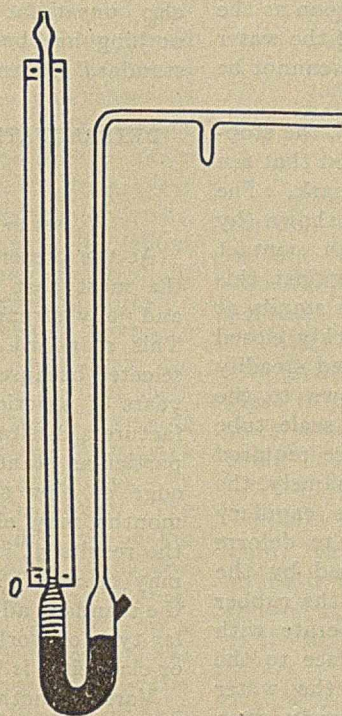


Fig. 2

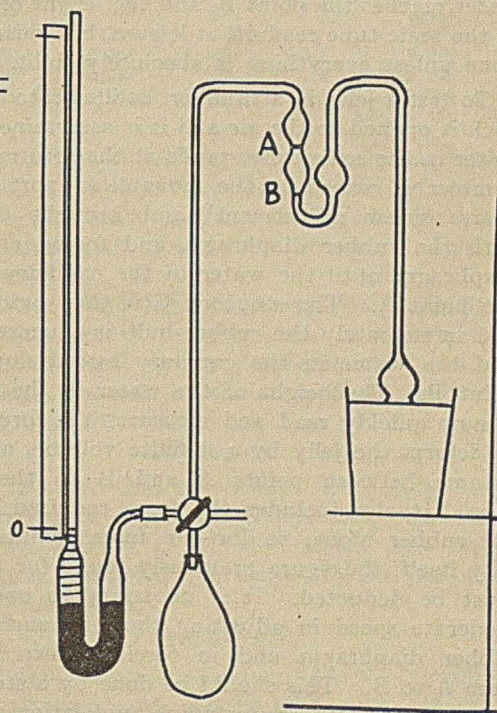


Fig. 3

The principal modifications are the use of mercury covered by water in the scale tube, and the use of air, instead of water, in the thistle tube, the mouth of which is covered with a thin, dental rubber diaphragm, as in the original Smith glue tester.

The use of mercury is a distinct improvement and has been followed in this laboratory for years. We do not readily see the advantage of the substitution of air for water in the thistle tube, especially since leaks are not so easily noticed in the rubber diaphragm or where it is attached to the thistle tube.

mercury in the instrument or of the whole instrument, the water will not always stand at the zero mark of the scale, as it should in starting a test. To overcome this trouble we made our scale movable in a vertical direction. With these modifications and methods of application we found we could properly grade our glues or glue jellies very closely and with comparative correctness.

## METHOD OF USE

Our instrument is kept permanently in a chill room connected with the laboratory and maintained close to 40° F., at which temperature the glue jellies are

<sup>1</sup> THIS JOURNAL, 5 (1913), 235.



tested; that is, the jellies are at the same temperature as the room and close to 40° F. Standard glues of various grades are always run at the same time, so temperature or other changes affecting the strength will act on the standards and jellies being tested in like manner. Enough water is put into the thistle tube and capillary so that when a flat glass plate is held firmly against the mouth of the thistle tube (covered with the rubber diaphragm) the water in the capillary will stand exactly at the upper mark A, the three-way stopcock C being opened to the air. Stopcock C is now closed to the air and opened to the rubber bulb D and scale tube E, the water in which was set to the zero mark. Pressure is now applied through the bulb (a screw clamp between the bulb and stopcock allowing only a steady, slow, and even pressure to be applied to the system) until the water in the capillary falls from the point A to the point B. The height of the water in the scale tube is now read off, and this measures the force required to deform the rubber diaphragm alone, and this force must be deducted from the tests on the jellies for the proper grading of the glues. If all joints of the instrument are tight, the stopcock C may be closed as soon as the water reaches the point B, and the height of the water in the scale tube read off at leisure, but this cannot be done unless everything is absolutely tight.

To test a jelly in a tumbler, as illustrated, the stopcock is opened to the air and it is ascertained that the water in the scale tube stands at the zero mark. The tumbler is placed on the movable support shown (by coarse screw adjustment) and brought in contact with the rubber diaphragm and forced against this diaphragm until the water in the capillary stands at the point A. The stopcock C is then quickly closed and immediately the rubber bulb is squeezed steadily and the water in the capillary forced down to the point B. The height of the water in the scale tube is now quickly read and measures the force required to deform the jelly by a definite volume, namely, the volume between points A and B on the capillary tube. It also includes the force required to deform the rubber alone, so for the force required by the jelly itself, the figure previously found for the rubber must be deducted. It is necessary to operate with moderate speed in adjusting the jelly surface to the rubber diaphragm and in forcing down the water from A to B. This should be done by a steady, even pressure, not by jerky or quick application of pressure on the bulb.

Used correctly this instrument is very sensitive and gives concordant results. Used incorrectly it is still sensitive, but will not give concordant results. The water in the capillary *must* stand at the point A when a flat glass or metal plate is held firmly against the mouth of the thistle tube covered with the rubber, as only in this way can we start with the jelly and rubber forming an approximately flat surface. It is assumed generally that if the mouth of the thistle tube is forced against the jelly surface until the water in the capillary stands at point A we are starting under like pressures each time. But this is not so. It

makes a difference whether we press into the jelly so that it forms a convex surface upwards into the rubber and then force down to a flat surface at the end, or whether we start with a level surface on the jelly and force the rubber downward into the jelly. In the first case the jelly itself tends to return to its original position and acts with the rubber, in the other case it continually works against the rubber.

An attempt was made to use an artificial jelly surface by stretching a rubber diaphragm over the mouth of the tumbler through the side of which we made a hole and attached a long pressure measuring tube. The tumbler was then completely filled with water and pressures through the thistle tube applied to the rubber surface. This did not work at all, for the rubber of the tumbler not only was deformed through the thistle tube, but bulged up between the center and sides and the pressures recorded were not at all proportional to the pressures applied. This same thing undoubtedly occurs to some extent in testing a glue jelly and occasions errors, but to no such extent as with a liquid like water acting on an elastic diaphragm. Working against a mercury surface was also unsatisfactory for several reasons. In short, nothing has been found to replace running a set of standard grades with each set of tests.

#### DETERMINATION OF THE TENSILE STRENGTH OF GLUE<sup>1</sup>

By George Hopp

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At the present time the testing of glue consists for the most part in the determination of the viscosity and jelly strength, as compared to a standard glue. This standard glue is usually one which has been selected because of the satisfaction it has given through years of practice. In the purchasing of glue, manufacturers hesitate considerably in departing from a particular standard, because of their lack of knowledge of how another glue will act. As six to ten months may elapse before the manufacturer knows the results, it is quite obvious that considerable risks may be run. The question also arises as to whether the standard adopted is what it should be for a particular type of work, whether it has the requisite strength or elasticity, is brittle or the reverse.

Various methods of measuring the tensile strength have been tried, such as gluing together wood or biscuit ware, or soaking paper in glue and determining the strength of the glues. These have been unsuccessful, due to the presence of so many variables, such as humidity, temperature, viscosity and temperature of glue, pressure at which it is applied at the joints, and condition of the joints, such as the amount of moisture, smoothness of surface, etc. Recognizing the fact that the most important test of a glue should be a knowledge of its actual strength and stretch or elasticity, and that equipped with such knowledge, standards could then be scientifically established for every phase of work, a new method has been evolved.

<sup>1</sup> Presented at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 2 to 6, 1919.



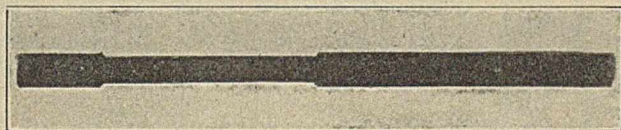
This work was originally done in 1913-1914, but owing to the fact that there were no suitable tensile machines at hand, the completion of the work was put off till this time.

#### METHOD

The method consists in melting the glue, pouring into molds, drying, cutting into strips of definite size, and obtaining the strength and stretch by pulling on a tensile machine.

Solutions of glue, 60 to 80 per cent by volume, are soaked for 12 hrs. and melted up at a temperature of 160° F. Great care is necessary at this point to avoid the formation of bubbles and foam.

The molds are of irons, 12 in. square and 1/4 in. deep. The surface of the iron is carefully polished, so that no impression of any kind will be left on the glue. It should be noted that the slightest imprint becomes strongly emphasized on the dried glue surface. The mold is just warm enough to prevent chilling of the glue, approximately 130° F. The glue solution is poured into the leveled molds and permitted to jell. Under summer conditions, approximately 5 hrs. are allowed. This sheet is then removed and permitted to dry on fine-meshed galvanized or tinned wire, raised so as to permit circulation of the air around the under surface of the glue. The glue sheet is continually reversed in order to obtain a flat surface without too much curling. As soon as the edges show signs of complete drying, they are cut to prevent curling and the formation of uneven spots and strains. This cutting consists merely of making a slit about 1/4 in. deep into the glue. By drying under slight pressure as soon as the glue appears partially dried, a perfect flat surface is obtained. It should be noted that the jelling and drying should take place undisturbed by air currents of any kind. The flat sheet is next cut into strips of definite size, preferably using a hot knife, as this is considerably quicker than using a very sharp cutter. They are then ground exactly on a grinding wheel or other suitable device. The center portion of the strip is then ground in, to insure breaking at center. The thickness of the strips and the widths were varied in order to check up the results. A suitable thickness averaged 0.1 in. and a suitable width 0.33 in. The overall length of the strip was about 7 in. and the ground portion at center about 2.5 in. A strip is illustrated in the accompanying photograph.



The tensile machine was an electrically driven Schopper with a capacity of 500 kg. The jaws on this machine are more nearly in alignment than on any other machine used. This is of vital importance, as the slightest deviation of the jaws will give poor results, due to the torsion and shear set up.

#### RESULTS

Using a hide glue, designated D-400, the following tests covering several parts of a shipment were made:

Thickness	Width	Kg. Break	Per cent Stretch	Tensile Strength Lbs. per Sq. In.
0.091	0.337	174	3	12,510
0.077	0.352	168	3	13,690
0.088	0.352	185	3.0	13,200
0.144	0.411	362	3.5	13,500
0.096	0.382	216	3.5	13,000
0.099	0.398	235	5	13,200
0.094	0.399	226	4.5	13,270
0.091	0.446	249	5	13,500
0.097	0.379	218	...	13,100
0.103	0.425	262	5.5	13,180
0.147	0.403	368	6	13,710
0.102	0.426	259	5.5	13,150
0.139	0.433	358	5	13,270
0.095	0.345	198	4.5	13,350
0.130	0.322	248	...	13,100
0.102	0.444	269	6	13,100
0.098	0.459	271	6.5	13,300
				Av., 13,240

The following are the results obtained with a glue designated D-300:

Thickness	Width	Kg. Break	Per cent Stretch	Tensile Strength Lbs. per Sq. In.
0.057	0.352	75	2	8,250
0.054	0.355	76	2	8,750
0.055	0.350	71	2	8,150
0.057	0.336	73	1.9	8,400
0.061	0.346	83	2	8,690
0.095	0.351	137	2.8	9,050
0.095	0.349	123	2.2	8,200
0.055	0.314	68	2	8,700
				Av., 8,523

A glue designated A-500 and taken from various portions of a shipment averaged 7,600 lbs. per sq. in. and 2 per cent stretch. A glue designated G-“Special” averaged 11,573 lbs. per sq. in. and 3.5 per cent stretch.

These glues were kept under standard conditions but pulled at room temperatures. They represent glues in commercial use of 3 different factories, and were selected because of the ease with which they could be obtained.

#### DISCUSSION

It will be noted that the results check very closely. Using varying widths and thicknesses, the tensile strength was always about the same. The stretch showed slight variation, due largely to the inaccuracy of the scale readings.

#### CONCLUSIONS

This method gives a feasible way of determining the exact strength and stretch of a glue, its elastic limit and other physical properties. It will permit manufacturers to determine exactly the physical character and properties of their glues, also to vary their processes and raw materials and determine the effect on the finished product.

This method will also permit the establishment of scientifically selected standard expressed in absolute units. At present it is not possible to compare the results of tests made by one laboratory with those of another, as the strength of solution, temperature, and manipulation are different. There are often exceptions to the fact that a glue with a high viscosity or jelly strength is stronger than a low viscosity glue



Certain factories can often meet viscosity and jelly requirements of a sample, but their glues will lack the strength and flexibility of glues from other factories having exactly the same jelly and viscosity.

At the present time work is being done to determine the following:

1—The relationship between the viscosity, jelly strength, tensile strength, and stretch.

2—The relationship between gelatin, chondrin, keratin, mucin, etc., and their effect on the strength and stretch of a glue.

3—Effect of heating and the length of time of heating on the strength and stretch of glues, both in liquid form and dried form.

4—Effect of the soaking of glues on their strength and stretch.

5—Effect of the age of glues on their strength and stretch.

6—Effect of dissolving a glue, drying, redissolving, etc., on the strength and stretch.

7—Effect of the addition of other substances to glue.

8—Effect of humidity and temperature on glue.

#### THE DETERMINATION OF IODIDE AND BROMIDE IN MINERAL WATERS AND BRINES<sup>1</sup>

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In a former paper<sup>2</sup> the authors recommended the permanganate method for the determination of iodide in mineral waters and brines. By the use of this method the iodide is oxidized to iodate by potassium permanganate in slightly alkaline solution and the iodate determined iodometrically by adding potassium iodide, acidifying with hydrochloric acid and titrating the liberated iodine with thiosulfate. This method gives very satisfactory results for the iodide content, but it does not leave the solution in a condition suitable for the determination of the bromide content.

In that paper the authors also showed that iodide may be quantitatively separated from large amounts of chlorides (up to 10 g. NaCl) and from amounts of bromide equivalent to not more than 0.3 to 0.4 g. bromine by liberating the iodine with ferric sulfate and removing it by distillation with steam. The iodine is absorbed in a potassium iodide solution and titrated with thiosulfate.

In a second paper<sup>3</sup> a method was proposed for the estimation of bromide in the presence of large amounts of chloride (10 g. NaCl). This method takes advantage of the selective oxidizing action of chromic acid, and the liberated bromine is removed by aspiration.

In the present paper some results are reported of determinations of the iodide and bromide content of mixtures of iodide, bromide, and chloride. These

results were obtained by combining the ferric sulfate method for iodide with the chromic acid method for bromide.

#### PROCEDURE

The procedure for the determination of iodide and bromide in the presence of chloride is as follows: The iodide should first be determined by the permanganate method, since more accurate results can be obtained by its use than by the use of the ferric sulfate method. Another sample is then taken which should contain not more than 0.1 g. of bromine as bromide or more than 10 g. of total salts. The iodine is removed from this sample by oxidizing with ferric sulfate and distilling the liberated iodine with steam according to the method described in the first paper. The iodine is absorbed in a potassium iodide solution and may be titrated with thiosulfate and the results used to check those obtained by the permanganate method. The residue in the distilling flask is emptied into a beaker, heated to boiling, and the iron precipitated with ammonia or sodium hydroxide. If sodium hydroxide is used, care must be taken not to add a great excess, though a slight excess will be taken care of later by the chromic acid. Any excess of ammonia will be removed during the subsequent evaporation. Filter off the iron hydroxide, wash with hot water, and evaporate to dryness or nearly so. The bromide is determined in the residue by the chromic acid method, the details of which were given in the second paper.

Calcium chloride does not interfere with either determination, but care should be taken when precipitating the iron not to precipitate too much calcium hydroxide by the addition of an excess of alkali, as otherwise the precipitate will be bulky and difficult to wash. This applies also to magnesium chloride if sodium hydroxide is used for precipitating the iron. If much magnesium chloride is present the solution may become acid during evaporation, due to hydrolysis. This may be guarded against by coloring the solution with methyl orange and adding a drop of sodium hydroxide solution if it becomes acid.

EXPT. No.	NaCl Taken G.	I as KI Taken G.	Br as		Error in I Det. G.	Error in Br Det. G.
			KBr Taken G.	I Found G.		
1	..	....	0.0600	....	0.0598	—0.0002
2	..	....	0.0800	....	0.0798	—0.0002
3	10	....	0.0800	....	0.0790	—0.0010
4	..	0.0803	0.0800	0.0794	0.0798	—0.0009
5	10	0.0803	0.0200	0.0792	0.0191	—0.0011
6	10	0.0803	0.0400	0.0794	0.0385	—0.0009
7	10	0.0402	0.0800	0.0397	0.0784	—0.0005
8	10	0.0803	0.0800	0.0792	0.0790	—0.0011
9	10	0.0803	0.0200	0.0795	0.0188	—0.0008
10	10	0.0803	0.0400	0.0798	0.0385	—0.0005
11	10	0.0803	0.0020	0.0805	0.0015	+0.0002
12	10	0.0803	0.0040	0.0798	0.0033	—0.0005
13	10	0.0803	0.0010	0.0792	0.0006	—0.0011
14	10	0.0402	0.1000	0.0396	0.0984	—0.0006

An inspection of the table will show that the results for iodide are all low with the exception of Expt. 11, which is probably due to an error of manipulation. Much better results can be obtained by the use of the permanganate method. The results for bromide are also low, but it is believed that this method will give more accurate results for the bromide content of such mixtures than any other method.

<sup>1</sup> Read before the Division of Water, Sewage and Sanitation, at the 58th Meeting of the American Chemical Society, Philadelphia, Pa., September 5, 1919.

<sup>2</sup> THIS JOURNAL, 11 (1919), 563.

<sup>3</sup> *Ibid.*, 11 (1919), 954.



# LABORATORY AND PLANT

## AUTOMATIC METHODS OF GAS ANALYSIS DEPENDING UPON THERMAL CONDUCTIVITY<sup>1</sup>

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### PRINCIPLE OF THE METHOD

When a wire, surrounded by a gas enclosed in a space the walls of which are at constant temperature, is continuously traversed by a constant electrical current, the wire rises in temperature until a state of equilibrium is reached, in which the continuous dissipation of thermal energy is equal to the electrical energy supplied. This dissipation of energy takes place in the four following ways:

- 1—By radiation.
- 2—By conduction through the connections to the ends of the wire.
- 3—By thermal convection, *i. e.*, by currents of gas circulating in the atmosphere surrounding the wire.
- 4—By conduction through the gas.

In case there is a flow of gas past the wire, heat is also carried away by the gas stream.

By a proper choice of conditions, all loss of heat except by conduction through the gas surrounding the wire may be reduced to a small portion of the whole. The equilibrium temperature attained by the wire will then depend mainly upon the thermal conductivity of the gas which surrounds the wire, being lower when the gas has a high conductivity and higher when the gas has a low conductivity. Since the coefficients of thermal conductivity of various gases and gas mixtures differ widely (Table I), the temperature of the wire will be determined by the composition of the surrounding gas. If, now, the wire has a high temperature coefficient of electrical resistance, its resistance to the passage of a constant current of electricity, depending as it does on the temperature of the wire, will have a value corresponding to the thermal conductivity, and therefore to the composition of the surrounding gas or gas mixture.

TABLE I—THERMAL CONDUCTIVITY OF GASES  
(From Smithsonian Physical Tables)

GAS	$k_l \times 10^4$
Air.....	0.568
Argon.....	0.389
Ammonia.....	0.458
Carbon monoxide.....	0.499
Carbon dioxide.....	0.307
Ethylene.....	0.395
Helium.....	3.39
Hydrogen.....	3.27
Methane.....	0.647
Nitrogen.....	0.524
Nitrous oxide.....	0.350
Oxygen.....	0.563

( $k_l$  is the heat in gram-calories flowing in 1 sec. through a distance of 1 cm. per sq. cm. for 1° C. drop in temperature.)

Inasmuch as electrical circuits are easily arranged for the rapid and very accurate determination or comparison of the resistance of such a wire, the quantitative determination of the constituents in a wide variety of gas mixtures whose qualitative com-

positions are known is possible by a simple and exceedingly sensitive method based upon the thermal conductivity of the mixtures.

### HISTORY OF THE METHOD

Schleiermacher<sup>1</sup> developed a method for determining the thermal conductivities of gases by utilizing the fact that a platinum or nickel wire stretched along the axis of a glass tube immersed in a water bath assumes a temperature, when heated by an electric current of constant strength, which depends upon the nature of the gas surrounding the wire. Goldschmidt<sup>2</sup> improved Schleiermacher's original method by comparing two wires of different lengths inclosed in silver capillaries inserted in a massive brass block. This improved method was used by Eucken<sup>3</sup> in his experiments on the thermal conductivity of gases. The use of thermal conductivity for gas analysis was probably first suggested by Leon Sowzee about 1880. Unfortunately a description of his method is not available since the report of the Prussian Fire Damp Commission, in which the only known reference to it occurs, gives no details. The Vereinigte Maschinenfabrik, Augsburg, patented in Germany<sup>4</sup> and England<sup>5</sup> the use of the thermal conductivity of a gas containing hydrogen to obtain an indication or a record of the variation in composition of a gas mixture. Several devices are specified, in one of which the resistance of an electrically heated wire in a closed tube is measured. This firm appears, however, to have been unsuccessful in applying the method. A. Koepsel<sup>6</sup> developed an apparatus for the determination and continuous indication of the hydrogen content of producer gas, which he stated was capable of easily detecting the presence of 0.001 per cent of hydrogen without the use of very refined measuring apparatus. He also proposed the application of his apparatus to the determination of carbon dioxide in flue gases and of methane in mine gases, but did not demonstrate experimentally the feasibility of his proposals. In its most improved form, Koepsel's apparatus consisted of four electrically heated wires exposed in pairs to the two gases to be compared, the two wires of each pair being connected to form opposite arms of a Wheatstone bridge. Koepsel's apparatus was particularly designed for use in a rather rapid gas stream and rather elaborate housings were arranged for the wires to shield them from the effects of convection. Siemens and Halske<sup>7</sup> improved Koepsel's apparatus by passing the gases going to the two sides of the bridge through tubes in the same metal block to bring them to the same temperature, and eliminated Koepsel's special housing for the wires.

<sup>1</sup> *Wied. Ann.*, **34** (1888), 623; **36** (1889), 346.

<sup>2</sup> *Physik. Z.*, **12** (1911), 417.

<sup>3</sup> *Ibid.*, **12** (1911), 1101.

<sup>4</sup> D. R. P. 165,349 (1904).

<sup>5</sup> Brit. Patent 15,706 (1904).

<sup>6</sup> *Ber. physik. Ges.*, **10** (1908), 814; **11** (1909), 237; *Z. Chem. apparat.*, **3** (1908), 377, 401; *Chem. Abs.*, **2** (1908), 3212; **3** (1909), 1108, 2646.

<sup>7</sup> D. R. P. 283,677 (1913).

<sup>1</sup> Published by permission of the Director of the Bureau of Standards.



In 1915 or 1916 the Sperry Gyroscope Company developed an apparatus similar to that of Siemens and Halske for detecting hydrogen in air. About the same time, but before learning of the work of this company, some investigations along the same line were begun at the Bureau of Standards but were abandoned because the delicacy of the indicating instrument required made it unsuitable for the immediate purpose in view. In the first months of 1917 a gas analysis apparatus utilizing the thermal conductivity principle was developed by a group of men at the University of California, was submitted to the Navy Department, and was tried out successfully for a number of purposes. In this instrument, which was extremely simple, two arms of the Wheatstone bridge consisted of wires stretched in the axes of metal tubes and exposed to the gases to be compared; the other two arms of the bridge were fixed external resistances. Prof. G. A. Shakespear<sup>1</sup> of Cambridge, England, has developed an apparatus similar in principle to that of the "University of California Hydrogen Detector," though of more compact construction, small platinum coils being substituted for the straight wires used in the earlier instrument.

As far as it is known, these investigators confined their methods to the use of a Wheatstone bridge with fixed resistances, depending upon the deflection of a galvanometer or millivoltmeter for indicating the relative resistance of the heated wires exposed to the gas to be analyzed and to a "standard gas," usually air.

About the first of January 1918, work was begun at the Bureau of Standards to utilize a thermal conductivity method in connection with a number of problems, and in addition to the schemes used by the other investigators, a number of other combinations were tried; and what is believed to be the most generally useful form of apparatus was arrived at by a process of elimination.

The history of this development is given in such detail in order to clear up a situation which might otherwise give rise to ill feeling. It is known that one of the recent experimenters believed himself the first to utilize heat conductivity measurements for making gas analyses until his apparatus was submitted to the Bureau of Standards for test, and a similar claim has been repeatedly made for another investigator, though not by the investigator himself.

In July 1918, at the request of the Nitrate Division of the Ordnance Department of the Army, the solution of the problem of the continuous analysis of the gases was undertaken for the analytical control of the synthetic ammonia plant being erected at Sheffield, Alabama. It was planned to use the thermal conductivity method for much of this work. At the time of the signing of the armistice when work on the ammonia plant was discontinued, the apparatus had been constructed and calibrated and was practically ready for installation in the plant.

The feasibility of using the method for the continuous analysis of the gases at different stages in the

separation of helium from natural gas was indicated by a few experiments. At the request of the Navy Department, this application of the method was developed, and apparatus for determining and automatically recording the percentage of helium in helium-nitrogen mixtures was built, calibrated, and installed in one of the helium extraction plants where it is reported to have given very satisfactory service for several months. Work has started on the construction of equipment for a 16-point analytical control apparatus for installation in another helium plant.

As opportunity has offered, experiments have been made to ascertain the applicability of the method to various important industrial processes. The uniform success which has been obtained indicates that its field of usefulness extends to a large number of operations and processes where a knowledge of changes in composition of gas mixtures is desired.

#### DESCRIPTION OF ELECTRICAL ARRANGEMENTS

**WHEATSTONE BRIDGE WITH INDICATOR**—In the device suggested by Koepsel<sup>1</sup> the indications of the changes in composition of a gas mixture were obtained in the following manner: Four wires of like material, cross-section, and length constituted the arms of a Wheatstone bridge. Two of the wires, forming opposite arms of the bridge, were placed together in a cell through which passed a current of the gas for analysis; the other two wires were placed together in a cell through which passed a comparison gas. Thus the differences in thermal conductivity of the gases in the two cells resulted in a maximum of unbalance of the bridge. An instrument calibrated to indicate directly the percentages of a constituent of the gas mixture of variable composition replaced the customary galvanometer. Since the resistance of the wire is very materially affected by changes in the current strength, the current must be carefully adjusted and maintained at a definite value. Koepsel used a battery as a source of constant current and avoided the use of an additional current-measuring instrument by providing a resistance coil between the battery and the bridge across the terminals of which the indicator could be switched. Current through the bridge was adjusted by means of a rheostat in the battery circuit until the deflection of the pointer of the indicator reached a certain mark on the scale. Fig. 1 shows diagrammatically the arrangement of the apparatus.

This arrangement, which is essentially the same as that developed by the other investigators mentioned, has many limitations. In order that the apparatus may detect small changes in the composition of the gas mixture, the indicator must have a high sensitivity, making it a necessarily expensive instrument, and since there must be a special scale for each gas and each range of gas percentages if the apparatus is to indicate percentages of the desired constituent directly, it becomes impracticable to use the same instrument for the determination of more than a very few constituents in a gas mixture and only over a

<sup>1</sup> U. S. Patent 1,304,208 (1919).

<sup>1</sup> *Loc. cit*



small number of ranges. Furthermore, the usefulness of apparatus for the analysis of gas mixtures whose composition is continuously changing is frequently dependent upon whether the apparatus can be arranged to provide a record of the changes in gas composition, to actuate control mechanisms, or to positively sound an alarm. For these purposes neither Koepsel's apparatus nor any other apparatus utilizing the same type of bridge arrangement is suited.

relative resistances of the two wires and of the presence and amount of one or more constituents of the gas mixture. Thus, the expensive specially calibrated galvanometer of small range or close scale is in effect replaced by a uniformly divided slide wire of very large range and wide open scale as the measuring instrument.

In order to determine when the current through the bridge just described is correctly adjusted, a second bridge is connected in series. Three arms of this bridge are fixed resistances, and the fourth arm is a wire, exactly similar to the wires of the first bridge, stretched along the axis of a gas-tight metal tube. Since the resistance of this wire constituting the fourth arm varies greatly with small changes of current, the current is adjusted very accurately when the bridge is balanced. By adjusting one of the fixed resistance arms, the bridge may be made to balance with any desired current strength. Both the conductivity apparatus and the current adjustor are placed in an electrically heated oil bath with thermostat. If desired, the same galvanometer may be used for both current adjustment and the conductivity bridge.

When the limit of variation of the desired constituent of a gas mixture is about 10 or 20 per cent, it has been found that by making the extension coils of the slide wire of the proper resistance, the position of the sliding contact with respect to a uniformly divided scale indicates directly and with considerable accuracy the percentages of the constituent when the bridge is adjusted to give no deflection of the galvanometer needle. Consequently the process of calibration consists of determining the values of the resistances which the two bridge extension coils should have for the constituent and gas mixture in question. The calibration of apparatus for the determination of 0-10 per cent carbon dioxide in air will suffice to illustrate a method for finding the value of the end resistances. Fig. 2 shows diagrammatically the arrangement of the apparatus.

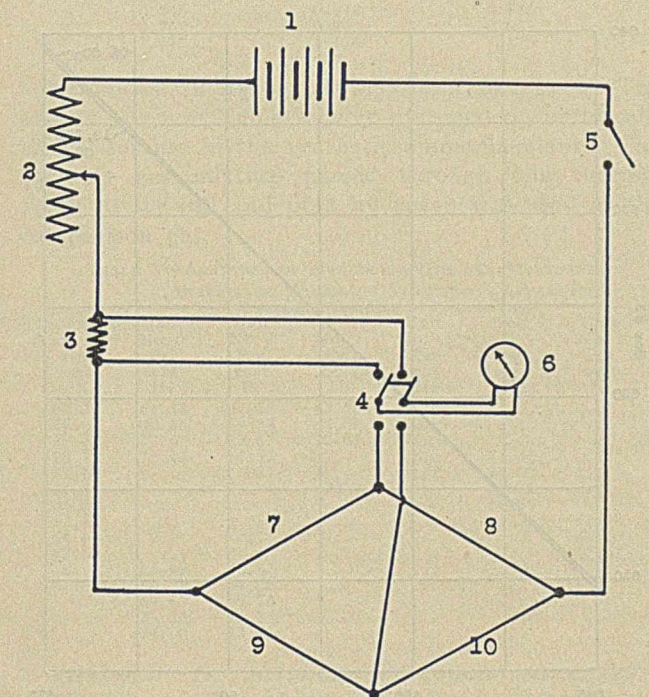


FIG. 1

- |                                     |                                      |
|-------------------------------------|--------------------------------------|
| 1—Battery                           | 5—Switch                             |
| 2—Rheostat                          | 6—Indicator                          |
| 3—Drop coil (shunt)                 | 7, 10—Wires exposed to gas mixture   |
| 4—Double knife, double throw switch | 8, 9—Wires exposed to comparison gas |

**BALANCED BRIDGE ARRANGEMENT**—These and other considerations led to the adoption of a balanced bridge arrangement in the method devised at the Bureau of Standards. Two wires, preferably of like material, cross-section, and length, stretched along the axes of two tubes in the same metal block, constitute two arms of a Wheatstone bridge. A Kohlrausch slide wire, such as is used in measurements of the conductivity of electrolytes, together with extension resistances ("end coils") form the other two arms. A portable, inexpensive, d'Arsonval needle galvanometer is used to indicate the balance point. The gas mixture to be analyzed is passed through the tube containing one of the wires; a comparison gas having a constant composition, such as air or hydrogen, and having a thermal conductivity of the same order as that of the gas mixture, or else the residual gas mixture after a constituent has been removed is passed through the tube containing the other wire. The bridge is balanced by moving the contact on the slide wire until the galvanometer gives a zero reading. After the apparatus has been calibrated the position of the contact on the slide wire is an indication of the

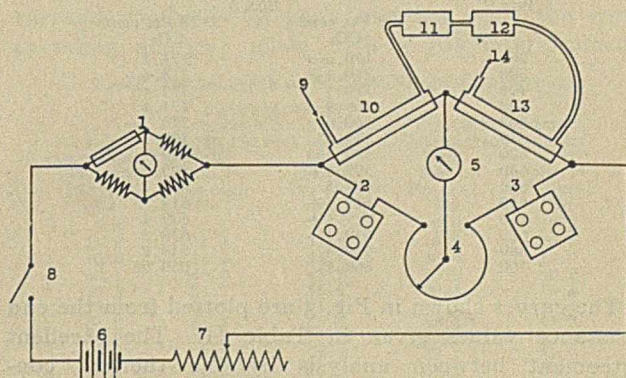


FIG. 2

- |                                 |                                   |
|---------------------------------|-----------------------------------|
| 1—Current adjustment bridge     | 8—Switch                          |
| 2, 3—Four dial resistance boxes | 9—Entrance for gas mixture        |
| 4—Slide wire bridge             | 10, 13—Thermal conductivity cells |
| 5—Galvanometer                  | 11—Carbon dioxide absorber        |
| 6—Storage battery               | 12—Drying tube                    |
| 7—Rheostat                      | 14—Exit for residual gas          |

With the contact of the slide wire bridge (4) set at zero, pure, dry air free from carbon dioxide is allowed to flow at a rate of about 20 cc. per minute through



the entrance (9) into the first conductivity cell (10), thence to a carbon dioxide absorber (11), and, after being dried over calcium chloride (12), passes through the second conductivity cell (13) and is discharged at (14). When the gas circuit is thoroughly swept out the switch (8) is closed, the setting of the rheostat (7) is varied until the current adjustment bridge (1) is balanced, and with one resistance box, *e. g.*, (2), set at a definite value, the other resistance box (3) is adjusted until the galvanometer (5) gives no deflection. Care must be taken that the current through the circuit is correct when the final adjustment is made. The settings of the two resistance boxes are noted and by increasing the resistance in (2) by regular steps and obtaining the corresponding values of (3) when the galvanometer (5) gives a zero reading, a series of values of correct end resistances is secured for the lower limit of carbon dioxide content.

A mixture of approximately 10 per cent carbon dioxide-90 per cent air is then prepared in a mercury-sealed gas-holder, carefully analyzed by volumetric apparatus, and then allowed to flow through the gas circuit of the thermal conductivity apparatus as described above for air. With the contact of the slide wire bridge (4) set at the scale division corresponding to the average of the volumetric analyses of carbon dioxide, another series of correct end resistance values for the upper limit of carbon dioxide content is secured.

Choosing the resistances of one box as ordinates and of the other as abscissae, end resistance curves corresponding to the lower and upper limits of carbon dioxide variation are plotted. The intersection of these curves gives the value of the end resistances to be used for this range of variation of the desired constituent in the given gas mixture.

Table II shows two series of end resistance values for a 0-10 per cent carbon dioxide-in-air calibration.

TABLE II—END RESISTANCE VALUES FOR CO<sub>2</sub>-IN-AIR CALIBRATION  
(Resistance in Ohms)

Box 2	0 Per cent CO <sub>2</sub>	Box 3 10.28 Per cent CO <sub>2</sub>
200	170.6—	174.7
300	270.4+	273.6+
400	370.1	372.45
500	469.9	471.4
600	569.6	570.2—
610	579.55	580.1
620	589.55	589.9
630	599.5+	599.8
640	609.5	609.7
650	619.5+	619.55
660	629.4+	629.4
670	639.4	639.3
680	649.4	649.2
700	669.4	669.05

The curves shown in Fig. 3 are plotted from the end resistance values given in Table II. The excellent agreement between analysis by the thermal conductivity apparatus and by careful volumetric analyses of several carbon dioxide-air mixtures is shown in Table III.

TABLE III—ANALYSES OF CO<sub>2</sub>-AIR MIXTURES

AVERAGES OF VOLUMETRIC ANALYSES Per cent CO <sub>2</sub>	ANALYSIS BY THERMAL CONDUCTIVITY APPARATUS (Uncorrected Reading of Instrument) Per cent CO <sub>2</sub>
0.00	0.20
2.65	2.68
4.85	4.85
7.12	7.14
10.28	10.28

By the use of a suitable switch the same slide wire, galvanometer, and current adjustor may be employed in connection with any desired number of thermal conductivity units. It is only necessary to provide special end resistance coils, which are easily and cheaply constructed, with each of the conductivity units to make the apparatus indicate directly the percentages of the constituents in the various gas mixtures for which the instrument has been calibrated.

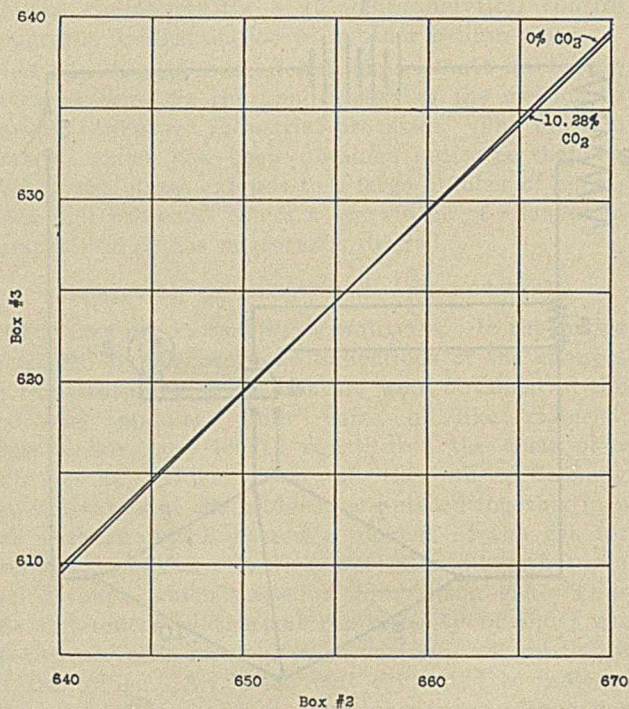


FIG. 3.

RECORDER—Unlike the devices used or suggested by others for gas analysis by methods depending upon thermal conductivity, the manually operated arrangement just described may be readily adapted to give a continuous record of gas composition, to actuate plant control mechanism, and to sound an alarm. For these purposes it is only necessary to substitute for the galvanometer and slide wire a recorder of the type manufactured by the Leeds and Northrup Company arranged to adjust automatically the position of a contact on a slide wire until a galvanometer gives no deflection, to draw a curve, or to otherwise provide a record of the position of the contact with respect to the slide wire, and, if desired, to operate mechanical or electrical devices of any suitable construction.

#### REPRESENTATIVE ANALYSES

In order to indicate the wide applicability of the thermal conductivity method which has been developed and to show the accuracy which has been obtained by its use a series of representative analyses is given below.

HYDROGEN IN HYDROGEN-AIR MIXTURES—The following results were obtained in the calibration of apparatus intended for use in determining the permeability of balloon fabrics to hydrogen. The gas mixture for analysis surrounded one wire of the thermal conductivity apparatus, and pure dry air the other.



TABLE IV—ANALYSES OF HYDROGEN-AIR MIXTURES  
ANALYSIS BY THERMAL CONDUCTIVITY APPARATUS

ANALYSIS BY GAS INTERFEROMETER Per cent H <sub>2</sub>	(Uncorrected Reading of Instrument) Per cent H <sub>2</sub>	Analysis from Calibration Curve Per cent H <sub>2</sub>
0.096	0.092	0.096
0.150	0.147	0.150
0.207	0.201	0.203
0.204	0.202	0.204
0.231	0.229	0.231
0.278	0.276	0.277
0.406	0.408	0.407
0.556	0.558	0.555
0.704	0.707	0.704
0.846	0.848	0.846
0.962	0.964	0.962
1.175	1.176	1.175

HYDROGEN IN HYDROGEN-NITROGEN MIXTURES—The results given in Table V were obtained on units calibrated for use in the synthetic ammonia plant. The variable gas mixture passed through one thermal conductivity cell and pure hydrogen was used as the comparison gas.

TABLE V—ANALYSES OF HYDROGEN-NITROGEN MIXTURES  
ANALYSIS BY THERMAL CONDUCTIVITY APPARATUS

AVERAGES OF VOLUMETRIC ANALYSIS Per cent H <sub>2</sub>	(Uncorrected Reading of Instrument)					
	Unit No. 6 Per cent H <sub>2</sub>	Unit No. 8 Per cent H <sub>2</sub>	Unit No. 9 Per cent H <sub>2</sub>	Unit No. 11 Per cent H <sub>2</sub>	Unit A Per cent H <sub>2</sub>	Unit No. 2 Per cent H <sub>2</sub>
67.58	67.58	67.58	67.58	67.58	...	...
69.63	69.85	69.88	69.69	69.69	...	...
72.67	72.67	72.67	72.67	72.67	...	...
75.43	75.44	75.52	...	...	75.41	...
74.00	...	...	...	...	74.00	...
71.40	...	...	...	...	71.40	...
66.50	...	...	...	...	66.50	...
42.48	...	...	...	...	...	42.48
44.65	...	...	...	...	...	44.75
47.62	...	...	...	...	...	47.62
...	65.99	65.87	...	...	...	...
...	66.22	66.28	...	...	...	...
...	69.63	69.75	...	...	...	...
...	71.30	71.35	...	...	...	...
...	74.00	73.93	...	...	...	...

NITROGEN IN NITROGEN-HYDROGEN MIXTURES—Table VI shows analyses made by a unit for the synthetic ammonia plant. Pure hydrogen was used as the comparison gas.

TABLE VI—ANALYSES OF NITROGEN-HYDROGEN MIXTURES  
ANALYSIS BY THERMAL CONDUCTIVITY APPARATUS

AVERAGES OF VOLUMETRIC ANALYSES Per cent N <sub>2</sub>	(Uncorrected Reading of Instrument) Per cent N <sub>2</sub>
3.43	3.43
5.30	5.35
8.60	8.60

CARBON DIOXIDE IN CARBON DIOXIDE-AIR MIXTURES—Refer to Table III.

CARBON DIOXIDE IN MIXTURES OF HYDROGEN, NITROGEN, AND CARBON DIOXIDE—In Tables VII and VIII are given the results obtained on apparatus

TABLE VII—ANALYSES OF CO<sub>2</sub>, HYDROGEN, AND NITROGEN MIXTURES (UNIT A)

Hydrogen in Residue Per cent H <sub>2</sub>	Nitrogen in Residue Per cent N <sub>2</sub>	Averages of Volumetric Analyses (Uncorrected Reading)	
		Per cent CO <sub>2</sub>	Per cent CO <sub>2</sub>
40	60	2.42	2.45
40	60	2.53	2.53
40	60	2.79	2.73
40	60	4.55	4.55
40	60	5.30	5.20
40	60	6.90	6.90
40	60	7.69	7.60
40	60	10.05	10.05
37.33	62.67	0.00	0.00
37.33	62.67	1.00	0.90
37.33	62.67	3.29	3.29
37.33	62.67	6.05	6.00
37.33	62.67	8.10	8.27
37.33	62.67	10.03	10.23
29.36	70.64	0.00	0.05
29.36	70.64	2.37	2.30
29.36	70.64	3.67	3.64
29.36	70.64	6.55	6.55
29.36	70.64	10.82	11.07

TABLE VIII—ANALYSES OF CO<sub>2</sub>, HYDROGEN, AND NITROGEN MIXTURE (UNIT B)

Hydrogen in Residue Per cent H <sub>2</sub>	Nitrogen in Residue Per cent N <sub>2</sub>	Average of Volumetric Analyses Per cent CO <sub>2</sub>	Analysis by Thermal Conductivity Apparatus (Uncorrected Reading) Per cent CO <sub>2</sub>
70	30	22.52	22.52
..	..	25.15	25.15
..	..	27.53	27.53
..	..	27.80	27.77

calibrated for the synthetic ammonia plant. The apparatus was arranged in the same manner as that described for the determination of carbon dioxide in air. It is to be noted in Table VII that, though the hydrogen-nitrogen ratio of the mixtures has been varied considerably, the analyses of carbon dioxide by the thermal conductivity method are almost unaffected.

AMMONIA IN MIXTURES OF AMMONIA, NITROGEN, AND HYDROGEN—Ammonia is determined by passing the gas mixture first through one conductivity cell, then through an ammonia absorber, and finally through the second thermal conductivity cell. In Table IX analyses made by two units are given. The residue after the removal of ammonia contained 75 per cent hydrogen and 25 per cent nitrogen.

TABLE IX—ANALYSES OF AMMONIA, NITROGEN, AND HYDROGEN MIXTURES  
ANALYSIS BY THERMAL CONDUCTIVITY APPARATUS

AVERAGES OF VOLUMETRIC ANALYSES Per cent NH <sub>3</sub>	Unit No. 10 Per cent NH <sub>3</sub>	Unit A Per cent NH <sub>3</sub>
0.27	0.39	0.30
2.55	2.52	2.42
2.63	2.65	2.62
5.26	5.20	5.10
5.78	5.63	5.55
8.03	7.96	7.85
8.48	8.36	8.28

HELIUM IN MIXTURES OF HELIUM AND NITROGEN—Helium in mixtures of helium and nitrogen is determined by passing the gas mixture through one thermal conductivity cell while hydrogen is passed through the other cell as a comparison gas. In Table X are shown the uncorrected readings made by a recorder when used in connection with two different thermal conductivity units together with the corresponding analyses made with a gas interferometer.

TABLE X—ANALYSES OF HELIUM-NITROGEN MIXTURES

GAS INTERFEROMETER ANALYSES Per cent He	UNCORRECTED RECORDER READINGS 0-100 Per cent Range		ANALYSES FROM CALIBRATION CURVES	
	Unit No. 1 Per cent He	Unit No. 2 Per cent He	Unit No. 1 Per cent He	Unit No. 2 Per cent He
9.8	7.1	7.5	9.8	10.0
19.8	21.3	22.5	19.8	20.7
30.0	34.3	34.3	30.0	29.9
40.1	45.0	45.7	40.0	40.2
50.3	54.7	55.0	50.3	50.3
58.4	60.9	61.4	58.3	58.4
63.6	64.7	65.0	63.6	63.3
71.8	70.7	71.2	72.4	72.3
82.7	77.2	77.7	82.7	82.7
87.7	80.2	80.7	87.6	87.6
88.8	81.0	81.8	88.9	89.5
90.5	82.0	82.4	90.8	90.5
93.1	83.2	84.0	93.3	93.1
	80-100 Per cent Range			
82.7	81.7	82.8	82.7	82.7
87.7	88.5	89.8	87.7	87.9
88.8	90.0	91.2	88.9	89.0
90.5	91.7	92.7	90.1	90.1
93.1	95.5	96.7	93.1	93.1

Although the direct readings taken from the recorder chart deviate considerably from the corresponding interferometer analyses, the discrepancies disappear



when the direct recorder readings are corrected by means of calibration curves.

#### CONSTRUCTION OF APPARATUS

**THERMAL CONDUCTIVITY UNITS**—The thermal conductivity units used in making the representative analyses cited in this paper are a product of evolution. In attempting to eliminate or avoid difficulties encountered in the early forms of construction the present form has resulted. Consequently, though an arrangement has been devised that works surprisingly well, future study will no doubt suggest further improvements.

In order to secure high sensitivity with a small consumption of energy it has been found that the

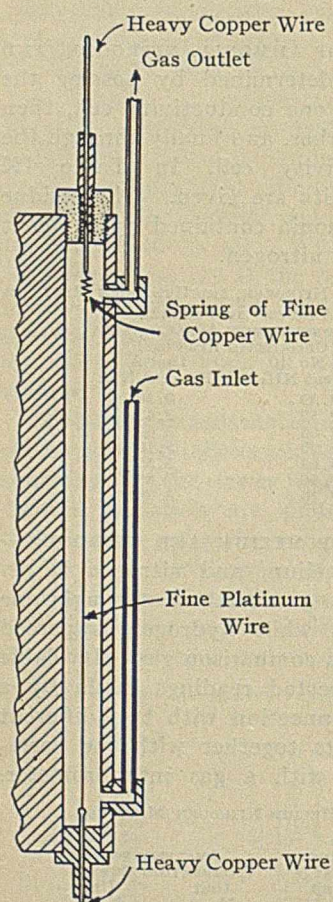


FIG. 4

wires exposed to the gas mixture for analysis and to the comparison gas should preferably be of small diameter and made from metals having a high temperature coefficient of resistance. Wires of nickel, tungsten, and platinum of various diameters and lengths have been successfully employed. Platinum, however, on account of its freedom from reaction with most gases, low thermoelectric power against copper, permanence of physical properties, and the ease of fusing it to supporting and connecting terminals, has been selected as the metal usually most suitable. The best diameter and length to choose for the wires depends upon the current strength to be employed and the sensitivity desired. Wires having diameters of 0.0254 and 0.0508 mm. (0.001 and 0.002 in.) and lengths of 15 and 10 cm. (6 and 4 in.) have given ample sensitivity with bridge currents of 0.15 ampere for the smaller wire and 0.30 ampere for the larger wire. In order to concentrate the resistance in the platinum wires, render the connections to the ends of the wires permanent and the junctions free from objectionable thermal e. m. f., copper wires are fused to each end of the platinum wire. To minimize the danger of the wire being displaced from its position when stretched along the axis of the tube forming the conductivity cell, one copper terminal is made of as heavy a wire as can be conveniently fused to the small platinum wire, and the other copper terminal is made of fine copper wire, which, after one end has been fused to

the small platinum wire, is wound into a helical spring and the free end is soldered to a heavy copper wire.

The gas chambers in which these platinum wires are stretched consist of two tubes in the same metal block, each provided with a gas inlet and outlet and bushings for supporting the wire stretched along the axis.

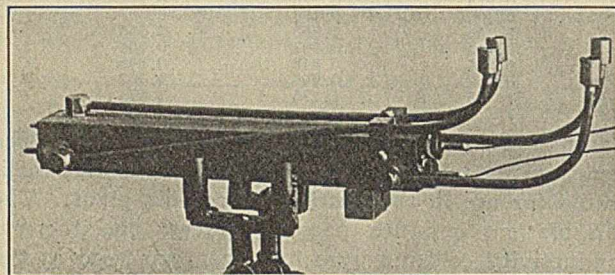


FIG. 5

Brass has been found a suitable metal where non-corrosive gases are used and the tubes are simply cylindrical holes drilled lengthwise through a square brass rod. To minimize the effect on the calibration of any possible displacement of the active wires and to reduce the power required to produce a given temperature difference between the wire and the wall of the tube, the diameter of the tubes or cells is made the maximum permissible. Experiments have shown that when the diameter much exceeds one centimeter convection currents cause unsteady readings. This unsteadiness appears rather suddenly as the diameter of the cell is increased, and the value of the limiting diameter is in somewhat striking agreement with the thickness of the stationary conducting gas film assumed by Langmuir<sup>1</sup> in his discussion of the loss of heat by conduction and convection from a cylindrical surface. Since the electrical arrangement used requires that the wires in the two cells be connected in series, it is necessary to insulate only one end of each wire from the metal block. This insulation is secured by Bakelite bushings screwed into one end of each cell and fitted with brass sleeves to which the wires may be

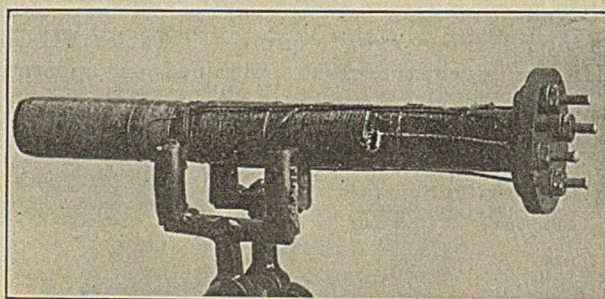


FIG. 6

soldered. The bushings are coated with Khotinsky cement to render the joints gas-tight. Fig. 4 shows a section through one of the cells with the active wire in place and Fig. 5 is a photograph of a complete unit.

<sup>1</sup> *Phys. Rev.*, 34 (1912), 401.



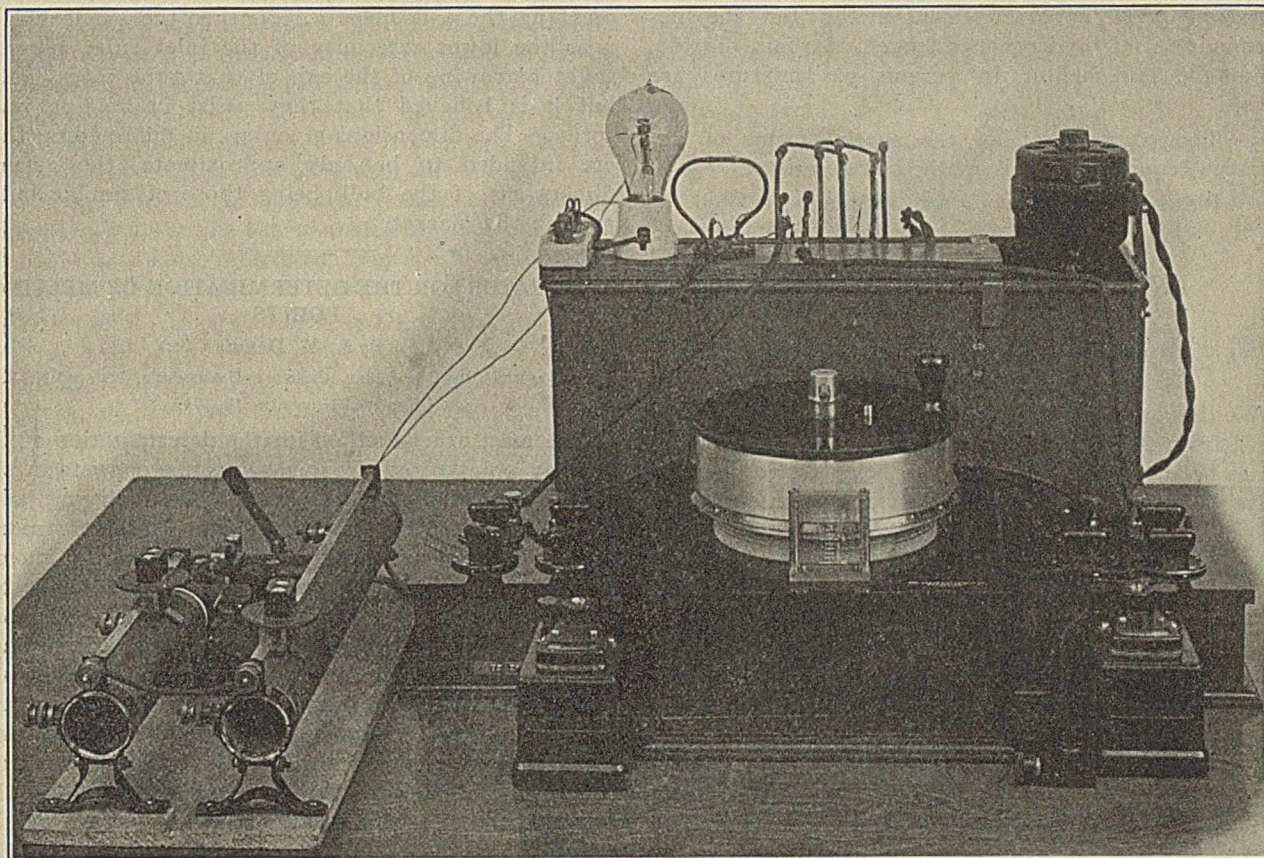


FIG. 7

**END RESISTANCE COILS**—After the values of the end resistances to be used with a thermal conductivity unit for the determination of a constituent in a gas mixture have been ascertained, as described in connection with the calibration of apparatus for the determination of carbon dioxide in air, the expensive resistance boxes are replaced by inexpensive coils. These coils are wound non-inductively on bobbins with advance or manganin wire, carefully adjusted to the proper resistances, and mounted for convenience in the oil bath together with the thermal conductivity units. A photograph of such a pair of end resistance coils is shown in Fig. 6.

An idea of the appearance of the manually operated apparatus as set up for experimental work on gas analysis by the thermal conductivity method may be had by inspection of the photograph shown in Fig. 7.

#### EXTENT OF STUDY OF METHOD AND PLANS FOR FUTURE WORK

Owing to the fact that loss of heat from the active wires in the conductivity cells takes place not only by conduction through the gas, but also to some unknown extent by convection, radiation, conduction through the terminals, and heating the stream of gas flowing past the wire, it has been considered inadvisable and not worth while to attempt to determine the mathematical relations which would make possible the calibration of the apparatus by calculation. Experience has shown that the results obtained by

empirical calibration are fully as reliable as those given by the apparatus with which the gas mixtures used in calibration are analyzed.

Although it is known in a general way that the readings are steady and reproducible when the rate of flow of the gas mixtures through the cells does not greatly exceed 20 cc. per minute, a careful study of the quantitative effect of changes in bath temperature and variations in bridge current is yet to be made. Plans for future work include the investigation of these effects, a study of the relative thermal conductivities of various gas mixtures, and further development of a number of devices which have already been applied in a preliminary way to the analysis of more complex gas mixtures than those cited, and to the application of the method to recording and automatic control work.

#### SUMMARY

1—A new form of apparatus for the continuous analysis of gas mixtures by an electrical method depending upon thermal conductivity is described.

2—Unlike the previous deflection methods of limited application, a balanced-bridge method applicable to the rapid and accurate determination of constituents in a wide variety of gas mixtures of industrial importance has been developed.

3—The adaptability of the apparatus for either manual or automatic recording operation is pointed out.



4—Representative analyses showing the results obtained with apparatus constructed for the determination of hydrogen, nitrogen, carbon dioxide, ammonia, and helium in various gas mixtures are given.

5—The construction of the special forms of apparatus required for this method is described.

6—Plans for future work are outlined.

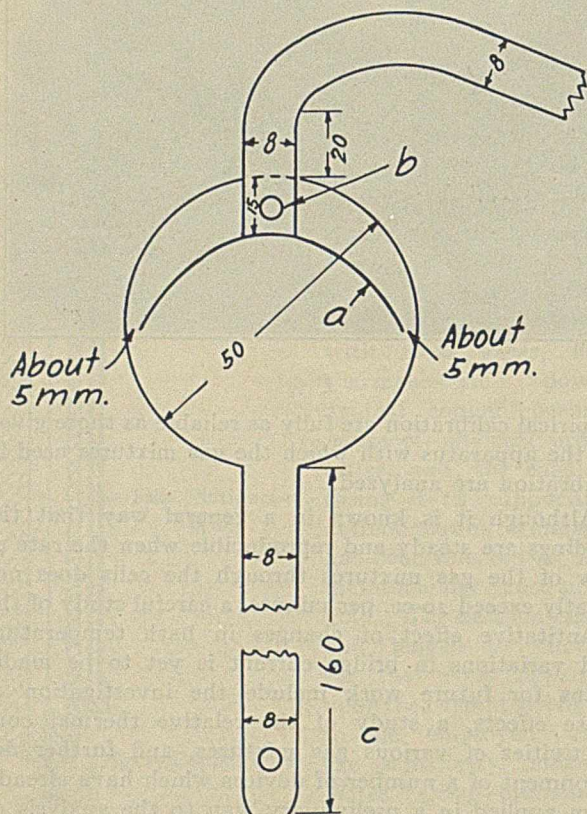
#### AN EFFECTIVE CONNECTING BULB

By C. M. Clark

THE CHATTAHOOGA CHEMICAL LABORATORY, CHATTAHOOGA, TENN.

Received November 5, 1919

We have frequent occasion in this laboratory to distil from flasks at a very rapid rate and have many times been troubled by the spray being carried over in spite of the best connecting bulbs we could obtain.



The writer was, in consequence, led to design the bulb herewith illustrated, which has on repeated trials and under the severest conditions fulfilled all requirements. It was first tested by half filling an Erlenmeyer flask with a 10 per cent solution of KOH and catching the distillate in water to which phenolphthalein has been added. Although the boiling was pushed until portions of the liquid were actually carried up into the bulb not a trace of color appeared in the catch water. Under such conditions all other bulbs on the market allowed spray to pass over, coloring the catch water red.

In the figure, *a* is a baffle-plate in the form of a deep watch glass, similar to that used in the "Pistorius" still. It reaches to within about 5 mm. of the sides

of the bulb and effectually prevents any spray from reaching the exit holes *b*. Neither the small hole *c* near the lower extremity of the inlet tube, nor the slight narrowing of the end of that tube is essential, but it is believed that they assist in holding back spray. The dimensions given are in millimeters; they are intended to be only approximate, the general dimensions of the bulb being those of similar bulbs now on the market.

#### APPARATUS FOR THE DETERMINATION OF MELTING POINTS

By L. M. Dennis

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Received November 19, 1919

The most exact method for the determination of the melting point of an organic substance consists in the observation of the melting point and solidifying point of the substance by direct immersion of the thermometer in the partly molten and partly solid mass.<sup>1</sup> This method is, however, frequently impracticable because at least 20 g. of material are necessary for the determination. It has therefore become customary to determine the melting point of an organic substance with approximate accuracy by placing a small amount of the material in a thin-walled glass tube about 1 mm. in diameter, fastening the tube to the side of a thermometer, and heating the thermometer and tube to the temperature at which the sample changes from the solid form to a clear and transparent liquid.

The heating of thermometer and sample tube is usually effected by immersing the two to proper depth in pure, concentrated sulfuric acid and then heating the acid until the melting point of the substance has been reached. It is apparent that if the acid that surrounds the bulb of the thermometer is unevenly heated, the readings may show considerable variation from the true melting point of the substance. For this reason it has been recommended<sup>2</sup> that the temperature be raised very slowly through the last 10°. It is preferable, however, to stir the sulfuric acid while it is being heated, and this can be accomplished either by mechanical stirring with a bent glass rod, or by means of the ingenious device of Olberg<sup>3</sup> in which the heating of the sulfuric acid causes the acid to circulate through the apparatus and thus tends to equalize the temperature throughout the liquid. This tube of Olberg has been simplified by Thiele.<sup>4</sup> The accuracy of a determination of the melting point of a substance with the Thiele tube is largely dependent upon the speed of flow that is set up in the sulfuric acid when the tube is heated. Acceleration of the circulation of the acid will naturally give better results. Very considerable errors that had been made by students in the determinations of the melting points with the Thiele tube seemed to indicate a too slow circulation of the acid.

The Thiele tube is heated at or just below the bend

<sup>1</sup> Lindenbaum, "Die Methoden der Organischen Chemie," I, p. 193.

<sup>2</sup> Wegscheider, *Chem.-Ztg.*, 29 (1905), 1224.

<sup>3</sup> *Repert. anal. Chem.*, 1886, 95.

<sup>4</sup> *Ber.*, 40 (1907), 996.



of the side loop.<sup>1</sup> The rise of the warm, specifically lighter acid through the upper side of this loop toward the main tube is, however, retarded by the slightness of the pitch of the tube and its relatively small diameter as compared with that of the upright portion of the tube. This slowness of movement of the acid causes marked differences of temperature of the upper and lower portions of the acid in the upright tube where the bulb of the thermometer and the substance are located, and also retards the even rise of temperature of the acid throughout the apparatus.

With the object of accelerating the circulation of the sulfuric acid and the equalization of temperature throughout the acid, with consequent increase in the accuracy of the results of the determinations, the writer made a tube of the shape shown in Fig. 1, and this and a Thiele tube were subjected to detailed and careful comparison by Mr. Raleigh Gilchrist. The tubes were filled with pure, concentrated sulfuric acid to a point about one cm. above the opening of the upper side-arm D. The temperatures were measured with two Anschütz thermometers which were first carefully compared to ascertain their variation. In the temperature readings that are given in the tables, correction is made for these variations. The thermometer T was so placed that its bulb was opposite the upper opening of the side-arm. The bulb of the other thermometer, S, was 4 cm. below that of T and was slightly to the left of the middle of the larger tube. The thermometers were similarly placed in the Thiele tube. The two tubes were heated with small Bunsen flames 2.5 cm. high, so placed that the tip of the flame was 4.5 cm. below the tube. The Thiele tube was heated on the lower side of the outer bend of the side-arm; the new form was heated slightly to the right of the point marked A. In making comparisons of the tubes, the Bunsen flame was placed in position and readings of the two thermometers were made at one minute intervals.

It will be seen from Table I that the Thiele tube, although it showed a much slower rise in temperature than did the new form, gave differences in the readings of the two thermometers that on the average were more than twice as great as those with the new form.

TABLE I—COMPARISON AT LOW TEMPERATURES

THIELE TUBE				NEW FORM OF TUBE			
Thermometer S °C.	Thermometer T °C.	Difference °C.	Duration of heating Min.	Thermometer S °C.	Thermometer T °C.	Difference °C.	Duration of heating Min.
22.6	24.8	2.2	1	22.6	23.8	1.2	1
22.8	25.0	2.2	2	24.6	26.6	2.0	2
23.0	25.4	2.4	3	26.4	27.6	1.2	3
23.2	26.0	2.8	4	27.4	27.8	0.4	4
23.6	26.2	2.6	5	28.0	28.6	0.6	5
23.8	26.2	2.4	6	29.2	30.8	1.6	6
24.0	26.2	2.2	7	30.6	31.8	1.2	7
24.2	26.4	2.2	8	31.6	32.4	0.8	8
24.4	26.4	2.0	9	32.0	33.0	1.0	9
24.6	26.6	2.0	10	32.8	33.8	1.0	10
24.6	26.8	2.2	11	33.4	34.2	0.8	11
24.8	27.0	2.2	12	33.8	34.6	0.8	12
Total rise of temperature in 12 min. .... 2.2°				Total rise of temperature in 12 min. .... 11.2°			
Average difference of thermometers ..... 2.29°				Average difference of thermometers ..... 1.05°			

With the beginning temperature in the neighborhood of 100°, Table II again shows that when the two

<sup>1</sup> A figure of the Thiele tube is also given in "Analyse und Konstitutionsermittlung organischer Verbindungen," by Hans Meyer, p. 95.

tubes are heated in as nearly the same manner as feasible with small gas flames, the Thiele tube shows much slower rise in temperature and nearly nine times as great an average difference between the two thermometers as does the new form of tube.

TABLE II—COMPARISON AT HIGHER TEMPERATURES

THIELE TUBE				NEW FORM OF TUBE			
Thermometer S °C.	Thermometer T °C.	Difference °C.	Duration of heating Min.	Thermometer S °C.	Thermometer T °C.	Difference °C.	Duration of heating Min.
98.2	106.2	8.0	1	98.8	100.0	1.2	1
99.0	106.2	7.2	2	102.2	103.8	1.6	2
99.6	107.6	8.0	3	106.0	107.0	1.0	3
100.2	108.4	8.2	4	108.4	109.0	0.6	4
101.2	109.0	7.8	5	111.2	112.2	1.0	5
102.0	110.0	8.0	6	113.8	114.6	0.8	6
102.6	110.6	8.0	7	115.4	115.6	0.2	7
103.0	110.0	7.0	8	117.0	117.6	0.6	8
Total rise of temperature in 8 min. .... 4.8°				Total rise of temperature in 8 min. .... 18.2°			
Average difference of thermometers ..... 7.77°				Average difference of thermometers ..... 0.87°			

Tables I and II show the differences of temperature during the heating of the sulfuric acid in that portion of the acid which surrounds the thermometer bulb and sample tube. As the melting point of the substance is approached, the accuracy of the determination will naturally be greatest if the acid in the wide portion of the tube is of the same temperature throughout. The slower the circulation of the acid, or, in other words, the greater the differences in temperature between the upper and lower portions of acid in the wide part of the tube, the greater will be the probability of error in the result of the determination.

To ascertain the completeness of the equalization of temperature in the two forms of tube when they were held within comparatively narrow ranges, Mr. Gilchrist made the measurements that are given in Table III.

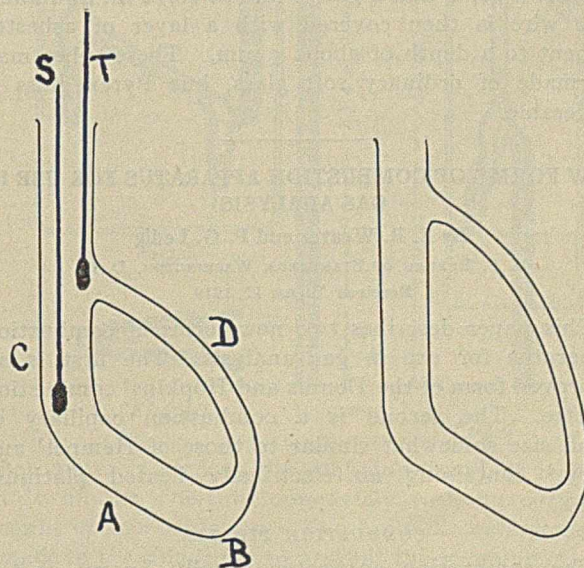


FIG. 1

FIG. 2

The form shown in Fig. 1 is entirely satisfactory for student use. However, at the suggestion of Prof. W. R. Orndorff, a tube of the shape shown in Fig. 2 has also been made. In this case the side-arm enters the large tube at a higher point, giving a taller column of sulfuric acid in the main tube. This permits the immersion of an Anschütz thermometer in the sul-



furic acid to such a depth that the top of the column of mercury in the thermometer is below the opening from the upper side-arm. The necessity of making correction for a portion of the mercury column above the sulfuric acid is thus obviated. A test of this form of tube has not as yet been made.

TABLE III—COMPARISON OF UNIFORMITY OF TEMPERATURE

THIELE TUBE				NEW FORM OF TUBE			
Thermometer S ° C.	Thermometer T ° C.	Difference ° C.	Duration of heating Min.	Thermometer S ° C.	Thermometer T ° C.	Difference ° C.	Duration of heating Min.
102.6	110.6	8.0	1	132.2	132.8	0.6	1
103.0	110.0	7.0	1	131.8	132.8	1.0	1
102.8	110.2	7.4	2	132.6	131.8	0.8	2
102.8	111.2	8.4	3	131.0	131.4	0.4	3
102.2	110.6	8.4	4	131.4	131.4	0.0	4
102.4	110.4	8.0	5	132.2	132.2	0.0	5
103.0	111.0	8.0	6	132.6	132.8	0.2	6
103.6	111.6	8.0	7	132.6	132.6	0.0	7
104.2	112.8	8.6	8	131.8	131.6	0.2	8
104.4	113.2	8.8	9	131.2	131.2	0.0	9
105.0	113.8	8.8	10	130.8	130.8	0.0	10
105.8	114.4	8.6	11	130.6	130.6	0.0	11
106.0	114.4	8.4	12	130.6	130.6	0.0	12
105.4	113.0	7.6	13	130.8	131.0	0.2	13
103.8	112.0	8.2	14	131.0	131.2	0.2	14
104.2	111.4	7.2	15	131.6	131.6	0.0	15
Maximum change of thermometer S.....		3.8°		Maximum change of thermometer S.....		2.0°	
Maximum change of thermometer T.....		4.4°		Maximum change of thermometer T.....		2.2°	
Average reading of thermometer S.....		103.8°		Average reading of thermometer S.....		131.55°	
Average reading of thermometer T.....		111.9°		Average reading of thermometer T.....		131.65°	
Average difference of thermometers.....		8.1°		Average difference of thermometers.....		0.1°	

The determination of melting points with this new form of tube is much more convenient and accurate if the tube is heated electrically rather than by a gas flame. To prepare the tube for electric heating, that portion of the tube marked A (Fig. 1), extending from B to the point where A turns upward, is first covered with a single layer of thin asbestos paper, and then wound with about ten turns of nichrome wire, No. 26, B and S gauge (about 0.016 in. in diam.). The wire is then covered with a layer of asbestos cement to a depth of about 5 mm. These tubes may be made of ordinary soft glass, but Pyrex glass is preferable.

## NEW FORMS OF COMBUSTION APPARATUS FOR USE IN GAS ANALYSIS<sup>1</sup>

By E. R. Weaver and P. G. Ledig

U. S. BUREAU OF STANDARDS, WASHINGTON, D. C.

Received August 12, 1919

This paper describes two new forms of combustion apparatus for use in gas analysis. The first is an improved form of the Dennis and Hopkins<sup>2</sup> combustion pipette. The second is a combustion capillary of small size somewhat similar to those of Hempel<sup>3</sup> and Levy,<sup>4</sup> containing an electrically heated platinum wire.

### COMBUSTION PIPETTE

The combustion apparatus usually known as the Dennis and Hopkins pipette, in which a platinum wire is placed in a pipette of sufficiently large size to hold all the sample to be burned, together with the oxygen necessary to burn it, has been made in many forms. In all of these, however, two leads are brought into

the bottom of the pipette, usually through a rubber stopper, and carried through the confining liquid to near the top of the bulb where they connect to the platinum wire. The unsatisfactory features of the apparatus are as follows:

1—It is difficult to make the stopper at the bottom of the pipette and the connections through it perfectly tight. The leakage of mercury is easily detected, but the pipette may be tight to mercury and allow air to leak in when its contents are under reduced pressure, and such a leak is very likely to be overlooked for a long time.

2—The weight of the mercury in such a pipette produces a serious danger of its forcing out the stopper and spilling the mercury.

3—In order to insulate the leads it is necessary to enclose one or both of them in glass tubes. The platinum wire is usually sealed through these tubes and the connection with the lead made inside the glass tube. This is a rather difficult form of construction and one hard to repair. The point at which the wire is sealed through the insulating tube is the most likely to crack in the whole apparatus, and such cracking is very likely to cause an undetected leak. If the leads pass through the glass and the active wire is crimped on, as is sometimes done, the junction usually has the highest resistance of any part of the circuit and is first to fail in use. A solder cannot, of course, be used in the presence of mercury. As the apparatus is frequently constructed, sufficient relative motion between the leads to distort or even break the platinum wire is possible.

4—The necessity of bringing two leads and a tube for the flow of the confining liquid out at the bottom of the pipette makes it difficult to support so heavy a weight without mounting it on a more or less bulky supporting frame.

5—In order to replace the platinum wire it is always necessary to completely drain the pipette and usually to disconnect it entirely.

The pipette shown in Fig. 1 was designed to overcome these difficulties. It is made of Pyrex glass, has a volume of between 125 and 150 cc., and is generally used with mercury as a confining liquid. The two short side-tubes through which the platinum wire is strung are slightly tapered and a cap made of soft glass is fitted to the outside of this taper. A detail of this construction is shown in Fig. 5. Through the end of this cap a heavy platinum wire is sealed. The wire is formed into a loop and the short end also sealed into the glass, as it has been found that this gives much greater strength and lessens the danger of breaking off the wire at the surface of the glass. It is usually best to have the long end of the wire, inside the cap, long enough to go up into the short side-tube at least half a centimeter, since the Khotinsky cement used in fastening on the cap may seal off the inner tube. For the coil which is strung through the pipette, 5 mil (0.125 mm. diameter) platinum wire has been found the best, though this is smaller than is commonly used. It has been found very satisfactory, however, because much less current is required to heat the wire to a dull glow than is necessary with heavier wire, and consequently the whole pipette is not heated to so high a temperature. A finer wire is not stiff enough to hold a good coil. About 7 in. of the wire are wound to a compact spiral on a large needle or heavy wire, leaving an inch or more of straight wire at each end. It is best to use a piece of brass or other stiff wire of small

<sup>1</sup> Published by permission of the Director of the Bureau of Standards.

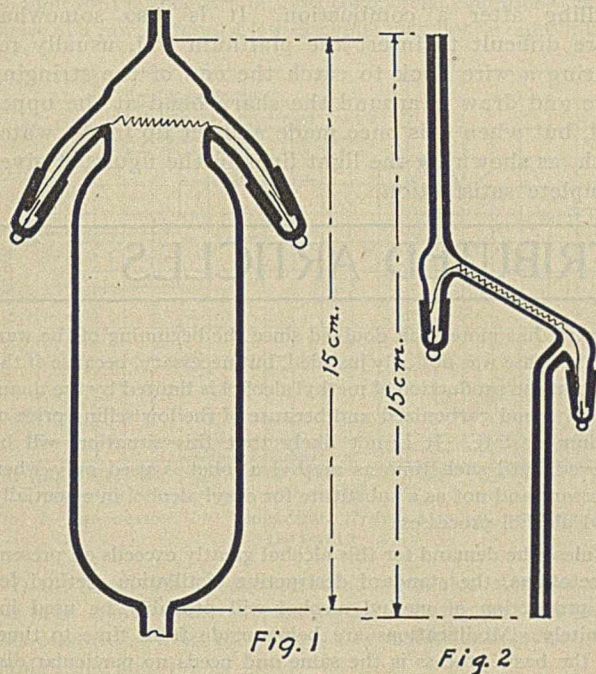
<sup>2</sup> *J. Am. Chem. Soc.*, 21 (1899), 398.

<sup>3</sup> *Z. angew. Chem.*, 25 (1912), 1841.

<sup>4</sup> *J. Soc. Chem. Ind.*, 31 (1912), 1153.



diameter to string this coil through the pipette. The projecting tips of the straight ends of the platinum wire are bent over the edge of the side-tube after the spiral has been pulled tight. The caps are then fastened on the side-tubes with Khotinsky cement, the whole cap and side-tube being gently warmed over the Bunsen burner until the cement flows smoothly in between.



When this cools, the Khotinsky holds the wire coil in place and fastens on the cap to make an air-tight joint. When the pipette is filled with mercury, the cups and side-tubes are filled too, and contact is made between the platinum wire in the cup and the heating coil.

This pipette gives satisfactory service over a very long period without renewal of the wire if care is used, but if the wire is broken or the coil gets too slack in the pipette, only a few minutes of work are necessary to replace it.

#### COMBUSTION CAPILLARY

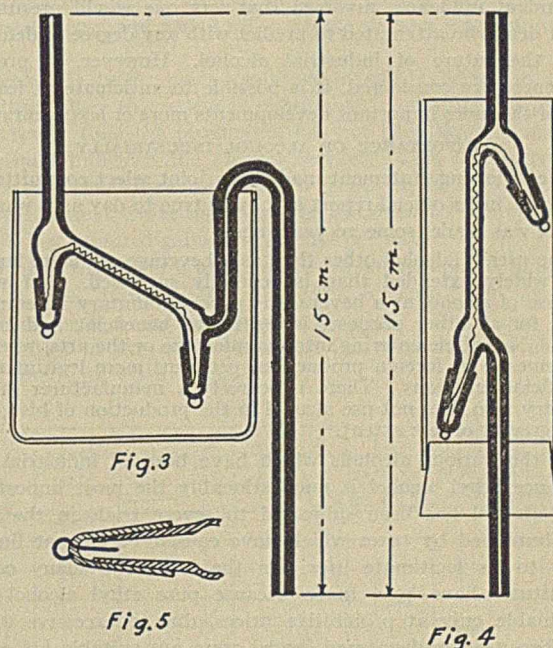
For the determination of small amounts of combustible gas in the presence of air, oxygen, or an inert gas, or for the determination of small amounts of oxygen in a combustible gas, there is a considerable advantage in the use of a combustion capillary similar to that devised by Levy.<sup>1</sup> The tube there described, in which a platinum wire is sealed into a quartz capillary by means of molybdenum leads, is very satisfactory in service; but the coil cannot be replaced if broken or burned out, and the device is expensive and difficult to obtain. A similar capillary devised by Levy, but having the lead wires passing through rubber stoppers, is very unsatisfactory from several standpoints. The capillaries of Hempel<sup>1</sup> and White<sup>2</sup> have objectionable rubber connections between the quartz tube and the glass tubes which carry the lead wires. The Hempel apparatus, at least, is also hard to

put together in good form, and must be rebuilt when either the wire or the rubber tubing requires replacement.

The three forms of combustion capillary described below are made of Pyrex glass. They are of a new type which has the following advantages:

- 1—The wire is easily replaced without disturbing the tube which may be permanently connected to the remainder of the gas analysis apparatus.
- 2—There are no rubber connections near the hot wire.
- 3—The tube is easy to make and to keep gas-tight.
- 4—It is not very difficult for an amateur glass blower to make and should be quite inexpensive if made commercially.

The simplest form is that shown in Fig. 2. The portion containing the coil has an inside diameter of about 3 mm. and the connecting capillary tubing has an inside diameter of about 1 mm. The platinum wire is made into a coil and drawn into place by the use of another wire, as in the case of the pipette previously described. The caps are made of soft glass exactly like those for the pipette and are fastened on with Khotinsky cement. The cap and side-tube are then partly filled with mercury to make electrical contact. Water or mercury may be used as the confining liquid, but mercury has a tendency to foul the tube with oxide in the course of several analyses.



When water is used as the confining liquid, it is better not to fill the side-tube completely with mercury on account of this oxidation. The bulb-like enlargement is made in the side-tubes to prevent the trapping of gas when the system is filled with water after an analysis, during which some of the water has evaporated from the side-tubes. In use, a double Hempel bulb or similar arrangement is connected to the lower end of the tube to confine the gas as it is passed back and forth.

The first form has proved satisfactory if carefully used, but if water is passed into it while it is hot from a combustion, it is likely to crack. Moreover methane

<sup>1</sup> *Loc. cit.*

<sup>2</sup> "Gas and Fuel Analysis," p. 49, McGraw-Hill, New York, 1913.



combustions, which require a very hot wire, have in one or two cases so softened the tube that it was bent and the platinum wire fused to the glass. This difficulty can be overcome by making the tube of quartz, but the advantages of using Pyrex glass are such that two other forms were developed in which the tube is kept immersed in a water bath.

Fig. 3 shows the first form. It is made exactly the same as that shown in Fig. 2 except that the lower capillary tube is sealed to the side of the lower side-tube and is made with a goose-neck curve which allows a beaker or similar water bath to be slipped up over the tube from beneath. If distilled water is used in this bath, no trouble is experienced from

electrolysis between the lead wires. The only objection to this form of tube, of which two have given satisfactory service over a period of several months, is that its awkward shape makes it fragile.

Consequently the third form shown in Fig. 4 was developed. It requires care in making to prevent the gas being trapped in the bends of the main tube when refilling after a combustion. It is also somewhat more difficult to insert the platinum coil, usually requiring a wire hook to catch the end of the stringing wire and draw it around the sharp bend at the upper end, but when it is once made and set up in the water bath, as shown by the light lines in the figure, it gives complete satisfaction.

## ADDRESSES AND CONTRIBUTED ARTICLES

### THE FUTURE OF INDUSTRIAL ALCOHOLS<sup>1</sup>

By B. R. Tunison

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#### INTRODUCTION

To attempt to predict the future of industrial alcohol is similar in many respects to making an attempt to foretell the outcome of a battle in a great war. The phases of the question are so numerous and complex and there are so many interdependent influences involved that any one would presume a great deal who attempted to predict with any degree of definiteness the future of industrial alcohol. However, if present influences are considered, it is possible to anticipate at least a few of the more important developments more or less accurately.

#### IMPORTANCE OF ALCOHOL INDUSTRIALLY

The following statement made by a joint select committee of Congress in its official report is fully as true to-day as it was the day it was made, some 20 years ago.

The uses of alcohol other than as a beverage are more largely and widely extended than is generally supposed. But while the use of alcohol as a beverage is purely voluntary its employment for all other purposes is legitimate, beneficial, and necessary. No article entering into manufacture or the arts, whether of domestic or foreign production, performs more legitimate or beneficial functions. There is scarcely a manufacturer in the country who does not use alcohol in the production of his goods to a greater or less extent.

Of the various alcohols which have been of industrial importance ethyl alcohol is unquestionably the most important. This alcohol has been subjected to severe trials in the past and burdened by taxes which have caused very great limitations to its legitimate uses. In the industries many costly substitutes have been made because pure ethyl alcohol was obtainable only at prohibitive prices, due to excessive taxes, and because the denatured grades were not suitable for many special purposes.

The importance of methyl alcohol industrially is second only to that of ethyl alcohol. There seems little doubt that this is due to commercial influences rather than to chemical reasons. In the United States it has been substituted for ethyl alcohol for a great many purposes for which it is not so well suited, because of the rules, regulations, and taxes surrounding the manufacture, distribution, and use of ethyl alcohol.

During and since the recent war the demand for methyl alcohol has greatly increased, due in part to the rapidly growing dye industry in this country and also to the enormous export demands because of its limited production in other countries.

<sup>1</sup> Read before the New York Section of the American Chemical Society, Chemists' Club, New York, N. Y., October 10, 1919.

Its price has more than doubled since the beginning of the war. This increase was not only justified, but necessary, because of the fact that the production of methyl alcohol is limited by the quantity of wood carbonized and because of the low selling price of calcium acetate. It is not likely that this situation will be relieved until such time as methyl alcohol is used only when necessary and not as a substitute for ethyl alcohol in essentially ethyl alcohol processes.

Unless the demand for this alcohol greatly exceeds all present expectations, the standard destructive distillation method for the production of methyl alcohol will probably be used indefinitely. Modifications are being made from time to time, but the basic process is the same and needs no particular discussion at this time.

The future development of the methyl alcohol industry in this country should be very rapid. As the chemical industries are progressing so rapidly, greater and greater quantities of this very essential alcohol will be used. If, as is hoped and expected, the United States becomes one of the largest exporters of dyes, chemicals, and allied products, the use of methyl alcohol will constantly increase. New uses will undoubtedly develop and as these new uses and new industries are expanded methyl alcohol will be of ever-increasing importance in this country.

While many other alcohols are of commercial significance in the United States, their production to any extent in this country has been very recent. Up to the time of the World war it was possible to import an adequate supply. During the war many substances in the production of which the higher alcohols are necessary could not be imported and the importation of the alcohols was almost completely cut off. This had the effect of greatly stimulating the production of these alcohols in the United States. Up to this time large quantities were discarded by many manufacturers because they were in admixture with other bulky waste materials, and their separation more or less difficult. Some manufacturers sold for solvent purposes mixtures of these alcohols, because they could not make the separation and compete with the alcohols brought into the United States from foreign parts.

The production, separation, and purification of higher alcohols is one of the new and rapidly growing industries of this country and should be supported by all users of these products to the greatest possible extent. It is hoped that this industry may survive any periods of depression that may be encountered and that the United States may be independent of any importation of these valuable and necessary alcohols.

The question of the production of ethyl alcohol is so important that it warrants our brief consideration at this time.



## PRODUCTION OF ETHYL ALCOHOL

**RAW MATERIAL**—The considerations which have determined the raw materials to be used in the manufacture of ethyl alcohol have been of a commercial nature rather than essentially chemical. At the present time the state of the art is such that from a chemical standpoint alcohol may be readily produced from nearly any available source of saccharine or starchy materials. To indicate the influence of local commercial conditions on the choice of a raw material a few examples may not be out of place.

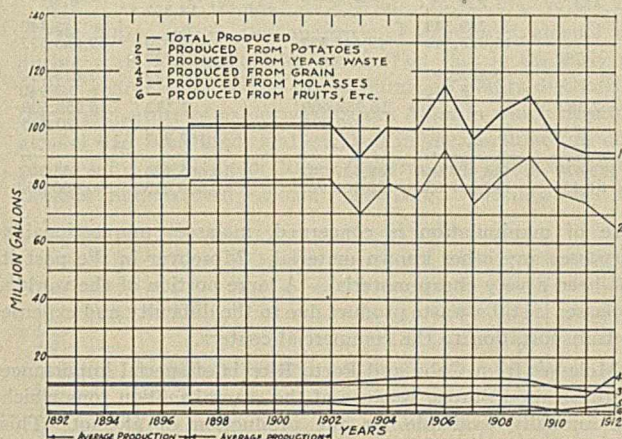


FIG. 1—PRODUCTION OF INDUSTRIAL ETHYL ALCOHOL IN GERMANY, 1892-1912

**In Germany**—The production of industrial alcohol in Germany has been a noteworthy example of the industry for some years. The production from the various raw materials is indicated in Table I and Fig. 1.

TABLE I—PRODUCTION OF ALCOHOL IN GERMANY (U. S. GALLONS)

YEARS	Potato Distilleries	Yeast Distilleries	Grain Distilleries	Molasses Distilleries	Fruit and Other Distilleries
1887/88-1891/92	59,826,000	10,190,000	3,065,000	3,330,000	686,000
1892/93-1896/97	63,362,000	10,370,000	3,615,000	3,620,000	898,000
1897/98-1901/02	81,730,000	10,900,000	4,750,000	3,190,000	872,000
1902/03	70,000,000	11,500,000	4,990,000	2,350,000	502,000
1903/04	80,420,000	12,060,000	6,204,000	4,410,000	608,000
1904/05	75,900,000	12,720,000	7,500,000	285,000	976,000
1905/06	92,820,000	12,670,000	7,000,000	219,000	819,000
1906/07	73,620,000	12,540,000	7,240,000	235,000	792,000
1907/08	83,100,000	12,770,000	6,440,000	272,000	1,005,000
1908/09	90,220,000	11,900,000	6,310,000	229,000	1,267,000
1909/10	77,600,000	9,460,000	6,150,000	1,936,000	1,005,000
1910/11	74,100,000	8,130,000	6,230,000	2,350,000	739,000
1911/12	66,000,000	7,925,000	13,100,000	3,380,000	898,000

Agricultural and labor conditions during the period indicated were such that potatoes were very cheaply produced and the saccharine material obtained therefrom on a per pound basis was relatively cheap, as compared, for example, with grains. Fruits had a higher value as food and the fruit wastes did not occur in sufficient quantities to materially affect the alcohol production. The molasses available in Germany was largely beet molasses and had a somewhat greater value as a feeding material than as a raw material for alcohol production.

**In France**—In France sugar beets and beet molasses have been used to a considerable extent as a raw material for production of alcohol. The production from the raw materials used is shown in Table II and Fig. 2.

TABLE II—PRODUCTION OF ALCOHOL IN FRANCE (U. S. GALLONS)

YEAR	RAW MATERIAL			Total	Per cent of Total Production in France
	Grain Distilleries	Molasses Distilleries	Beet Distilleries		
1908	9,560,000	12,620,000	33,300,000	55,500,000	81.5
1909	9,420,000	12,600,000	20,960,000	52,800,000	82.7
1910	12,200,000	13,350,000	31,200,000	56,800,000	90.0
1911	17,450,000	13,300,000	27,800,000	57,600,000	90.0
1912	23,300,000	12,300,000	42,800,000	78,400,000	89.3
1913	11,340,000	16,000,000	41,200,000	68,600,000	88.0
1914	21,700,000	9,940,000	12,370,000	35,700,000	81.5
1915	9,400,000	6,980,000	21,100,000	38,800,000	74.0
1916	17,560,000	4,100,000	11,900,000	33,600,000	81.6

**In Switzerland**—Consul W. P. Kent of Berne, Switzerland, has stated<sup>1</sup> that calcium carbide is being developed in Switzerland as a source of alcohol. He stated that installations were started with about 20,000 horse power minimum and 30,000 horse power maximum (summer time), which would produce from 7,500 to 10,000 tons of alcohol per annum. Calcium carbide is produced by the usual electric furnace method and acetylene from the calcium carbide by the action of water. Two methods are used in the production of alcohol from the acetylene:

1—Acetylene is hydrogenated by catalytic means and ethylene is produced. The ethylene is dissolved in sulfuric acid, and alcohol and sulfuric acid are formed upon saponification.

2—Acetaldehyde is produced catalytically from the acetylene. The acetaldehyde is oxidized to acetic acid or reduced to alcohol, by means of catalyzers. Great care is used in the selection of a catalyzer in order to eliminate numerous complicated side reactions which are liable to occur.

The production of alcohol by such processes as just described of course necessitates a very cheap source of power. This is available only in such places as Switzerland and Norway, and alcohol made by this means could not compete with alcohol of vegetable origin if such could be produced cheaply in those countries.

**In the United States**—In the United States many different materials are used with more or less success. In some portions of the country corn and corn wastes are effectively and economically utilized. These products afford a convenient and easily handled source of alcohol. For many years maize was the chief source of industrial alcohol in the United States, and undoubtedly corn and maize will continue to be a very important source.

Alcohol is being made from waste sulfite liquor in some localities where this waste is obtainable in quantity. Up until the present time the production has not been large enough to materially influence the alcohol market. This is largely due to the fact that there are very few places in the United States where sufficient waste liquor can be obtained to produce more than a few hundred gallons of alcohol per day.

So far as the writer is aware there are but two plants in operation in the United States producing alcohol from sawdust, and these have a relatively small production. There seem to be possibilities in the development of the production of alcohol from wood and wood waste.

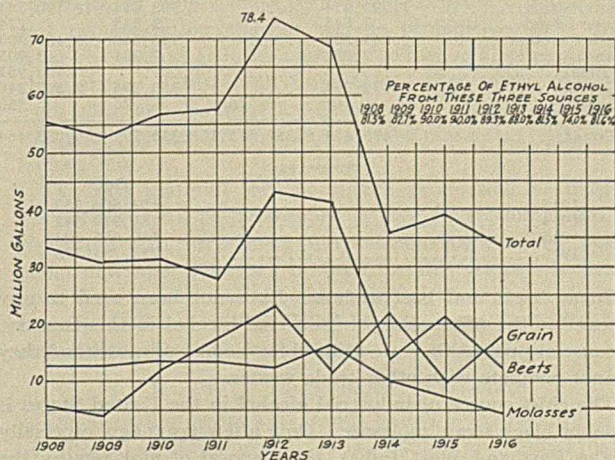


FIG. 2—PRODUCTION OF ETHYL ALCOHOL IN FRANCE

During the last two years very little alcohol has been produced from the various grains. This is largely due to the fact that the grains are in demand as food materials at a high figure. In this country the only time it is feasible to use grains for the

<sup>1</sup> Commerce Reports, 102 (1917), 426.



TABLE III—RAW MATERIALS FOR PRODUCTION OF ALCOHOL IN THE UNITED STATES

STATES AND TERRITORIES	RAW MATERIALS						TOTAL	
	Corn Bu.	Rye Bu.	Malt Bu.	Other Materials Bu.	Molasses Gal.	D. S. L. Gal.	Bu.	Gal.
California	1,656	..	..	..	13,223,826	..	1,656	13,223,826
Connecticut	1,767	1,622	1,311	..	..	..	4,700	..
District of Columbia	79,309	..	122,013	69,909	236,433	..	271,231	236,433
Hawaii	..	..	..	..	7,700	..	..	7,700
Illinois	8,130,912	29,770	880,135	44,993	4,219,519	..	9,085,810	4,219,519
Indiana	2,637,875	48,036	206,883	50,942	..	..	2,943,736	..
Kentucky	2,200,183	87,771	282,019	36	..	..	2,570,009	..
Louisiana	9,594	132	1,000	2,986	226,980	..	..	226,980
Maryland	130,522	2,734	5,176	..	33,214,705	17,620,539	13,712	50,835,244
Massachusetts	2,690	2,418	1,272	..	34,626,539	..	138,432	34,626,539
Michigan	..	..	..	..	13,118,139	..	6,380	13,118,139
Missouri	13,967	1,986	2,444	..	..	5,290,164	..	5,290,164
Montana	..	..	..	..	..	..	18,397	..
New York	342,150	37,712	69,158	..	249,340	..	449,020	245,340
Ohio	578,451	3,170	63,324	784	11,025,740	28,637,832	645,729	39,663,572
Pennsylvania	295,793	7,116	33,213	..	7,873,097	..	363,122	7,873,097
Rhode Island	..	..	..	7	5,942	..	7	5,942
South Carolina	..	..	..	2,382	..	16,958,609	2,382	16,958,609
Texas	..	..	..	..	..	20,098	..	20,098
Wisconsin	119,676	26,397	21,729	..	..	..	167,802	..
TOTAL	14,544,545	248,864	1,689,677	172,039	118,027,960	68,527,242	16,665,124	186,555,202
TOTAL FOR FISCAL YEAR 1917	33,973,268	3,375,439	4,239,677	81,435	112,497,633	78,462,969	40,669,819	190,960,602

production of industrial alcohol is when a crop has been damaged in some manner so that it cannot be utilized as a food material.

Labor and agricultural conditions have been and are such in this country that potatoes cannot be grown economically enough to compete with trade wastes as sources of alcohol.

Fruit in this country is so valuable as food that it cannot be used as a source of industrial alcohol. Fruit wastes do not occur in sufficient quantity to supply even a centrally located distillery and transportation of such products is of course not feasible.

TABLE IV—PRODUCTION OF ALCOHOL IN THE UNITED STATES

STATES AND TERRITORIES	1918			1917 Total Production Gal.
	From Materials other than Fruit Gal.	Fruit Brandy Gal.	Total Production Gal.	
California	8,727,694	5,295,952	14,023,646	17,851,482
Connecticut	23,527	2,924	26,451	132,055
District of Columbia	749,517	..	749,517	608,812
Hawaii	3,935	..	3,935	14,016
Illinois	49,679,940	140	49,680,080	79,320,617
Indiana	15,820,031	10,899	15,830,930	43,361,276
Kentucky	12,604,703	3,734	12,608,437	36,441,778
Louisiana	24,406,539	..	24,406,539	26,545,832
Maryland	26,746,386	..	26,746,386	24,965,321
Massachusetts	10,873,375	..	10,873,375	12,511,238
Michigan	752,745	..	752,745	819,908
Missouri	77,026	2,501	79,527	289,661
Montana	186,248	..	186,248	244,773
Nebraska	..	..	..	2,938,594
New Jersey	..	51	51	54,494
New Mexico	..	..	..	315
New York	10,540,421	4,480	10,544,901	13,856,054
Ohio	3,277,485	36,618	3,314,103	10,114,573
Pennsylvania	7,293,914	..	7,293,914	12,190,764
Rhode Island	2,845	..	2,845	224
South Carolina	943,568	..	943,568	1,159,309
Texas	7,281	..	7,281	13,905
Virginia	..	..	..	122,957
Wisconsin	759,294	..	759,294	2,527,249
Wyoming	..	26	26	260
TOTAL	173,476,474	5,357,325	178,833,799	286,085,464

SUMMARY

	Gal.
1913	193,606,258
1914	181,919,542
1915	140,656,103
1916	253,283,273

Sugar beets and beet molasses have not been used to any extent for the production of industrial alcohol in this country, because at present they are utilized for feeding purposes and they cannot compete with other trade wastes.

The chief source of industrial alcohol in the United States is cane, or black strap, molasses. Only a few years ago, especially in the East and West Indies, the disposal of molasses by the sugar mills was a serious trade waste problem, but it is now very largely used the world over as a raw material for alcohol manufacture. The conversion of molasses into alcoholic liquor, especially into rum, is an old enterprise. West India rum has been famous in New England for more than 200 years. But the use of molasses in large quantities for industrial alcohol production is a development of the last few years. As far as

ease of manipulation is concerned, molasses unquestionably surpasses any other known material. Moreover in the past it has been a very cheap material. A large portion of the world's molasses is still a waste product due to the difficulty and expense of transportation to the commercial centers.

Molasses from Cuba and Porto Rico is of special importance to the United States, because of the several million tons which are annually available for the production of alcohol. This molasses is gathered from the various producing mills in barges, steam lighters, hundreds of tank cars, and in some few cases barrels and hogsheads, and taken to the large storage tanks which are located at deep water shipping ports. Tank steamers, such as are used for the transportation of petroleum, are used to carry the molasses from these storage points to various plants in the United States: in Boston, New York, Baltimore, New Orleans, and other points. In addition to the above-mentioned sources of molasses large quantities are obtained from the cane sugar refineries located in the southern and southeastern parts of the United States.

The various raw materials used and the quantities of alcohol obtained from them are indicated in Tables III and IV.

The figures are, of course, totals and include the beverage alcohol production.

The total production of ethyl alcohol in various countries from 1908 to 1912 is shown graphically in Fig. 3.

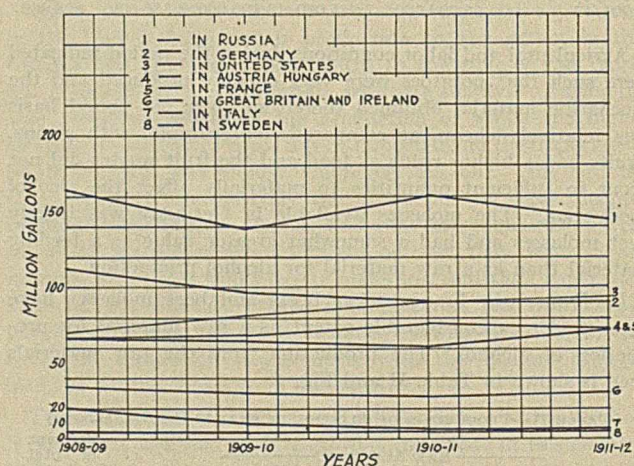


FIG. 3—PRODUCTION OF ETHYL ALCOHOL, 1908-1912

PRODUCTION OF DENATURED ALCOHOL

In the last few years the production and consumption of denatured alcohol in the United States has increased very greatly, as shown by Table V and Fig. 4.



TABLE V—PRODUCTION OF DENATURED ALCOHOL IN THE UNITED STATES

Fiscal Yrs.	Denaturing Ware-houses	Completely Denatured Wine Gal.	Specially Denatured Wine Gal.	Total	
				Wine Gal.	Proof Gal.
1907	8	1,397,861	382,415	1,780,276	3,084,950
1908	12	1,812,122	1,509,329	3,321,451	5,640,331
1909	12	2,370,839	2,185,579	4,556,418	7,967,736
1910	12	3,076,924	3,002,102	6,079,027	10,605,870
1911	14	3,374,019	3,507,109	6,881,129	11,682,887
1912	14	4,161,268	3,933,246	8,094,515	13,955,903
1913	21	5,233,240	4,608,417	9,831,658	16,953,552
1914	25	5,213,129	5,191,846	10,404,975	17,811,078
1915	23	5,386,646	8,599,821	13,986,468	25,411,718
1916	33	7,871,952	38,807,153	46,679,108	84,532,253
1917	44	10,508,919	45,170,678	55,679,597	93,762,422
1918	49	10,328,454	39,834,561	50,163,016	90,644,722

The falling off in the quantity of denatured alcohol used during 1918 is accounted for by the fact that in the early part of the year several of the larger munition plants discontinued the manufacture of explosives for the Allies in which denatured alcohol had been used, and engaged in the manufacture of explosives for the United States Government, using principally tax-free undenatured alcohol. The army specifications were

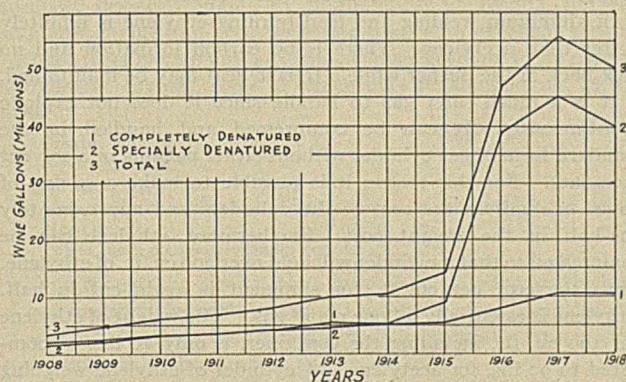


FIG. 4—PRODUCTION OF DENATURED ALCOHOL IN THE UNITED STATES

subsequently changed and denatured alcohol was used exclusively in those plants. The quantity of denatured alcohol used during the year in the manufacture of explosives and for other war purposes was approximately 52,847,117 proof gallons.

#### NEW SOURCES OF RAW MATERIALS

Upon the demand for alcohol and the price at which it is sold will depend the new raw materials used for its production. Of the possible raw materials which have been proposed may be mentioned the following:

**ARTICHOKE**—The tubers of the artichoke contain on an average about 14 to 15 per cent of directly fermentable levulose and about 2 per cent of starch which can be converted into sugar. It is possible that these tubers may be used to some extent where they may be easily grown.

**SORGHUM**—This plant contains from 6 to 15 per cent of levulose and crystallizable sugar. It deserves considerable attention because of the readiness with which it is grown, the extent of the territory in which it flourishes, and the large amount of sugar which certain varieties contain.

**FRUITS AND FRUIT WASTES**—Fruits and fruit wastes may be used for the production of alcohol, but in most cases the fruits are too valuable to use for such purposes and the wastes do not occur in quantities sufficient to maintain a distillery. Transportation of these wastes is not possible.

**BERRIES**—While many different berries have been used for the production of alcoholic beverages, in most cases it will not be possible to use them for the production of industrial alcohol, because of their greater value as food.

**AGAVE AND CACTUS**—While various specimens of the agave and cactus have been used in Mexico and the southwestern parts of the United States for the production of alcoholic beverages, they have not been used as a source of industrial alcohol. It is known that the juice of these plants contains large amounts

of sugar at times and there is reason to believe that they may be used industrially. Recently some experimental work has been done on the so-called "Sotol" plant of Mexico, with promising results. It is possible that these plants may be used to some extent for the production of industrial alcohol.

**CASSAVA**—The root of the cassava contains about 25 per cent of starch which may be utilized for the production of alcohol. A few years ago the Bureau of Chemistry determined that an average of 40 gal. of alcohol per ton of roots could be obtained. If these roots can be cheaply grown this is a promising possibility.

**GARBAGE WASTE**—This material has been suggested as a source of industrial alcohol. That this cannot be a factor in the production of industrial alcohol we can readily see when we consider that the material is poor in fermentable substance and would be very expensive to handle.

**PRODUCTS OF COAL DISTILLATION**—The report of the British Inter-Departmental Committee on the Production and Utilization of "Power Alcohol" states that sufficient ethylene can be obtained from the gas works and coke-oven gases of Great Britain to yield annually up to 150,000,000 gal. of 90 per cent alcohol. It is believed that steps are being taken to utilize these wastes which may become important.

In other countries there are many raw materials not yet used extensively which will probably become important sources of industrial alcohol. In tropical regions there are large numbers of plants from which alcohol can be readily produced. As an example may be mentioned the Nipa palm which flourishes in the Far East, India, Africa, etc. Concerning the utilization of this plant for the production of alcohol, the following statement has been made:<sup>1</sup> "There are over 100,000 acres of Nipa swamp now available in the Archipelago (Philippines) of which about 90 per cent have never been touched, and it is estimated that the untapped swamp area of the islands would yield 50 million gallons of alcohol every season."

In addition to the requirement of a sufficient percentage of fermentable matter to give a yield which will pay for the handling, etc., of the latter, there must also be an abundant, concentrated, and stable supply of raw material. Few people realize that a small alcohol plant of even 100 gal. daily capacity will consume every day approximately 200 gal. of molasses, or one ton of shelled corn, or 4 tons of potatoes (about 15 per cent starch), or 7 to 8 tons of sweet apples (about 12 per cent sugar). A concentrated supply must be available because otherwise labor and transportation charges become prohibitive. The supply must be constant as it does not pay to run an alcohol plant only at intervals. This is an important factor when considering fruit, corn, or other wastes. Questions of whether the material will stand transportation and storage are often determining factors.

The raw material for the future production of industrial alcohol then will be determined largely by economic and local conditions. In this country, as conditions are modified, possibly many new materials may be used for this purpose, depending upon these conditions. For some time to come, however, in this country molasses will probably be the most important raw material for the production of industrial alcohol.

#### MANUFACTURE FROM RAW MATERIALS

The alcohol industry as it stands to-day is the result of a very long period of development. The methods and apparatus used have become pretty well standardized and no revolutionary changes or developments are anticipated. Many minor improvements are being made from time to time, but as far as the distillation and rectification are concerned the principles involved have been carefully worked out and apparatus designed which correctly applies them.

<sup>1</sup> Pure Products, 14 (1918), 286.



A large amount of research is being directed towards the processes of fermentation and we may hope for many developments from this phase of the industry. Paralleling this development, better systems of plant control will undoubtedly be put into practice. While the yields obtained at present are good, better results are being searched for.

#### USES OF INDUSTRIAL ALCOHOL

As far as the influence of the industries in which alcohol is used is concerned, the use of alcohol in the future should develop fully as rapidly as the industries themselves. If this were the only, or even the most important factor, as it is concerning most commodities, the prediction of the future would be a relatively simple matter. Most of the newly developed chemical industries in this country have come to stay and they will require an ever-increasing quantity of alcohol.

While most of the uses of alcohol will be developed more or less parallel with the development of the chemical and allied industries, there are a few the expansion of which will be more or less revolutionary in character.

**SOLIDIFIED ALCOHOL.**—One of the very important uses to-day, and one that is becoming increasingly so, is that of solidified alcohol. Alcohol burns with a smokeless flame and does not carbonize like kerosene, so that even at the present time it is a very satisfactory fuel, and its only rival for chafing dishes, coffee percolators and such articles is electricity. In regions where electricity is not available solidified alcohol is a very important fuel for cooking purposes. It is also used very extensively during vacation trips to the mountains, or the seashore. It is very convenient to handle and is without dirt, smoke, or odor, and is an economical fuel, and undoubtedly the use of alcohol in this form will increase to a very marked degree.

**PURIFICATION OF TURPENTINE.**—One of the newer developments is the utilization of alcohol in the separation and purification of gum-turpentine. The present method of heating and making the crude products at the turpentine camps results in rosin of which only a small part is of good color. It has been found that the crude gum-turpentine is readily soluble in high proof denatured alcohol. It is then possible to remove such foreign matter as particles of wood, twigs, insects, etc., which have been found largely responsible for coloring the finished products. Subsequent distillation of the gum-turpentine-alcohol solution results in a separation of the alcohol heads containing a small percentage of turpentine, which functions as a denaturant and at the same time produces a product which can be used again as a solvent. The turpentine fraction is clear and marketable as a high-grade product and the rosin residue is colorless and of the highest grade. The loss of solvent at present is approximately 6 per cent and developments indicate that it may become lower. When using this process it is possible to centralize the processing of the gum-turpentine and thus reduce operating expense to a minimum. The increase in quantity of high-grade products, the very small losses involved, and the small cost of operation incurred by this process indicate that it will become of very great importance in the turpentine industry.

**ETHYLENE.**—Another relatively new development in the utilization of alcohol is in the catalytic production of ethylene. Ethylene has been considered as a substitute for a cutting and welding gas in the place of acetylene. From comparative tests ethylene is found to have many advantages over acetylene. As far as the heat of combustion is concerned ethylene has a slightly higher value. In cutting and welding iron and steel ethylene has among others the following advantages:

1—It has a high ratio of carbon to deterrent element like hydrogen, overcoming the tendency to retard combustion and slow the cutting.

2—It has a rapid rate of combustion, preventing loss of temperature from absorption and conduction of heat by the metal being cut.

3—It will help to maintain an oxidizing effect fast enough to overcome any tendency to burn too deeply or chill the surfaces of the metal being cut.

4—It will produce a rapid rate of cutting with a low consumption of gas making a high efficiency possible from the regular operators of the torch.

5—It shows a decrease of labor and gas consumption with increase of thickness of metal cut, making the cutting of thick metal as economical as thin metal.

6—It possesses a remarkable degree of lightness of the charged cylinders, providing a valuable portability and ease of movement of the gas supply about a shop.

7—It is a non-poisonous, safe gas, without objectionable odor.

8—It has a clearly outlined cone in the flame, without which a workman would be badly handicapped.

In the working of copper it has been impossible to make a satisfactory weld with acetylene, because of the formation of carbon and the consequent blistering in the weld. Perfect copper welds have been obtained with ethylene.

In aluminum welding and lead burning ethylene is infinitely better than acetylene. There is no carbon formation and no kick back in the burner while a terrific heat may be maintained.

It is a much safer gas to handle since it does not explode spontaneously. It can be compressed and handled in the standard form of gas cylinder without either packing or a solvent (acetone). For this reason it is possible to make a saving of about one-half in the cost of the cylinders and approximately one-half in the freight cost. Furthermore, ethylene can be compressed so that a cylinder will hold over 200 cu. ft. of ethylene. Thus the cost per cu. ft. for shipment is again cut in half. Ethylene is essentially a "one man" gas. The weight of ethylene per 100 cu. ft., including the container, is only 40 lbs. as compared to 90 lbs. for acetylene. A cylinder of ethylene weighing between 80 and 85 lbs. contains approximately 210 cu. ft. of gas and can be easily handled by one man.

Ethylene is being sold at the same price as acetylene. It may be used in an ordinary welding torch, but preferably one with a mixing chamber. A change in the size of the tip is also desirable.

In addition to the use of ethylene as a cutting and welding gas it has been found to have considerable value for lighting and heating purposes where electricity is not available.

In the chemical industries we all realize that with a source of cheap, pure ethylene it will be possible to manufacture a great number of synthetic and chemical products.

**INTERNAL COMBUSTION ENGINES.**—Probably the use of alcohol which in the future will have the greatest significance is its use as such, or in admixture with other compounds, as a fuel for internal combustion engines. For years volumes have been written about the use of alcohol in such engines. In Germany where the supply of petroleum products was inadequate and where alcohol was a relatively cheap commodity, large quantities were used for automotive and power purposes. In France alcohol was used extensively for automotive engines. In the United States considerable has been said about using alcohol for automobiles, farm machinery, etc. Vivid word pictures have been drawn of the farmers taking their waste products and processing them in their small plants and then doing everything from sawing wood to doing the family washing with alcohol as a source of power. However, the petroleum supplies in this country have been so abundant and available at such reasonable prices that these word pictures have remained as such and the farmers continue to buy gasoline with which to operate their automobiles and small engines. This has been the case since the agitation for alcohol production by the farmers in 1906-7.

Recently, however, conditions have suddenly changed. The Geological Survey tells us that in about 3 years the peak in a curve showing petroleum production will be reached and after



that time there will be a gradual decline in petroleum production over a long period of years.

Not only will the production of petroleum continually decrease, but the present indications are that the demand for petroleum products will increase in the next few years even more rapidly than ever. The increase in the number of automobiles, trucks, tractors, and airplanes during the last 5 years has been remarkable. If the increase from now on is only a small percentage of what it has been we must soon obtain a new supply of explosion engine fuel, and while our supply of petroleum is limited in quantity, the raw materials for the production of alcohol are around about us in inexhaustible quantities. At this time the utilization of alcohol for power purposes appears very significant.

While it will probably be several years before alcohol alone will be used as a motor fuel, alcohol in admixture with other fuels is now being produced and marketed in some localities of this country. The alcohol fuels as used at present do not require a specially designed engine to obtain optimum results, but the standard engine and accessories are used with excellent results. Alcohol fuels have many advantages over gasoline when used in automotive engines, some of which may be summarized as follows:

- Greater mileage
- No preignition
- No knocking
- More uniform application of power during power stroke of the piston
- More power
- Power more completely under control

Even though the alcohol has a lower B. t. u. value for the heat of combustion, the combustion of the fuel is more complete and greater efficiencies are obtained. This is especially true in slow speed engines such as motor trucks.

Alcohol fuels have also been developed for airplane use. In this regard the *Journal of the Society of Automotive Engineers*<sup>1</sup> says in part:

A new alcoholic fuel, consisting of alcohol, benzol, and ether, is about to take the place of the high-grade airplane gasoline, which has previously been used in the Air Mail Service.

The advantage of this fuel lies in the resulting cleanliness of the engines, reduction in the cost of upkeep and its burning cooler than gasoline, which to some extent overcomes the objection to a high-compression engine when operating at low altitudes. It requires about four-fifths as much of the new fuel for any given distance and altitude. This gives greater flying radius to the planes and will enable the DeHaviland Fours to cover the New York to Cleveland route, a distance of 430 miles, in a non-stop flight. It reduces the probability of forced landings by keeping the spark plugs and the engine cylinders clear of carbon deposits and accumulations of oil.

As the realization of the advantages of the above-mentioned fuels becomes more universal, and as the quality of the gasoline becomes poorer due to the diminishing petroleum supply, and as the price of gasoline rises, alcohol will be used in greater and greater quantities. In anticipation of this great increase in the demand for alcohol for power purposes we should take active steps to assist in the development of the alcohol industry so that an adequate supply of alcohol may be at hand when needed.

#### PROBABLE EFFECT OF LEGISLATION ON THE FUTURE OF INDUSTRIAL ALCOHOL

The effect of the passage of the industrial alcohol bill in 1906 had a marked effect on American industries in which alcohol is used. Many new enterprises were undertaken and those in operation were greatly extended. Since 1907 these industries using alcohol have prospered and grown until the United States instead of being one of the smallest nations from an industrial alcohol point of view has now become one of the greatest world influences. The increase in the production of

industrial alcohol since 1907 is summarized in the last two columns of Table V.

While the progress made by the alcohol industry has been little short of remarkable, our chemical industries which depend very largely on a supply of cheap alcohol have not prospered as have those in some other countries. The bill of 1906 was an important step forward, but the manufacture, sale, distribution, and use of alcohol was so surrounded with rules and regulations that many manufacturers did not use it and the normal growth of the industry was never approximated.

When the present prohibition bill was being discussed and prepared the very existence of the industries using industrial alcohol was threatened. The Commissioner of Internal Revenue with very great foresight and with a realization of the necessity of a supply of alcohol for industrial purposes, the use of which should be as free of restrictions as possible, prepared and presented the section of the prohibition bill relating to industrial alcohol. The assistance given by the Internal Revenue Bureau in showing the necessity of industrial alcohol, and the absolute dependence of some industries on a supply of alcohol, to those concerned with the making of our laws was a coöperation with the chemical and allied industries which should be known and appreciated by every chemist. A great deal of credit should be given to members of the present Congress who took time to learn facts concerning some of the industrial phases of the alcohol question, with the result that the industrial alcohol section of the bill was passed.

Now that a new law is to govern the alcohol industry we may feel certain that the Internal Revenue Bureau will proceed in the same just manner in drafting the rules and regulations which are to surround the manufacture, sale, distribution, and use of alcohol. We may feel confident that the Government's taxable interests will be protected and at the same time the manufacturers will be given the greatest possible freedom in the use of alcohol for industrial purposes. Due to the attitude of fairness of the Internal Revenue Bureau it is not too much to expect that we shall have alcohol under such conditions as will be an added stimulus to the progress of our rapidly growing chemical industries.

Aside from the natural growth of the alcohol industry care should be taken to foster its development. We are told that we should maintain a policy of preparedness throughout the coming years. Now that the whiskey distillery is to be a thing of the past where could we look for a supply of alcohol in case of war? In the recent war the distilleries came to the rescue and in case of another war we must have industrial alcohol plants in operation which could immediately supply large quantities of a most necessary product.

It has been said that the future increased production of alcohol could be attained by persuading the farmers to produce alcohol in small agricultural distilleries in a manner similar to those in operation in Germany. There are several reasons why this is not likely to take place. Labor is very much higher in this country than in Germany. The farmers of this country have become accustomed to production on an extensive scale rather than in an intensive manner, and are not likely to be satisfied with the results of a small distillery. In order to obtain satisfactory results the fermentation must be carefully controlled and the average farmer does not possess sufficient technical training to do this effectively. The manipulation of an alcohol plant is difficult except to the technical man. These farm installations would necessarily be small units because of the limited quantity of raw material available, the cost of the installation would be high, the labor cost would be excessive, the output would be small, and the unit cost of production would be so high that the farmer could buy alcohol cheaper than he could make it. Our case is quite different from that of Germany where these conditions do not exist, and where

<sup>1</sup> *J. Soc. Auto. Engrs.*, 5 (1919), 207.



the industry has been subsidized by the government. Without such subsidy and government pressure it is the writer's opinion that the farmers of this country are not likely to produce alcohol for industrial purposes for some time.

Alcohol is essentially a cheap commodity and should be treated as such. To-day this is not the case, and the manufacturer may be partly to blame for this condition. If manufacturers using alcohol in quantity would buy alcohol as they buy other commodities in tank car lots and not require shipment in expensive cooperage or steel drums, they would help to remove the impression that alcohol is such a valuable product. The ordering, billing, shipping, returning, crediting, etc., of these packages is a nuisance as well as a source of considerable expense. Alcohol is the only product in the history of the United States which has been taxed several hundred per cent of its value. This has given the impression that industrial alcohol is also extremely valuable. As the uses of alcohol are developed and as it becomes used more extensively for such purposes as motor fuel, it is not too much to expect that it should be handled in much the same manner as petroleum and its products. Great Britain is beginning to realize that alcohol is essentially a cheap product and should be treated as such. The British Inter-Departmental Committee on the Production and Utilization of "Power Alcohol" recommends in part the following:

As the price of alcohol for power and traction purposes, to which we propose the name of "power alcohol" should be given, must be such as to enable it to compete with gasoline, it is essential that all restrictions concerning its manufacture, storage, transport, and distribution should be removed so far as possible, consistent with safeguarding the revenue and preventing improper use, and that cheap denaturing should be facilitated.

Finally, we are of opinion that steps to facilitate the production and utilization of power alcohol in the United Kingdom can in no circumstances be taken, nor arrangements for such development carried into effect, unless provisions and alterations of the kind we recommend in our report are made in *advance* of the time when an acute recurrence of high prices for motor fuels may otherwise call for action too late for it to be effective.

Why should not the common carriers in this country as well as our Government recognize that industrial alcohol is a 50 cent commodity and not one of \$5.00 value? Why shouldn't manufacturers assist in bringing about such a recognition by using the specially denatured alcohols wherever possible and not be frightened by such words as "permit," "bond," and similar words identified with the Internal Revenue Bureau. The Commissioner of Internal Revenue is the friend of the manufacturer using alcohol and not his enemy. He will endeavor to assist you and not retard your progress. Over 40 formulas of specially denatured alcohol have been authorized for use in the manufacture of over 350 articles in addition to many class authorizations. The Commissioner of Internal Revenue will consider the authorization and the extension of the use of any formula in new industries or will consider the authorization of new formulas, if existing formulas are not applicable. As long as the Government's taxable interests in the alcohol are protected it may be freely used by the industries.

The requirements of the Internal Revenue Bureau surrounding the use of specially denatured alcohol are neither difficult nor prohibitive. The papers may be easily prepared. If you are a user of alcohol why shouldn't you take advantage of your privileges and assist in the stabilization and progress of our American chemical industries?

If the United States builds up a large industrial alcohol production and fosters its growth and development the industries will benefit and there is no question but that this nation as a world power will be stronger. During peace the nation will be strong because of its industries; during war these industries using alcohol will be instantly available as the media for the production of war materials.

#### CONCLUSIONS

In the preceding paragraphs it has been possible to mention only a few of the probable influences affecting the future of industrial alcohols, and to attempt to indicate in a general manner their effect on the future. Now that the realization of the necessity of industrial alcohol is becoming more general, legislation favorable to manufacturers is taking place and the industries using alcohols are developing rapidly. May we not look forward to the time when the industry of making industrial alcohols in the United States will be of such a magnitude as to be a great national asset under all conditions?

### RECONSTRUCTION ASPECTS OF SOME CHEMICAL INDUSTRIES IN THE UNITED STATES<sup>1</sup>

By Edward Gudeman

CHICAGO, ILLINOIS

It often takes a major operation to save the life of a person or of a nation. The glorious outcome of the war has not alone saved the life of our own people, but has been the salvation of the world, has amputated the heads of autocracy, sounded the death-knell of the cankerous German confederation, and in doing so, has saved the life of the German nation as well.

This heroic military operation brought to life the American chemists, who two years ago were hardly known by name, but who stand to-day before the world, the equal of our fellow-chemists in any of the four quarters of the globe. The horizon is still too near and the time elapsed too short to tell what they have done to help attain a mastery in the air, on the earth's surface, and below its depths, on land and sea. This must be left for coming historians of chemistry. We are now in the critical convalescent after-period of this military operation, and whether we fully recover and become economically, socially, and industrially stronger—in fact whether we survive or not, for the benefit, welfare, and prosperity of ourselves and coming generations—depends on what we do now, on our reconstruction program, its construction and operation. It is not with us a question of readjustment or of rehabilitation or of reclamation, but of reconstruction. Our reconstruction means *construction* of a structure that will justly and fairly meet our present and coming social and industrial problems. It cannot be a plan of one or a few and we cannot expect to erect it within a day or a few weeks. To-day we can only and must lay its foundation stone, anchored firmly on a bedrock of justice and equality.

The statement that "Money wins wars" has been shown to be false. It is loyal man and woman power, abroad and at home, inside and outside of uniforms, to whom the victory must be credited. No class, no profession, no sect can be singled out, and what I claim for our profession, others may claim for theirs.

Chemistry helped to create our powerful navy and our new merchant marine, and we must do our best to see that neither of them remains at a standstill. To-day the United States flag is flying on all the seven seas and never again will leave them, with or without a "Liberty of the Seas" whatever that may be or mean. To-day every land- and water-way is open to us, and must be kept so for us to take our place and have our share in the world's commerce.

Let us see to what extent our war allies of yesterday have become serious trade rivals, or perhaps friendly trade enemies. We all know that at the time of signing the armistice large stores of all kinds were accumulated and isolated in the Southern Hemisphere and in the Far East. Immediately with the release from military duties of a very large number of ocean carriers, these stores became accessible and available, and are now

<sup>1</sup> Address before the American Institute of Chemical Engineers, Chicago, Ill., January 15, 1919.



floating back through the old commercial channels into their former markets. Great Britain and France are taking advantage of these national opportunities, perhaps national obligations, and are cutting away from the United States, except so far as pre-war contracts and immediate necessity compel them to still deal with us. Commodities bought by foreign nations, and still in our country, are being resold to us at reduced prices.

Within thirty days of the signing of the armistice, negotiations were completed between Argentina and Great Britain and France for the whole wheat crop of that country, and, to facilitate payment, Argentina is said to have loaned \$240,000,000 for two years to England and France. Sugar is on its way to England from Java and Australia. We shall hold the bag for the Cuban crop as we guaranteed a fixed price for it, anticipating that England and France would take not less than 40 per cent thereof. Argentina and Australia are now shipping meats to Great Britain at 6 to 10 cents per pound below our export prices.

Great Britain is an asset to the Southern Hemisphere and the Far East, and the United States a liability. Friendship and commerce mix even though preference is a matter of dollars and cents, of francs, shillings, and marks. Therefore to protect ourselves, whether chemists or not, our reconstruction program must extend beyond the boundaries of our own country and we must make part thereof the underlying principle announced to his country by the British Minister of Reconstruction, that "the problem of reconstruction begins with shipping." Great Britain had a Minister of Reconstruction for many months before the signing of the armistice. What have we done and what should we do? We have appointed uncountable commissions, committees, and federal, state, county, and city trade boards, many unable to see beyond their own local horizon, or their own individual interests, some even tainted with political aspirations.

Anyone, whether chemist or not, can enumerate many problems that our reconstruction program should solve, and I have picked out a few, such as:

1—It is the moral and patriotic duty and obligation of the reconstruction program to put back into industry or commercial life, every soldier and sailor who has in any way become incapacitated, directly or otherwise, due to the war.

2—Our reconstruction program must give protection, assistance, and encouragement to all newly established and all newly developed American industries, many of which, if not directly chemical, have a chemical underlying basis. Of course, those which are exclusively war industries can be ignored.

3—The program must be one which will conserve our natural resources and our raw materials. Finance must come to the assistance of industries, not alone from private sources, but from our Government, through loans, subsidies, and otherwise. We must have protection, perhaps through regulated and controlled federal corporations. Mr. Frank A. Vanderlip stated "we must make great shipments of foodstuffs, of raw materials and in a lesser measure of manufactured products immediately after the war, and they cannot be paid for by equal shipments of goods to this country. Payment must be made to us through bonds and securities." This is very true from the viewpoint of his profession, finance, but it does not go far enough. For the protection of our industries we should be most conservative in the shipment of our raw materials, especially from our natural resources, and more liberal credits should be extended to American manufacturers to enable them to export our manufactured products. We should never again ship our raw materials in carload lots, and take them back in manufactured form, in ounce packages; and be forced, moreover, to compete with them both in foreign markets and at home due to liberal credits even now extended to our war allies and trade rivals.

The rank of a country among the nations is dependent on its ownership, control, and conservation of raw materials. The break through at Chateau Thierry sealed Germany's doom, not because it was a great military victory, but because it drove the opening wedge into Germany's commercial position, driving the Huns from necessary raw materials. Deprived only of two chemical essentials, iron and coal, a new Germany, whatever its geographical and political boundaries may be, will never again

be a menace to the world, never again can gain a commercial ascendancy, and will remain a minor power among the nations.

4—Health problems of to-day are distinctly chemical, and must have a strong plank in our reconstruction platform, not alone as to the prevention of disease, but as to protection against disease. We must incorporate therein the problems pertaining to sanitation, housing, vital statistics, birth registration, and many other social health reforms. We must have interstate health regulations as a regular thing, not only as an emergency call. A national health administration should be organized, not on strictly medical lines, but on lines similar to those of our past Food Administration, primarily educational, bringing health teachings into every household.

5—It is an absolute necessity that our reconstruction program include a strong educational policy. We should have compulsory education as a fact, and the education must be in English. We must obtain real Americanization of all our foreign-born citizens, of our immigrants, of our illiterate mountain white population, and of our negroes.

6—Transportation problems must be settled in our reconstruction program, and as all transportation, whether of commodity or speech, depends on fuel or electric power, the chemists are vitally interested in the correct solution of this problem.

7—Economic equality is one of the greatest and most important problems to be considered in our reconstruction program. Chemists are only a small part of the mass that must try to settle the questions as to labor and capital. Women are to-day acting as chemists in many industries, and the records show that as to capability and efficiency they are equal to the men. We are directly involved in the settlement of the replacement of man's labor by woman's labor, and *vice versa*. This great problem of the replacement of the one by the other can only be justly solved through individual selection, based on personal considerations, and it cannot be at the expense of man or woman as a class.

Economic equality is of international scope and will affect political and diplomatic questions, may eliminate reciprocity treaties, and most favored nation acts; will affect taxes and duties, and can regulate immigration and emigration; may bring about an international day of labor, with a minimum wage scale. It is of such magnitude that it can be a consideration of the Treaty of Peace, and may even become the preamble of the Constitution and By-Laws of a coming League of Nations.

Considering some of these reconstruction problems from the narrower viewpoint of the chemical profession, let us see where we stand and what we can do to become a force in solving some of these problems.

Nothing pleases a child more than to be able to say "I told you so," and I admit being childish. At the meeting of the AMERICAN CHEMICAL SOCIETY in April 1914, I asked: "What is the standing of the chemists in the United States, not as we see ourselves but as others see us? In fact do we, as a profession, exist at all? Is it not time (1914) for the chemists to get the recognition due them and to place the profession on the high plane it should occupy? In Illinois the only two scientific professions that can be followed by anyone without a license, permit or qualification to practice, are those of cooks and chemists."<sup>1</sup> To-day we are known and stand on a high plane, but as to qualifications and requirements there has been no change; in fact, many are acting to-day as chemists who hardly know what the word means, and who have had no training at all, not even a diploma from a school of correspondence. We have no legal or official standing, and consequently no professional responsibility; are not in the same class as other professional men, such as druggists, doctors, lawyers, engineers, or ministers.

Now is the time, right now, to start something, while we are still in the public eye, when blame or credit is given us for at least having been involved in the war's activities. To-day the public and the press are still impressed with the "supreme" importance and value of chemistry, if only as a war science. We have a strong numerical and very influential backing of the other unrecognized profession, that of the cooks, who have been so dependent on us, through our scientific control of their

<sup>1</sup> THIS JOURNAL, 6 (1914), 689.



raw materials. No one will forget the slogan, "Food will win the war." We backed the cooks, some 20,000,000 kitchen soldiers, and taught them more about foodstuffs and feeding in 18 months than they had been taught during our whole previous existence from 1492. Chemical terms such as calories, proteins, fats, carbohydrates, and vitamins are to-day common household words, and even in the kindergarten they know what the chemical verb "to hooverize" means.

In September 1914 I spoke to the Chicago Association of Commerce<sup>1</sup> and in December before this Institute<sup>2</sup> using the same title for my paper as of to-day, without the word "Reconstruction." Conditions have not changed, except in so far as temporary war measures have, for the time being, modified them, and unless we can make many of these temporary measures permanent, we shall drop back into the same old rut. The statement of the past will apply to our future, that "In many cases, not all, Federal requirements so restrict us as to actually prohibit us from manufacturing and going into competition with, and replacing imported articles. Patent, trademark, and copyright acts, with internal revenue acts, stand in the way, firmly braced by the Sherman Act. You can convince yourselves of this by a study of those (chemical) industries in the United States where this interference, this prevention is not felt." The "Trading with the Enemies" Act gives some relief, annulling temporarily our patent and trademark acts, but this is limited, requiring a government license, with safeguards to protect the former owners, as to profits and royalties. Restrictions under the Sherman Act, the Interstate Commerce Act, the Foods and Drugs Act, and others, are partly annulled, for the time of the war, and perhaps for a limited time thereafter, but some of these privileges are already withdrawn, and many of those active in their enforcement have received very complimentary letters of thanks for past services. Powers delegated to the War Finance Corporation, Emergency Fleet Corporation, War Industries Board, War Labor Board, Food, Fuel and Railroad Administrations, Chemical Warfare Service, and other similar bodies should not be given up, without substituting something, at least during the reconstruction period.

The Webb-Pomerene act is a permanent measure and no doubt will be of commercial importance, although it sets up a dual standard for doing business. It nullifies the Anti-Trust acts, allowing combinations or trusts in the United States, but only as far as export trade is involved. If it is a good business policy to allow trusts or combinations to obtain a foreign market, why will it not be of equal benefit to allow regulated and controlled combination at home to enlarge and hold the home market? Would this not enable us to meet foreign competition at home better, especially if we protect manufactured products and raw materials? In effect this act allows us to combine to dump our goods in certain foreign markets, but in no way prevents foreign goods from being dumped on us. This dual standard for doing business may bring about the conditions that we can re-import, duty-free, exported manufactured products, at a lower price than we can get them at source of manufacture. This act may become a boomerang if not a Frankenstein.

At the Paris Economic Conference, as early as September 1916, a plan was considered by the Allies to restrict the markets of the Central Powers, and it was proposed to deprive German industries of raw materials, by conserving for themselves "their natural resources and establishing social arrangements to facilitate interchange of these resources." Commercial history shows that national trade wars are as destructive as military wars, but that measures of defense are necessary to prevent a nation's manufactured products from discrimination. We have magnified or exaggerated the importance to us of

foreign markets for our raw materials, as compared with our manufactured products. If we are a market for the whole world, why not make the world our market for our manufactured products? We can do so by restricting exports of and interchanging of raw and natural resources. We must consider the opposite of a Webb-Pomerene Act, an Anti-Dumping Act, adequate to protect American business from unfair foreign methods, and this is more of a necessity for the chemical industry than any other.

It was a chemical war, and must be a chemical reconstruction. As to some of the exclusively chemical problems before us, only a superficial and very narrow review can be attempted. Individual cases cannot now be considered, such as changing a synthetic phenol factory into one making rose oil or vanillin extract, and the rebuilding of a flexible glass works into one making non-losable golf balls, is of minor importance.

Our most important chemical reconstruction should come in the teaching of chemistry, not alone in our universities and colleges, but in our preparatory schools. Dr. Henry S. Pritchett, President of the Carnegie Foundation for the Advancement of Teaching, in the preface to Prof. Chas. R. Mann's bulletin on "A Study of Engineering Education, 1918," very forcibly says:

In the half century that has passed, this course of study (engineering) has been overlaid with a number of special studies intended to enable the student to deal with the constantly growing application of science to industries. While the original plan remains as the basis of the teaching in the four-year engineering curriculum, the course given in most schools has been greatly modified in the effort to teach special subjects. As a result, the load upon the student has become continually heavier and bears unequally in different places and in different parts of the course. In addition there is a wide-spread feeling that under this pressure the body of the students will fail to gain, on the one hand, a satisfactory grounding in the fundamental sciences; and on the other hand, do not fulfill the expectations of engineers and manufacturers in dealing with the practical problems with which they are confronted on leaving the engineering schools.

Have not the last two years shown that this applies as strongly to chemistry as to engineering? We all know that before the war there was a strong fence dividing the theoretical, research, and pure chemical scientist from the practical, commercial, and industrial chemist. A few outsiders, some connected with technical schools, some in large laboratories, and some individuals, straddled the fence, seldom doing anything to bring the two factions together, often in fact finding it to their advantage to keep them apart. Commercialization of chemical science was abhorrent to many teachers of chemistry. The pre-war German chemical preëminence was due not to any super-capability or super-mentality of her teachers of chemistry or other sciences, but to their utilizing and developing *industrially* every advance in scientific knowledge. No one can point out any new fundamental discoveries made by German scientists within the past forty years. Laws of chemistry and physics were discovered by Dutch, Norwegian, English, French, Italian, Japanese, Canadian, and American men of science. Many scientific discoveries have been claimed by the Germans, who simply appropriated them, and developed and applied them in their industries.

But the change in our teaching cannot be confined to our educational institutions, it must also embrace our employers and employees. We must develop a scheme for adequate training of workers so that their versatility, efficiency and productivity will be increased—a mutual advantage to capital and labor. We must have an association between labor and capital, not alone practical and physical, but also mental and educational.

We had different classes among our chemists some two years ago, and the contempt shown each other by the members of these factions was mutual. There is to-day no chemical labora-

<sup>1</sup> Chicago Assoc. Commerce, *Bulletin*, October 1914.

<sup>2</sup> THIS JOURNAL, 7 (1915), 151.



tory, collegiate or university, corporation or private, whose chemical staff was not temporarily badly disrupted by the war. These chemists, men and women, are returning to their pre-war industries and activities, and they will insist on a radical reconstruction in the chemical curricula of our institutions.

We are sure that our new mutual comrade in chemistry, Prof. X. Y. Z., having served our Government, perhaps having perfected a new waterproof coating for airship wings, assisted by a former stock-yards chemist, or having supervised the making of deep-sea bombs, with the help of a fermentation expert, or reconstructed a gas mask, coöperating with a fuel specialist, will not be satisfied to accept for a doctor's degree a thesis describing a new compound containing 23  $^{13}$ /<sub>19</sub> carbon radicals, arranged in a parabolic curve, and some 57 varieties of salts made therefrom, all ready to be nicely hidden away in the cellar of the chemical museum of his Alma Mater; nor will the practical chemical cuss run and dodge, or take the other side of the street, when he sees Prof. X. Y. Z. in the far distance. This war has conclusively shown us that "real genuine research work and chemical manufacturing experience go together, are absolutely dependent on each other, and are the foundation of the development of chemical industries."

Reconstruction in the teaching of medicine must also come, and it must follow chemical lines. Recent medicine is directly chemical. Preventive medicine depends on products originated and produced in the chemical laboratory, whether a direct active medicinal preparation, or a serum or anti-toxin. Internal medicine is based on chemical and microscopic analysis, which may consist in a blood count, analysis of stomach contents, or the making of a direct coal-tar color in testing the urine by the indol reaction. The first treatment of the wounded on the battle-field is chemical, involving the use of iodine, and in the base hospital the Carrel treatment with the Dakin solution depends on the action of chlorine. The excellent health conditions in our Army and Navy, at our camps and cantonments were due to chemical means, even if camouflaged under the name of sanitary methods.

Little authentic information is at hand as yet in regard to the damage done through the illegal, brutish, foul and inhuman use of poisonous and noxious gases. Prospective medicine will have to consider the effects caused by these chemical death-dealing and health-destroying gases, but it is not improbable that within a reasonable time these and other chemical gases may be used legitimately as health-restoring agents, especially against infectious diseases of the respiratory organs, and diseases transmitted to the system through nose or mouth. It may be wrong to draw the conclusion, but the suggestion is not out of place, that the relatively small number of cases and fatalities, due to influenza, among the allied troops on the Western front, was partly due to the peculiar antiseptic character of the atmosphere there, both on account of the poisonous antiseptic gases used in warfare and also the gases generated by the ammunition exploded. This view is supported by the meager circumstantial evidence that at the second battle of the Marne, the turning point of the war, the German advance was retarded for 10 to 15 days, due to the epidemic of influenza among her troops. *The allied nations did not make use of intensive gas warfare at that time.* A little, not very authentic, data is also at hand, indicating that workers in plants making war gases, mainly chlorine, were more immune against infectious diseases than others in the same communities, and the same thing seems to be indicated for workers in other chemical industries where active gases pollute the atmosphere.

According to Dr. F. Tweddell,<sup>1</sup> "certain occupations are comparatively free from the curse of phthisis and others are immune to it. On page 98 of Fishberg's 'Pulmonary Tuberculosis' we find, 'coal dust prevents the development of phthisis, and that

miners are less subject to phthisis than all other occupied males.'" Dr. Tweddell suspected sulfurous acid gas as the cause of this immunity. In this connection many of us remember our grandmother's treatment for whooping cough and throat troubles, sending the children to coal-gas works to play in that polluted, antiseptic atmosphere. Many cases could be cited to show how dependent present-day medicine is on chemistry, its products and processes, and that a reconstruction in medicine and its teachings must be along chemical lines.

A chemical influence in the reconstruction of teaching some underlying principles of law can also be anticipated, if only so far as to have expert testimony confine itself to actual facts, rather than to precedents based on the scientific fancies and follies of the legal profession.

An inoculation of some chemistry into theology is also not out of place, especially in calling attention to the fact that the right way of living, the use of certain foods, and the abstaining from others, and certain religious sanitary laws are based on chemical facts.

Agriculture, production, distribution and conservation of the products of the soil, is strictly a chemical consideration, and we should be active in solving the many agricultural problems that our reconstruction program will have to consider. We never again should become as extravagant in the use of our food products as we were before the war. The economical principles we were taught and practiced should remain a part of our national custom. Four pounds of sugar per month will satisfy the most fastidious sweet tooth and a return to about 90 pounds per capita per year will again be wilful waste. We got along very well when using a greater amount of cereals, rice, cornmeal, home-grown vegetables, and lesser amounts of meats. The Government has recognized the necessity of stabilizing our agricultural industries through guarantee of a minimum price for crops grown during 1919. We need only consider one real chemical industry to show the chemist's interest and place as to agriculture.

Before the war we depended on foreign nations for the greatest part of our nitrogen and potash fertilizers. We had phosphoric acid in the form of natural phosphate rock. This natural raw material we exported in very large amount, shipping it as ballast from southern ports. We took part of it back, after the phosphoric acid had been made available, paying a high price for this simple chemical transmutation. Although we did use some of this phosphate rock ourselves, we never exported any phosphate fertilizers. As to nitrogen fertilizers, we actually paid tribute to foreign nations, and furnished the dividends for the British niter trust. Our main nitrogen fertilizers came from Chile, as guano, often after having crossed the ocean twice, with stop-overs at Hamburg or Bremen, or English or Dutch ports.

Potash salts we had none except those imported. We used many in our industries and laboratories, which we have since replaced with sodium salts. This unnecessary use of potassium compounds was fostered through the propaganda of the German alkali trust. To-day we are home producers of potash salts. Are we going to construct a program that will enable these home industries to continue?

We can anticipate a surplus manufacture of nitrogen and potash fertilizers and other similar products, and must develop a system that will take care of these industries. We cannot kill off the development of our potash deposits and potash kelp industry, nor should our potash recovery in steel mills and cement plants be given up. While our steel and cement industries will live and prosper without a recovery of potash salts from their flue gases, we cannot allow them to waste these products. Nothing definite can be stated to-day as to our natural potash deposits, as they are still in the initial stage of development. The kelp potash industry is undergoing a radical

<sup>1</sup> *Medical Record*, December 21, 1918.



change, and the potash recovered is becoming a by-product, the main products being those obtained from "wood" distillation and iodine recovery. Our nitrogen fixation plants will have to compete with the by-product coke industry and the gas-house nitrogen plants.

We must bear in mind that these new nitrogen and potash plants give us really *new* sources of supplies, commercially non-existent before the war. Our production, therefore, is in excess over the world's pre-war production and we have substituted our own products for those we formerly imported. We must look for a greater production right along and must enlarge our home consumption to take care of our own products of manufacture. We cannot expect any export business, as our lessened imports will increase available stocks in foreign countries. The nitrogen products from our government nitrogen fixation plants are to be taken care of by the Nitrate Division, through the Department of Agriculture, who expect to sell during 1919 about 300,000 tons of nitrate of soda to farmers, at cost price on board of cars at loading points. With a proper reconstruction program, our fertilizer industries can also be free from importations. The proper protection of our nitrogen industries has direct bearing on our dye industry, as its source of raw material is the same, coal-tar and coke industries. In 1917 we manufactured an excess of dyes over our American needs, and exported United States dyes of the class formerly imported to the value of \$11,709,287, exceeding in value our pre-war importations, although we did not equal our former imports in tonnage or in variety of colors. The 1918 value for dyes exported is given at \$16,921,288. The total amount of dyes made in the United States for 1917 is valued at \$57,639,901.<sup>1</sup>

The industrial chemist must not forget that to-day we are perhaps the only credit nation on the surface of the earth. We cannot expect to export products which we formerly imported, without seriously affecting trade balances, especially if at the same time we curtail our imports through home manufacture. To maintain a necessary equilibrium in trade we must buy as well as sell, we must create liabilities to make good our assets. In all cases we shall have to meet strong competition from our former war allies, our present friendly trade enemies, who are to-day ignoring as far as they can our agricultural products, going to the Southern Hemisphere, Australia, the Far East, and Africa for wheat, sugar, meats, leather, wool, cotton, and other products of the soil. To uphold our agricultural industries and those depending thereon, we must have protection, free trade, or a tariff for revenue, the proper financial assistance, a correct government control, regulation or ownership, or at least a partial government partnership in industry, with or without subsidies. We must look to our own home market for our greatest demand and must protect this so as to hold it absolutely against all outsiders, and we must have a fair return on invested labor and capital. Under no condition can we allow our country to become a dumping ground for any foreign commodity, notwithstanding that the Webb-Pomerene act allows us to do to others what we don't want them to do to us.

These requirements hold good for many chemical commodities: products from our glass, china, porcelain, clay, and ceramic industries; it holds good for our coal-tar industries, dyes, colors medicinal preparations, flavors, perfumes, solvents; it does not differ for our food, fuel, petroleum, rubber, wood, textile, or paper industries; it applies to our metal and alloy industries; in fact, to every industry where chemistry is or can be involved. It applies to every industry whose products we formerly imported, and for all products for which we have found a suitable United States substitute.

It will not be many months before the devastated territory of France and Belgium will be rehabilitated and again productive.

<sup>1</sup> U. S. Tariff Commission Census of Dyes and Coal-Tar Chemicals, Information Series 6.

America is helping not alone with food and money, but is actually rebuilding many industrial concerns, on American foundations and according to American methods. What the American Army and Navy did in France is bearing fruit among the people of France and Belgium and they have learned a lesson through observation. They see before them the marvelous picture of a community of about 2½ million people created within 18 months, everything, people and supplies brought from 3000 miles away. Future history will designate this work of our Army and Navy (and much of it was chemical) as the eighth wonder of the world, greater than the combined seven wonders of past history. These American methods of doing things are incorporated in the rehabilitation schemes of those countries and their application will greatly shorten the time of recovery, bring them so much more quickly into a productive stage, and so much sooner into the world's markets and into competition with all their war allies. American steam shovels, portable saw mills, traction engines, and many chemical reclamation tools are used in the French and Belgium reconstruction programs, which call for extension of canals, new water- and roadways, reforestation and every other means to make their lands more productive and fitter places to live in. This has been a chemical war and will be a chemical restoration. We chemists cannot again be given places on the last row of the top gallery, in this great world's show. We have been actors, in the spotlight, holding the center of the stage. We are to-day strong and our existence is recognized, and we stand where we stood five years ago. "The chemists of the United States have the brains and the ability and the gumption to make it—not 'Made in America' but 'Made in the U. S.' All the chemists in the United States need and all they again ask for is the opportunity, fair play and an open field, not fenced in with adverse legislation."

We have now the opportunity and can do our part in seeing that chemical industry allows every one of our returning fighting boys to return to his pre-war activities. We have the chance to do our part in the reconstruction of our educational systems. We must be an active force in solving problems as to health and social economic conditions. We must be a strong factor in the development of our agricultural interests and the industries depending thereon. We must enforce our views for the protection of all new and newly developed chemical industries. We must exert ourselves in regard to the conservation and utilization of our raw materials and our natural resources, and in the production and distribution of our manufactured products. We must become a vital part in a real chemical reconstruction program.

## NITROCELLULOSE FROM WOOD PULP<sup>1</sup>

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### INTRODUCTION

Considerable interest was aroused in 1915 by press notices that the Germans were successfully using wood cellulose in place of cotton cellulose in the manufacture of smokeless powder. Due to shortage of cotton in Germany caused by the blockade it was absolutely necessary for them to turn to this source for nitrating material. It can be readily appreciated that with the enormous supplies of short-fibered cotton available in this country for nitrating purposes there appeared to be no possibility whatsoever that this country would be placed in the position of having insufficient cotton for smokeless powder manufacture, or have to figure on the use of wood cellulose to supplement its supply of cotton. The object of this paper is to

<sup>1</sup> Read before the New York Section of the American Chemical Society, Chemists' Club, New York, N. Y., November 7, 1919.



give some idea as to how the Government and the powder manufacturers together were prepared to meet this emergency, which actually arose a few months before the signing of the armistice.

#### HISTORICAL

The attempt to produce nitrocellulose from wood dates back to the year 1865. During that year an article appeared by a Prussian artillery officer, Edward Schultze, in which was noted the preparation from wood of an explosive and propellant. In the years 1870 and 1871 Volkmann improved this so-called "white powder" by gelatinizing the nitrocellulose with an ether-alcohol solution. Although this Schultze powder was not suitable for military purposes, it attained a great degree of popularity as a sporting powder.

The United States, as is generally known, was the last of the world powers to adopt smokeless powder for military use in place of black powder. As the nitrocellulose used in foreign smokeless powders was at that time made from cotton, and as there was an aversion to the use of wood cellulose, it was quite natural that the smokeless powder industry in this country should have been initiated with the use of cotton cellulose as the raw material for nitrocellulose.

Carl Schwalbe<sup>1</sup> states:

Up to the present time nitrocellulose used in different industries has been prepared almost entirely from cotton, although wood cellulose in sufficient amount and in a suitable physical condition has been available at a lower price; furthermore, the wood cellulose is produced in Germany while cotton must be imported. Nevertheless the more expensive cotton is given preference because pure nitrocellulose is more easily obtained from pure cotton cellulose and because the existing impurities in the wood cellulose in all probability exert an influence on the stability and on the process of stabilization.

By far the most important reason why wood cellulose was not used in place of cotton for smokeless powder manufacture was the question of chemical stability of the powder during storage. This has always been a very vital problem, affecting as it does the safety of officers and men of our Army and Navy, besides the safety of property. While there are various stability tests at elevated temperatures which give an indication, in a relatively short time, of the stability of a powder and afford a very useful criterion of the quality, its stability is largely gauged by what occurs in actual storage. Inasmuch as too little has been known regarding the chemical stability of smokeless powder under various conditions, and since diphenylamine was not adopted by this country as the stabilizing agent until 1909, it can be appreciated that government officials have been very reluctant even to consider a change to some source of cellulose other than cotton, without the most thorough investigation, covering observations of powders during many years of storage under varying conditions.

As far back as 1909, however, a survey of the subject indicated the possibility of economy due to the lower cost of wood pulp as compared with cotton. At this date several articles had been published in the literature, largely by the Germans, which were not particularly encouraging as regards the use of wood cellulose, indicating lower yields of nitrocellulose, greater consumption of acid, and questionable stability. In 1915, when it was announced that the Germans were successfully using wood cellulose for nitrocellulose in place of cotton, it was planned in our laboratories to nitrate on an experimental scale a few hundred pounds of wood cellulose, purify the product, and manufacture the same into powder for ballistic and chemical tests, although it was not thought then that there would ever come a time when the amount of cotton available in this country would be less than the amount required for war purposes.

Great difficulty was met in obtaining wood pulp suitable for nitrating purposes. Although a great many samples were

obtained from various manufacturers, all indicated insufficient purification treatment, severe overbleaching, and entirely unsuitable physical condition. However, with a great deal of difficulty, due largely to unsuitable physical condition of the wood pulp, about 50 lbs. each of sulfite and soda-wood pulp were nitrated in laboratory equipment, the nitrocellulose purified according to government specifications, and made into 0.30-cal. and 1-pounder powder. The results of stability and ballistic tests were very encouraging. The unsuitable quality of wood cellulose from the standpoint of chemical purity and composition as compared with cotton and the unsuitable physical condition impressed us with the need of large-scale experiments in order to obtain data regarding manufacturing difficulties in the nitration of the wood pulp, purification of the nitrocellulose, and its manufacture into powder.

During 1917 the development of the problem of using wood cellulose on a large scale was greatly facilitated by the fact that specially purified soda and sulfite wood pulp were obtained in suitable quantity.

Acknowledgment is here made of the assistance given by the West Virginia Pulp and Paper Company of New York in preparing at our request special lots of purified soda and sulfite wood pulp; and also of the assistance given by the Forest Products Laboratory, of Madison, Wis., in furnishing us information in regard to available supplies of wood and the purification treatment received by the various woods in the preparation of commercial wood pulps.

#### BRIEF OUTLINE OF SMOKELESS POWDER MANUFACTURE

The first step in the process of manufacture of smokeless powder is the purification of the cotton by digestion with 2 per cent caustic soda solution for 4 or 5 hrs. at 75 to 80 lbs. steam pressure. The digester charge is then blown over into a wash tank to remove the waste liquor. The cotton is bleached with a very weak solution of bleaching powder and washed several times with water. It is then run through squeeze rolls to remove a part of the moisture, picked to open up the fiber, and finally run through the cotton driers until the moisture content is reduced below one per cent. It is next nitrated for about 30 min. in a mechanical agitator, dumped into a centrifugal wringer, and the excess of acid removed by wringing for about 3.5 to 4 min. The nitrocellulose, containing about a pound of acid for every pound of nitrocellulose, is then dumped into a drowning basin containing a large quantity of water and thence pumped to the boiling tubs. It receives a preliminary boiling treatment in acidulated water for about 40 to 45 hrs. which serves to hydrolyze and break down the unstable esters, especially the sulfuric esters; is then pulped by means of boilers or Jordan engines; and after pulping receives a poacher boiling treatment, divided into several treatments of a total duration of 12 hrs. The purified nitrocellulose is then wrung in centrifugal wringers until the moisture is reduced to about 30 to 33 per cent. This operation completes the preparation of the nitrocellulose for smokeless powder manufacture.

The next process in the manufacture of smokeless powder is the removal of the water in the nitrocellulose by means of alcohol. A charge of wet nitrocellulose is formed into a block in a hydraulic press by means of low pressure of about 250 lbs. per sq. in. About 50 lbs. of alcohol are pumped through a perforated screen in the head of the press, and as it passes down through the nitrocellulose displaces the water. To remove the excess alcohol the block of nitrocellulose is pressed under a pressure of about 3,000 lbs. per sq. in. until it contains the desired amount of alcohol, as predetermined by weighing a block of dehydrated nitrocellulose and marking the size on the tell-tale.

The blocks are then sent to a block breaker, consisting of a revolving screen of very heavy wire containing balls which break

<sup>1</sup> *Z. angew. Chem.*, 1 (1914), 662.



up the block into small pieces. The charge of nitrocellulose is then placed in the mixer and treated with sufficient ether so that to every 100 lbs. of nitrocellulose there are about  $33\frac{1}{3}$  lbs. of alcohol and  $66\frac{2}{3}$  lbs. of ether. Diphenylamine, which is used as a stabilizing agent, is previously dissolved in the ether. The mixer is provided with brine refrigeration to prevent loss of solvent. The mixing of the nitrocellulose requires about 30 min. at the end of which time a mealy looking colloid is obtained. This is formed into a block about 12 in. in diameter and 16 in. high, at a pressure of about 3,000 lbs. Three blocks of this size constitute a charge for the finishing press, where the colloid is pressed through dies and granulated on cutting machines into grains of various sizes, according to the caliber of the gun. The grains then receive a treatment of several days in order to remove and recover the excess of solvent. The powder is next treated in a tank of hot water at about  $55^{\circ}$  C. for several days until the solvent content is reduced to the amount desired, when the excess of water is removed by air drying. The powder is then taken to large blending towers and in lots of 100,000 lbs. each given a very thorough blending. Ballistic and chemical tests are finally made on representative samples of the lot of powder.

#### NITRATION OF WOOD PULP ON LARGE SCALE—PRELIMINARY EXPERIMENT

The processes of manufacture in which it was expected that the use of wood-pulp cellulose would give difficulty were in the nitration and purification of the nitrocellulose and in its dehydration and colloidizing. Early in 1918 it was decided to nitrate about 1,200 lbs. of sulfite wood pulp in the regular nitrating equipment in order to obtain some information about the difficulties which would be met in nitrating wood pulp in regular factory installation. This wood pulp, in crepe paper form, gave considerable difficulty in nitration. There was a tendency of the nitrated material to gelatinize during nitration, and owing to this and to the shortness of the fiber the wringing of the acid from the nitrocellulose in the centrifugal wringer gave great difficulty. The yield of nitrocellulose was very low, only 1.2 lbs. of nitrocellulose per pound of wood cellulose, as compared with 1.5 lbs. when nitrating cotton. The consumption of acid was high, due to difficulty in wringing the acid from the nitrocellulose. Considerable difficulty was also found in the dehydration of the nitrocellulose. Due to the very finely divided condition of the material it required almost 50 per cent longer to pump the alcohol through and to remove the excess alcohol. It was found, as was expected, that the viscosity of the wood pulp nitrocellulose was much lower than that prepared from cotton, the amount of solvent required for colloidizing being about 8 per cent less than when using nitrocellulose prepared from cotton.

The above experiment impressed us with the serious mechanical difficulties involved in the use of wood pulp for nitrocellulose and the necessity for a thorough chemical comparison of wood cellulose with cotton cellulose.

#### COMPARISON OF COTTON CELLULOSE AND WOOD CELLULOSE

As previously stated, wood pulp prepared for paper making is not sufficiently purified or in a suitable physical condition for nitrating.

With reference to the specifications for purified cotton for nitrating purposes the War Department requires it

To be bleached cellulose prepared for nitrating which will be obtained by purifying unspun cotton waste or suitable short-fibered commercial cotton and thoroughly washing to remove the purifying material or salts.

To contain not more than 0.4 per cent of ether extractive matter, not more than 0.8 per cent of ash, and not more than traces of lime, chlorides and sulfates, nor more than 7 per cent KOH-soluble material.

To be of absorbent and uniform character, clean and free from such lumps as will prevent uniform nitration; to contain as

little as possible of felted fiber and dust, technically known as "fly," to be free of motes, flues, sweepings, hull fiber, or other foreign matter.

Moisture must not exceed 7.0 per cent.

It may be of interest to note that these specifications were very misleading to pulp manufacturers, many of whom furnished samples of wood pulp which complied with the specifications as regards ether extractive matter and ash, but did not appreciate that the specifications were meaningless when applied to anything but cotton. The specifications in regard to KOH-soluble matter were not incorporated in the government specifications until the latter part of 1918, although this specification had been used for many years by us. While these specifications were sufficient to cover the purity of cotton cellulose they were unsuitable for wood cellulose.

Wood belongs to the widely distributed class of lignocelluloses of which jute probably represents the purest form. In addition to lignocellulose as the main constituent, hemicelluloses, *i. e.*, the pentosans, are always present in wood; while resins, gum, tannin, carbohydrates, dyes, ethereal oils, etc., occur as secondary constituents. Wood pulp requires a much more drastic cooking treatment than cotton cellulose to remove these impurities. Moreover, the purified wood cellulose is considerably different in chemical composition from cotton cellulose. This difference is strikingly brought out by the method of Jentgen, extensively used by Cross and Bevan, of dividing cellulose into three types:  $\alpha$ , which is insoluble in cold 17 to 18 per cent caustic soda solution when treated for 30 min.,  $\beta$ , that portion which is dissolved by the caustic solution but reprecipitated on the addition of acetic acid, and  $\gamma$ , that portion which is dissolved by caustic but not reprecipitated on the addition of acid. Applying this method of analysis to purified cotton such as is used in smokeless powder manufacture, we find that it consists of approximately 94.5 per cent  $\alpha$ -cellulose, 3.0 per cent  $\beta$ -cellulose, and about 2.5 per cent  $\gamma$ -cellulose. If the cellulose is very strongly bleached, severely digested, or both, the tendency is to increase the  $\beta$ -cellulose; for example, a long cooked and well bleached cotton gives 81.0 per cent  $\alpha$ -cellulose, 16.0 per cent  $\beta$ -cellulose, and 3.0 per cent  $\gamma$ -cellulose. We know that in cotton cellulose the  $\gamma$ -cellulose is lost during nitration but that the  $\alpha$ - and  $\beta$ -celluloses are converted into nitrocellulose. As shown by Piest and others overbleaching gives nitrocellulose more difficult to stabilize, so that specifications for cotton for smokeless powder manufacture prescribe that overbleaching is to be avoided.

The chemical composition of the cellulose in soda and sulfite wood pulps differs from cotton cellulose. For example,

	$\alpha$ -Cellulose Per cent	$\beta$ -Cellulose Per cent	$\gamma$ -Cellulose Per cent
Unbleached sulfite pulp from spruce...	85.0	7.0	8.0
Mildly bleached sulfite pulp from spruce.....	87.0	3.0	10.0

It will be noted that sulfite pulp, bleached or unbleached, usually contains about 8 to 10 per cent of  $\gamma$ -cellulose which is of no value for nitrating purposes. Small-scale digestions in the laboratory indicated that most of the  $\gamma$ -cellulose and a portion of the  $\beta$ -cellulose in sulfite pulp, either bleached or unbleached, could be removed by digesting with caustic soda solution under pressure, and that the resulting product would approach more closely to the composition of cotton cellulose. This is shown by the following comparison:

	$\alpha$ -Cellulose Per cent	$\beta$ -Cellulose Per cent	$\gamma$ -Cellulose Per cent
Purified cotton.....	94.5	3.0	2.5
Mildly bleached sulfite from spruce, digested 5 hrs., 80 lbs. pressure, 2 per cent caustic soda solution.....	95.0	2.0	3.0
Unbleached sulfite from spruce, re-digested as above.....	94.5	2.0	3.5

Soda pulp, on the other hand, naturally contains less  $\gamma$ -cellulose because of the alkaline digestion treatment, but the amount



of  $\beta$ -cellulose is very much higher than in the case of sulfite wood pulp, even in the unbleached material.

	$\alpha$ -Cellulose Per cent	$\beta$ -Cellulose Per cent	$\gamma$ -Cellulose Per cent
Bleached soda pulp from Jack pine....	67.0	26.0	7.0
Same, redigested 5 hrs., 80 lbs. pressure, 2 per cent caustic soda solution....	85.0	13.0	2.0
Unbleached soda pulp from Jack pine..	86.0	10.0	4.0
Same, redigested with caustic soda solution, under pressure as above...	84.0	14.0	2.0

The unbleached soda pulp contains considerable colored products which are not removed by digestion with caustic soda solution and give a very dark nitrocellulose. Furthermore, these colored products give a very dark smokeless powder. If the soda pulp is bleached and redigested with caustic soda, the color of the pulp is very materially improved, owing to the removal of the colored products which are made more soluble in alkali by chlorination. Briefly, while sulfite and soda pulps as prepared for paper manufacture are not purified sufficiently for nitrating purposes, further digestion under pressure with caustic soda solution, preceded in the case of soda pulp by a mild bleaching to improve the color, will give a cellulose more nearly approximating the chemical composition of cotton cellulose, except that the soda pulp will have a higher  $\beta$ -cellulose content than cotton.

Another important difference between wood and cotton cellulose is the length of the individual fibers. Second cut linters will have a maximum length of about 25 mm., a minimum of 0.80 mm. and an average of about 4.6 mm. Hull shavings will average about 2.4 mm. with a maximum of 8.00 mm., depending on how much linters have been removed from the seed, and a minimum of 0.5 mm. Wood fiber from coniferous woods will usually have an average length of about 2.5 to 4.0 mm. Reference has already been made to the difficulty experienced in the nitration of a small lot of sulfite wood pulp in regular factory equipment. It should be appreciated that the factory apparatus for nitration, purification of nitrocellulose, and its manufacture into powder are the result of a gradual development covering a period of more than 20 years. A number of years ago experiments demonstrated considerable mechanical difficulties in using hull shavings alone, due to the tendency of the short fiber to dry harsh, and the difficulty of wringing out the excess acid after nitration. It was found at that time that the addition of 30 per cent or more of the linters to the hull shavings greatly facilitated the drying of the short fiber in suitable physical condition for nitration, and also the wringing out of the excess acid. For a number of years previous to the war it was the standard factory practice to use a mixture of linters and hull shavings for nitrocellulose.

We have already referred to the unsuitable physical condition of wood pulp for nitrating purposes. Cellulose in harsh paper form cannot be nitrated as the acid does not readily penetrate the material. It is necessary that the cellulose be absorbent and preferably in fibrous form or very thin sheets, in order that the proper nitration of the cellulose can be obtained. While it was appreciated that wood pulp prepared in tissue or crepe paper form could be dried in fairly soft condition for nitration, this would have involved the manufacture and installation of special machinery by the paper manufacturers, and, furthermore, the nitrating equipment in our factories was not suitable to handle wood pulp in this form.

Numerous experiments were made in an endeavor to produce a softer wood cellulose, and one which could be dried in bulk like cotton, by digesting the wood under milder conditions than used for paper making purposes. While this produced a slightly softer fiber, the difference was too slight to be an important factor. It was accordingly decided to experiment on the use of wood pulp by blending it with cotton fiber before use.

Accordingly a number of factory experiments were made with mixtures of cotton fiber and sulfite or soda wood pulps. It was found that at least 50 per cent of wood pulp could be used in the mixture without seriously affecting any operation in the manufacture of smokeless powder. This was very gratifying in view of the magnitude of smokeless powder manufacture.

The idea as finally developed was to obtain carefully purified sulfite pulp from paper manufacturers, prepared largely from spruce, or mildly bleached soda pulp prepared from Jack pine wood instead of a deciduous wood, as the latter appeared to be less suitable for nitrating purposes. The preparation of this material by the wood pulp manufacturers would have required no important change in their process of manufacture. It was proposed to consider this as raw material equivalent to linters or hull shavings and to digest it simultaneously with linters and hull shavings under pressure for about 5 hrs. at 80 lbs. pressure, with approximately 2 per cent caustic soda solution. In the case of unbleached sulfite wood pulp we expected a loss on digestion of about 20 to 25 per cent and in the case of mildly bleached soda pulp a loss of only 10 to 15 per cent. The purified mixture of cotton and wood cellulose was to be dried, nitrated, and purified in regular factory equipment.

Following preliminary experiments which showed that the mixing of about 50 per cent linters with the wood pulp eliminated almost entirely the difficulties in connection with the manufacture of nitrocellulose and powder from the wood pulp, arrangements were made with the Government to manufacture 100,000 lbs. of 3 in. field gun powder from a mixture of 50 per cent sulfite wood pulp and 50 per cent linters. This mixture of sulfite wood pulp and linters was digested in regular factory equipment. The wood pulp was received in rolls about 24 in. in width and was simply cut longitudinally and charged into the kier together with the linters. The hull shavings were omitted in the experiment in order to obtain more accurate figures with reference to the yield of purified wood cellulose and the yield of nitrocellulose from the wood cellulose portion of the mixture. The mixture of linters and wood cellulose gave practically no difficulty in nitration; the acid recovery was normal and the yield of nitrocellulose was about 1.50 lbs. per pound of cellulose, as compared with about 1.52 lbs. for cotton. This indicated a yield of about 1.48 lbs. of nitrocellulose per pound of sulfite wood cellulose.

The nitrated material received the regular government purification treatment. The pulping of the nitrocellulose was found to require slightly shorter time due to the shorter wood fiber. The stability of the nitrocellulose was found to be very satisfactory indeed.

This material was made up into 3 in. field gun powder. In the manufacture of the nitrocellulose into powder it was found that the nitrocellulose dehydrated about 10 per cent more slowly, a difficulty which could be taken care of in the manufacture by the installation of additional dehydrating presses to make up for the loss in output. The nitrocellulose required slightly less solvent, due to the fact that the viscosity of the nitrocellulose from the wood pulp was lower than the viscosity of the nitrocotton.

The general conclusion reached from the manufacture of this 100,000 lbs. of nitrocellulose and of powder from the mixture of linters and wood pulp was that there were no serious difficulties which could not be overcome. The ballistic tests of the finished powder showed the product to be the equal of powder made from nitrocotton.

#### SHORTAGE OF COTTON

Up till about July 1918 it was not anticipated that any wood pulp would be needed, in spite of the enormous production of smokeless powder proposed for the balance of 1918 and for 1919, with the Old Hickory Plant at Nashville and the Nitro Plant beginning to come into operation. On the first of August,



however, the Department of Agriculture forecast a most serious drop in cotton yield owing to the drought. A conference was immediately called at Washington by the War Industries Board and it was pointed out that owing to the cotton shortage, instead of using a mixture of 70 per cent linters and 30 per cent hull shavings, it would be necessary to use a mixture of about 63 per cent linters, 23.5 per cent hull shavings and 13.5 per cent wood pulp, with the prospect that the amount of wood pulp would have to be very materially increased. The program of the War Department figured on the use of 150,000 tons of sulfite wood pulp for the ensuing year, or 500 tons per day, approximately 10 per cent of the output of sulfite wood pulp.

On the basis of the large-scale experiment above described it was recommended that preliminary to putting the entire manufacture of smokeless powder in this country on the cotton-wood pulp basis, we should immediately manufacture 1,000,000 lbs. of powder each, from a mixture of 50 per cent linters, 25 per cent hull shavings and 25 per cent sulfite wood pulp, and a mixture of 50 per cent linters, 25 per cent hull shavings and 25 per cent soda wood pulp.

At the time of the signing of the armistice, all preparations were made for the nitration of the material. The order for the manufacture of the powder was immediately cancelled, so that we were prevented from obtaining very valuable data as regards yields and manufacturing difficulties, and a satisfactory comparison of sulfite and soda wood pulp.

We believe that a very satisfactory powder could be made from either sulfite or soda wood pulp and that the advantages or disadvantages of these two materials as regards difficulty in manufacture, yields, etc., could be obtained only from large-scale manufacture such as we recommended. We also wished to place ourselves in a position so that we could use either soda or sulfite wood pulp in case of shortage of material.

With reference to the kind of wood, coniferous woods appeared to be more suitable because of longer fiber, so that in the case of sulfite pulp we were specifying largely spruce and, in the case of soda pulp, Jack pine. On the advice of government experts and private manufacturers we confined our attention almost wholly to the use of coniferous woods. Information from Germany indicated that they were probably using sulfite wood pulp and examination of several samples showed two to be entirely spruce and the third about 75 per cent spruce and 25 per cent pine. Other information stated that soda and sulfate wood pulps were being used by Germany and it is quite possible that all three kinds were finally used.

#### USE OF WOOD CELLULOSE FOR NITRATING IN PEACE TIME

In regard to the use of wood cellulose in peace times in the manufacture of smokeless powder, we have previously referred to the reluctance on the part of government officials to change from cotton to another form of cellulose, due to the serious question of chemical stability. In view of the enormous stocks of smokeless powder now in storage and the ample supply of cotton fiber for nitrating purposes, all smokeless powder for many years will continue to be made from cotton.

Relative to the use of wood pulp for industrial purposes other than explosives, there is a possible field. We believe that experiments in the past have in many cases failed because the investigator was satisfied to nitrate wood pulp as obtained from the paper trade and prepared for paper making purposes. We believe that better success can be obtained by giving the pulp an additional purification treatment so that the final cellulose will more closely approach cotton cellulose in chemical characteristics. This additional treatment naturally adds to the expense of the material.

Nitrocellulose prepared from wood pulp will be found to be of lower viscosity than cotton cellulose and might be of advantage for use in those industries where such a nitrocellulose is desired. As we have pointed out, however, there are serious

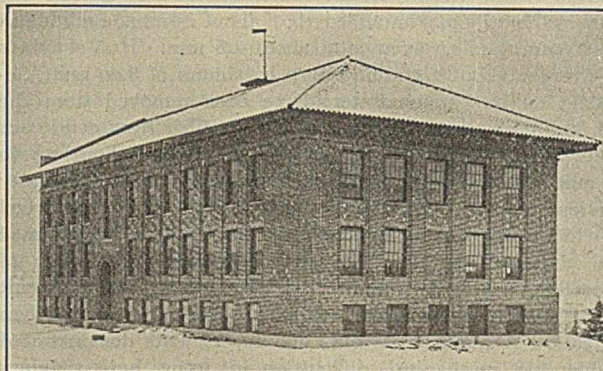
difficulties in connection with the nitration of wood pulp alone, due to the physical character of the material and shortness of the fibers. These difficulties would of course have to be solved in order to make nitrocellulose from wood cellulose a serious competitor of nitrocellulose from cotton.

#### NEW CHEMISTRY BUILDING AT MONTANA STATE COLLEGE

The formal dedication of the new chemistry building of the Montana State College, at Bozeman, took place on January 14, 1920, in connection with the inauguration of Alfred Atkinson as president of the college.

The dedicatory address on "Chemistry in Our Agriculture and Mechanic Arts Colleges" was given by Prof. William F. Coover, head of the department of chemistry at the Iowa State College. Formal dedication was made by Chancellor Elliott, of the University of Montana.

Prof. E. A. Duddy, of the State College, outlined the history of the department of chemistry, which in 25 years has become one of the leading departments of the college. Its work began in 1894 in a frame building, used also as a skating rink, with a heating system which required the use of overshoes and overcoats in the classroom. Frequently from two to four classes recited in the room at once under different instructors.



NEW CHEMISTRY BUILDING, MONTANA STATE COLLEGE

Other addresses were made by President Atkinson of the State College, Director F. B. Linfield of the Montana Experiment Station, Dr. W. F. Cogswell of the State Board of Health, Prof. Lester J. Harzell of the Montana State School of Mines, Prof. W. G. Bateman of Montana University, Prof. C. C. Todd of Washington State College, W. H. Andrews, chemist for the Three Forks Portland Cement Co., Lyndall P. Davidson, chemist for the Anaconda Copper Mines' reduction department, and A. P. Stark, senior in chemistry at the college.

The new building, completed and equipped at a cost of \$175,000, takes the place of one burned in 1916, and is believed to be one of the most up-to-date in the West. In many important features it is similar to the chemistry building at the Iowa State College.

It provides quarters for the department of chemistry and chemical engineering at the State College, research offices and laboratories for the Montana Experiment Station, water and food laboratories for the State Board of Health and an oil laboratory for the State Oil Commission.

The building has an efficient ventilating system for classrooms, offices, and laboratories, and a separate system for the laboratory hoods. The plumbing system provides pipe lines for hot and cold water, distilled water, drainage, compressed air, vacuum, and hydrogen sulfide. These pipe lines are placed in furred ceilings in each hallway and additional connections to all these lines can be made as future needs require. Alternate and direct current circuits are provided for each laboratory.



## WILLIAM H. NICHOLS MEDAL AWARD

A unique situation was presented at the Chemists' Club, New York City, on the evening of March 5, 1920, in that the Nichols Medal for the first time was awarded to a previous recipient. For his brilliant paper on "The Arrangement of Electrons and Atoms in the Molecule," published in the June 1919 issue of the *Journal of the American Chemical Society*, Dr. Irving Langmuir was awarded the medal, following a unanimous vote by the jury of the award. Dr. William H. Nichols, the founder of the medal, made the presentation in a thoughtful address and in his usual felicitous manner. In his address of acceptance, Dr. Langmuir presented a general outline of the octet theory of valence. An unexpected feature was added to the evening by the exhibition of a series of models constructed by Mr. Leffert Lefferts illustrating the structure of molecules of well-known compounds according to the theory.—[EDITOR.]

### MEDAL PRESENTATION

By William H. Nichols  
GENERAL CHEMICAL CO.,  
NEW YORK, N. Y.

The President of the SOCIETY regretfully finds himself unable to be with us to-night, and assume his privilege of presenting the SOCIETY'S medal to Dr. Langmuir. I have been asked to act in his place, and assume the honor with pleasure tinged with regret that our highest official should not be here to add dignity to the occasion. He has, however, written a most appreciative letter.

There are at least two points in which I can claim personal resemblance to Dr. Langmuir. We were both born in Brooklyn and we both, when we were rather young, attended public school, a course which I wish more American boys pursued. After that you know his history, where he was educated, the splendid work he did, and how, finally, after his graduation at Göttingen, he took up teaching at Stevens Institute. I am going to ask his permission and yours to take his career as a scientist for granted and to make use of an important decision of his, without in the slightest degree any criticism of it, to point a moral to what I consider one of the vital questions before the American people to-day. Dr. Langmuir began with teaching, and you can all imagine what kind of a teacher he must have made, and what kind of a teacher he would have

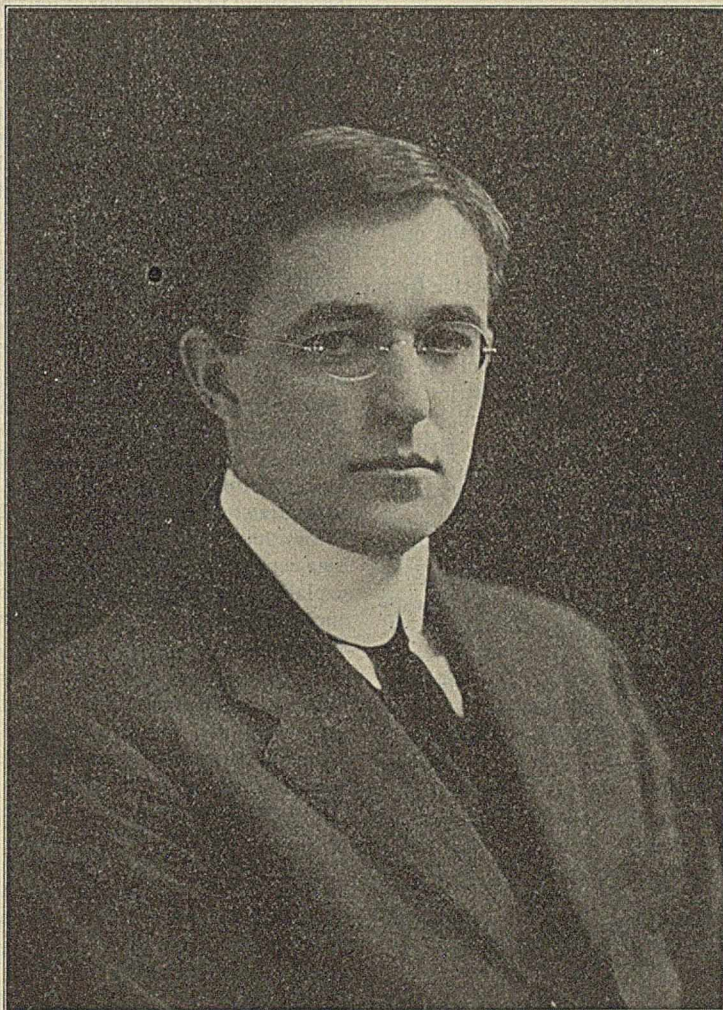
grown to be, and you can therefore understand something of the great loss to this country which his giving up the teaching profession must have produced. Fortunately, he made good in other directions, and I do not doubt that his contribution to the world has been greater in the field which he has chosen than it would have been had he continued as a teacher of young men. But it frequently happens that such is not the case. I have seen men, young, splendid men, lured away from professional work into the industries, and I have seen them get

smaller and smaller as time went on, and the output of the laboratory they abandoned dwindle as to quality because their places had been inadequately filled. Now you understand I am not saying these things in criticism of Dr. Langmuir, but I want to use the fact as a pretext to point out something which I consider of tremendous importance.

We have had in this SOCIETY for several years a committee known as the Committee on Co-operation between the Universities and the Industries. The chairman of that Committee is now the President of the SOCIETY, and he has put a great deal of intelligent work into the operations of the Committee. I have been and am one of its members. I have been studying the work of that Committee, realizing, as all of us must realize, the absolute necessity, in one way or another, of getting a closer relationship between the universities and the industries. The lack of coöperation may possibly be likened to the invention of the

sewing machine. You remember how difficult that was until some smart fellow happened to think that he could put the eye near the point of the needle. Then we had the sewing machine. Now this Committee has been working from below up with the idea of founding fellowships rather than providing for what the Fellows were to receive when they get to college. I have come to feel that it is more important to look out for the professor who is going to teach the Fellow, although fully appreciating the importance of the other point of view.

As you think of it thus, you see that the position of professor in the university or technical school (or for that matter, of teacher in all branches down to the primary school) has not in point of reward kept pace with the times; and the temptation which



IRVING LANGMUIR, WILLIAM H. NICHOLS MEDALIST, 1919



comes to a perfectly capable man to yield to the allurements of industry and leave his professorial work is strong, and his embracing the opportunity thus offered is by no means discreditable. Now the industries in holding out the lure cannot realize that they are "killing the goose that lays the golden egg." The case before us to-night is the antithesis of this, which shows that every rule has its exceptions. We must think of some way to make it to the interest of the best men in the profession to teach chemistry, or the science of chemistry will suffer here in comparison with other countries, and, as all of the industry of the country rests first upon the chemical industry and that in turn upon the chemist, this means that the effect will be shown in all of our industries if we do not find some way to induce the best men to remain. What is even more important is to induce the best men on graduating to prepare to fill the places which become vacant.

Now how can that be done? It is a problem of finance. Many of the colleges and universities have had, are having, or are going to have drives to make large and much needed additions to their endowment funds, and yet if they get all they ask there will not be enough in my judgment to carry out the idea which I have tried to express, of making the teaching of chemistry at our universities and colleges not only one of the most delightful employments, one of the most heartening and loving labors, which it is now, but also one of the most attractive from the point of view of the welfare of the incumbent himself as well as that of his family. This thought had led me to the conclusion that the relation between the universities and the industries must be shown in the readiness of the industries willingly, gladly, and from a sense, if you please, of enlightened selfishness, to see to it that the good chemical professors shall be adequately and even more than adequately provided for for the present, and also for the future by annuities for old age and insurance in case of death. I believe that this is the need, to take hold of the question from the professorial end, and not from that of the student alone. Of course, there will be an abundance of splendid students. How are they going to be met when they get to the college, and what kind of teaching are they to receive?

I think a plan is being devised, which is not yet advanced sufficiently to be made public, which will solve this question. I believe that before the year is out we shall see that this problem is in a fair way of solution, and that successful professors of chemistry will not only find themselves in one of the most delightful of all occupations, teaching, but that they will also be well paid. When that comes about we can all congratulate ourselves that our industries will continue to lead the world.

The gentleman who is to receive the SOCIETY'S medal to-night is so well known to every one of you that words from me are unnecessary. This is not his first appearance. He received the medal in 1915. This will be his second recipiency and in this his case is so far unique. Moreover, the paper for which the Committee has awarded the medal had the distinction of being read twice at the same meeting. When at Buffalo it created such a sensation that he was asked to read it again, which he did. I do not recall another instance of this. It shows that instead of the vote of the Committee it was the vote of the SOCIETY; in fact, a real referendum.

Dr. Langmuir, in carrying out the very agreeable duty which is assigned to me, I do so with the utmost pleasure and with the right hand of fellowship. One of your qualities that we like, almost better than any other, is that you are always prepared to give credit to those who have been assisting you and to share with them the honor. It adds very much to my pleasure in presenting this medal to think that there are others who aided and assisted you in your splendid work, and that you generously make that claim in their behalf.

## THE STRUCTURE OF ATOMS AND ITS BEARING ON CHEMICAL VALENCE

By Irving Langmuir

GENERAL ELECTRIC CO., SCHENECTADY, NEW YORK

According to the well-established Rutherford-Bohr theory, all the positive electricity in an atom is concentrated in a *nucleus* at its center. The dimensions of this nucleus are negligibly small compared with those of the rest of the atom, its diameter being of the order of 0.00001 of that of the atom. The charge on the nucleus is an integral multiple of the charge of an electron but of course opposite in sign. The remainder of the atom consists of electrons arranged in space about the nucleus, the normal number of such electrons (called the *atomic number*) being equal to the number of unit positive charges on the nucleus, so that the atom as a whole is electrically neutral. If the number of electrons in the atom exceeds the atomic number we have a negatively charged atom or ion while in the reverse case a positively charged atom or ion results. The atomic number of any element has been found to be equal to the ordinal number of the element in the periodic table. Thus hydrogen has the atomic number one, helium 2, lithium 3, carbon 6, neon 10, chlorine 17, nickel 28, silver 47, cerium 58, tungsten 74, radium 88, and uranium 92. The atomic numbers can be determined experimentally from the X-ray spectrum so that we are not dependent upon the periodic table for our knowledge of these numbers.

Bohr, Sommerfeld, and others have developed an extensive and very successful theory of spectra upon the hypothesis that the electrons in atoms are in rapid rotation in plane orbits about the nucleus in much the same way as the planets revolve around the sun. Stark, Parson, and G. N. Lewis on the other hand, starting from chemical evidence, have assumed that the electrons are stationary in position. It should be noted that Bohr's theory has had its greatest success when applied to atoms or ions containing only one electron and that it seems incapable of explaining the chemical or ordinary physical properties of even such simple elements as lithium, carbon, or neon.

The two theories can, however, be reconciled if we consider that the electrons, as a result of forces which they exert on one another, rotate about certain definite positions in the atom which are distributed symmetrically in three dimensions. Thus for atoms containing only a single electron the chemical theory is in agreement with Bohr's theory. But for an atom such as neon the eight electrons in the outside layer would revolve around positions which are located about the nucleus in the same way that the eight corners of a cube are arranged about the center of the cube. This structure is not inconsistent with those parts of Bohr's theory which have received experimental confirmation. In fact, Born and Landé,<sup>1</sup> starting from Bohr's theory and without knowledge of Lewis' work, arrived at exactly this conception of the structure of atoms (*i. e.*, the cubic atom) from a study of the compressibility of the salts of the alkali metals.

The atomic numbers and the properties of the inert gases furnish us with a clue to the arrangement of the electrons within atoms. The low boiling point, the high ionizing potential, the chemical inertness, etc., of helium prove that the arrangement of the electrons in the helium atom is more stable than that in any other atom. Since this atom contains two electrons we must conclude that a pair of electrons in the presence of a nucleus represents a very stable group. It is reasonable that with elements of higher atomic number there should be an even greater tendency for this stable pair of electrons to form about the nucleus. There are two sets of facts which furnish conclusive experimental evidence that this stable pair exists in all atoms above helium.

In the first place, the properties of lithium, beryllium, etc.,

<sup>1</sup> *Verh. d. phys. Ges.*, 20 (1918), 210.



show that in these elements also the first two electrons are held firmly while the remainder are easily detached. Thus, lithium readily forms a univalent positive ion by the detachment of one of the three electrons in its neutral atom. The divalence and other properties of beryllium prove that there is little or no tendency for a second stable pair of electrons to surround the first pair.

In the second place, the absence of irregularities in the observed K and L series of the X-ray spectra of the various elements proves that there are no sudden changes in the number of electrons in the innermost layers of electrons about the nucleus. From these two sets of facts, as well as from other evidence, we may take it as a fundamental principle that the arrangement of the inner electrons undergoes no change as we pass from elements of smaller to those of higher atomic number.

The properties of neon indicate that its atoms are more stable than those of any other element except helium. Since the atomic number is 10, and the first 2 electrons form a stable pair about the nucleus as in the helium atom, it follows directly that the other eight electrons arrange themselves in a second layer or shell possessing a very high stability. If these 8 electrons revolved about the nucleus in a single circular orbit or ring, as would be suggested by Bohr's theory, there is no apparent reason why there should be any very great difference in stability between rings having 7, 8, or 9 electrons. On the other hand, we readily see that the geometrical symmetry of the arrangement of the 8 electrons at (or rotating about) the 8 corners of a cube would not only account for a high degree of stability but for the fact that an arrangement of 7 or 9 electrons would have no such stability. Chemical considerations and Born and Landé's work on compressibility also lead us to this spatial arrangement of the electrons. We shall refer to the stable group of 8 electrons by the term *octet*. From the principles already enunciated it is clear that in the atoms of all the elements above neon the inner electrons are arranged in the same way as those of neon.

From the atomic numbers of the inert gases we are thus able to determine the number of electrons in the various layers or shells of electrons which exist in the atoms. The results are summarized in Table I.

TABLE I—DISTRIBUTION OF ELECTRONS IN THE VARIOUS SHELLS

Shell	Number of Electrons	Inert Gas Corresponding to Completed Layer
1st shell.....	$2 = 2 \times 1^2$	He 2
2nd shell, 1st layer.....	$8 = 2 \times 2^2$	Ne 10
2nd shell, 2nd layer.....	$8 = 2 \times 2^2$	Ar 18
3rd shell, 1st layer.....	$18 = 2 \times 3^2$	Kr 36
3rd shell, 2nd layer.....	$18 = 2 \times 3^2$	Xe 54
4th shell, 1st layer.....	$32 = 2 \times 4^2$	Nt 86

Thus the xenon atom with an atomic number 54 contains 54 electrons arranged as follows: Close to the nucleus are two electrons which constitute the first shell. This is surrounded by the second shell which contains two "layers" of 8 electrons each. The third shell, which in the xenon atom is the outside shell, contains 18 electrons.

An examination of the numbers of electrons in the layers (Table I, 2nd column) shows that they bear a simple mathematical relation to each other, namely, that they are proportional to the squares of the successive integers 1, 2, 3, and 4. This is to be looked upon as perhaps the most fundamental fact underlying the periodic arrangement of the elements. It is significant that in Bohr's theory these same numbers, 1, 4, 9, 16, etc., play a prominent part. Thus the energies of the electron in the various "stationary states" are proportional to  $1, 1/4, 1/9, 1/16$ , etc., and the diameters of the various possible orbits in Bohr's theory are proportional to 1, 4, 9, 16, etc. In Bohr's theory the various stationary states correspond to different number of quanta (Planck's quantum theory), the innermost orbit corresponding to one quantum, the second orbit to two quanta, etc. We should thus consider (Table I) that the

electrons in the 1st shell are monoquantic, those in both layers of the 2nd shell are diquantic, etc. It is interesting that Born and Landé from quite other evidence have concluded that the outermost electrons of the chlorine atom (2nd layer of the 2nd shell) are diquantic instead of triquantic as was at first assumed.

The foregoing theory of the arrangement of electrons in atoms explains the general features of the entire periodic system of the elements and is particularly successful in accounting for the position and the properties of the so-called 8th group and the rare earth elements. It also serves to correlate the magnetic properties of the elements.

Let us now consider the bearing of this theory of atomic structure on the phenomena of chemical valence. The outstanding feature of the theory is that there are certain groups of electrons, such as the pair in the first shell and the octet in the second, that have a remarkable stability. Those atoms in which all the electrons form parts of such stable groups (*viz.*, the inert gases) will have no tendency to change the arrangement of their electrons and will thus not undergo chemical change. Suppose, however, we bring together an atom of fluorine ( $N = 9$ )<sup>1</sup> and an atom of sodium ( $N = 11$ ). Ten electrons are needed for the stable pair in the first shell and the octet in the second shell, as in the neon atom. The sodium atom has one more electron than is needed to give this stable structure while the fluorine atom has one electron too few. It is obvious then that the extra electron of the sodium atom should pass over *completely* to the fluorine atom. This leaves the sodium atom with a single positive charge while the fluorine becomes negatively charged. If the two charged atoms or ions<sup>2</sup> were alone in space they would be drawn together by the electrostatic force and would move as a unit and thus constitute a molecule. However, if other sodium and fluorine ions are brought into contact with the "molecule" they will be attracted as well as the first one was. There will result (at not too high temperature) a space lattice consisting of alternate positive and negative ions and the "molecule" of sodium fluoride will have disappeared. Now this is just the structure which we find experimentally for sodium fluoride by Bragg's method of X-ray crystal analysis. There are no bonds linking individual pairs of atoms together. The salt is an electrolytic conductor only in so far as its ions are free to move. In the molten condition or when dissolved in water, therefore, it becomes a good conductor.

The case of magnesium ( $N = 12$ ) and oxygen ( $N = 8$ ) is similar except that two electrons are transferred from the magnesium to the oxygen atom. The resulting ions have their electrons arranged exactly like those of the neon atoms and the ions of sodium and fluorine. Therefore, the crystalline form of magnesium oxide and sodium fluoride should be identical, and this prediction of the theory has been confirmed experimentally by Dr. A. W. Hull by the X-ray method. Because of the much greater forces acting between the ions as a result of the double charges, the stability of the magnesium oxide is much higher than that of the sodium fluoride. This is manifested by the high melting point, low conductivity, low solubility, and hardness of magnesium oxide.

Phosphorus ( $N = 15$ ) and sulfur ( $N = 16$ ) have, respectively, 5 and 6 electrons more than neon, and are thus capable of giving up these numbers of electrons. If these elements are brought into contact with an excess of fluorine (which because of its

<sup>1</sup> We will denote the atomic number of an element by  $N$ .

<sup>2</sup> It is convenient and it has been customary with many physicists to speak of a charged atom or molecule as an ion, irrespective of whether or not the particle is able to wander under the influence of an electric field. The writer has used the term in this way in his recent publications. This practice is very distasteful to many physical chemists and is apt to be misunderstood by them. Nevertheless, it seems to me probable, especially in view of the recent work of Milner and Ghosh, that it will be desirable to abandon the physical chemists' definition of the ion and to apply it to all charged atoms or molecules. The ion which wanders may then be referred to as a "free ion."



proximity to neon has a particularly strong tendency to take electrons) all the extra electrons pass to fluorine atoms. Thus a sulfur atom will supply electrons to 6 fluorine atoms and will form the compound  $\text{SF}_6$ . The force acting between the fluorine ions and the central sulfur ion is still electrostatic in nature but it must be nearly 6 times greater than the force between sodium and fluorine ions. Furthermore, the 6 fluorine ions would surround the sulfur ion so that there would be little stray field of force. Therefore, we should not expect sulfur fluoride to be salt-like in character but to consist of very stable molecules having weak external fields of force and, therefore, readily existing in the form of a gas. As a matter of fact this extraordinary substance has these properties developed to such a degree that it is an odorless and tasteless gas with a boiling point of  $-62^\circ$ . Phosphorus pentafluoride, as would be expected from its less symmetrical structure, is a gas having greater chemical activity.

The fluosilicate ion  $\text{SiF}_6^{--}$  has a structure exactly like that of the sulfur fluoride molecule since the number and arrangement of the electrons are the same. This is clear if we consider that the atomic number of silicon is 14 while that of sulfur is 16. Thus if we should replace the nucleus of the sulfur atom in a molecule of sulfur fluoride by the nucleus of a silicon atom, without disturbing any of the surrounding electrons, we would have removed two positive charges and would obtain a negative ion with two negative charges of the formula  $\text{SiF}_6^{--}$ . In the presence of potassium ions we would then have the familiar salt potassium fluosilicate. The theory is thus capable of explaining typical complex salts. In fact, it is applicable to the whole field of inorganic compounds covered by the work of Werner, and helps to simplify the theory of such compounds. There is no time, however, to go into this subject.

The simple theory of atomic structure which we have discussed thus far explains perfectly what has usually been called "the maximum positive and negative valence." The maximum positive valence represents the number of electrons which the atom possesses in excess of the number needed to form one of the particularly stable configurations of electrons. On the other hand, the maximum negative valence is the number of electrons which the atom must take up in order to reach one of these stable configurations.

For example, magnesium has a positive valence of two, since its atomic number is 12 while that of neon is 10. Sulfur has a positive valence of 6 since it has 6 electrons more than neon; but it has a negative valence of two because it must take up two more electrons before it can assume a form like that of the argon atom.

It is clear, however, that this theory of valence is not yet complete.<sup>1</sup> It is not applicable to those cases where we have usually taken valences of 4 for sulfur, or 3 and 5 for chlorine, etc. But more especially it does not explain the structure of organic compounds and such substances as  $\text{H}_2$ ,  $\text{Cl}_2$ ,  $\text{O}_2$ ,  $\text{N}_2\text{H}_4$ ,  $\text{PCl}_3$ , etc.

J. J. Thomson, Stark, Bohr, and others had suggested that pairs of electrons held in common by two adjacent atoms may function in some cases as chemical bonds between the atoms, but this idea had not been combined with the conception of the stable groups of electrons or octets. G. N. Lewis, in an important paper in 1916, advanced the idea that the stable configurations of electrons in atom could share pairs of electrons with each other and he identified these pairs of electrons with the chemical bond of organic chemistry. This work of Lewis has been the basis and the inspiration of my work on valence and atomic structure.

As a result of the sharing of electrons between octets, the number of octets that can be formed from a given number of electrons is increased. For example, two fluorine atoms, each having seven electrons in its outside shell, would not be able to

<sup>1</sup> The theories of Kossel, Lacomblé, Teudt, etc., which have recently been proposed in Germany, have not advanced beyond this point and are therefore very unsatisfactory as a general theory of valence.

form octets at all except by sharing electrons. By sharing a single pair of electrons, however, two octets can be formed since two octets holding a pair in common require only 14 electrons. This is clear if we consider two cubes with electrons at each of the eight corners. When the cubes are placed so that an edge of one is in contact with an edge of the other a single pair of electrons at the ends of the common edge will take the place of four electrons in the original cubes. For each pair of electrons held in common between two octets there is a decrease of two in the total number of electrons needed to form the octets.

Let  $e$  represent the number of electrons in the outside shell of the atoms that combine to form a molecule. Let  $n$  be the number of octets that are formed from these  $e$  electrons, and let  $p$  be the number of pairs of electrons which the octets share with one another. Since every pair of electrons thus shared reduces by two the number of electrons required to form the molecule it follows that  $e = 8n - 2p$  or

$$p = \frac{1}{2}(8n - e).$$

This simple equation tells us in each case how many pairs of electrons or chemical bonds must exist in any given molecule between the octets formed. Hydrogen nuclei, however, may attach themselves to pairs of electrons in the octets which are not already shared. For example, in the formation of hydrogen fluoride from a hydrogen atom and a fluorine atom there are 8 electrons in the shells ( $e = 8$ ). We place  $n = 1$  in the above equation and find  $p = 0$ . In other words, the fluorine atoms do not share electrons with each other. The hydrogen nucleus having given up its electron to the fluorine atom attaches itself to one of the pairs of electrons of the fluorine octet, and thus forms a molecule having a relatively weak external field of force. As a result, hydrogen fluoride is a liquid of low boiling point instead of being salt-like in character.

The equation given above is applicable to all types of compounds. For example, if we apply it to substances such as sodium fluoride, sulfur fluoride, or potassium fluosilicate, which were previously considered, we find in each case  $p = 0$ . In other words, there are no pairs of electrons holding the atoms of these compounds together. On the other hand if we consider the compound  $\text{N}_2\text{H}_4$ , we find  $p = 1$ . Since there are only two octets the pair of electrons must be between the two nitrogen atoms while the hydrogen nuclei attach themselves to pairs of electrons of the nitrogen octets. It can be readily shown that this simple theory is in fact identical with the accepted valence theory of organic chemistry and leads to the same structural formulas as the ordinary theory in all those cases where we can take the valence of nitrogen to be 3, oxygen and sulfur 2, chlorine and hydrogen one. In other cases such as those where quinquivalent nitrogen has been assumed, the new theory gives results different from the old but in each case in better agreement with the facts.

The theory indicates a series of new relationships between certain types of substances which I have termed *isosteric* substances. For example, it indicates that the molecules of carbon dioxide and nitrous oxide should have nearly identical structures and this is borne out by the extraordinary similarity in the physical properties of these gases. Nitrogen and carbon monoxide constitute another pair of gases which are similarly related. The same theory also points out a number of previously unsuspected cases of similarity of crystalline form (isomorphism).

It is clear that in the past the term valence has been used to cover what we may now recognize as three different types of valence, as follows:

1—Positive valence: the number of electrons an atom can give up.

2—Negative valence: the number of electrons an atom can take up.

3—Covalence: the number of pairs of electrons which an atom can share with its neighbors.

It is recommended that only for valences of the covalence



type should definite bonds be indicated in chemical formulas. One of the particular advantages of the present theory is that it becomes easy to distinguish between covalence and the other types and thus to predict with certainty in what way electrolytic dissociation will occur if at all.

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## FOREIGN INDUSTRIAL NEWS

By A. McMILLAN, 24 Westend Park St., Glasgow, Scotland

### SYNTHETIC AMMONIA

According to *Comptes Rendus*, Professor D'Arsonval has made an important communication in the name of M. Georges Claude to the Academy of Science, Paris. M. Claude recently showed that it was not only possible, but very easy to produce and turn to industrial uses, pressures of 1,000 atmospheres and more. He has now succeeded in applying these pressures in the synthetic production of ammonia. Hitherto this has been done only in Germany, but M. Claude has surpassed the German scientists in that under the new conditions the combination of hydrogen and nitrogen takes place with such intensity that a very small apparatus is capable of a considerable output. He proposes shortly to show to the members of the Academy a tiny apparatus capable of producing daily 200 l. (44 gal.) of liquid ammonia. Whereas Haber could get only 0.33 g. of ammonia per catalytic gram, M. Claude gets 10 g.

### ISOTOPES

An important communication, says the *Chemical News*, on the spectra of isotopic lead and thallium was brought before the Royal Society, London, by T. R. Merton. Interferometer measurements of the principal line in the spectra of ordinary lead and in lead from pitchblende showed that, in the latter case, the line was less refrangible by 0.0050Å., whereas the lead from Ceylon thorite was more refrangible by 0.0022Å. Thallium, which occurs in pitchblende in very small proportion, was examined by alloying it with pure cadmium and was found to give a line more refrangible than that of ordinary thallium by 0.0055Å.

### MANGANESE IN BRAZIL

According to a report from the Bank (British) of South America, stocks of manganese ore continue to accumulate and as there is practically no demand for export, there appears to be little immediate likelihood of a demand occurring under peace conditions. The only solution likely to relieve this situation would seem to be the construction of suitable electric smelting apparatus for the reduction of the ore in Brazil. The stocks in hand at Rio de Janeiro on November 12 amounted to 256,279 tons.

### ARTIFICIAL HONEY

During the war large quantities of artificial honey were used in Germany. The products of different firms did not show much uniformity, a thin fluid being much in evidence. According to an article in *Schweiz. Chem. Zeit.*, 1919, for the production of the thick variety, the water content and the degree of inversion of the cane sugar are matters of great importance. The water content must not exceed 22 per cent and the inversion must be complete to within 5 per cent. The inversion proceeds best when at least 0.1 per cent of formic acid is present. The strongly acid taste of the product is removed by neutralizing with sodium carbonate. On economic grounds, thick artificial honey is an indispensable product in Germany, and it is recommended that legislation be introduced to prevent confusion between natural and artificial honey.

### METAL-MELTING FURNACE

A feature of the Alldays-Charlier metal-melting furnace is that no crucible is employed. It consists of a heavy steel-plate cylinder with cast-iron end plates, carried on two cast-iron standards provided with ball bearings. The cylinder is lined with refractory firebricks or cupola bricks, and the burner is arranged to play through one of the trunnions. The firing is by either oil or gas, and pouring is effected by tilting. As regards melting losses two sets of tests gave the following results: One set consisted of two tests made on gun-metal in the foundry of a large French factory. In one case the charge consisted of 541 lbs. of old copper, 99 lbs. of tin, and 20 lbs. of zinc, and the weight of metal poured was 645 lbs.; in the other the charge was 574 lbs. of old copper, 66 lbs. of tin and 20 lbs. zinc, and the poured weight 647 lbs. The melting loss was thus round about 2 per cent in both cases. Less favorable results were obtained by a foundry in the Haute Saône which specializes in "Parisien bronze." Old metal containing 65 per cent of copper and 35 per cent of zinc was melted; in one test the weight of the pour was 418 lbs. against 440 lbs. of charge, the melting loss being 5 per cent; while in the other 623 lbs. were poured from a charge of 660 lbs., the loss being 5.6 per cent. When the same metal was melted in an ordinary crucible furnace of the natural draught type the melting loss on a charge of 220 lbs. was 11 lbs. or 5 per cent. It is claimed, however, for the Charlier furnace that the elimination of expensive crucibles effects considerable economy and that its large production means reduced labor charges.

### THE GREAT WAR EXHIBITION

According to *Engineering*, the Crystal Palace, London, will reopen in May next with the Great War Exhibition in which will be included a section relating to general and electrical engineering and another devoted to the petroleum industry. It is understood that the whole of the available space on the main floor of the building has been taken by the government for naval, military, and aircraft exhibits in connection with the war, but that some 50,000 sq. ft. of space are available for manufacturers wishing to exhibit in the engineering and electrical sections. The oil section will be accommodated in two large exhibition halls in the Palace grounds facing the terraces, one hall to be devoted to stand exhibits, and the other to machinery in motion. The special features of the oil section will be oil field supplies and general equipment, transport and distributing facilities, oil systems of power and domestic use, Diesel and other oil engines, exhibits by distributing and importing companies and government exhibits. All particulars of the exhibition, which will remain open till October, can be obtained from the General Manager, Crystal Palace, London.

### ZINC-MANGANESE ALLOY

Among the new German alloys is one of zinc with not more than 4 per cent of manganese which is claimed to be about twice as hard as zinc itself and to have 50 per cent greater resistance to impact, while unlike other zinc it can be forged when warm as well as stamped and rolled. The alloy can serve as a substitute for copper, bronze, and brass.



### THE MANUFACTURE OF GLUE

While the chemistry of gelatin, says *Nature*, 2623, 611, has been investigated with much care, less attention has been given to that of glue. Both gelatin and glue are hydrolytic products of the collagen present in hides, but they represent different phases of the hydrolysis, and the details of the manufacture of glue have largely been kept secret. Crucial points are, first, the stage at which the process of hydrolysis must be stopped and the degree of concentration necessary in order to obtain a glue solution which will "set" to a jelly, and, next, the method of drying this jelly into a finished glue. A low temperature has been considered necessary for successful drying, and also for avoiding bacterial action during the process. Hence the making of glue in warm countries, such as India, has hitherto not been found practicable. A paper by Mr. K. C. Srinivasan, of the Department of Industries, Madras, on the "Manufacture of Glue in the Tropics," describes how the foregoing points have been investigated by the Department and the difficulties overcome. It is claimed that by a study of the chemical principles involved, and by laboratory and factory experiments, the details of manufacture have been successfully adapted to the climatic conditions prevailing even in the hottest parts of India.

### SOURCE OF NITRATE OF AMMONIA

Prof. Garelli, of Turin, has lately been carrying out experiments with a view to the extraction of nitrate of ammonia from surplus stock of explosives. According to an article in *La Nature*, the explosive is allowed to stand with a fixed quantity of water in special vessels. A concentrated solution of nitrate of ammonia is thus formed and is separated by decantation. Powdered peat is then added to this solution and the product obtained after mixing and drying is a species of manure called "nitric peat." This material, which has the appearance of a blackish powder, gave the following analysis: Water, 17.8 per cent; ash, 18.8 per cent; nitrate of ammonia, 42.8 per cent; organic matter 20.6 per cent. Tests for ascertaining its value as a fertilizer have been carried out and the results show that its action is nearly equal to that of nitrate of soda.

### ANHINGA

Anhinga, according to *Engineer*, 129, 39, is a Brazilian plant which is chiefly found along the banks of the rivers of Para, whose slow current allows a soft mud bed for its roots. The plant constitutes a raw material from which cellulose for the manufacture of linen paper is obtained, and may also under chemical treatment be transformed into artificial cotton fiber. A new process for dissolving the fibers has been discovered which transforms them into very fine white fibers. These resemble cotton fiber of high quality but are considered to be superior to it inasmuch as the lines of the anhinga fiber are straight and parallel. It is said that the state of Para is capable of producing 100,000 tons of fiber for export annually and a mill already in operation has a daily output of about 10 cwt.

### FUEL OILS IN SWEDEN

According to a report from Sweden, the Swedish Board of Trade has issued a report on the regulation of the market for imported fuel oils, and sulfite alcohol used as a motor fuel. The Board points out that the conditions for the distribution of liquid fuel are at present unfavorable, the chief inconvenience being that the Swedish fuel market is dependent on a small number of oil trusts. To remedy this evil, the Board recommends the acquisition by Sweden of foreign oil wells, the construction of a fleet of tank steamers of her own and the erection of oil refineries. For the transport of oil to Sweden two boats with an aggregate dead weight capacity of 12,000 tons are considered sufficient. The total cost of the two vessels is estimated at about Kr. 10,800,000.

### TIN SOLDER SUBSTITUTES

In view of a continued shortage of tin in Germany, an article in the *Metallbörse* discusses the possibility of discovering suitable solder substitutes. During the war a considerable reduction in the proportion of tin in soft solders was permitted and such tin as was used was not taken from fresh ingots but from waste, ashes, and scrap. The proportion of tin varied between 25 and 40 per cent; that of lead was considerably increased and careless manufacturers often produced harmful results. Cadmium alloys, which, as a rule, are used only for very low smelted solders, came greatly into favor but such substitutes as this have no particular durability. Cadmium solder may be suitable in theory but is generally useless for practical purposes. The employment of solder substitutes in the gas meter industry has proved a failure, resulting in cracking on cooling, splitting on the slightest tension, and leaking in the seams of plates, etc. For dry gasometers antimony solder may be used with advantage but in other cases the employment of substitutes for tin has led to endless difficulties.

### SULFUR IN BENZENE

According to a report in *Chemical Trade Journal*, 66 (1920), 200, the Aeronautical Inspection Department undertook investigations as to the effect of sulfur in benzene on various metals, at the request of the National Council of Commercial Motor Users and the Petroleum Executive. Four samples of benzene were supplied stated to contain 0.1, 0.2, 0.4, and 0.8 per cent of sulfur, respectively. Chemical investigation showed that sulfur was nearly all present as carbon disulfide. The metals used were copper, brass, phosphor bronze, aluminum, and cast iron. After prolonged contact with benzene containing 0.8 per cent sulfur, a slight discoloration of the surface took place in copper and brass. In the presence of moist air also, slight action appears to take place but no measurable difference occurred in the weights of the metal before and after the action. The iron and steel rusted in the moist air but there was no evidence of formation of any oxidation products of sulfur. A sample of the deposit found in carburetors of aéro-engines consisted of a grayish white powder decidedly alkaline to litmus and containing metallic particles. The particles consisted of aluminum with traces of copper, zinc, and iron. The remainder of the deposit was sodium silicate with traces of carbonate and sulfide, the latter probably impurities present in the sodium silicate used for doping the carburetors. There is, therefore, no evidence that the sulfur in benzene has any corrosive effect on carburetors.

### ARTIFICIAL WOOL

According to the *Chemical Trade Journal* which quotes from a contemporary, considerable interest is being shown in Germany in a patent recently taken out on the manufacture of artificial wool. In principle the process consists of compressing wool scraps which cannot be otherwise utilized, shreds, ends, short fibers, and washing waste, and soaking them in a viscous solution of cellulose or one of its compounds with a small percentage of glue. The product thus obtained is cut into thin sheets or strips which can be treated after the manner of paper yarn. The Germans claim that the yarn thus obtained possesses all the properties of real wool. It is made waterproof by treating it in the process of manufacture with compounds of chromium and later with formaldehyde and tannin after which the product will resist the action of boiling water. The addition of glycerin gives it additional flexibility. The process is now being modified by the mixture with the woolen waste of a certain percentage of paper pulp, and treatment of the product with sulfuric acid and zinc chloride. The addition of various other compounds gives it the necessary flexibility and makes it waterproof.



## NOTES AND CORRESPONDENCE

### BRITISH NITROGEN PRODUCTS COMMITTEE REPORT

The final report of the Nitrogen Products Committee, Munitions Inventions Department, Ministry of Munitions of War of Great Britain, has been issued. It is a voluminous document of 323 pages with charts and tables covering several years' careful study of the nitrogen problem. It contains a summary of the pre-war position of the world's nitrogen industry, of the pre-war nitrogen industry of the United Kingdom; a discussion of power production and power requirements for nitrogen fixation; a study of costs and other economic features of established processes, and a discussion of partly developed processes, recent inventions, and research undertaken by the Nitrogen Products Committee and others. It goes into the effect of the war on the nitrogen industries and on future problems; depicts the post-war aspects of the nitrogen problem, and the post-war requirements of the United Kingdom. It also discusses the nitrogen resources of the United Kingdom and the establishment of nitrogen fixation in other parts of the British Empire. It closes with a summary of conclusions and general observations on nitrogen in agriculture; the world's demand and production; relative costs of synthetic and non-synthetic processes; the international situation after the war; and ends with a series of recommendations based essentially on provisions for the national safety of Great Britain, but also carefully considered from an industrial standpoint. Especial credit for this report is known to be due to Dr. J. A. Harker, director of the Nitrogen Research Section.

It is understood that these recommendations were made during the war period when it was advisable for England to develop every possible means for procuring nitrogen, exactly as was the case in the United States. The recommendations were confirmed by the Committee after the war was completed, as it seemed still desirable to have this information. Up to the present time, however, while the synthetic method is being developed, nothing has been done towards the development of a cyanamide plant in England, and it seems improbable that a cyanamide plant will be built in that country.

The recommendations of the Committee, under the chairmanship of H. E. F. Goold-Adams, are as follows:

#### RECOMMENDATIONS

In making the recommendations set out below, the Committee has been guided by the following salient facts:

(a) The sources of supply of combined nitrogen in the United Kingdom must be considerably increased if the existing and prospective home demands are to be met and the pre-war scale of exportation maintained.

(b) The existing sources of supply of combined nitrogen in the United Kingdom proved wholly inadequate for meeting the war demands.

(c) The risks and costs of importation during the war are very serious.

(d) Combined nitrogen (as cyanamide or ammonium sulfate) can be obtained by synthetic processes at a cost, at the factory, which is less than half the market price of combined nitrogen from other sources, pre-war conditions being taken as the basis in each case.

(e) Concentrated nitric acid can be made by synthetic processes for about half the pre-war cost by the standard retort process through Chile nitrate.

(f) The ammonia oxidation process provides a means whereby, during a state of war, the importation of Chile nitrate would be rendered unnecessary.

(g) The world's demand for combined nitrogen appears to double every ten years. The increased production during the war has not been more than the normal rate of increase during peace.

(h) The actual consumption of combined nitrogen for agriculture in the United Kingdom has practically doubled during the war, and there is certain to be a further increase.

(i) No very large increase in the output of by-product

ammonia in Great Britain in the immediate future seems probable.

The following measures are recommended by the Committee as a minimum provision for safeguarding the future and for meeting a portion of the growing home demand for various nitrogen products.

#### CALCIUM CYANAMIDE PROCESS

The Committee recommends that this process should be established in Great Britain without delay, by private enterprise (supported, if necessary, by the government) or as a public work on a scale of manufacture sufficient to give an output of about 60,000 tons of cyanamide per annum, equivalent on the basis of combined nitrogen to about one-eighth of the present home production of ammonium sulfate. The necessary electrical energy should be obtained from water power in Scotland or from a large steam-power station, such as the capital power stations proposed by the Board of Trade Committee on Electric Power Supply.

On the basis of pre-war prices plus 50 per cent, the approximate capital outlay at a water-power site would be £1,680,000, inclusive of the capital cost of the hydro-electric development, the latter being taken at £40 per e. h. p. (£53.6 per kw.) of maximum demand. If the factory were erected at the site of a large steam-power station, the approximate outlay would be £800,000, inclusive of the proportionate cost of the power plant. The chemical sections for either site would cost about £445,000 of the above totals.

The approximate cost, at the factory, of raw cyanamide (19.5 per cent N) would be as follows:

SOURCE OF POWER	Price of Electrical Energy per Kw. Year	FACTORY COSTS PER METRIC TON Raw Product (19.5 Per cent N)	Combined Nitrogen as Raw Cyanamide
	£	£	£
Water.....	4	6.4	32.8
Steam (with coal at 11s. 3d. per ton).....	5.6	7.0	36.0

The factory costs include interest on the capital outlay for the power section of the plant, but not on the chemical section, and are exclusive of any royalties.

The Committee suggests that it might prove desirable to arrange for a certain proportion of the calcium carbide produced to be utilized as such instead of converting it into cyanamide.

Plans and working drawings of the chemical sections can be obtained for a reasonable consideration. Moreover, as the result of the action taken upon the Committee's Interim Recommendations, considerable progress was made with the plans and specifications for a suitable steam-power station designed as the nucleus of a possible capital power station. These plans and specifications are available.

The Committee is authorized to state that the Board of Agriculture is of opinion that the establishment of this process would be beneficial.

#### SYNTHETIC AMMONIA (HABER) PROCESS

The Committee recommends that the synthetic ammonia process should be established forthwith on a commercial unit scale and extended as rapidly as possible, as a post-war measure, up to a minimum manufacturing scale of 10,000 tons of ammonia (equivalent to 40,000 tons of ammonium sulfate) per annum. The Billingham factory, begun by the government early in 1918 for the manufacture of synthetic ammonia and ammonium nitrate, should be completed and utilized for the purpose if such a course is practicable.

The evidence on the question of capital cost is somewhat uncertain, but the outlay for a factory of the capacity indicated,



on the basis of pre-war prices plus 50 per cent, would probably amount to £600,000 if ammonia were the end-product, or to £780,000 if the factory were laid out for the manufacture of synthetic ammonium sulfate.

#### AMMONIA OXIDATION PROCESS

Early in the war, a full-scale installation was erected on the Thames by private enterprise, supported by the government, the older method of oxidation being utilized. A long series of experiments carried out at the Research Laboratory of the Department resulted in the design of a simple and modified plant which has been installed on a trial scale at several works in the country. The designs of the ammonia oxidation section of the government ammonium nitrate factory referred to above were based upon the results obtained in these research and trial operations.

The Committee recommends that an ammonia oxidation plant should be completed with the synthetic ammonia factory already referred to on a scale sufficient to produce about 10,000 tons of strong nitric acid (95 per cent) or its equivalent in nitrates, per annum; and that the plant should be designed to utilize either synthetic or by-product ammonia as a raw material.

Upon the basis of pre-war prices plus 50 per cent, the estimated capital outlay for an installation having a capacity of 10,000 tons of strong nitric acid (calculated as 100 per cent) per annum would be £120,000.

The process, when thoroughly tested, will probably lead to the ultimate abandonment of the older retort method of producing nitric acid from Chile nitrate, and also to the abandonment of niter pots in chamber sulfuric acid manufacture.

#### BY-PRODUCT AMMONIA INDUSTRIES

For conserving and increasing the output of combined nitrogen from existing by-product ammonia industries, for securing the better utilization of the national resources in coal, and for reducing the consumption of raw coal as a fuel, the committee recommends that:

(1) Encouragement should be given to facilitate and to ensure the replacement of non-recovery coke ovens by recovery ovens at a much more rapid rate than hitherto, in order that the reserves of coking coal shall be utilized to the maximum advantage.

(2) It should be made incumbent on all gas works which now contribute to the home supplies of ammonia nitrogen to put into practice the various simple expedients that have been proved to result in a considerable diminution in the loss of ammonia by volatilization or otherwise.

(3) In collaboration with the responsible organization of the gas industry, a comprehensive scheme of ammonia recovery and collection (including distribution of sulfuric acid) should be devised, which would be economically applicable to as many as possible of the smaller works where ammonia is at present allowed to run to waste. Wherever it can be shown that no financial loss would be incurred by any works included in the scheme, the works should be required to cooperate.

(4) In collaboration with the responsible organizations of the by-product ammonia industries, definite standards of efficiency in ammonia recovery should be formulated (based upon the qualities of coal in use and upon good average practice realized under the conditions in force at by-product recovery installations of various classes and sizes), and all works contributing towards the home supplies of ammonia should be required to conform to appropriate standards of recovery.

(5) Encouragement should be given to all efforts to extend the use of coal gas and coke in the place of raw coal for domestic purposes, for steam-raising, and for other industrial requirements.

#### TRIAL OPERATIONS AND RESEARCH

With regard to the trial in the United Kingdom of various nitrogen fixation and recovery processes, and to the general

question of research upon the nitrogen problem, the Committee makes the following recommendations:

#### *Hausser (or Explosion) Process*

The Committee recommends that plans and estimates should be prepared for a plant of the smallest size sufficient for the thorough testing of the modified process by which nitric acid and power can be obtained simultaneously. In the events of the estimates proving favorable, a company possessing a coke-oven installation of the necessary capacity, or other suitable supply of gas, should be encouraged to erect the plant and carry out the trials.

#### *Cyanide Processes*

The barium process, using externally heated retorts, is at present under trial in this country.

The Committee recommends that steps should be taken to test, by means of a trial plant on a small works scale, other modifications of the processes, and especially internal electrical heating. The problem of devising a continuous process for the production of ammonia should be thoroughly investigated at the same plant.

#### *Low Temperature Carbonization of Coal*

This subject is under investigation by the Fuel Research Board.

The Committee recommends that any commercial plant erected or about to be erected in this country should be thoroughly tested. In the national interests, the trials should be organized and carried out by the government.

#### *Sulfate Recovery Processes*

The Committee recommends that the possibilities of sulfate recovery processes which avoid the use of sulfuric acid should be thoroughly investigated on a commercial scale, and encouragement and assistance should be given to companies and undertakings willing to carry out the trials for the benefit of the by-product ammonia industries as a whole.

#### *Research*

The Committee recommends that the researches on the nitrogen problem initiated during the war should be continued after the war under the auspices of the government for the general benefit of the country; and that the results of the researches carried out up to the present date should be edited and published at the earliest possible moment as a further Appendix to this Report, subject to such reservations as may be considered necessary by the government.

#### IMPERIAL SUPPLIES

The Committee recommends that a coördinated policy should be framed for safeguarding the future nitrogen requirements of the Empire, and this should be done by an Imperial Authority.

As the information already available concerning the future prospects and possibilities in the different portions of the Empire will need to be supplemented before such a policy can be framed, the Committee suggests that advisory bodies should be set up to examine the local aspects of the nitrogen problem and to furnish the Imperial Authority with the requisite data.

#### OTHER RECOMMENDATIONS

In connection with other aspects of the Committee's inquiry, the following additional matters arise:

#### *Oxygen and Other By-Products from Nitrogen Plants*

The Committee recommends that:

(1) All liquid air nitrogen plants used in connection with nitrogen fixation installations in the United Kingdom should be operated to the maximum advantage for supplying the growing home demand for oxygen and argon.

(2) The imposition of restrictive trade agreements—such as have been operative at nitrogen fixation installations abroad—which prevent the recovery and utilization of by-product oxygen from nitrogen plants, should not be permitted in this country.



## STATISTICS

The Committee recommends that comprehensive annual statistics for the nitrogen industries of the United Kingdom and for the home consumption of coal by various classes of users and industries should be officially collected and a detailed summary of the figures published. Strong representations on this subject should be made to the government departments concerned. The Committee suggests that this question is also worthy of the attention of the overseas governments.

## SPECIAL OBSERVATIONS

As far as the United Kingdom is concerned, nitrogen fixation and allied processes will constitute a new "key" industry. The Committee is of the opinion that the initiation and development of the industry will require the active support of the government.

HYGIENIC CONTROL OF THE ANILINE DYE INDUSTRY IN EUROPE<sup>1</sup>

The above is the title of a valuable report for the United States Department of Labor after inspection and study in an official capacity by one of America's ablest and most distinguished specialists in occupational diseases and hazards, Dr. Alice Hamilton.

The development of the coal-tar industries in the United States has carried with it the incident dangers, which were better recognized and more thoroughly controlled in Germany than in any other country. For obvious reasons this might have been suspected, but the fact was verified by inspection of works in England, France, Germany, and Switzerland. In drawing special attention to this subject it may be permitted to make reference to the need of an Institute for Industrial Hygiene in the United States.

In the words of Dr. Hamilton, "One of our difficulties in controlling occupational poisoning in American dye works has been our ignorance of the effects on the human body of the many substances which are encountered and we have had to depend on published reports from Germany and England to tell us where to look for danger and how to provide against it."

"The precautions taken in German plants are very elaborate—based on accidents which have actually occurred among workmen, or on careful animal experiments to determine the effects of a new substance." The Germans are in a position to give most valuable information for "even though they may no longer have any cases of poisoning, they can tell of their experience in earlier years when little was known about the effects of the new compounds till some workman or chemist had developed poisoning from them." The German was thus well versed in toxic substances long before he dishonored himself in breaking a solemn contract by using "poison gases" in warfare.

In this connection it may be stated that there is an urgent need for publication of an immense amount of valuable research carried on by the Chemical Warfare Service of the United States Army and Navy, which now appears to have little chance to reach the light of day. The data on the physiological factors and changes involved in fatigue also are of immense value.

While disclaiming an exhaustive treatment of the subject, Dr. Hamilton nevertheless considers in this brochure benzene, aniline, the nitrobenzenes and nitrotoluenes, chlorobenzenes, and various cyclic derivatives, among them the nitrated phenols, nitranilines, and phenylenediamines. Dimethylsulfate, phosgene, arsine, and the chromates are also considered.

Inhaled benzene vapors attack the centers in the central nervous system which control respiration, heart beat and heat

<sup>1</sup> Monthly Labor Review (December 1919) of the Bureau of Labor Statistics, United States Department of Labor.

production. Aniline workers develop bladder tumors. Many poisoned, by DNB, for example, appear outwardly as if very drunk. Dinitrochlorobenzene often delays its effect, with a slow onset of symptoms. Dermatitis comes from all nitro- and amino-derivatives of benzene. The above simply indicates a few of the numerous items of importance in the monograph.

Cleanliness of plant, ventilation, conditions for refreshment, and medical supervision are dealt with. Of these we have much yet to learn and more to put into practice. During the war the pressure drove existent plants to intense systems for super-production. The exigency of the situation brought about the hasty construction of many plants for the production of chemicals of great variety needed in the prosecution of the war. The speed factor did not admit providing all the preventives for the protection of the workers in some instances, and in others the transientness of contracts caused little concern for the welfare of labor. These and other factors associated with the enormous and permanent development of the chemical industry have caused thinking chemists to give the subject most serious consideration. In numerous instances every precaution known has been applied in American plants, yet there is a strong hope that something worth while might be done in a large constructive way to better conditions of labor in the industry.

There are plenty of good people in the chemical industries and there are farsighted corporations seeking means to improve the situation, but the evils of the conditions first serve to call attention to their existence. Efforts to palliate are evident in the provision of medical attention. It is good business to have healthy labor, but the motive is primarily humanitarian. Multiplication and adaptation of education for the workers call for teachers with qualifications peculiar to the needs.

The mephitic odor of the laboratory covers a maze of mystery in the lay mind and is held responsible for numerous maladies. The word "chemical" is sinister. Where chemical processes are in operation, especially when fumes or vapors, even though the latter be only steam, are seen to escape from the works, there grows a feeling of suspicion, often with reason, and all kinds of sickness are attributed to these "poisonous substances." If the writer may judge from numerous letters as well as his own experience in "fume matters," even the members of the medical profession are not free from these sensations. And well they may not be in many instances, for even though the action of many chemicals in varying degrees of concentration is known, the insidious effects of many more are absolutely closed books. They require most diligent study and painstaking research.

The program would call for the coöperation of the chemist, but not his direction. It calls for special training in the field of preventive medicine, which involves social welfare. An excellent, but limited, program of training given at a few institutions is available for those who seek, but the industries inform us that suitably trained men and women cannot be had in adequate numbers to meet the demands.

I speak without authority, but I assert with assurance that the chemical industry will welcome and coöperate with the separate efforts for the improvement of conditions. But it will go much further if those efforts be coördinated. So I venture to say that it would appear desirable to extend the present localized opportunities for instruction and tie them together in one main institute for research, advanced and special instruction, and for coördination.

CHARLES BASKERVILLE, *Chairman*

Committee on Occupational Diseases  
in the Chemical Trades, American  
Chemical Society



### DIVISION OF INDUSTRIAL COÖPERATION AND RESEARCH OF THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY

As a result of the activity incident to raising its Eight Million Dollar Endowment Fund, the Massachusetts Institute of Technology has established a form of relationship with certain commercial organizations which bids fair to become an important factor in that much discussed and highly desired coöperation between educational institutions and the industries of the country. This relationship is set forth in a contract between individual industrial organizations and the Institute of Technology, under which the industry pays an annual retaining fee to the Institute, in return for which the Institute assumes certain definite obligations of such a character as it is clearly in a position to meet.

To execute the obligations thus incurred, the Institute has set up a separate organization known as the Division of Industrial Coöperation and Research. The adjective *industrial* limits the noun "coöperation" rather than "research," because the research work made possible by the new contract plan is not limited to industrial investigations. While originally the funds obtained under the contract were applied to the Endowment Fund of the Institute, those which accrue from contracts obtained since March 1 will be devoted to the support of the Division and to research, largely in fundamental science. This work will naturally be carried on largely at the Institute, but not necessarily so. The industrial progress of the community depends upon an active and productive interest in research everywhere, and it is the intention of the Division to support investigations of promise wherever found.

Obviously the most insistent demand which the industries make upon the universities is for men trained to solve the many problems with which these industries are confronted. Unfortunately many manufacturers in America still suffer from the erroneous idea, seldom met in Germany but very prevalent here, that only those men specifically trained in a definite industry can be of any help in promoting the progress of that industry. A soap manufacturer still asks for a man trained in soap-making; a tanner still seeks for a man who has had a "course in tanning." If a new factory is to be put into operation along old and well-tried lines, it is clearly to the advantage of the owner to obtain a man who can do well those things which have already been done by others. For this purpose, however, a man with factory experience and not a man who has just graduated from the university is indicated. If, however, it is desired to do better something which is already reasonably well done, the greatest promise lies not in the man who has spent his time while at the university in learning the specific details of this one industry, as these details were then known and practiced, but rather in a man whose whole time was spent in acquiring a sound knowledge of science and, so far as possible, a training in the application of science to existing problems, whatever the field in which those problems may have been found.

Hence the Institute does not agree to train men peculiarly fitted for any particular industry or to organize courses of study for the development of specialists. This special knowledge can best be obtained by working in that particular industry under a superintendent who knows well the technique of the industry. A year's factory experience in a well-administered plant will superimpose upon a man's scientific foundation a structure which will enable him not only to follow the old methods, but to determine the underlying reasons for their existence, and hence to form a rational plan for trying to improve on them. Someone has said that a practical man is one who continues to make the same old mistakes; a scientific man makes mistakes but they differ from time to time.

But for those industries which want to advance, something more must be done than to supply them a man with the regular

four-year undergraduate course of study. The application of science and scientific method not only to the search for new knowledge and new processes but also to rational improvement of old processes is susceptible to organized effort, and is generally spoken of as "training for research." The method is the same whether the object be to establish some underlying principle or generalization without reference to its immediate application (pure science); or whether the problem be a search for knowledge concerning a definite reaction in order that the yield of a particular process may be thereby increased (applied science).

Some men work more enthusiastically on one type of problem and some on the other; and obviously the training can be as effective in one case as in the other. It is the quality and fundamental character of the work which counts, and not the interval of time which may be expected to elapse before the results may find utility.

The Division of Industrial Coöperation and Research will foster both types of investigation. The work in fundamental science will be supported by the retainer fees paid by contracting companies, while studies in applied science will be paid for by the contractor for whom the work is done; obviously, in the latter case the results obtained become the property of the contractor meeting the expenses of the investigations.

As previously stated, it is not the intention of the Division to limit its contractors to the facilities of the Institute. It has already been found advantageous to divert a large number of queries submitted under this plan to consulting engineers throughout the country, and it is expected that the practice will be greatly increased.

That wonderfully effective coöperation which in Germany was brought about by the interposition of governmental authority over both the universities and the industries must in America be effected by some such voluntary plan of mutual helpfulness as this contract provides. The Technology Plan will doubtless be improved upon as time goes on, but even to-day it has elements of great promise.

WILLIAM H. WALKER, *Director*

Division of Industrial Coöperation and Research

MASSACHUSETTS INSTITUTE OF TECHNOLOGY  
CAMBRIDGE, MASS., March 16, 1920

### PURCHASE OF GERMAN BOOKS AND PERIODICALS

*Editor of the Journal of Industrial and Engineering Chemistry:*

It may be worth noting in the JOURNAL that the Leipzig Book Dealers Association has agreed upon an arrangement whereby it is practically impossible to purchase German books and periodicals in any way that will even approximately take advantage of the current exchange rate of the German mark. A great many chemists have ordered German publications in the hope that in some way this could be done. How this works out may, however, be indicated by the following:

All German publishers now ask 100 per cent over the published price of all books and in addition to this the Leipzig Association adds 400 per cent for all publications for export (on January 15 this was 340 per cent). Furthermore they insist on payment at the rate of \$9.00 per 100 Marks regardless of the current rate of exchange.

I have inquired into this carefully and I find that most of the importers are really getting a very small profit. The difficulty is at the other end. For example, a recent small book, publishers' price 20 Marks, actually sold to the American purchaser through a well-known New York importer for \$4.00.

The working agreement of the Leipzig Association does not apply to secondhand books. It is possible for private individuals in Germany to purchase publications there at 100 per cent over



the published price. No established dealer or importer will undertake to purchase books in this manner for American clients on account of the penalty of being excluded from all further purchases from any of the members of the German Publishers Association at Leipzig if discovered. A notice sent out by the Leipzig Association on January 15, 1920, states that government coöperation in this matter has been sought, but it is not clear that the government is actually a party to this arrangement. Certain German publishers and dealers bill their goods only in foreign money at substantially the equivalent of price increment agreed upon by the Leipzig Association.

A set of Liebig's *Annalen* advertised by a well-known German dealer at 14,000 Marks was sought by an American importer who cabled his Leipzig agent to purchase at this figure. In due time reply by letter was received to the effect that the price would be 21,000 Marks. A second cable to purchase brought the belated reply that the price would be 42,000 Marks, at which point negotiations were dropped.

This may give your readers some notion of the situation and indicate that the idea of purchasing German books at this time at a low figure on account of the exchange rate is largely a delusion.

25 WEST 43rd STREET  
NEW YORK, N. Y.  
March 2, 1920

B. T. BROOKS

#### CHEMICAL MARKET IN INDIA

*Editor of the Journal of Industrial and Engineering Chemistry:*

In one of your more recent issues, as I recall, you requested your readers to send in notes which might be of some interest to the rest of the subscribers to your magazine. It has recently fallen to my lot to purchase, or at least try to purchase, chemicals and chemical apparatus. The situation as I find it is that the country has but few houses supplying these things and, due to the war, low stocks. What we do get is Japanese and though these goods had a clear field they have failed utterly to capture the trade or gain the confidence of the Indian manufacturers and the buyers in the many colleges here. Just now supply houses are offering the remnants of their pre-war stocks of German goods, which are being bought in preference to Japanese goods. This is true of many kinds of buyers.

It occurs to me, therefore, that this country offers a peculiarly good place for American goods. I am constantly being asked the names of factories producing this or that thing of a chemical nature and it would seem to me that with the desire of the Indian purchaser to obtain American material some of our factories might well try to place their articles here. Thus far, needless to say, one cannot get an American chemical or piece of apparatus without importing it himself, but he can get and will be forced to accept German or other articles unless there is greater ease of purchase of American goods than at present. With the tremendous industrial development which India is making, and the far greater development which will be one of the not far-

distant results of the war, this country will be a market worthy of some serious consideration.

I trust that this little note may be of some interest to the manufacturers at home. It is given for what it may be worth, but with the certain knowledge that the field is open and that American goods will receive a ready welcome here.

LAHORE, INDIA  
January 12, 1920

HAROLD S. BENNETT

#### CALIFORNIA INSTITUTE OF TECHNOLOGY

Throop College of Technology at Pasadena, California, has recently changed its name to the California Institute of Technology, in order to denote its transformation from a college of local significance to a scientific school of national scope. The institution has recently received two gifts of \$200,000 each to form permanent endowments for the support of research in physics and chemistry, and a gift of \$800,000 for general maintenance on condition that the new endowment be increased to a total of \$2,000,000. Gifts totaling \$280,000 have been received for the construction of new buildings, including an auditorium, physical and chemical laboratories, and a laboratory for aeronautical research.

#### CHANDLER LECTURE

The Chandler Lecture will be given on April 27 by Dr. Willis R. Whitney at Havemeyer Hall, Columbia University, on "The Littlest Things in Chemistry."

The Lectureship, which was inaugurated at the time of the celebration of the Fiftieth Anniversary of the founding of the School of Mines, in 1914, was allowed to lapse during the period of the war, and is now being reestablished.

#### SQUALENE: A HIGHLY UNSATURATED HYDROCARBON IN SHARK LIVER OIL—CORRECTION

In the article of the above title [THIS JOURNAL, 12 (1920), 63] the following correction should be made:

Page 71, 1st col., 19th line, "benzene" should be written "benzene."

TOKYO, JAPAN

MITSUMARU TSUJIMOTO

#### THE DETERMINATION OF CELLULOSE IN WOODS—CORRECTION

In my article under the above title [THIS JOURNAL, 12 (1920), 264] the following correction should be made:

Page 267, 1st col., 12th line, "sufficient 3 per cent sodium hydroxide solution added" should read "sufficient 3 per cent sodium sulfite solution."

BERKELEY, CALIFORNIA

WALTER H. DORE

## SCIENTIFIC SOCIETIES

#### ALL READY FOR THE ST. LOUIS MEETING

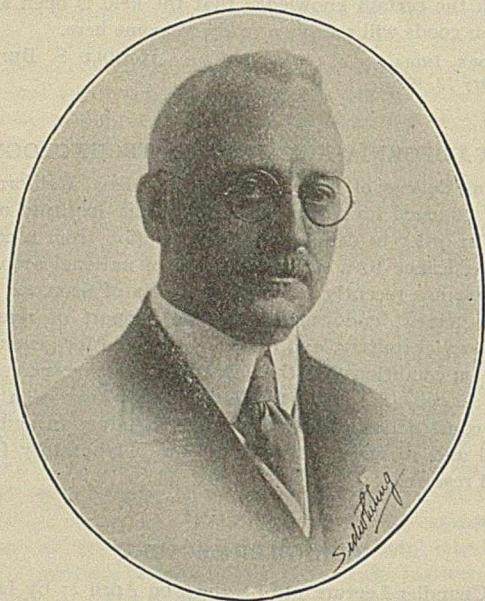
Special Correspondence to the JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY by JOHN WALKER HARRINGTON

ST. LOUIS, MARCH 25—All signs indicate that the largest meeting of the AMERICAN CHEMICAL SOCIETY ever held will be that of the spring of 1920. If the enthusiasm and interest of the St. Louis Section can accomplish this alone that goal has already been reached. The St. Louisians never fail in anything which they undertake, and the members of this enterprising western Section of our SOCIETY represent the spirit of progress which has always characterized this city of leaders and pioneers.

That the members of the AMERICAN CHEMICAL SOCIETY will respond in legions to the invitation of our hosts of Missouri is shown by the many reservations which are being made at the hotels of the city. Arrangements have been made for special trains from neighboring centers, and it is expected that with the coming of April 12 there will be from two to three thousand chemists assembled for the sessions of the SOCIETY. Owing to the forethought of the Section in this city and of that of the University of Missouri, at Columbia, which joins with it as host, nothing has been omitted for making the stay of the visiting delegation an experience which they will remember with delight in years to come.



From a technical point of view, this Spring Meeting of 1920 promises to be an epoch-making one in this era of reconstruction. The fruits of victory have been gathered; the country is seeking to return to the norm of industry and commerce, and



CHARLES E. CASPARI

upon the chemists there rests especially the responsibility for bringing the activities of the nation back to their accustomed channels. In order that the chemical industry may do well its part in the national service, it is natural that it should consider well the matter of legislation. The address, therefore, of Mr. E. P. Costigan, of the Tariff Commission, will have a special bearing. Its subject, "Chemical Industry and Legislation," shows that Federal officials are considering very seriously the status of the chemical manufacture.

As shown by the program, the activities of the sessions begin to stir on the afternoon of Monday, April 12, when the Council Meeting will be held, and in the evening when the Council will be entertained at dinner at the University Club. When the visiting members assemble at the General Meeting in the Hotel Statler they will be officially welcomed by the Honorable Henry W. Kiel, mayor of St. Louis. The response will be given by Dr. W. A. Noyes, of the University of Illinois, the president of our SOCIETY. Following the speech of Mr. Costigan, the editor of the *Journal of Industrial and Engineering Chemistry* will speak of the responsibilities of chemistry in this important crisis of our national history.

Among the papers that will be delivered at the afternoon session of the General Meeting on the same day will be "The Prediction of Solubility," by J. H. Hildebrand; "Selenium Oxide, a Neglected Inorganic Solvent," by Victor Lenher; and "Studying Plant Distribution with Hydrogen Ion Indicators," by E. T. Wherry.

It is an index of the sturdy growth of the AMERICAN CHEMICAL SOCIETY that two new Sections will be formed at this meeting.

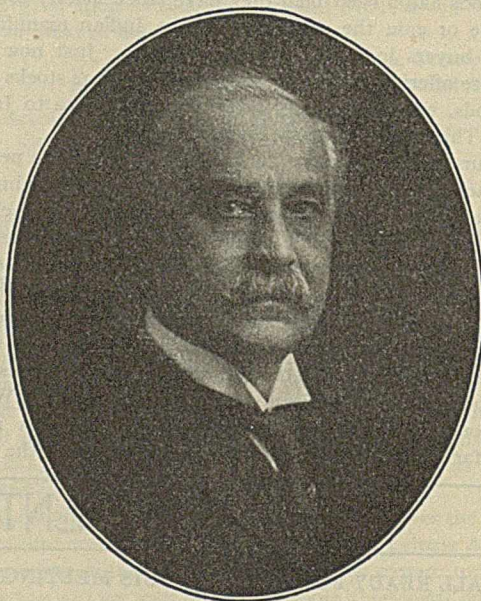
A Section of Leather Chemistry will come into being for the purpose of establishing a forum for the discussion of the chemistry of leather manufactures and other closely allied industries. No more appropriate place for founding so important a Section could have been selected than St. Louis, which by its traditions is an entrepôt of the cattle industry. In these days it is indeed a problem for mankind to find out how it shall be shod, and the new processes of tanning which the leather chemists are developing are destined to receive unusual attention. In the Organic Division the paper of Dr. John Arthur Wilson

on "Vegetable Tanning as a Colloid Chemical Process" is also in line with the latest researches in the useful art honored by St. Crispin.

For the first time in the history of the SOCIETY a Section of Sugar Chemistry will meet; and it is also significant that it should be founded in this flourishing city which is within easy reach of the sugar-cane districts of the sunny South, as well as of the beet fields of Nebraska. There will be exhibited at the meeting of this Section the new American-made saccharimeter.

Those of us who were at Philadelphia will remember how the "Baby Section"—Dyes—burst out of its bright-hued swaddling clothes. It refused to take its assigned place in the Blue Room of the Bellevue-Stratford, nor would it be content either with the Green or the Red Room. Many did not believe that it would have more than 25 or 30, but when the veracious Secretary, Mr. R. Norris Shreve (the adjective is applied on account of his St. Louis birth), insisted that he could fill the Palm Garden, there were many doubters. The high count at the meeting of the Dye Section last year, however, was 385, and it is likely that this infant—which has now become a giant—will show an even larger growth next month and will be promoted to the rank of a Division. Many important papers in this Section have already been prepared.

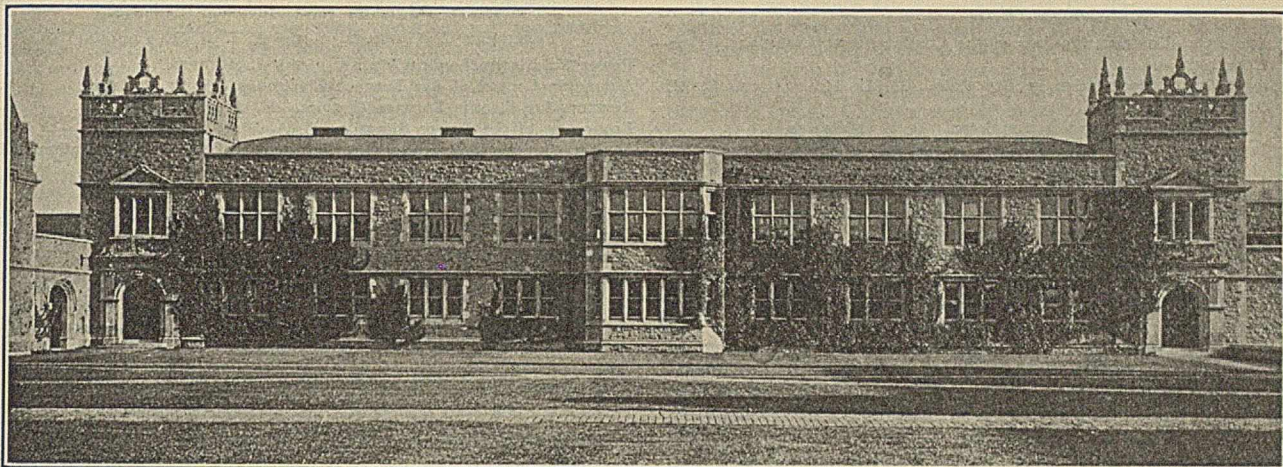
Word comes from the office of the Secretary in Washington that there are also likely to be notable contributions from the Divisions of Agricultural and Food Chemistry, Biological Chemistry, Fertilizer Chemistry, Industrial and Engineering Chemistry, Organic Chemistry, Pharmaceutical Chemistry, and also in those Sections devoted to the Chemistry of Rubber and of Water, Sewage and Sanitation. The newspapers of St. Louis have already taken a deep interest in the approaching meeting, and have given liberal advance notices of it. On account of the many subjects of interest which will come before the various Sections, it is desirable that synopses of papers which have a



F. W. FRERICHS

popular appeal be sent as soon as possible to the A. C. S. News Service, care of the AMERICAN CHEMICAL SOCIETY, 35 East 41st Street, New York City, which, in cooperation with the Publicity Committee of our local Section and the St. Louis Convention and Publicity Committee, will use its best endeavors to see that full information is supplied to the press. In these synopses the relation of the subjects of the papers to every-day life or to the future of an industry should be stressed. Papers dealing with public policy, national affairs, the general welfare, food supply,





BUSCH HALL, CHEMICAL LABORATORY, WASHINGTON UNIVERSITY, ST. LOUIS

living and housing conditions, or with the announcement of discoveries and the results of original research, are always desirable in popular form. To insure their effective use for both the lay and technical press, they should be in hand not later than ten days in advance of the meeting.

The local committee is planning a series of excursions to the various chemical plants of St. Louis. This will give all the members an ample opportunity for seeing the leading drug center of the West, which leads the country in the production of synthetic pharmaceuticals, alkaloids, and anesthetics. This also is an important center for ceramics, glass, paint, lead, and zinc. These last two important metals are mined not very far from the city in whose industries they are notable factors. Among the plants to be visited are those of the Laclede By-Products Coke Company, the Monsanto Chemical Works, the Laclede-Christy Clay Products Company, the Standard Oil Company Refinery at Wood River, Illinois, and the Illinois Glass Company at Alton, Illinois.

The social features of the St. Louis meeting are eloquent of the spirit of hospitality which characterizes this city. On the evening of April 13 there will be a theater party for the ladies, and at the Liederkrantz Club a smoker for men. Several automobile tours have also been planned.

Washington University grouped about its stately quadrangle waits to welcome the Knights of the Retort. Dr. Ball, of its department of chemistry, will officially keep the census of the Spring Meeting. He was discovered to-day blending the ingredients of a new adhesive which will keep delegates attached to one place until they have been duly registered and badged. He and Dr. L. McMaster, former chairman of the St. Louis Section, are busy in the well-equipped laboratories of the institution training ambitious youth, and let us hope future members of the A. C. S. Their students include some of the gentler of the species which shows there is a chemical career for woman in the ever-progressive State of Missouri. There is no more beautiful region in all the academic world than the campus and the neighboring Forest Park in which the fine old University is set like a jewel. It is in sight also of the Museum of Art, the Jefferson Memorial, and other structures which adorn this wonder city of Laclede. Your correspondent to-day, making an automobile tour of the metropolis of the West through the kindly invitation of Dr. Nickell, saw many signs of the charm which comes to the valley of the Mississippi when Mother April makes all things new. They were pledges of the time when the parks and the famous Shaw Botanical Garden will surpass Kew at its best. It is well worth crossing the continent to be here in April and, as most of us, owing to the central position of St. Louis, will

not have to go more than half as far, to miss this Spring Meeting would be to let slip an alembic of good fortune.

The local committees in charge of the arrangements for the St. Louis meeting are: Edward Mallinckrodt, Sr., Honorary Chairman, Executive Committee; Charles E. Caspari, Chairman, Executive Committee; F. W. Frerichs, Chairman, Finance Committee; L. F. Nickell, Chairman, Publicity Committee; Gaston DuBois, Chairman, Excursion Committee; H. A. Carlton, Chairman, Hotels Committee; T. R. Ball, Chairman, Registration and Information Committee; A. C. Boylston, Chairman, Transportation Committee; E. J. Sheppard, Chairman, Banquet Committee; F. W. Sultan, Chairman, Smoker Committee; and Mrs. C. E. Caspari, Chairman, Ladies' Entertainment Committee. These committees are assisted by: F. W. Russe, P. A. Shaffer, Herman Schlundt, Edward Mallinckrodt, Jr., Jules Bebie, B. M. Dugger, H. M. Whelpley, J. W. Marden, P. C. Brooks, E. R. Cramer, J. M. Olin, R. S. Sherwin, A. D. Alt, George Lang, Jr., J. T. Ragsdale, Jr., O. H. Pierce, D. B. Conrad, E. R. Harding, A. W. Becker, W. E. Buck, H. E. Wiedemann, F. B. Langreck, W. R. Teeters, R. R. Barlow, L. J. Chamberlain, J. D. Robertson, L. McMaster, C. C. Luedeking, Hamilton Merrill, W. F. Monfort, W. S. Ritchey, and F. E. Turin of the St. Louis Convention and Publicity Bureau.

#### AMERICAN PHARMACEUTICAL ASSOCIATION

During the Annual Convention of the American Pharmaceutical Association, to be held in Washington, D. C., on May 4 to 10, 1920, the Scientific Section will hold its meetings on Thursday, Friday, and Saturday, May 6, 7, and 8. Those desiring to read papers before this section should submit them to the Secretary, Dr. A. G. DuMez, Hygienic Laboratory, U. S. Public Health Service, Washington, D. C., not later than April 1.

#### AMERICAN ELECTROCHEMICAL SOCIETY

The Spring Meeting of the American Electrochemical Society will be held in Boston, Mass., April 8, 9, and 10, 1920, and promises to be unusually attractive and interesting.

The annual business meeting, at the Massachusetts Institute of Technology on April 8, will be followed by the address of the retiring president, W. D. Bancroft, on "Contact Catalysis," a symposium on "Colloids," and other interesting papers. The evening will be occupied by a smoker.

The meetings on April 9 will be joint sessions with the American Institute of Electrical Engineers.

There will be an excursion to the General Electric Plant at Lynn on Friday afternoon, April 9, and to the Huff Electrostatic Separator Company on Saturday afternoon, April 10.



## PROGRAM OF PAPERS

- A Criticism of Kohlrausch's Law of the Conductivity of Solutions. GUY CLINTON.  
 Hydrogen Ion Concentration in Dry Cells. H. D. HOLLER AND L. M. RITCHIE.  
 The Reactions of the Lead Accumulator. D. A. McINNES.  
 Arc Images in the Chemical Analysis. WM. ROY MOTT.  
 The Soderberg Self-baking Continuous Electrode. J. W. RICHARDS.  
 Synthetic Electric Furnace Pig Iron. CHAS. A. JELLER.  
 Step Induction Regulators for Electric Furnaces. PHILIP B. SHORT.  
 The Electric Furnace as Applied to Metallurgy. CLARENCE J. WEST.  
 The Electric Brass Furnace, Its Evolution. H. M. ST. JOHN.  
 The Electric Furnace in Iron and Steel Metallurgy. L. B. LINDEMUTH.  
 Reduction of Manganiferous Silicate Slags. EDW. F. KERN.

## Symposium: Colloid Chemistry

- Colloid Chemistry and Electrochemistry. W. D. BANCROFT.  
 Settling Problems. W. A. DEANE.  
 The Adsorption of Arsenious Oxide by Metastannic Acid and the Purification of Spent Electrolyte. T. R. BRIGGS AND W. J. BARTLETT.  
 Grafitic Acid, a Colloidal Oxide of Carbon. G. A. HULETT.  
 Determination of Grafitic Carbon. A. C. FIELDNER.  
 Selective Adsorption by *Bacillus pyocyaneus*. WM. C. MOORE.

## Symposium: Electrically Produced Alloys

W. D. BANCROFT, *Presiding*

- Fundamental Problems in Alloys Research. H. E. HOWE.  
 Recent Developments in the Ferro-Alloy Industry. R. J. ANDERSON.  
 The Manufacture of Ferro-Alloys in the Electric Furnace. C. B. GIBSON.  
 Ferrovandium. B. D. SAKLATWALLA.  
 Stellite. ELLWOOD HAYNES.  
 Properties of Ferrosilicon. F. A. RAVEN.  
 Ferromanganese. E. S. BARDWELL.  
 The Manufacture of High Speed Steel in the Electric Furnace. R. C. MCKENNA.  
 Ferrocerium. ALCAN HIRSCH.  
 Ferrozirconium. THEODORE SWANN.  
 Ferrophosphorus. B. C. KLUGH.  
 Nichrome and Other Special Alloys. R. M. MAJOR.  
 Electric Properties of Nickel Iron Alloys. T. D. YENSEN.  
 Electrical Properties of Titanium Alloys. M. A. HUNTER.  
 Nitrogen Fixation by the Silent Discharge. C. F. HARDING.

## Symposium: Power for Electrochemical Industries

PRESIDENT C. TOWNLEY, of the A. I. E. E., *Presiding*

- Power for Electrochemical Plants. JOHN L. HARPER.  
 Automatic Control of Arc Furnace Electrodes. JOHN A. SEEDE.  
 Reactors for Electric Furnace Circuits. H. A. WINNE.  
 Economics of the Power Situation. C. A. WINDER.  
 Electric Furnace Power from the Standpoint of the Central Station. E. A. WILCOX.  
 The Electric Furnace and the Central Station. H. L. HESS.  
 Location of a New England Electrochemical Plant. C. T. MAYNARD.  
 Water Powers of the Pacific Coast. F. F. FOWLER.  
 Power Development in Scandinavia. J. W. BECKMAN.  
 Power Problems from the Standpoint of the Furnace Operator. W. G. BERLIN.

## CALENDAR OF MEETINGS

- American Electrochemical Society—Spring Meeting, Boston, Mass., April 8 to 10, 1920.  
 American Drug Manufacturers' Association—Annual Meeting, Hotel Biltmore, New York, N. Y., April 12 to 15, 1920.  
 Technical Association of the Pulp and Paper Industry—Spring Meeting, Hotel Waldorf and Hotel Astor, New York, N. Y., April 12 to 16, 1920.  
 American Pharmaceutical Association—Annual Convention, Washington, D. C., May 6 to 8, 1920.  
 American Chemical Society—Fifty-ninth Meeting, Hotel Statler, St. Louis, Mo., April 12 to 17, 1920.  
 National Foreign Trade Council—Convention, San Francisco, Cal., May 12 to 15, 1920.  
 National Fertilizer Association—27th Annual Convention, White Sulphur Springs, W. Va., week of June 21, 1920.  
 American Institute of Chemical Engineers—Semi-Annual Meeting, Montreal, June 21 and 22; Ottawa, June 23; Shawinigan, June 24 and 25; La Tuque, June 26, 1920.

## WASHINGTON LETTER

By J. B. McDONNELL, Union Trust Building, Washington, D. C.

Efforts to have the embargo section of the Longworth dye bill eliminated when that measure is again called up in the Senate for consideration will be made by opponents of the bill.

The bill was called up in the Senate and debated for a day just before the Treaty of Peace was again taken up. No action was taken on the bill, however, a small filibuster staged by western senators as a means of forcing the Senate Finance Committee to act on other tariff measures pending before it preventing any final action. Following its action in reporting favorably the dye bill, Republican members of the Senate Finance Committee determined to "smother" in committee the other tariff measures which had been passed by the House, including the surgical and scientific instruments bill, the magnesite, zinc, and pearl button bills.

The committee conclusion met with strong opposition from western senators in whose states were located the interests affected by the bills it had been determined to smother, and when the dye bill was taken up on the Senate floor they were open in their condemnation of the committee's procedure, and the dye bill was debated for the day without any action being taken. The calling up of the Peace Treaty the next day under agreement has prevented further consideration of it up to this time.

Responding to the urging of textile interests of New Hampshire, Senator Moses of that state has introduced an amendment to the committee bill which would eliminate the embargo section entirely. In its stead it would permit all dyes to be imported under the duties assessed in the tariff section of the committee bill, plus a duty sufficient to make the price of the imported dye equal to the "fair wholesale price" of the same dye when manufactured in this country.

The fair wholesale price, the bill states, means the "lowest wholesale price sufficient to insure the maintenance in the United States of the production of such article by an efficient plant operated on a scale reasonably adequate to supply ordinary domestic demands." This price is to be determined by the Secretary of the Treasury.

The Moses amendment, which is pretty much an anti-dumping measure, would assess duties as follows:

(b) In addition to the duties provided in Section 500 of this Act, there shall be levied, collected, and paid, during the period of three years beginning from the date of the enactment of this amendatory Act, upon any article enumerated in Group II or Group III of such section, if at the time of importation of such article a like article or a satisfactory substitute therefor is manufactured and offered for sale in the United States on a substantial commercial scale, an additional duty equal to the difference between (1) the sum of the dutiable value of such article, ascertained as provided in subdivision (R) of Section 111 of the Act entitled "An Act to reduce tariff duties and to provide revenue for the Government, and for other purposes," approved October 3, 1913, plus all non-dutiable costs, charges, and expenses incident to the importation thereof, and any duty thereon imposed by Section 500 of this Act, and (2) the fair wholesale selling price in the United States of a like article or satisfactory substitute therefor of domestic manufacture, plus 20 per centum of such wholesale selling price in the United States: Provided that subdivision (2) exceeds subdivision (1).

The amendment goes on to prohibit false labeling and provides penalties for incorrectly labeled dyes brought into this country.

Senator Moses has expressed the belief that in spite of the confidence expressed by supporters of the committee measure, it will meet with considerable opposition when called up on the floor again, and declared that Senators Dial, Keyes, Henderson, Pittman, Sherman, Poindexter, himself, and some others, are opposed to the committee bill. Senator Keyes, it is said, intends to offer several amendments.

With Republican and Democratic leaders supporting the committee bill, Senator Watson, of Indiana, in charge of the measure, is confident of its passage without material amendment. When it will be called up again depends upon the course of the treaty debate. It is planned to bring the dye bill up as soon as the treaty is disposed of.

Acting against commercial bribery practiced by dealers in



dyestuffs and chemicals, the Federal Trade Commission has issued complaints against 22 concerns. In ten cases the commission has issued a report, dismissing one complaint—that against the Commonwealth Color & Chemical Company, of New York—and issuing orders to "cease and desist" from commercial bribery against the other nine concerns whose cases have been tried. The concerns named in complaints issued by the Federal Trade Commission are F. E. Atteaux & Company; Dearborn Chemical Company; The Commonwealth Color and Chemical Company, and Herbert L. Wittnebel, a salesman of that concern; the Federal Color & Chemical Co.; William Mohrmann; John Campbell & Co.; Holliday-Kemp Co., Inc.; A. Klipstein & Company; Geigy Company, Inc.; C. Bischoff & Company, Inc.; The Heller & Merz Company; The Franklin Import & Export Company, Inc.; New York Color & Chemical Company; Louis Rosenthal, doing business under the name and style of the United Chemical & Color Company; Andreykovicz & Dunk, Inc.; Arkansas Distributing Company; Pennsylvania Salt Manufacturing Co.; L. Richardson, H. Smith Richardson and L. Richardson, Jr., co-partners, doing business under the name of The Vick Chemical Company; The Arabol Manufacturing Co.; Bosson & Lane; and Ricco Company, Inc.

All of the concerns named in the complaints issued by the Commission are accused of giving gratuities "consisting of cigars, meals, drinks, entertainment and in some instances small presents," while several of them are also accused of giving and offering to give to employees of both customers and prospective customers and competitor's customers "sums of money." With the commercial bribery in mind, with which the dyestuff and chemical business in this country was infested prior to the war, it is not surprising that a number of dealers are yet to be found who cling to the methods which in the past have been so effective as one of the means used to prevent the development of a real dye industry here. It is interesting to note, however, that none of the larger American manufacturers of dyestuffs or chemicals have been included among those against whom complaints have been issued by the Commission.

Concerns against which the Commission found its complaint to be justified and issued orders to cease and desist include F. E. Atteaux & Co., Inc.; Federal Color & Chemical Company; William Mohrmann; John Campbell & Company; Holliday-Kemp Company, Inc.; A. Klipstein & Company; Geigy Company, Inc.; C. Bischoff & Co., Inc.; and The Arabol Manufacturing Company.

While the Senate has been debating the Treaty, after having taken it up once more for consideration, the House has latterly been engaged in consideration of the army reorganization measure. As did the new army bill reported by the Senate military committee, the House bill provides for a separate Chemical Warfare Service. Prominent members of the House, however, consider the bill defective in that it does not define the duties of the Chemical Warfare Service, and therefore the General Staff, which has openly opposed a separate Chemical Warfare Service, would be enabled practically to kill the Service if it so desired. Representative Dent has announced that he will seek to guard this point in the legislation. The action of the House committee in making this Service a separate department of the Army was early forecast. There seems little doubt but that the House will sustain the committee and that when the army reorganization is finally worked out the importance of chemistry, not only in peace, but in warfare also, will have been recognized by both House and Senate, and the Chemical Warfare Service set up as a separate and distinct department of the Army. Any other action would be surprising to those in charge of the measures in both bodies.

It is interesting to note in this connection that Major General William L. Sibert, who had been chief of the Chemical Warfare Section of the Army, has been replaced by Lt.-Col. Amos Fries. Colonel Fries was put in charge of the Chemical Warfare Service of the American Expeditionary Forces and served in that capacity in Europe until the end of hostilities. Upon his return to this country he resumed his former rank and was recently put in charge of the Service in this country.

Dr. S. W. Stratton, chief of the Bureau of Standards of the Department of Commerce, appeared before the Senate Appropriations committee recently and urged that the Senate reverse the action of the House in reducing the appropriations asked for by the Bureau to carry out its important work. Like many other departments and bureaus, the Bureau of Standards was struck by the program of economy determined upon by the Republicans in Congress, and its appropriations for the ensuing year were cut even below the sums granted in the last appropriation bill for the current year. The importance of the work of the Bureau was emphasized to the committee by Dr. Stratton, who asked that the Senate at least restore the appropriation to the amount granted this year.

March 15, 1920

## OBITUARIES

### FRANCIS C. PHILLIPS

Dr. Francis Clifford Phillips died at his residence, 144 Ridge Ave., Ben Avon, Pa., on Monday, February 16, 1920, of influenza-pneumonia, passing away in the same peaceful manner which characterized his life.

He was born in Philadelphia, April 2, 1850, the son of William S. and Fredericka Ingersoll Phillips. He received his early education at home from an unusually capable and devoted mother. In 1864 Dr. Phillips studied at the Academy of the Protestant Episcopal Church in Philadelphia, and in 1866 entered the University of Pennsylvania, where he obtained his A.B. From 1871-1873 he studied under Regimus Fresenius at Wiesbaden, Germany. During the latter year he was private assistant to Prof. Fresenius. He then spent a year at the Polytechnic School at Aachen (Aix-la-Chapelle). Here he was associated with Prof. Landolt. Professor Phillips was unable to complete his studies abroad because of the poor health of his father. He returned to America, and during the following year became instructor in chemistry at Delaware College. In 1875 he was appointed to the teaching staff of the University of Pittsburgh, then the Western University of Pennsylvania,



FRANCIS C. PHILLIPS

where he taught for forty years, retiring as head of the department in 1915. For many years he taught chemistry, geology, and mineralogy. In 1878-1879 he also lectured to the students in the Pittsburgh College of Pharmacy. In 1879 he received the degree of A.M. from the University of Pennsylvania, and in 1893 the Ph.D. from the same institution. In June 1919 Dr. Phillips received the honorary degree of Doctor of Science from the University of Pittsburgh.

He was married in 1881 to Sarah Ormsby Phillips, daughter of Ormsby Phillips, a former mayor of Allegheny.

In 1915 Dr. Phillips retired from active service in the University of Pittsburgh under the pension system of the Carnegie Foundation. On this occasion his colleagues and the alumni tendered him a banquet at which he was presented with a check for one thousand dollars for a little vacation with "Mother" Phillips. Professor Phillips was so solicitous of the safety of his dear wife, that though they intended visiting the Bermudas and the Panama Canal Zone, the war, with the possible accompanying danger in ocean travel, caused him to postpone the trip which was never realized. At the banquet, the fruit of his labors as a teacher was in plentiful evidence, including such men of prominence as George H. Clapp, J. M. Camp, Walther Riddle, C. M. Johnson, Charles Kellar, Charles Dinkey, W. A. Hamor, Jacob Rosenbloom, the late Robert Carnahan and many others. It was an evening of tribute to one who had so conscientiously given to former students all that he possessed as a teacher.

Since 1915 he had been engaged continuously in research and writing in a laboratory provided by the Mellon Institute.



During the recent war he conducted researches on gases in cooperation with the Gas Warfare Service.

Dr. Phillips was an authority on natural gas, in which field he held international recognition. In 1904 he published the "Methods of Analysis of Ores, Pig Iron and Steel Used by the Chemists in the Pittsburgh Region," and in 1913 a "Textbook of Chemical German," of which a second edition appeared in 1916. At the time of his death Dr. Phillips had two other books well under way, one on the "Life and Work of Joseph Priestley," the other on "Qualitative-Gas Reactions."

Dr. Phillips was a member of the following societies:

Phi Kappa Sigma Fraternity since 1867.  
 Engineers' Society of Western Pennsylvania since 1880.  
 American Association for Advancement of Science since 1887.  
 American Institute of Mining Engineers since 1892.  
 American Chemical Society since 1894.  
 American Philosophical Society since 1894.  
 Phi Lambda Upsilon Fraternity since 1919.

Dr. Phillips was a member of the Chemists' Club of New York City and the University Club of Pittsburgh. He had been a member of the Council of the American Chemical Society since the organization of the Pittsburgh Section in 1903.

As the Rev. Henry H. Forsythe so aptly said at the funeral service, Professor Phillips' life was characterized by many great virtues. Though recognized throughout the world for his researches on gases, he was simple and plain as a child and never refused another his counsel on any matter. He possessed a modesty rarely seen in men and walked always in a humble spirit. He loved and bore the love of his many pupils, who though they might have had a difference in the classroom invariably learned to love the dear professor. He was the gentlest and kindest of men. As in his scientific researches he realized that the strictest observance of truth and honesty was necessary for the accurate solution of nature's secrets, so in life he walked uprightly and knew not of untruth or subterfuge.

Beside his widow, Mrs. Sarah Ormsby Phillips, Dr. Phillips leaves two sons, Clifford S. and Frederick I. Phillips.

ALEXANDER SILVERMAN

#### DAVID SHEPARD PRATT

The sudden death of Dr. David Shepard Pratt, on January 28, 1920, was a distressful misfortune to the world of science. While his published researches in the field of organic chemistry had been looked upon as achievements and had accordingly directed attention to his rare ability as an investigator, yet some of his most important accomplishments—some of those which had resulted from his contact with industry—were still awaiting preparation for publication at his hands, and at the time he succumbed to pneumonia he had just finished the development of several processes of undoubted value to agriculture. His end was very premature, and it is with an acute sense of bereavement that this occasion is taken to pay a brief word of tribute to so esteemed and efficient an associate.

David Shepard Pratt, son of Charles Manville and Louise Hale (Woodford) Pratt, was born at Towanda, Pa., on September 20, 1885. He was graduated at Cornell University with the degree of A.B. in 1908, and remained there as a Fellow in Chemistry until 1911, when he received the degree of Ph.D. He then joined the staff of the Bureau of Chemistry in Washington, D. C., as assistant chemist, but remained there only a short time, being soon selected as Chief of the Organic Division of the Bureau of Science in Manila, P. I. His chemico-economic studies in the Philippines were varied and valued, and he also acted as a member of the Pure Food and Drug Board. In 1914 he returned to the States to accept a professorship of chemistry at the University of Pittsburgh, and he successfully occupied that chair and the headship of the organic department of the School of Chemistry for three years—until he was made an Assistant Director of

the Mellon Institute of Industrial Research. On January 1, 1920, after three years' service, he resigned at the Institute and was arranging to enter consulting chemical practice in St. Louis, Mo., at the time of his fatal illness.

From his student days at Ithaca, Pratt's studies in organic chemistry were characterized on the one hand by philosophical breadth of view, and on the other hand by extreme accuracy of statement. It was to the combination of these characteristics with highly skillful experimentation that the value of his research work was due. The combination is seen prefigured in his reports of researches on various problems in the domain of tropical chemistry; it is noted as realized in his published investigations on phthalic acid derivatives; but it became most effective at the Mellon Institute, where Pratt enjoyed broad opportunities to apply, in the inquiries of the Industrial Fellowships under his supervision, his profound knowledge of organic chemistry and his familiarity with research methods. His scientific spirit, his keen critical insight, and his exhaustive control of sources of information gained and held the high respect of all and played a prominent part in establishing the success of the system in operation at the Institute. To the members of the Institute the tidings of his death came with a deep sense of loss, for, in addition to being an efficient research executive, he was a lovable man.

W. A. HAMOR

#### LEONARD MERRITT LIDDLE

The untimely death, on February 21, of Dr. Leonard Merritt Liddle, a Senior Industrial Fellow of the Mellon Institute of Industrial Research, was an irreparable loss not only to the friends whose privilege it had been to know so scholarly and amiable a man, but to the interests of the chemical profession in general. To some, perhaps, it may seem to be extravagance to refer to the irreparability of any such loss to chemistry; for in the great work of the world we are inured to see the ranks close up as heroes fall by the way, and when we come to calculate the sum of actual accomplishment, in our gratitude over the estimable results obtained we rarely perceive the importance of those innumerable unrealized possibilities upon which, in the nature of things, we can place no just evaluation. It is inevitable that this should be so. There is, however, a viewpoint from which it may be reasonably insisted that the work which rare and original minds fall short of achieving because of brevity of life does never actually get done at all. No doubt similar work is carried out; but the performance of it is in an entirely different order of causation; and while there may be a resemblance of equivalence, the fact is evident that, from the sum of human effort, an indefinite amount of rich and fruitful life has been lost. True as this is in the case of pure science, it is still more obviously true in applied science or in industrial research. This becomes clear when we consider that the work of a keen, skillful scientific investigator is artistic, the peculiar product of endless complexities of individual characteristics—personal poise, mental tone, and method of thought.

In the case of Leonard Merritt Liddle, none except the friends who knew the treasures of his highly trained, acute mind, as revealed in intimate association and in familiar conversation, are likely to realize how great is the loss which chemistry has sustained in his death. His end was very premature indeed, and it is with a deep sense of bereavement that this occasion is taken to pay a brief word of tribute to him.

Leonard Merritt Liddle, son of Stockwell and Belle Watts Liddle, was born at Mt. Vernon, Iowa, on September 11, 1885. He received his preparatory education at Cornell College Academy and was graduated in 1902. He then entered Cornell College, from which institution he received the degree of B.S. in 1906. It was while a student at Cornell that he became



interested in chemistry; and after serving as principal of the Delhi, Iowa, public school during the year 1906-7, he matriculated as a graduate student in organic chemistry in the Sheffield Scientific School of Yale University. While at "Sheff," he brilliantly pursued organic research under the direction of Prof. Henry L. Wheeler, centering his inquiries on the pyrimidines; and the following is a list of the joint papers of Wheeler and Liddle:

"Researches on Pyrimidines: Thio Derivatives of Uracil and the Preparation of Uracil in Quantity," *Am. Chem. J.*, 40 (1908), 547-558.

"Researches on Pyrimidines: Synthesis of Uracil-4-Acetic Acid," *J. Am. Chem. Soc.*, 30 (1908), 1156-1160.

"Researches on Halogen Amino Acids: Iodine Derivatives of Paratoluidine; 3,5-di-iod-*p*-Aminobenzoic Acid," *Am. Chem. J.*, 42 (1909), 441-461.

Liddle's graduate minor was in physiological chemistry (under Prof. Frank R. Underhill) and he maintained a lifelong interest in biochemical subjects. During the summer of 1908 he was the expert analyst of the Referee Board "Poison Squad" stationed at New Haven, under the supervision of Dr. Russell H. Chittenden, and immediately after he received his Ph.D. in June 1909, he joined the research staff of the Connecticut Agricultural Experiment Station. He spent one productive year there with Dr. Thomas B. Osborne, in investigatory work on the constitution of the proteins, and the results of his studies were reported in the following papers:

"Notes on the Analysis of Edestin and Zein," *Am. J. Physiol.*, 26 (1912), 295-304.

"Separation and Estimation of Aspartic and Glutamic Acids," *Am. J. Physiol.*, 26 (1912), 420-426.

In September 1910 Liddle went to Grinnell College as instructor in chemistry, and three years later he accepted an Industrial Fellowship at the Mellon Institute of Industrial Research, where he spent the remainder of his short life in conducting techno-chemical investigations on vegetable and animal

oils, fats, soaps, and perfumes. His researches on the refining and hydrogenation of vegetable oils (1913-16) were conspicuously valuable from an industrial standpoint, and his later work on soaps (1917) and perfumes (1918-1920) showed consistently high research ability and also resulted in the development of several processes of commercial importance. Liddle published the following papers after becoming a member of the Mellon Institute:

"Iron Carbonyl in Water Gas," *J. Ind. Eng. Chem.*, 8 (1916), 89-90.

"Occurrence of Azelaic Acid as a Product of the Spontaneous Oxidation of Fats" (with B. H. Nicolet), *J. Ind. Eng. Chem.*, 8 (1916), 416-417.

Soon after coming to the Institute, Liddle found his place and at the time of his demise he was indisputably among the foremost—in efficiency and loyalty—of the Industrial Fellows. Constantly capable of research of high order, and always alert to give generously of his profound knowledge of his specialties, he was nevertheless keenly eager for new facts and suggestions and in readiness for the revision of his opinions; and to this unflinching freshness of spirit—a candid professional vivacity which inspired ready esteem—was joined a sagacity which, naturally great, had been strengthened by a decade most fruitful in research experience. To those members of the Institute who enjoyed his friendship even in a slight way the sense of loss has been keen, for he was a very lovable man. At the time of his death he was serving as president of the Robert Kennedy Duncan Club, the organization of the Industrial Fellows of the Institute.

It has been a bitter thing to lose a research chemist of this mold, just in the prime vigor of life, and at a time when his constantly growing investigational power seemed to be making research, ever easy and pleasant to him, generously recognitory of his devotion to it. To have known such a man is an experience one cannot forget. To have had him pass away is a real misfortune to science.

W. A. HAMOR

## PERSONAL NOTES

Dr. A. S. Loevenhart, professor of pharmacology and toxicology at the University of Wisconsin, was elected president of the Pharmacological Society at the annual meeting held in Cleveland recently.

On the alumni lectureship in chemistry, Oberlin College has had Dr. W. D. Bancroft, chairman of the division of chemistry, National Research Council, lecturing on "Colloid Chemistry," and Mr. Marsh, of the Hercules Powder Co., lecturing on "High Explosives."

Dr. Chase Palmer, chemist of the U. S. Geological Survey, has resigned to accept a position as chief chemist in the fuel oil department of the Southern Pacific Co., Bakersfield, California.

The Chemists' Club has devised a plan by which depreciated German marks may be disposed of at the price originally paid. Those interested should communicate with the Club secretary, J. R. M. Klotz, 120 Broadway, New York City.

Dr. A. A. Noyes has resigned as professor of theoretical chemistry and director of the research laboratory of physical chemistry at the Massachusetts Institute of Technology to undertake similar duties at the California Institute of Technology.

Mr. W. L. Savell, chemical superintendent and later general superintendent of the Metals-Chemical, Ltd., plant at Welland, Ontario, and Mr. J. G. G. Frost, chief chemist and later chemical superintendent of the same firm, have opened a consulting office at Cleveland, Ohio.

Mr. Neil Kershaw, formerly with Procter & Gamble Co., Ivorydale, Ohio, as a member of the chemical division, has taken a position as chemist with the American Hominy Co., of Indianapolis, Indiana.

Mr. Howard Grafton, formerly chemist at the Marietta Plant of the Lavino Furnace Co., Marietta, Pa., has accepted a position with the Trumbull Steel Co., Warren, Ohio.

Mr. George A. Rankin, who resigned from the Chemical Warfare Service last July, recently returned to the Geophysical Laboratory to undertake further researches on cements.

Capt. Ross H. Dickson, Ordnance Department, U. S. A., has been recently discharged from active service and is now with the Standard Oil Co. of New Jersey as chemical engineer in the manufacturing development department.

Mr. Oscar R. Smith has resigned as assistant chief chemist of the Standard Chemical Co., Canonsburg, Pa., and has been appointed chemist and engineer of tests for the B. R. & P. Railway Co., United States Railroad Administration, stationed at DuBois, Pennsylvania.

Dr. Phoebe A. T. Levene, of the Rockefeller Institute for Medical Research, in New York, was elected associate member of the Société Royale des Sciences Médicales et Naturelles of Brussels, on December 1, 1919.

Dr. Paul Sabatier (Toulouse) and Dr. Pierre Paul Emile Rous (Paris) have been elected honorary members of the British Royal Institution.

Dr. Francis E. Engelhardt, milk inspector and city chemist of the City of Syracuse, N. Y., has retired. Dr. Engelhardt came to Syracuse as chemist for the Onondaga Salt Co., and while filling this position he was appointed state chemist for the Onondaga Salt Springs reservation. He became an expert on the salt deposits of this part of the state, and as such has had his share in building up the salt industry of the country. In 1877, while still holding his position with the Salt Company, Dr. Engelhardt became chemist for the City of Syracuse.

Mr. John J. Carty, president of the American Telephone and Telegraph Co., delivered a lecture on "Science and the Industries," which is the first of a series which will be held under the auspices of the National Research Council in Washington, D. C., for the purpose of stimulating interest in broad, scientific research, and to emphasize the vital connection between so-called scientific and industrial research.

Prof. Chas. H. LaWall, of the Philadelphia Section of the American Chemical Society, has received the honorary degree of Doctor of Pharmacy from the University of Pittsburgh, Pittsburgh, Pennsylvania.





Mr. Edward G. Harris has left the National Aniline & Chemical Co., Buffalo, N. Y., and has taken a position with the Niagara Sprayer Co., Middleport, New York.

Mr. H. B. Rodeman, formerly Captain in the Ordnance Department, U. S. A., stationed in Washington, D. C., has been for a number of months head of the chemical laboratory of the Duratex Co., Newark, New Jersey.

Miss Elizabeth A. Trembath, formerly with the H. J. Heinz Co., Pittsburgh, Pa., and more recently during the war emergency with the Atlas Powder Co. at their Reynolds Plant, is now process chemist for the Roessler & Hasslacher Chemical Co., at their bleaching station, Perth Amboy, New Jersey.

Mr. E. B. Clark has left the laboratories of the Ward Baking Co., and has taken charge of the laboratories of the Omaha Flour Mills Co., Omaha, Nebraska.

Mr. H. H. Tebault, formerly in the scientific department of the Pompeian Oil Co., now has charge of the business of Mead Johnson & Co., of Evansville, Ind., on the Pacific Coast, including Hawaii.

Mr. L. M. Tanner, formerly in the Chemical Warfare Service, accepted a position as superintendent of States Metal Co., Inc., Mellenville, N. Y.

Mr. C. L. McDonough has left the employ of the American Alloy Chemical Co., as chemist, and has associated himself as chemist for the Warman Steel Castings Co., of Los Angeles, California.

Mr. Frantz F. Berg, formerly chief chemist with the Burroughs Bros. Manufacturing Co., Baltimore, Md., is now in charge of the pharmaceutical division of E. R. Squibb & Sons, Brooklyn, New York.

Mr. Samuel C. Levy has severed his connections as plant inspector for the Pennsylvania State Highway, and has become research chemist for the Barrett Manufacturing Co., Shady-side, New Jersey.

Mr. R. L. Comstock has left the employ of the Brown Co., Berlin, N. H., and has entered that of the Warner Chemical Co., Carteret, N. J., as plant chemist.

Mr. J. F. Johnson, formerly chemical superintendent of the Procter & Gamble Co., Cincinnati, O., has been appointed head of the chemical division of Lever Bros. Co., Cambridge, Mass.

Mr. B. F. Twining resigned as laboratory director of the Nixon Nitration Works, New Brunswick, N. J., to accept a position with the Barrett Company, Frankford, Philadelphia, Pennsylvania.

Dr. Warren K. Lewis has been appointed head of the course of chemical engineering at the Massachusetts Institute of Technology, succeeding Dr. William H. Walker who has also resigned as chairman of the administrative committee of the Institute in order to devote his entire time to the new division of industrial cooperation and research. Prof. H. P. Talbot has been appointed chairman of the administrative committee.

Mr. Gordon MacLean, formerly superintendent of the Mantle Factory, Welsbach Co., Gloucester, N. J., has become factory superintendent for Richard Hellmann, Inc., manufacturers of pure food products, Long Island City, New York.

Dr. Henry Leffmann, of the Philadelphia Section of the American Chemical Society, has been elected a member of the faculty of the Philadelphia College of Pharmacy as special lecturer on research.

Mr. Carleton W. Reade, formerly chief chemist for the Delta Chemical Co., Wells, Delta Co., Mich., is now with the Marinette and Menominee Paper Co., Marinette, Wis., as chemist.

Mr. M. Harold Smith has resigned from the department of metallurgical research at the University of Utah, Salt Lake City, Utah, and has accepted a position as smoke abatement demonstrator for the Smoke Investigation Commission of Salt Lake City, Utah.

Mr. Russell B. Stoddard, formerly in the research laboratory of the National Aniline and Chemical Co., Buffalo, N. Y., is now research chemist for the Palatine Aniline & Chemical Co., Poughkeepsie, N. Y.

Mr. Philip Drinker, formerly in the technical section of the Air Service, A. E. F., is now with the Buffalo Foundry & Machine Co., Buffalo, N. Y.

Mr. I. W. Hazard resigned as laboratory director of the Gibbs Preserving Co., Baltimore, Md., to take a similar position with the Red Wing Co., of Fredonia, New York.

Mr. Frederic J. Le Maistre has severed his connection with E. I. du Pont de Nemours & Co., Wilmington, Del., and has entered into partnership with Wallace P. Cohoe, of New York.

Dr. George D. Van Epps, formerly nonferrous and oil chemist for the Continental Motors Corp., Muskegon, Mich., is at present analytical chemist for the Citro Chemical Co., Maywood, N. J.

Mr. Harris D. Hine line resigned as research engineer with the Westinghouse Electric & Manufacturing Co., to become a member of the faculty of the Science School of the Carnegie Institute of Technology, department of chemistry, Schenley Park, Pittsburgh, Pennsylvania.

Mr. Ralph W. Bridges, formerly research chemist with the International Nickel Co., and more recently metallurgical chemist in the U. S. Navy, Bureau of Steam Engineering, accepted a position in the technical department of the Aluminum Co. of America, New Kensington, Pennsylvania.

Mr. S. L. Calechman resigned as manager of the chemical and dyestuff department of the Nemours Trading Corp., to enter business for himself, with office in New York City, under the firm name of S. L. Calechman & Company.

Mr. John P. Trickey, instructor in chemistry at Northwestern University prior to service in the Chemical Warfare Service, recently accepted the position as research chemist for the Miner Laboratories, Chicago, Illinois.

Mr. Byron A. Soule, formerly chemist at the Dye Works of E. I. du Pont de Nemours & Co., Carney's Point, N. J., is now first assistant to the director of the chemical laboratory of the University of Michigan, Ann Arbor, Michigan.

Mr. Melvin B. Danheiser closed his relations as general manager of the Park Chemical Co., Cincinnati, O., and accepted a position with the Imperial Drop Forge Co., Indianapolis, Ind., as metallurgical engineer.

Dr. Frank C. Whitmore resigned his position at the University of Minnesota and is now a member of the chemistry department of Northwestern University, Evanston, Illinois.

Mr. Nathan Smith, formerly connected with the R. J. Strassenburgh Co., Rochester, N. Y., as chief chemist, is now chief chemist and superintendent of the plant of the Drug Products Co., New York City.

Mr. Simon Collier left the Boston Belting Co., Boston, Mass., where he was a rubber chemist on factory research and chemical problems, to return to the Bureau of Standards, Washington, D. C., to assume charge of the Rubber Chemical Section, Mr. S. E. Epstein having resigned because of illness at home.

Mr. Edward S. Chapin, of Boston, sailed recently for Paris to succeed Mr. H. G. Stephenson as the representative of the Textile Alliance in the matter of securing vat dyes from Germany.

Dr. R. L. Jickling has resigned as research chemist with the Newport Co., Carrollville, Wis., to enter the employ of the National Aniline & Chemical Co., Inc., New York City.

Mr. Albert E. Goldstein, formerly assistant chemist in bituminous materials with the Bureau of Standards, Washington, D. C., has accepted a position as assistant chemist with the Barber Asphalt Paving Co., Madison, Illinois.

Mr. Frank L. Wright resigned as metallographist with the New Departure Manufacturing Co., to take a position as factory metallurgist with the Atlas Ball Co., Philadelphia, Pa.

Mr. Murray J. Etkins, 21 years old, of 1352 Forty-sixth St., Brooklyn, N. Y., assistant chemist at the Picatinny Arsenal, six miles north of Dover, N. J., was killed recently while trying to board a moving train.

Mr. C. L. Wiswall, for several years connected with the Hercules Powder Co., in various capacities, especially in relation to solvent recovery work, recently accepted a position as production engineer with the Vulcan Detinning Co., Sewaren, New Jersey.

Mr. Paul Pierce recently resigned as chemist and metallurgist for Savage Arms Corp., Utica, N. Y., to go with the Nordyke-Marmon Co., of Indianapolis, Ind., as chief chemist.

Mr. Charles H. Plantz, formerly connected with the Brass Rolling Mill of J. B. Wise, Inc., Watertown, N. Y., as chief chemist, has accepted a position in the engineering department of the Edison Lamp Works of the General Electric Co., at their Harrison Plant.

Mr. H. Rossbacher has assumed the duties of chief chemist of the Chicago Paving Laboratory and will supervise the routine work of the laboratory in addition to continuing investigations on the technical utilization of bituminous materials which he has been conducting for this organization since last August.

Mr. A. R. Stevenson has severed his connections with Schaar & Co., and is now with the Deegan Supply Co., Chicago, Illinois.



Prof. Carl Raymond McCrosky, formerly instructor in qualitative analysis at Oregon Agricultural College and later at the College of the City of New York, now has charge of qualitative analysis at Syracuse University, Syracuse, N. Y.

Prof. Leon A. Congdon, who was chief chemist of the State Division of Foods and Drugs of the Kansas State Board of Health and who later served in the Sanitary Corps of the U. S. Army, has taken charge of the quantitative work at Syracuse University, Syracuse, New York.

Mr. N. Gales has resigned as chemist at the United States Naval Engineering Experiment Station, Annapolis, Md., and is at present in business with his brother in New York City where they are representing the Trico Chemical Co., Inc., of Buffalo, New York.

Prof. Charles Wilson Easley, who was, respectively, associate professor, professor, and head of the department of chemistry at the University of Maine, is at the present time professor of chemical engineering at Syracuse University, taking charge of the new department of chemical engineering.

Mr. Maximilian Toch has been appointed adjunct professor of industrial chemistry in the new course at Cooper Institute, New York City.

Mr. Chauncey D. Snow, formerly assistant director of the Bureau of Foreign and Domestic Commerce, and until recently commercial attaché representing the Department of Commerce in Paris, has been appointed manager of the Foreign Commerce Department created by the National Chamber.

Mr. Harvey R. Dunbar has resigned as chemist with the United States Bureau of Mines, Washington, D. C., to become chemist with the Stonega Coke & Coal Co., Appalachia, Virginia.

Mr. Ben E. Watkins, formerly government airplane inspector at Aviation Repair Depot, Montgomery, Ala., now holds a technical position with the Aluminum Company of America, Maryville, Tennessee.

Mr. Arthur Myron Breckler, vice president and chief chemist of the Eflorose Sugar Co., Cincinnati, O., was killed in an automobile accident on January 28, 1920. Mr. Breckler was a native of Bay City, Mich., and was in his thirty-fifth year. He was a graduate of Drake University, and had also studied for one year at the University of Chicago.

Dr. Harry A. Curtis has resigned as professor at Northwestern University, Chicago, to accept a position with the International Coal Products Co., New York City.

Mr. Albert Hugh Bryan, chief chemist of the firm of Arbuckle Brothers, New York City, and a member of the American Chemical Society, died on January 20, 1920, of influenza, in his forty-sixth year.

Dr. Charles J. Moore, associate professor of chemistry at Hunter College, New York City, has been granted a leave of absence for one year to undertake important research work in the government laboratories in Washington, and Dr. Minnie A. Graham, who has been librarian and abstractor for the research department of the General Chemical Co., New York City, has been appointed as substitute for Dr. Moore.

The Chemical Society of the College of the City of New York is pleased to announce the following lectures for the Spring semester of 1920 to be delivered in the Doremus Lecture Theatre: HELIUM, March 29, at 4.00 P.M. Dr. R. B. Moore, chief chemist, U. S. Bureau of Mines. (Lantern); ROMANCE OF LEATHER MAKING, April 13, at 4.00 P.M. Mr. R. S. Bonsib. (Lantern); WATER SUPPLY AND DRAINAGE IN WAR CANTONMENTS, April 21, at 4.00 P.M. Prof. D. D. Jackson, administrative head, department of chemical engineering, Columbia University. (Lantern); MODERN COKE AND GAS MANUFACTURE (Kopper's Process) April 29, at 4.00 P.M. Mr. E. L. Crowe. (Lantern and Cinema); CHEMICAL NAVAL WARFARE, May 7, at 4.00 P.M. Prof. James Kendall, professor of chemistry at Columbia University; FIGHTING FIRE-FIREFOAM, May 14, at 4.00 P.M. Mr. F. A. Epps, chief engineer, the Foamite Firefoam Co. (Experimental, cinema, and lantern.)

Mr. James Gayley, recipient of the Perkin Medal of the SOCIETY OF CHEMICAL INDUSTRY in 1913 for his work in chemical metallurgy, died at his home in New York City on February 25, 1920.

Mr. H. R. Cary has resigned his position as director of Control Laboratories with E. R. Squibb & Sons to accept the position of general manager with A. L. van Ameringen, Perfumer, of New York City.

Miss Clara Bracken McMillen, of Ft. Wayne, a junior in Indiana University, was awarded the Alpha Chi Sigma Scholarship Medal in the competitive examination for 1919.

Mr. Ralph L. Dodge received appointment as junior chemist for the War Department a short time ago and is now employed as research assistant at the Fixed Nitrogen Research Laboratory, American University, Washington, D. C.

Mr. Albert D. Brokaw, formerly associate professor of economic geology and mineralogy at the University of Chicago, has opened a New York office in coöperation with four partners, under the name of Brokaw, Dixon, Donnelly, Garner & McKee, for the practice of his profession as consulting geologist.

Mr. Harry C. Helmlinger, who was formerly with the Dye Products and Chemical Co., of Newark, as chief chemist, recently became associated with the Monsanto Chemical Works in St. Louis, where he is assisting the president.

Dr. H. S. Riederer, who was formerly engaged in work on commercial chemical development at the Experimental Laboratories of the Atlas Powder Co., is now with the United Color & Pigment Co., Newark, New Jersey.

Mr. LeRoy Weil, formerly with the General Motors Corp., research laboratory, Detroit, Mich., and for a short time with the Gillespie Motor Co., of Paterson, N. J., is at present with the Hyatt Roller Bearing Co., Harrison, N. J., in the engineering department.

Mr. A. L. Meader has resigned as chemist with the Bureau of Chemistry, Food and Drug Inspection Station, New Orleans, La., to accept the position of chemist for the Southern Tobacco Products Co., Clarksville, Tennessee.

Mr. C. Harvey Sorum is now a member of the instructing staff in the department of chemistry and chemical engineering at the Massachusetts Institute of Technology, Cambridge, Massachusetts.

Mr. M. E. Pennington, formerly chief of the food research laboratory, Bureau of Chemistry, U. S. Department of Agriculture, has become manager of the research and development division of the American Balsa Co., Inc., of New York City.

Dr. Horace C. Porter, formerly of the Bureau of Mines, and later chemical engineer with the Koppers Co., Pittsburgh, and the Chemical Service Laboratories, Inc., Philadelphia, has opened an office and laboratory in Philadelphia, Pa., for consulting work in fuel testing and research.

Mr. Holland B. Slusser has resigned as chief chemist of the Hodgman Rubber Co., Tuckahoe, N. Y., to become vice president and treasurer of the Pocono Rubber Cloth Co., Trenton, New Jersey.

Dr. E. C. Sullivan has been elected vice president of the Corning Glass Works, Corning, New York.

Mr. H. M. Williams has severed his connection with the Remington Arms U. M. C. Co., to accept a position as research engineer with the General Motors Corp., Dayton, Ohio.

Mr. Alfred Watt has left the S. S. White Dental Manufacturing Co., Price Bay, N. Y., where he was employed as chemist and assayer and has accepted a position as analytical and plant research chemist in the chlorine division of the Brown Co., Berlin, New Hampshire.

Dr. Clifford J. Rolle, formerly instructor of physical and organic chemistry at the University of Cincinnati, Cincinnati, Ohio, has become research chemist for the Van Schaack Bros. Chemical Works, Chicago, Illinois.

Mr. A. J. Walker has left the Federal Rubber Co., where he was employed in the chemical laboratory, to become chief chemist for the Archer Tire & Rubber Co., of Minneapolis, Minnesota.

Mr. Hugo H. Sommer, formerly instructor in agricultural chemistry at the University of Wisconsin, Madison, Wis., is now with the Northern California Milk Producers' Association in charge of the chemical, bacteriological, and testing department.

Dr. E. P. Wightman, formerly connected with Parke, Davis & Co., Detroit, Mich., has accepted a position as research chemist with the Eastman Kodak Co., Rochester, New York.

Mr. Richard D. Leitch, after eleven months of enforced inactivity following his discharge as 2nd Lieut. in the U. S. A., has accepted an appointment as junior explosives chemist with the Bureau of Mines Experiment Station, Pittsburgh, Pennsylvania.

Mr. H. Stirling Snell has severed his connections with E. I. du Pont de Nemours & Co., where he was located at their dye works at Carney's Point, N. J., and has taken a position as chemist for William Heap & Sons, Grand Haven, Michigan.

Mr. Merrill C. Hart, formerly senior assistant chemist for the Puget Sound Navy Yard at Bremerton, Wash., is at present research organic chemist for the pharmacology department of the University of Minnesota, Minneapolis, Minnesota.



## INDUSTRIAL NOTES

The Bureau of Chemistry of the U. S. Department of Agriculture has announced that decomposition in the materials used in the manufacture of catsup and other tomato products can be detected in all cases by the Howard microscopic count method. Chemical tests are not always effective in detecting spoilage resulting from mold. The Howard method has been used by the Government for over ten years, and is used by many commercial chemists and by practical catsup manufacturers in controlling the purity of their product.

The *Scientific American* reports that an English chemist has demonstrated on a laboratory scale a process which may revolutionize the methods of rubber manufacture, if it proves as successful on a commercial scale. At present the latex must be converted into rubber within 24 hours from the time it is extracted from the tree, to prevent putrefaction, but when treated by the new process it is said that it can be kept for any time. It now takes at least a fortnight to bring raw rubber into condition for transmission to the market, but the new process reduces this time to 30 minutes.

According to *Oil, Paint & Drug Reporter*, gums, among which the most valuable are the tragacanth, are produced from small shrubs which grow all over the mountains of Mesopotamia, Southern Persia. The gums are collected by rather crude methods, and no organized trade appears to exist, due largely to the difficulties of transportation and the wild and unsettled nature of the country.

The U. S. Bureau of Chemistry is investigating the use of domestic sumac, which grows wild in the United States, as a vegetable dyeing and tanning material in place of Sicilian sumac which is now used largely by American dyers on account of its deeper color. Experiments are now being conducted to determine whether an extract of equally good color can be obtained from the domestic sumac, which brings a much lower price than the imported material.

The manufacture of synthetic nitrogen on a large scale has been commenced in Sweden under a new process invented by a Swedish engineer. The company's output is now on the market, and large quantities of ammonium sulfate are being sold.

A problem confronting the granite industry in Vermont is the utilization of the immense quantities of waste granite which have collected. A small amount is used for road construction, and the use of crushed granite for bricks is being investigated. There is a considerable potash content in Barre granite, in silicate form, but recovery on a commercial scale has not been undertaken. Pulverized granite may be used in abrasive soaps, for poultry grit, and as filler in roofing and fertilizer materials, but sufficient markets have not yet been established to consume any quantity of granite, and the Bureau of Mines would welcome suggestions regarding any phase of the problem.

The American Library Association, cooperating with the Special Libraries Association, is planning a movement to further installation of technical and special libraries in industrial and manufacturing plants and associations. The National Paint, Oil and Varnish Association, National Wholesale Druggists' Association, American Drug Manufacturers' Association, International Association of Master Painters, Lithopone Institute, American Dye Manufacturers' Association, American Dye Institute, Shellac Association, American Pharmaceutical Association, American Chemical Society, and American Petroleum Institute will also cooperate in the movement.

The American Pharmaceutical Association has available a sum amounting to about \$450 to be expended after October 1, 1920, for the encouragement of research. This amount either in full or in fractions will be awarded in such manner as will in the judgment of the A. Ph. A. Research Committee produce the greatest good to American Pharmaceutical research. Investigators desiring financial aid in their work will communicate with H. V. Arny, chairman, A. Ph. A. Research Committee, 115 West 68th St., New York, N. Y., before May first, giving their record and outlining the particular line of work for which the grant is desired. The committee will make recommendations to the Association at its meeting in Washington, May 3 to 8, 1920, when the award or awards will be made.

The House has passed the Agricultural Appropriation Bill, including the item of \$77,000 for investigating the chemical and physical character of road materials, and \$192,000 for continuing the government kelp potash plant at Summerland, California. \$80,000 worth of potash produced there has been sold, and the plant is now self-supporting.

The Fairbanks, Alaska, Station of the Bureau of Mines has recently completed two series of tests to determine the comparative steaming value of Alaska lignite and spruce wood, and the resistance of lignite to weathering when stored in piles in the open. The results showed that pound for pound, the value of spruce wood lay between two samples of lignite used, but that a cord of wood is equivalent to more than a ton of lignite. The weathering tests showed that the average loss in weight-through weathering was about 6 per cent, and that the weathering after 14 months was only slightly more than after one month. In large piles, only the surface to a depth of about 5 in. would be affected.

*Dyestuffs* reports results of tests made for the British Admiralty with a combination of coal and petroleum fuel oils for steamship fuel. It has been found possible to combine 35 per cent of pulverized coal with 20 per cent of binder and 45 per cent of oil, thereby saving considerable cost and obtaining increased heat values per barrel.

The U. S. Bureau of Chemistry at Washington has announced that the work on photosensitizing dyes begun during the war for the Bureau of Aircraft Production has made possible the preparation in the United States of all the recognized types: pinaverdol (including Orthochrome T), cyanine, pinacyanol and dicyanine; and of a new type useful for astrophotographic work. The Color Laboratory of the Bureau will place its experience at the disposal of any manufacturer who wishes to prepare these dyes for the American market, and is now prepared to supply the dyes to users at a price fixed by the Secretary of Agriculture.

The Chartered Chemical Works has recently been incorporated in Delaware with a capital of \$1,000,000 for the manufacture of chemicals.

The American Clearing Co. is planning the erection of a turpentine plant at Green Cove Springs, Florida, for the extraction of pine oil, turpentine, and rosin from light wood and stumps.

According to *Commercial Fertilizer*, the recent discovery of large deposits of nitrates and potash in the Poison Hills of Prieska, South Africa, will prevent the necessity of depending on the deposits in Germany and in Chile which have heretofore had almost a monopoly. It is believed that this discovery will prove the most powerful factor in the economic reconstruction of South Africa.

The Bureau of Mines reports the discovery of an extensive asbestos claim in Arizona. The deposit is on an Indian Reservation, and under the present law development work cannot be undertaken. The American Consul at Chungking, China, reports the discovery of asbestos in Yachow and the mountains around Ta-chien-lu, although mineral specialists have not yet investigated the value of the discovery. An asbestos plant in Italy has recently begun the manufacture of asbestos pipe from Portland cement and asbestos, which is said to be lighter than iron pipe, acid proof, and corrosive proof and able to bear 450 pounds pressure per square inch. The cost of manufacture is said to be one-third of the net selling price of English iron pipe. The same company has also begun the manufacture of wall tile similar in composition to the pipe.

The U. S. Bureau of Soils has recently issued a statement in regard to work actually done and in contemplation in connection with investigating within the United States the possible sources of supply of nitrates, potash, and other natural fertilizers. The experimental plans outlined include the utilization of the cyanamide as a fertilizer, converting the war munitions plants to fertilizer factories.

In view of the almost inexhaustible supplies of earth pigments, ochers, and oxides, the manufacture of paints in South Africa promises to develop into an industry of considerable importance. Formerly all the ingredients were imported, and the local output is still far from sufficient to meet demands.

The Bureau of Mines has published a comprehensive mining glossary, covering both technical and local usage in various branches of the mining industry, as well as over two thousand Latin-American mining terms and many obsolete terms which are frequently met in research work. The glossary (Bulletin 95 of the Bureau of Mines) may be obtained from the Superintendent of Documents, Government Printing Office, Washington, D. C., at a cost of seventy-five cents.

The American Forestry Association, after a long investigation, has announced the phenomenon of sugar growing on fir trees in the Province of British Columbia. This sugar is extremely sweet and is valued at \$66 a pound.



According to *Oil, Paint & Drug Reporter*, Sweden has imported dyes heavily since the signing of the armistice, chiefly from England. America has also a dye market in Sweden. The German market is apparently closed as a source of supply although before the war it was the source of practically the total supply. During the war Sweden succeeded in producing some satisfactory dyes, but at almost prohibitive prices.

The British sugar shortage, according to the *Journal of the Society of Chemical Industry*, has led to the proposal to form a company capitalized at £1,000,000 for the manufacture in England of sugar from sugar beets. A large factory is to be built, and it is hoped that the enterprise will be sufficiently remunerative to encourage the growing of sugar beets on a large scale, making the country more self-supporting in regard to the sugar supply, and also benefiting agriculture. Before the war more than 90 per cent of England's sugar supply was imported from foreign countries.

Arthur D. Little, Inc., Cambridge, Mass., is issuing a Biographic Series of pamphlets, the first three numbers of which have been printed and distributed. The subjects covered in these numbers are "Chemical Warfare," "The Production of Alcohol from Sulfite Waste Liquors," and "Industrial Research." Further pamphlets in course of preparation are: "The Automobile and Tractor at the Front," "The Electric Furnace," "Industrial Laboratories," "Molasses," "The Chemical Action of Light," "Woods and Fibers used as Paper Making Materials."

According to the *Journal of Commerce*, 47 companies were organized in January 1920 for the manufacture of chemicals, drugs, and dyes, an increase of 32 over December 1919. The total capitalization was \$17,943,000 as compared with \$20,145,000 for December. Nine companies were capitalized at \$1,000,000 or more.

The U. S. Department of Internal Revenue has recently announced that by securing a permit, chemical and physical laboratories may use a special denatured alcohol tax-free. This denatured alcohol is made up as follows: To each 100 gal. of 95 per cent grain alcohol are added 10 gal. of pure wood alcohol of specific gravity of not more than 0.810 at 60° F.

Damage of considerably over \$100,000 was caused by a fire which broke out recently at the Works Laboratory of Toch Brothers at Long Island City, N. Y., destroying the Dry Color and Chemical Works, together with several tons of fine chemicals which cannot be replaced at this time. This is the first extensive fire which Toch Brothers have had in seventy-two years.

The Tariff Commission has recently made a report to the Senate Finance Committee on the cost of manufacturing dyes in this country, embodying the results of a special investigation of the costs of thirty typical coal-tar products in 1918 and 1919. The report shows that the cost of producing these dyes in this country is from two to five times greater than the German selling prices before the war, but that they are lower than the prices at which the reparation dyes were offered, figuring the mark at par.

At the annual meeting of the stockholders of National Aniline & Chemical Co., Inc., held Feb. 16, 1920, the following directors were elected: Orlando F. Weber, H. H. S. Handy, W. N. McIlravy, W. G. Beckers, L. C. Jones, C. S. Lutkins, Henry Wigglesworth, W. J. Matheson, T. M. Rianhard, R. C. Taggesell, and F. M. Peters. The following officers of the company have been appointed: President, O. F. Weber; Vice Presidents, W. G. Beckers, J. W. Newlean; Acting Treasurer, W. H. West; Secretary, H. F. Atherton; Assistant Treasurers, H. S. Trott, T. S. Baines; Assistant Secretary, R. V. Mahon. A quarterly dividend of one and three-fourths per cent on preferred stock has been declared for the period ending March 31, 1920.

The Textile Alliance, Inc., announced on March 9, that 543 separate consignments of German dyestuffs have been held up at Rotterdam by the longshoremen's strike. 438 additional consignments are ready for shipment at the factories and will be forwarded as soon as strike conditions permit, or when arrangements can be made for shipment via another port.

The House bill relative to American participation in carrying out the trade-mark convention of Buenos Aires has recently been passed by the Senate, having been unanimously favored by the Patent Committees of both House and Senate. This legislation does not affect the domestic rights of anyone, but enables manufacturers to register their trade-marks in this country for the purpose of complying with legislation in foreign countries, which necessitates registration in the United States as a preliminary for such foreign registration. Under the present law, trade-mark pirates in foreign countries can register trade-marks of American manufacturers and thus levy blackmail upon them.

The National Aniline and Chemical Co., Inc., has withdrawn from the jobbing of all goods not of its manufacture, and will confine sales efforts to products of its own factories: dyestuffs, intermediates, food colors, coal-tar drugs, and related products.

Financial statements of the Semet-Solvay Co. and the By-Products Coke Corporation, of Syracuse, N. Y., which have just been issued show that neither company was able to pay the dividends declared from the net earnings during 1919 and that it was necessary to draw upon the surplus in order to pay the dividends. Strikes during 1919 caused losses of approximately \$750,000 to the Semet-Solvay Co. H. H. S. Handy is president of both companies.

The report of the Bureau of Chemistry of the U. S. Department of Agriculture on the stocks of turpentine and rosin in the storage yards and in the hands of large dealers and jobbers throughout the country on February 2, 1920, shows a decided decrease since March 31, 1919, and indicates that the stocks will be even smaller on March 31, 1920. Stocks of some concerns manufacturing paint and varnish have not been included, but will be included in a later report covering stocks in the hands of consumers. The following table shows a comparison of stocks on hand:

	March 31, 1919	February 2, 1920
Turpentine, casks.....	131,288	50,926
Rosin, barrels.....	430,724	262,289

The Bureau of Mines of the Department of the Interior has made a thorough investigation of the effect of the removal of gasoline on the value of natural gas, and has reached the conclusion not only that the gas is not impoverished, but that removal of the gasoline is an actual benefit to the consumer. A gallon of gasoline burned in gas is worth about 1<sup>3</sup>/<sub>4</sub> cents while to the automobile owner it is worth about 30 cents, and from this source several hundred million gallons of gasoline are secured yearly which is more valuable than straight-run gasoline on account of its low boiling point. When gasoline is not removed, it causes great difficulty by condensation in the pipe lines, which results in large leakages and consequent lowering of pressure. Installation of gasoline plants eliminates most line trouble and gives better service to the public.

Representatives of the chemical and engineering societies and industries at a hearing before the House Committee on Rules on February 24, 1920, urged prompt consideration of the Nolan Bill for reorganization of the Patent Office. They stated that the Patent Office is demoralized on account of the low salaries paid, and that inventors and scientists are seriously discommoded by long delay in action and by action without proper knowledge. Without the efficient cooperation of the Patent Office which would be insured by the Nolan Bill, the American dye and chemical industries will be helpless in the face of French and German competition, according to the representative of the Manufacturing Chemists' Association, inasmuch as delay in action on American patents would admit foreign chemicals without restriction for a six months' period, probably giving them a continuous market here. The Nolan Bill has since passed the House and is awaiting action by the Senate.

At the invitation of the Federal Trade Commission, about 200 manufacturers and dealers in nitrated cellulose, pyroxylin, plastics, fibriloid, viscoloid, xylonite, etc., and also manufacturers and dealers in pyroxylin plastic products from all parts of the country participated in a trade practice submittal in Washington on March 8, 1920. This conference was for the purpose of defining practices which are considered unfair and of assisting in the elimination of methods which those engaged in the business themselves recognize as unfair, and did not mean the squashing of the separate proceedings now on the commissioner's docket.

Arrangements have been made by the Shipping Board whereby sufficient tonnage will be furnished to take care of the importation of Chilean nitrate needed as fertilizer for next season's crops. Approximately 270,000 tons will be brought to this country before July 1, and some of the shipments have already started.

After considerable discussion between importers and refiners, brokers, and others, the New York Produce Exchange has adopted regulations regarding soy-bean oil as the basis of trading by the Exchange.

The House of Representatives has set the appropriation for the next fiscal year of the Bureau of Foreign and Domestic Commerce at the same figure as last year. This is a substantial increase in the amount recommended by the House Committee on Appropriations, but is considerably less than the amount estimated by the Department as necessary for the proper carrying on of its work.



# GOVERNMENT PUBLICATIONS

By NELLIE A. PARKINSON, Bureau of Chemistry, Washington, D. C.

**NOTICE**—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Commerce Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Commerce Reports mailed daily is \$2.50 per year, payable in advance, to the Superintendent of Documents.

## CONGRESSIONAL COMMITTEES

**Dyes and Dyeing.** Public Resolution 21. 1 p. Approved November 19, 1919. To continue control of imports of dyes and coal-tar products.

**Weights and Measures.** House Report 477. 1 p. December 4, 1919. In re House Resolution 408, to accompany House Resolution 408, providing for consideration of House Resolution 9755, to establish standards of weights and measures for the following wheat-mill and corn-mill products, namely, flours, hominy, grits, and meals, and all commercial feeding-stuffs, submitted by Mr. Campbell.

**Zinc Ore.** 37 pp. November 17, 1919. Hearings before subcommittee on House Resolution 6238, to provide tariff and obtain revenue in connection with metal contents of zinc ore.

**Glass.** 71 pp. December 12 and 13, 1919. Hearings before subcommittee on House Resolution 7785, to provide revenue for Government, to establish and maintain in the United States the manufacture of laboratory glassware, laboratory porcelain ware, optical glass, and scientific and surgical instruments.

**Magnesite.** Part 2. 146 pp. January 13, 1920. Hearings before subcommittee on House Resolution 5218.

## NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

**Power Characteristics of Fuels for Aircraft Engines.** Report 47. 35 pp. 1920. (From Fourth Annual Report.)

(1) **Power Characteristics of Aviation Gasoline.** H. C. DICKINSON, W. S. JAMES, E. W. ROBERTS, V. R. GAGE AND D. R. HARPER, 3D.

(2) **Power Characteristics of Sumatra and Borneo Gasolines.** E. W. ROBERTS.

(3) **Power Characteristics of 20 Per cent Benzol Mixture.** E. W. ROBERTS.

The following are from the Fifth Annual Report:

**Glues Used in Airplane Parts with Lists of References.** Compiled by S. W. ALLEN AND T. R. TRUAX. Report 66. 28 pp. 1920. Prepared by Forest Service in coöperation with University of Wisconsin.

**Effect of Kiln Drying on Strength of Airplane Woods.** T. R. C. WILSON. Report 68. 69 pp. 1920. Prepared by Forest Service in Coöperation with the University of Wisconsin.

## TARIFF COMMISSION

**The Acids of Paragraph 1 and Related Materials Provided for in the Tariff Act of 1913.** Tariff Information Series No. 13. 85 pp.

The report is divided into eight parts:

Part 1—*Summary*, in which are cited the portions of the tariff act of 1913 covering the commodities considered in the report, together with a brief summary of the salient facts presented in the more detailed discussion of the individual industries.

Part 2—*The boric acid and borax industry* has been investigated and detailed information pertinent to the tariff is presented.

This includes the uses to which the products are put, the methods and process of manufacture, statistics for domestic production, imports and exports, and available information on domestic competition and international trade.

Part 3—*The citric acid industry* is discussed in a similar manner and special reference made to the foreign and domestic sources of the raw material and the tariff problem involved.

Part 4—*The formic acid industry* has been given like treatment and attention directed to its peculiar tariff problem.

Part 5—*The gallic, pyrogallic, and tannic acids industry* includes the manufacture of three of the acids which are closely related in their raw material and methods of manufacture.

Part 6—*The lactic acid industry* has been given similar treatment with particular reference to new commercial developments and the tariff considerations involved.

Part 7—*The oxalic acid industry* is discussed and all available information in relation to the tariff has been summarized.

Part 8—*The tartaric acid and cream of tartar industry*, like the industries discussed in Parts 3 and 5, depends upon imported raw materials. Detailed information bearing on this and other factors of tariff significance is presented.

## SMITHSONIAN INSTITUTION

**International Catalog of Scientific Literature, Year Ending June 30, 1919.** 5 pp. From Report, 1919.

## PUBLIC HEALTH SERVICE

**Botulism from Eating Canned Ripe Olives.** CHAS. ARMSTRONG, R. V. STORY AND ERNEST SCOTT. Public Health Reports, 34, 2877-2905 (December 19, 1919).

**Health Hazards in the Industries of Niagara Falls, N. Y.** P. M. HOLMES. Public Health Reports, 35, 1-20 (January 2, 1920). The twenty-one industrial plants covered by the report are divided into the following groups: abrasives, chemicals and gases; electrodes and carbon; metals and alloys; miscellaneous. Facts concerning hygienic conditions brought out by this survey indicate that industrial health hazards are preventable.

**Treatment and Disposal of Sewage.** H. B. HOMMON, J. K. HOSKINS, H. W. STREETER AND H. H. WAGENHALS. Public Health Reports, 35, 101-31 (January 16, 1920). Brief descriptions of methods, processes, and structures used in the treatment and disposal of sewage in the United States, with bibliography.

**Prevention of Stream Pollution by Dye and Intermediate Wastes.** E. J. CASSELMAN. Public Health Reports, 35, 167-85 (January 23, 1920). Investigations were made as to properties and disposal treatment of several wastes of the dye and intermediate industries. Laboratory studies were made of the properties of toluidine waste, nitrobenzene waste, mono-ethyl-aniline waste, dye wastes, and lagoon liquor. Treatments that were successful on a 300-gal. scale and that were cheaper than evaporation were found for the lagoon liquor, the mono-ethyl-aniline wastes, and the lake-forming dye wastes; and a method of treating toluidine waste was indicated in laboratory experiments. Specific treatments of wastes were recommended to the Chemical Company of America, based on their proposed production schedule.

**Botulism. Protective Measures and Cautions from the U. S. Bureau of Chemistry, Department of Agriculture.** Public Health Reports, 35, 327-30 (February 13, 1920).

**Studies of the Treatment and Disposal of Industrial Wastes.** Made under the supervision of EARLE B. PHELPS. (3) **The Purification of Tannery Wastes.** HARRY B. HOMMON. Bulletin 100. 133 pp. Issued November 1919.

**Studies of Methods for the Treatment and Disposal of Sewage.** Made under the supervision of EARLE B. PHELPS. **The Treatment of Sewage from Single Houses and Small Communities.**



L. C. FRANK AND C. P. RHYNUS. Bulletin 101. 117 pp. Issued December 1919. The report includes data obtained from operating testing stations at two tanneries. It gives the process of tanning hides and wastes at the two tanneries studied, descriptions of the testing stations, treatment of the wastes, the volume, analysis, and disposal of sludge. Construction data are also given for a plant to treat wastes from a tannery of 100 hides per day capacity, the effect of the tannery wastes studied on nearby water, the effect of untreated and purified tannery wastes on fish life and a bibliography of tannery wastes treatment.

#### HYGIENIC LABORATORY

Drugs. A. G. DUMEZ. Bulletin 119. 316 pp. Paper, 20 cents. November 1919. This is a digest of comments on Pharmacopeia of United States of America (8th and 9th decennial revisions), and National Formulary (3rd and 4th editions), calendar year 1916.

#### BUREAU OF INTERNAL REVENUE

Opium. Regulation 35 relating to importation, manufacture, production, compounding, sale, dispensing, and giving away of opium or coca leaves, their salts, derivatives, or preparations thereof, under Act of December 17, 1914, as amended by Sections 1006 and 1007 of revenue act of 1918, and under Section 1008 of the said Revenue Act of 1918. Revised November 1919. 78 pp.

#### GEOLOGICAL SURVEY

Copper Deposits of Ray and Miami, Arizona. F. L. RANSOME. Professional Paper 115. 192 pp. Paper, 75 cents.

Sunset-Midway Oil Field, California. Part II. Geochemical Relations of Oil, Gas, and Water. C. S. ROGERS. Professional Paper 117. 103 pp. Paper, 15 cents.

Ground Water in Lanfair Valley, California. D. G. THOMPSON. Water Supply Paper 450-B. Contributions to the hydrology of the United States. 22 pp. Issued January 15, 1920. This was prepared in coöperation with the Department of Engineering of the State of California.

Gold, Silver, Copper, Lead, and Zinc in the Eastern States in 1918. J. M. HILL. Separate from Mineral Resources of the United States, 1918, Part I. 8 pp. Issued January 26, 1920.

The total value of the output of gold, silver, copper, lead, and zinc in the eastern states was \$27,517,488 in 1918, a decrease of over 26 per cent from the production in 1917. The total value is distributed among the various metals as follows: Gold, \$14,352; silver, \$106,585; copper, \$4,411,058; lead, \$366,243; zinc, \$22,619,250.

As will be seen from the following table, the decrease in value is due to large decreases in the output of gold, copper, lead, and zinc, and reflects the condition of mining in the eastern states.

MINE PRODUCTION OF GOLD, SILVER, COPPER, LEAD, AND ZINC IN THE APPALACHIAN STATES, 1917-18

	1917	1918	Increase or Decrease
Ore sold or treated, short tons.	2,709,201	2,355,688	-353,513
Gold.....	\$29,227	14,352	-14,875
Silver, fine ounces.....	106,450	106,585	+135
Copper, pounds.....	18,061,068	17,858,535	-202,533
Lead, pounds.....	6,879,053	5,158,329	-1,720,724
Zinc, pounds.....	312,566,805	248,563,192	-64,003,613
TOTAL VALUE.....	\$37,521,025	27,517,488	-10,003,537

Gold, Silver, Copper, Lead, and Zinc in Nevada in 1918. V. C. HEIKES. Separate from Mineral Resources of the United States, 1918, Part I. 48 pp. Issued January 27, 1920.

Gold, Silver, Copper, Lead, and Zinc in Montana in 1918. C. N. GERRY. Separate from Mineral Resources of the United States, 1918, Part I. 38 pp. Published February 26, 1920.

Gold, Silver, Copper, Lead, and Zinc in New Mexico and Texas in 1918. C. W. HENDERSON. Separate from Mineral Resources of the United States, 1918, Part I. 26 pp. Published February 19, 1920.

Gold, Silver, Copper, Lead, and Zinc in Arizona in 1918. V. C. HEIKES. Separate from Mineral Resources of the United States, 1918, Part I. 40 pp. Published February 20, 1920.

Gold, Silver, Copper, Lead, and Zinc in California and Oregon in 1918. C. G. YALB. Separate from Mineral Resources of the United States, 1918, Part I. 55 pp. Published February 24, 1920.

Gold, Silver, Copper, Lead, and Zinc in Idaho and Washington in 1918. C. N. GERRY. Separate from Mineral Resources of the United States 1918, Part I. 51 pp. Published February 27, 1920.

Gold, Silver, Copper, Lead, and Zinc in Colorado in 1917. C. W. HENDERSON. Separate from Mineral Resources of the United States, 1917, Part I. 57 pp. Published March 1, 1920.

World's Production of Petroleum. Unnumbered circular, dated January 22, 1920, giving the world's production of petroleum by countries for 1918 and total estimated production since 1857.

Our Mineral Supplies. H. D. McCASKEY AND E. F. BURCHARD, Geologists in Charge. Bulletin 666. 278 pp. Paper, 20 cents. This is a composite of about 30 articles which have appeared separately and have been reviewed previously in THIS JOURNAL.

Mineral Resources of Alaska, Report on Progress of Investigations in 1917. G. C. MARTIN and others. Bulletin 692. 400 pp. Paper, 50 cents. The separate parts of this bulletin have been reviewed from time to time as they appeared.

Bibliography of the Metals of the Platinum Group. Platinum, Palladium, Iridium, Rhodium, Osmium, Ruthenium, 1748-1917. J. L. HOWE AND HOLTZ. Bulletin 694. 558 pp.

Porcupine Gold Placer District, Alaska. H. M. EAKIN. Bulletin 699. 29 pp.

Analysis of Silicate and Carbonate Rocks. W. F. HILLEBRAND. Bulletin 700. 285 pp. 1919. A revised and enlarged edition of Bulletin 422.

Deposits of Manganese Ore in Costa Rica and Panama. Papers by J. D. SEARS. Bulletin 710-C. Contributions to Economic Geology, 1919, Part I. 31 pp. Issued December 30, 1919. The manganese deposits thus far discovered in Costa Rica are all on the Nicoya Peninsula, on the Pacific Coast. They are widespread but mostly of low grade or small extent.

The ore bodies are intimate mixtures of various oxides of manganese. Some of the ore is amorphous and moderately soft, with enough pyrolusite to leave a black "smut" on the fingers; some is very hard and distinctly crystalline. The iron content is generally negligible. The silica is usually not in chemical combination with the ore but is mechanically mixed and occurs in grains and fine stringers. The manganese oxides are found mostly in pockets or troughs along the contact between the red metamorphic rocks and the light-colored sediments.

#### BUREAU OF THE CENSUS

Census of Manufactures, Mines and Quarries, Petroleum and Natural Gas, 1919, Instructions to Special Agents. Prepared under the supervision of E. F. HARTLEY. 80 pp. 1919.

#### BUREAU OF MINES

Report of Committee on Standardization of Petroleum Specifications, Fuel Administration, December 1918. Fuel Administration, Oil Division, Bulletin 2. 10 pp. Reprinted December 1919. This report concerns the standardization of oil specifications.

Report of Committee on Standardization of Petroleum Specifications, Fuel Administration, Effective November 25, 1919. Fuel Administration, Oil Division, Bulletin 3. 11 pp. Reprinted December 1919. Paper, 5 cents. This report concerns the standardization of gasoline specifications.



**Removal of the Lighter Hydrocarbons from Petroleum by Continuous Distillation, with Especial Reference to Plants in California.** J. M. WADSWORTH. Bulletin 162. Petroleum Technology 45. 162 pp. Paper, 50 cents. Issued October 1919. The purpose of this bulletin is to describe the methods of constructing and operating representative types of plants in California used for removing the light hydrocarbons from petroleum by continuous distillation, such plants being commonly known in the trade as topping or skimming plants. Limitations for recording data at the different plants made impossible the determination of a heat balance for each. The author confined himself to a comparison of the relative efficiencies of the distilling apparatus alone by the following method:

$$\text{Efficiency of stills and settings} = \frac{\text{Heat theoretically required}}{\text{Heat content of fuel consumed}}$$

**Waste and Correct Use of Natural Gas in the Home.** S. S. WYER. Technical Paper 257. 23 pp. Paper, 5 cents. Issued January 1920.

#### BUREAU OF STANDARDS

**Recommended Specifications for Turpentine (Gum Spirits and Wood Turpentine).** Circular 86. 11 pp. Paper, 5 cents. Issued February 16, 1920. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification Standardization, October 27, 1919.

These specifications apply both to the turpentine which is distilled from pine oleoresins, commonly known as gum spirits or spirits of turpentine, and to turpentine commonly known as wood turpentine, which is obtained from resinous wood, whether by steam or by destructive distillation. When ordering under these specifications, the purchaser shall specify whether (a) gum spirits or (b) wood turpentine, is desired. When wood turpentine is specified, it may be stated whether steam or destructively distilled wood turpentine shall be furnished.

Turpentine shall be pure and conform to the following requirements:

**APPEARANCE**—Shall be clear and free from suspended matter and water.

**COLOR**—Shall be "standard" or better.

**ODOR**—Shall be characteristic of the variety of turpentine specified and, if desired, shall conform to the odor of the sample agreed upon.

	Max.	Min.
Specific gravity, 15.5/15.5° C.....	0.875	0.862
Refractive index at 20° C.....	1.478	1.468
Residue after polymerization with 38 N H <sub>2</sub> SO <sub>4</sub> :		
Gum spirits:		
Volume (per cent).....	2.0	
Refractive index at 20° C.....		1.500
Wood turpentine:		
Volume (per cent).....	2.5	
Refractive index at 20° C.....		1.495
Initial boiling point at 760 mm. pressure.....	160° C.	150° C.
Distilling below 170° C. at 760 mm. pressure (per cent).....		90

**Recommended Specifications for Zinc Oxide, Dry and Paste.** Circular 87. 8 pp. Issued February 16, 1920. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification Standardization, January 12, 1920. Zinc oxide may be ordered in the form of dry pigment or paste ground in linseed oil. Purchases shall be made on the basis of net weight.

The pigment may be American process zinc oxide, made direct from the ore, or French process zinc oxide, made from spelter. The contract shall state which kind is desired.

The color and color strength when specified shall be equal to samples mutually agreed upon by buyer and seller.

The pigment shall meet the following requirements:

	Max. Per cent	Min. Per cent
Coarse particles retained on Standard No. 200 screen <sup>1</sup> ...	0.0	..

<sup>1</sup> The No. 200 screen is the same as the screen formerly known as 200-mesh.

	American Process Max. Per cent	Min. Per cent	French Process Max. Per cent	Min. Per cent
Zinc oxide.....	..	98	..	99
Total sulfur.....	0.2	..	0.1	..
Total impurities, including moisture.....	2.0	..	1.0	..

The paste shall be made by thoroughly grinding the above pigment with pure raw or refined linseed oil. The paste shall not cake in the container and shall break up readily in oil to form a smooth paint of brushing consistency.

The paste shall consist of:

	Max. Per cent	Min. Per cent
Pigment.....	86	82
Linseed oil.....	18	14
Coarse particles and "skins" (total residue left on No. 200 screen based on pigment).....	0.5	..
Moisture and other volatile matter.....	0.5	..

**Recommended Specifications for Leaded Zinc Oxide, Dry and Paste.** Circular 88. 8 pp. Issued February 16, 1920. Prepared and recommended by the United States Interdepartmental Committee on Paint Specification Standardization, January 12, 1920. Leaded zinc oxide, frequently known as "leaded zinc," consists of zinc oxide and varying amounts of lead compounds. It may be ordered in the form of dry pigment or paste ground in linseed oil. Purchases shall be made on the basis of net weight.

The pigment may be high-leaded zinc oxide or low-leaded zinc oxide. The contract shall state which kind is desired. The color and color strength when specified shall be equal to samples mutually agreed upon by buyer and seller.

The pigment shall meet the following requirements:

	Max. Per cent	Min. Per cent
Coarse particles retained on Standard No. 200 screen <sup>1</sup> .....	0.0	..

<sup>1</sup> The No. 200 screen is the same as the screen formerly known as 200-mesh.

	High-leaded Max. Per cent	Min. Per cent	Low-leaded Max. Per cent	Min. Per cent
Zinc oxide (ZnO).....	..	60	..	93
Water soluble salts.....	1.0	..	1.0	..
Total impurities, including moisture.....	1.5	..	1.5	..

The balance to be normal or basic lead sulfate.

The paste shall be made by thoroughly grinding the above pigment with pure raw or refined linseed oil. The paste shall not cake in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall consist of:

	Max. Per cent	Min. Per cent
Pigment.....	88.0	..
Linseed oil.....	..	12.0
Moisture and other volatile matter.....	0.5	..
Coarse particles and "skins" (total residue left on No. 200 screen based on pigment).....	0.5	..

**Thermal Expansion of Insulating Materials.** W. H. SOUDER AND PETER HIDNERT. Scientific Paper 352. 31 pp. Paper, 10 cents. Issued December 27, 1919.

**Constants of Radiation of a Uniformly Heated Inclosure.** W. W. COBLENTZ. Scientific Paper 357. 7 pp. Paper, 5 cents. Issued January 16, 1920. The object of the paper is to give experimental data on atmospheric absorption as it affects the measurements of the radiation constants.

**Efflux of Gases through Small Orifices.** EDGAR BUCKINGHAM AND J. D. EDWARDS. Scientific Paper 359. 43 pp. Paper, 10 cents. Issued January 28, 1920. A theoretical and mathematical discussion.

**Methods for Computing and Intercomparing Radiation Data.** W. W. COBLENTZ. Scientific Paper 360. 8 pp. Paper, 5 cents. Issued January 31, 1920.

**Distribution of Energy in the Spectrum of an Acetylene Flame.** W. W. COBLENTZ. Scientific Paper 362. 13 pp. Paper, 5 cents.



Preparation and Reflective Properties of Some Alloys of Aluminum with Magnesium and with Zinc. R. G. WALTENBERG AND W. W. COBLENTZ. Scientific Paper 363. 5 pp. Paper, 5 cents. Issued February 12, 1920.

Materials and Methods Used in the Manufacture of Enameled Cast-Iron Wares. H. T. STALEY. Technologic Paper 142. 158 pp. Paper, 20 cents. Issued December 20, 1919. It is the purpose of this paper to deal with the materials and methods employed in the manufacture of enameled cast-iron ware. Methods of calculating enamel formulas and examples of the composition of enamels of various types are given. The defects in enamels, their causes, and the remedies to be employed in overcoming them are discussed from both the theoretical and practical standpoints. Manufacturing methods and equipment used in compounding the enamels, making and preparing the castings, and applying the enamels are also discussed. The treatise deals with the enameling of cast iron by the dry, or American, process.

A Study of the Deterioration of Nickel Spark-Plug Electrodes in Service. H. S. RAWDON AND A. I. KRYNITZKY. Technologic Paper 143. 16 pp. Paper, 10 cents. Issued January 22, 1920.

Properties of American Bond Clays and Their Use in Graphite Crucibles and Glass Pots. A. V. BLEININGER. Technologic Paper 144. 52 pp. Paper, 10 cents. Issued January 28, 1920.

#### DEPARTMENT OF AGRICULTURE

Quality and Value of Important Types of Peat Material. A. P. DACHNOWSKI. Department Bulletin 802. 40 pp. Paper, 10 cents. Issued December 29, 1919. A classification of peat based upon its botanical composition and physical and chemical characteristics that will aid in solving the problem of peat utilization.

A Chemical Study of the Ripening and Pickling of California Olives. R. W. HILTS AND R. S. HOLLINGSHEAD. Department Bulletin 803. 24 pp. Paper, 10 cents. Issued January 24, 1920.

Beet-Top Silage and Other By-Products of the Sugar Beet. J. W. JONES. Farmers' Bulletin 1095. 24 pp. Paper, 5 cents. Issued December 1919.

Report of the Chemist for the Fiscal Year 1919. 24 pp. Issued December 1919.

#### Articles from the Journal of Agricultural Research

Effect of Lime upon the Sodium Chloride Tolerance of Wheat Seedlings. J. A. LECLERC AND J. F. BREAZEALE. 18 (January 2, 1920), 347-56.

Cyanogenesis in Sudan Grass: A Modification of the Francis-Connell Method of Determining Hydrocyanic Acid. PAUL MENAUL AND C. T. DOWELL. 18 (January 15, 1920), 447-50.

Invertase Activity of Mold Spores as Affected by Concentration and Amount of Inoculum. NICHOLAS KOPELOFF AND S. BYALL. 18 (February 16, 1920), 537-42.

#### COMMERCE REPORTS—JANUARY 1920

Research associations in England under the government's plan, whereby \$4,866,500 are made available for industrial research, are becoming more frequent in connection with a number of the industries, and the formation of associations for refractory materials, nonferrous metals and Scottish shale oil has been sanctioned. (Pp. 1-2)

Phosphate deposits have been discovered in the eastern part of Holland and about 30 tons of 15 to 20 per cent phosphate are being produced daily. (P. 44)

The outlook for American dyes in Japan is said to be very good. (P. 55)

The German aluminum factories, many of which were installed during the war, may be doomed to failure unless measures are taken to protect the industry from foreign competition, and unless a way is found for maintaining the use of aluminum as a substitute for copper. (Pp. 73-4)

The beginning of work at the largest sulfite pulp factory in Finland is announced. (P. 85)

The olive oil industry in Palestine suffered a severe setback during the war from which it will take at least 50 years to recover. A description of the manufacture of olive and sesame oils is given. (Pp. 140-1)

A native millet is being used in the Camarines, Philippine Islands, as a substitute for rice. If it is found upon analysis to be thoroughly suitable for food it is believed that the rice problem of that province will be solved. (Pp. 163-4)

Huddersfield is now the most important center of coal-tar color making in Great Britain. A large trade is also done in chemicals, the most recent development being the production of drugs formerly obtained from Germany. (Pp. 169-70)

The entire stock and output of the Greek emery mines at Naxos have been turned over to the French government. (P. 189)

A shortage of phosphates in France is shown in the following table:

SUPERPHOSPHATES	Average Annual Consumption in Normal Years		Average Annual Supply, 1915-1919	
	Amount Tons	Phosphoric-Acid Content Tons	Amount Tons	Phosphoric-Acid Content Tons
Mineral.....	1,950,000	273,000	750,000	105,000
Bone.....	100,000	16,000	50,000	8,000
Bone, dissolved..	10,000	1,000	5,000	600
Slaggy lava.....	390,000	55,000	60,000	8,400
Natural.....	100,000	14,000	20,000	2,800
TOTAL.....	.....	359,000	... ..	124,800

A plan of partition and distribution is urged, whereby at least 80,000 tons per month shall reach France. (P. 219)

The United States has been allotted 379,635 lbs. of Taiwan camphor for the first three months of 1920. (P. 225)

A report of the wood pulp industry in Japan is available. (P. 236)

Advantages and uses of cement fiber as a by-product in cement plants in the United States are pointed out. (P. 283)

The production of camphor in Japan is being encouraged. (P. 312)

The Welsh tin plate trade is experiencing a period of great prosperity consequent upon the diminution of American competition, but a new tin plate competitor is gradually coming to the front in Japan. (P. 384)

The prospects of the palm oil industry are given, showing the average yield of nuts, source and recovery of the oil, trade in kernels, and the scope for new enterprises. (Pp. 386-8)

The phosphate reserves of the Island of Nauru in the Pacific are said to be sufficient to meet the demands of the world for 200 years. The quality of the phosphate is said to be of the highest grade. (P. 427)

The rubber industry in Brazil is considered to be in a critical condition because of the competition of oriental rubber. (P. 454)

Several plants for refining oils for export are being installed in China. (P. 462)

Attention is called to the growing demand for artificial fertilizers created by the agricultural development in China and Japan. (P. 462)

A new dye-making process was exhibited and explained at a recent meeting of the Inventors' Union in London. The colors are fluorescent and are derived from inorganic matter. (P. 467)



Zinc production in Belgium continues to increase slowly in spite of the scarcity and exceedingly high prices of raw materials. (P. 535)

A prospective shortage of tin plate in South Wales is reported, due to the serious lack of material for that industry. (P. 543)

The production of nitrate in Chile during November 1919 decreased almost 50 per cent as compared with that of November 1918. The exports showed even a greater decrease. (P. 559)

Extensive deposits of magnesite and talc which are reported in Manchuria have a great potential value. The magnesite is for the most part too pure to use for making bricks without the admixture of 7 to 8 per cent of iron. (P. 578)

Crude bitumen and limestone highly impregnated with bitumen occur in deposits around Hit, Mesopotamia, in almost unlimited quantities. Before these products can be put upon the market the limestone needs to be burned to make lime and the bitumen refined by boiling. The secrets of both processes are at present known only to local experts. (P. 587)

The International metric system is the sole legal standard of weights and measures in Mexico under the Mexican Law of Weights and Measures. (P. 588)

Swedish imports of chemicals and dyestuffs have been rather large and regular since the armistice was signed. Sweden produced some chemicals during the war with rather satisfactory results but the prices were almost prohibitive. Germany, as a source of supply, has been replaced by England and America. The American chemicals which will most readily find a market are wood rosin, rosin oil, heavy gas-tar naphtha, dead oil, crude carbolic acid, shingle-stain oil, and creosote oil. The prospect for American dyestuffs is regarded as very good. A list of the importations of chemicals in 1917 is given. (Pp. 615-6)

The situation in the German chemical trade is regarded as unsatisfactory because of the continued scarcity and increased prices of coal, soda, glue, spirits, and other materials. (Pp. 615-6)

The Bohemian glass industry has succeeded in maintaining its activity by indefatigable work and perseverance. There is a great shortage of coal and raw materials. Extreme difficulty is experienced in securing soda and potash, and oftentimes even insufficiency of limestone and Saxon sand becomes threatening. (Pp. 624-5)

The importation of good lime to Peru, both hydrated and unslaked, would be a profitable enterprise. (P. 628)

#### SPECIAL SUPPLEMENTS ISSUED IN JANUARY

BRITISH INDIA—53a and 53b TUNIS—72a CHINA—55a

#### STATISTICS OF EXPORTS TO THE UNITED STATES

BRITISH INDIA—Sup. 53a and 53b	CHINA—Sup. 55a	TUNIS—Sup. 72a
Hides and skins	Castor oil	Chemicals
Indigo	Hempseed oil	Drugs
Paraffin wax	Peanut oil	Hide cuttings and glues
Bauxite, crude	Perilla oil	Glue stock
Chemicals, drugs and dyes	Soy-bean oil	Caraway seeds

#### COMMERCE REPORTS—FEBRUARY 1920

All restrictions have been removed from the exportation of graphite in Madagascar. (P. 646)

Argentina's trade in chemicals is reviewed, particular attention being paid to caustic soda, soda ash, chloride of lime, silicate of soda, sulfur, rosin, paraffin wax, white arsenic, chlorate and bichromate of potash, sulfuric acid, glacial acetic acid, pharmaceutical drugs, dyes, and colors. Prior to the war most of Argentina's heavy chemicals were imported from Europe but during and since the war the majority of them have come from the United States. With the advent of peace, the chemical market in Argentina is said to have entered upon a more stable condition. (Pp. 651-2)

The Connaught tunnel is to be lined with concrete, which is

part of the electrification scheme to do away with smoke nuisance. It is also expected that the lining of concrete will eliminate the water seepage which has caused so much trouble and expense. (P. 653)

The Pan-American Financial Conference during its sessions in Washington in January 1920 endorsed the metric system in the following resolution:

XIV. *Resolved*, That the conference recommends that the metric system of weights and measures be universally employed, and that, pending the attainment of that end, articles weighed and marked and shipping documents prepared according to the system of weights and measures now prevailing in the United States, should be accompanied with statements giving the equivalents under the metric system. (P. 661)

Large quantities of sugar that had been withdrawn from human consumption in Germany during the war were used in the manufacture of glycerin for war purposes. A description of the process of production is given. (Pp. 676-7)

Sheet-glass shortage in Canada has become very acute. (P. 684)

The legal measures and weights of Bulgaria are all based upon the metric system. (P. 687)

The production and sale of salt in Ecuador is monopolized by the government which enjoys a good profit therefrom. (P. 696)

Announcement is made of an agreement between Estonia, Lithuania, and Lettland, on the one hand, and the National Metal and Chemical Bank on the other, whereby the bank takes over and disposes on a commission basis of the entire flax output of the countries named. The arrangement on the British side is that the National Metal and Chemical Bank goes into partnership with the local governments to aid the restoration of trade and production. It is reported that the agreement will result also in the reopening of the Provodnik rubber factory at Riga. (P. 709)

A deposit of mollusc marl has been discovered in Finland which is estimated to contain 125,000 tons of usable marl. Chemical analysis shows calcium oxides and 80 per cent carbonate of lime rendering the deposit important as fertilizer. (P. 712)

The French government intends to conduct a campaign in favor of the utilization of crude oil. A concrete oil pipe line is planned from Havre to Paris. (P. 725)

As a result of an official test with benzene as a motor fuel, it is hoped that benzene, together with alcohol, will solve the all-important problem of finding a cheap British-made fuel for power. (P. 725)

The Finnish government is reported to be contemplating the purchase from Germany of machinery for a superphosphate and sulfuric acid plant. (P. 732)

Kwantung Province in China is turning out over 5 per cent of the entire world's production of bismuth. (P. 738)

A revival of the Japanese trade in isinglass is reported. (P. 738)

An account is given of the oil-bearing seeds and nuts of Guatemala, of the castor oil exports, of the oil yield of cohune nuts, and of the difficulties in collecting nuts. (Pp. 778-9)

An oil development in Argentina is rumored. (P. 787)

Two important factors in the development of the Algerian phosphate industry are the improvement of railroads and the methods of extraction. (Pp. 788-9)

To encourage the introduction of home-made articles, a permanent Scandinavian Industry Exposition is contemplated in Copenhagen. (P. 829)

The German Potash Syndicate reports the total production of actual potash for 1919 at 946,000 short tons, of which 264,000 tons were sold abroad, the remainder meeting about 41 per cent of home requirements. A new increase in the schedule of prices of potash for domestic consumption of about 45 per cent is



announced. The only present legal limitation over the syndicate's export activities provides that prices for foreign sales shall not be below the maximum domestic price. The possibility of dumping large amounts of potash in America at low prices seems rather remote, especially in view of the fact that the present production is far below normal. (Pp. 835-8)

A process for producing a cattle food from seaweed is announced from Denmark. The analysis shows the food to be nourishing and the cattle eat it willingly. It can be mixed with oil cakes. (P. 845)

The British Research Association for the Woolen and Worsted Industry is contemplating removing its headquarters from Leeds to Bradford where it will begin with the establishment of a chemical laboratory and a physics laboratory, followed as soon as convenient by the opening of engineering experimental workshops. (Pp. 846-7)

The increasing demand from the United States for supplies of camphor from South China is leading to a very marked activity on the part of all interests concerned in the trade. It is planned to eventually place the industry on a modern basis by the introduction of modern methods. (P. 859)

Magnesite is not available for export from Austria, as the production is limited owing to the shortage of coal. (P. 865)

A customs decision of Chile requires that the formulas of certain pharmaceutical preparations should specify not only the ingredients but also their respective proportions. (P. 881)

A bibliography on petroleum in Latin America has been prepared by the Bureau of Foreign and Domestic Commerce. (P. 905)

It is reported that Canton, China, is to manufacture white newsprint paper to meet the demand of local newspapers. (P. 917)

A new process of making artificial silk is reported in France. The product is known as silk cellulose, is claimed to possess great brilliancy, a remarkable solidity and durability, a touch similar to silk, and absolute imperviousness to water. At the same time, it is no more endangered by fire than the ordinary natural silks. The methods of determining artificial silk and the chemical reactions of various artificial silks are given. (Pp. 920-3)

There is a great demand for copper, leather and other materials in Austria. (P. 925)

A market for wire cloth used in the paper and pulp industry of Finland is reported. (P. 927)

A report on mineral oil as a factor in the United States trade is given, including statistics of imports and exports for the past six fiscal years. Particular emphasis is laid upon the demand for petroleum products during the past few years. (Pp. 932-7)

The necessity of conserving and restocking the timber lands of Quebec is noted. The importance of keeping the raw material for the use of the Canadian pulp and paper plants is emphasized. It was even intimated that the Provincial Government might have to limit the cut of the Quebec forests for the exclusive use of Quebec mills. (P. 943)

The tanning industry in Tientsin, China, is growing rapidly. (P. 966)

The development of the rubber industry in the Island of Hainan, China, promises to be a notable success. (P. 980)

Experiments have been undertaken with a view to utilizing the valuable deposits of white clay in the Ballarat District of Australia. It is believed that most of these deposits are especially suitable for conversion into white earthenware. (Pp. 1000-1)

The revival of the Belgian plate and window-glass industry is said to continue in spite of the shortage of industrial fuel and certain raw materials, notably sodium sulfate, and the transportation difficulties. (Pp. 1006-7)

The final official estimate of India's 1919-1920 indigo crop is 21 per cent below the final 1918-19 figures as to area and 15 per cent below as to yield. (P. 1013)

The International metric system is the sole standard of weights and measures in Switzerland. (P. 1016)

The Latvian government has passed a law introducing the metric system, but the old Russian system of weights and measures may be used where necessary until January 1, 1926. (P. 1017)

Since the armistice the production of potash in Alsace has increased by more than 300 per cent. The following statistics show the production of crude potash by months for the 11 months ending November 30, 1919:

	Tons		Tons
January.....	22,968	August.....	...
February.....	37,793	September.....	40,263
March.....	45,817	October.....	78,878
April.....	52,774	November.....	74,484
May.....	53,196		
June.....	52,916	TOTAL.....	508,138
July.....	49,049		

The mines now in operation in Alsace alone are capable of supplying the world's needs. The transportation crisis in France complicates the shipment of potash to the United States. (Pp. 1027-9)

The stocks of mercury at Idria are accumulating and the following statistics are given for the production of Tuscan and Idrian mercury:

The production of mercury (in tons) in the Monte Amiata (Grosseto-Siena) districts was as follows in the years 1915-1918: Ore—1915, 110,612; 1916, 132,524; 1917, 113,782; metal—1915, 985; 1916, 1,093; 1917, 1,071; 1918, 1,038. Total production of Tuscan and Idrian mercury (metal) in 1918 was: Monte Amiata (Grosseto-Siena) districts, 1,038 tons; Idria (estimate of the Italian Bureau of Mines), 600 tons; total, 1,638 tons. (Pp. 1040-1)

An account of the Galician mineral oil industry is given. The most recent survey gives the petroliferous survey as 47,000,000 tons. The raw petroleum of this district during recent years has given the following approximate results upon refining: 14.8 per cent gasoline; 15 per cent illuminating oil; 33 per cent gas oil; 15.7 per cent oil fats or lubricating oil; 5.5 per cent paraffin; and between 3 and 6 per cent coke, with a small percentage of waste product. Its calorific value is given at 10,770 cal., as compared with 5,000 to 6,000 cal. in coal of good quality. (Pp. 1042-7)

British export licenses for the exportation of all coal-tar products, excepting finished dyestuffs, but including aniline oils and salts, granted before January 1, 1920, are revoked, effective February 21, 1920, but renewal applications will be considered for special cause. (P. 1057)

There is a great need for raw materials in Riga among which the following may be mentioned:

NATIVE	FOREIGN	
Gypsum	Glass sand	Phosphate
Chalkstone	Porcelain clay	Pitch
Animal fats	Chalkstone	Resin
Bones	Dyestuffs	Oil seed
Turpentine	By-products for dyes	Pyrites
Linseed	Crude oil	Rubber goods
Paper and wood pulp	Salts	Tanning material
	Acids	Soda

(Pp. 1084-8)

The extraction of oil from Kauri-gum peat promises to become the most important industry in New Zealand. (P. 1095)

The Polish salt deposits are capable of much more intense exploitation and under proper development could probably become the base of several branches of the chemical industry. The salts of Galicia are composed chiefly of kainite and sylvite. (Pp. 1097-9)



## BOOK REVIEWS

**The Outlook for Research and Invention.** By NEVIL M. HOPKINS. George Washington University. x + 241 pp. and 6 photogravure portraits. D. Van Nostrand Co., New York, 1919. Price, \$2.00.

One of the striking characteristics of the great war was the important rôle which chemistry, and especially the chemistry of poison gases, played in it. Equally striking was "the sudden recognition of the fundamental importance of chemical research by governments, leaders of industry and the man in the street." Because of this a book which states as its purpose "to stimulate a more general interest, not so much in what has been known as Yankee invention, but in the broader and more comprehensive American research, and to add a tiny mite towards lifting its efficiency through indicating its charm, its national worth at this time, the educational requirements to those new in the field and to point out the reasons for loss in efficiency through misconception, intense duplication work and the many snares and pitfalls awaiting the unwary inventor," should be of interest to the chemist and the general reading public as well. Hopkins accomplishes his purposes by quotations from well-known scientists and by drawing upon his own broad experience both before and during the war. After discussing the spirit of research and men of research, he takes up the question of education, some seemingly impossible problems which have been successfully solved, and then discusses industrial research in the factory. The closing chapter relates to the making and protecting of inventions. That the book is written from the viewpoint of a physicist and engineer is seen in the appendix, which lists "problems awaiting solution." According to this list, the following chemical problems await solution: chemical data for the engineering solution of problems, synthetic diamonds and foods, catalysts and the fixation of nitrogen. It would seem that there are many other industrial chemical problems that might well have been included, for the uninitiated may easily have the idea upon glancing through this list that chemistry is practically a closed art.

Perhaps the lesson we need most to learn to-day is found in the chapter on "Research in the Factory." By the use of such examples as the development of nitrocellulose and smokeless powder, the adding machine, and the high tension electrical magneto, Hopkins points out the relation of the investigator in pure science to industrial research. "While it is extremely interesting and upbuilding to think in terms of atoms and molecules, it is equally important to think in terms of large quantities of the chemical compounds that enter into reactions." This research may be carried out in one of three ways. The most expensive, but not the most costly, is the equipment of factories with modern research laboratories and the employment of able specialists. While this method can be used only by large companies, such as the General Electric Company, there are few instances where such laboratories have failed to become self-supporting and to add very materially to the quality and quantity of factory production. And when such laboratories are not abundantly self-supporting, they fail of their purpose. A second method is that proposed by the late Professor Duncan, involving the use of industrial fellowships at universities. The plan has been installed at the Universities of Kansas and Pittsburgh, and has proven satisfactory in many cases. There is only one danger attending this scheme; the university atmosphere pervades it, and the manufacturer who is willing to spend a certain sum of money, but who desires results quickly, often finds the work dragging over a long period of time. The third method, and probably the one most suited to the majority of manufacturing concerns, is to utilize the commercial laboratories which are scattered throughout the country. These are

equipped with men and apparatus capable of solving practically all the industrial problems submitted to them. Since it is their livelihood, they aim to secure results as quickly as is consistent with an adequate solution of the problem, and while the finer scientific points of the investigation must of necessity be left unsolved or turned over to university men who have time for such work, the manufacturer secures information which enables him to install or correct his process and to maintain his production at a desired standard, both of quality and of quantity.

The only disappointing chapter is the one on American War Research. There we looked for some of the problems and achievements of our Chemical Warfare Service and the other scientific organizations which contributed so materially to the success of our army abroad. All we find, however, is the organization of the National Academy of Sciences and the National Research Council.

The book will prove interesting reading to those who desire to know more of that fascinating subject "research," especially as related to physics and engineering.

CLARENCE J. WEST

**Colloids in Biology and Medicine.** By H. BECHHOLD. Translated by J. G. M. BULLOWA. xiv + 464 pp. D. Van Nostrand Co., New York, 1919. Price, \$5.00.

In view of the rapidly increasing recognition of the importance of colloid chemistry, a translation of Bechhold's book is a thing to be thankful for. Although the book deals primarily with biology and medicine, the facts that are given is in many cases of very general application and are consequently of interest to many different readers. The relation between the reactions of gelatin and the processes of tanning is self-evident. The relation of leather to hides is very similar to that of vulcanized to raw rubber. Ore flotation depends on the stabilization of bubbles by selective adsorption. The dyeing of tissues and the dyeing of textiles involve the same general principles. The emulsions of the druggist enable us to duplicate and understand some of the reactions of protoplasm.

The author treats his subject under four main heads: introduction to the study of colloids; the biocolloids; the organism as a colloid system; toxicology and pharmacology. Under the first subdivision, the titles of the chapters are: what are colloids? surfaces; size of particles, molecular weight, osmotic pressure, conductivity; phenomena of motion; consistency of colloids; optical and electrical properties of colloids; methods of colloid research. Under biocolloids the chapters are entitled: carbohydrates; lipoids; proteins; food and condiments; enzymes; immunity reactions. Under the general heading of the organism as a colloid system, we have the special subdivisions: significance of the colloidal condition for the organism; metabolism and the distribution of material; growth, metamorphosis, and development; the cell; the movements of organisms; blood, respiration, circulation and its disturbances; absorption; the nerves.

Bechhold's views in regard to starch and iodine, page 135, are very interesting.

On account of their great surface development, the adsorptive capacity of starches is very great. As has been said, when they swell they adsorb water, dyes, etc. A very characteristic adsorption compound is formed with iodine. Iodine is the best known reagent for starches; by it they are stained blue. It was formerly believed that iodine and starch united chemically; W. Biltz (Küster) showed that it is merely an adsorption. According to the degree of adsorption, iodine solution is blue, red, orange or yellow, inasmuch as the starch solution acts as a protective colloid (W. Harrison). There are in addition varieties of starch which give at once a brownish red or a wine-red color with iodine. Inulin and lichenin are colored yellow by iodine.



It is worth noting, page 178, that there is a wide-spread error that stale bread has lost water and is desiccated. This is not true; the crumbling consistency of stale bread is due to a shifting of the water within the loaf; the starch grains transfer water to the albuminous framework. J. R. Katz studied this problem and found that bread kept fresh longer at 50° to 100° as well as below -10° (best in a current of air); in other words, there is a balance of swelling in starch and gluten which corresponds to that of fresh bread. At from 0° to 25° stale bread is the stable form. Staling is a particularly reversible process; dry rolls are made fresh by heating them. This is an old expedient frequently employed. The results of Katz' research on keeping bread fresh at low temperatures deserves the attention of the trade.

WILDER D. BANCROFT

**Les Amandes et L'Huile de Palme.** By E. BAILLAUD AND A. STIELTJES. xxiv + 346 pp. Institut Colonial de Marseille, 5, Rue Noailles, Marseille, France, 1920.

This volume of 350 pages gives a very complete view of the palm kernel oil industry up to the breaking out of the World War. It goes largely into the conditions of the industry in England, Germany, and France, considering carefully the returns from manufacturing, the capacity of the factories, problems connected with the importation of palm kernel nuts, transportation, the difference in favor of Germany at the breaking out of the war, and the actual situation of the fat and oil industry in England and France at the present time. It also contains a chapter showing the means which should be taken to develop the industry.

The greater part of the book consists of a translation of the report of the Committee on Edible Oil Producing Nuts and Seeds presented to both houses of Parliament by command of His Majesty, June 1916. This Committee made a careful investigation of the industry as it existed in England, and the report gives the minutes of the evidence taken. This translation occupies 255 pages.

The second part of the book goes very carefully into the preparation of the fruit from which the palm oil is obtained. It contains numerous descriptions and cuts of machinery used.

The third part of the book goes very particularly into the mechanical treatment for producing palm oil at Togo and Cameroun.

Part 4 of the book consists of world statistics of fats and oils compiled in 1913. The book is recommended particularly to those interested in the production and manufacture of this special product.

DAVID WESSON

**Analysis of Babbitt.** By JAMES BRAKES. 169 pp. Allen Book and Printing Co., Troy, New York, 1919. Price, \$2.00.

The author states in his preface that this is a small practical book on the analysis and manufacture of Babbitts and that it includes reactions and data, as well as references to many methods of analysis and an extensive bibliography on metallurgical engineering. The data comprise, for a small volume, a remarkably extensive compilation of information on the history, properties, occurrence, metallurgy, uses, and compounds of the metallic elements used in bearing metals; namely, antimony, tin, lead, and copper. These statements are evidently taken from a wide range of sources and indicate an exhaustive search of the literature. They are, however, quoted one after another as a series of notes with nothing to indicate their relative importance. Many references are given, but in many more cases the reader is left to judge of the authenticity and value of the information. Unfortunately, there are many inaccuracies and misstatements.

Very extensive information is given on the occurrence of minute quantities of tin, lead and copper in food products. Undue importance is given to the poisonous nature of the metals

and statements are quoted which appear not to have been fully authenticated.

The title of the book is, in fact, not broad enough to cover its scope, and, although there are separate analytical methods given for each of the four metals, there is no complete method for quantitative analysis of Babbitt metal described consecutively. The methods described are not those generally accepted as desirable.

The bibliography covers references to over one thousand different articles and publications, both in English and other languages, and apparently includes all that was found relating in any way to the analysis and metallurgy of these four metals. This is a valuable list of references and it includes a great many relating to the analysis of Babbitt. However, much the larger number refer to articles with little relevancy to this subject, including many on the detection of traces of metals in food products and in relation to medical diagnosis. With a bibliography of such broad scope it is difficult for one studying a special subject to find the references particularly relating to it.

The last chapter, entitled "Babbitt Metal," contains notes on manufacture, mixing and sampling of Babbitt. Two formulas are given here as No. 1 and No. 2 Babbitt, which are stated to be all that are usually necessary for ordinary work. These are not generally accepted as such.

The book contains grammatical and typographical errors, although the latter are probably no more frequent than would be expected in a first edition from a non-technical press. The arrangement and type of titles and sub-heads is poor.

W. A. COWAN

**Annuario per le Industrie Chimiche e Farmaceutiche—Anno I, 1918.** Compiled for the Committee on the Chemical and Pharmaceutical Industries by Professors NASINI, VILLAVECCHIA, CAPPELLI AND MENEGHINI with the collaboration of Doctors FANTACCHIOTTI AND SILVESTRI. Published by the Minister of Industry, Commerce and Labor. vi + 555 pp. Tipografia Operaia Romana Coöperativa, Via Porta Cavallegeri 7A, Rome, Italy, 1919.

Very tangible evidence of the tremendous increase in Italy's chemical production during the war period is presented by this directory of her chemical and pharmaceutical industries, which is the work of the committee organized in November 1915 to cooperate with the Minister of Industry, Commerce and Labor in the war work of the industry. Data collected from manufacturers by this committee were so obviously of commercial value that it was decided to put it into permanent form. The directory gives an alphabetical list of the manufacturers, their addresses, and the products they manufacture; a similar list of chemical and pharmaceutical products with their makers; statistics of average monthly production, and of imports and exports from 1911 to 1918.

These Italian facts and figures, set down in black and white, remind us very forcibly that the United States is not the only country which has expanded her chemical industry during the war and foretell, too, the keen competition that there will be for chemical and drug products in all of the world's markets. Italian sulfur, argols, tartaric and citric acids, vegetable oils, and citrus essences have long held their place in these markets; but to-day Italy is producing lead acetate, nitric and hydrochloric acids, caustic soda, benzene, and naphthalene (to mention but a few of the products of her war expansion) all far in excess of her domestic requirements.

This carefully arranged directory will surely be of value to the Italian chemical industry in its sales efforts, for it makes immediately available just the information of sources and of output which both the foreign and domestic buyer is continually seeking.

WILLIAM HAYNES



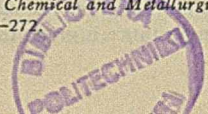
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- Boiler Management: Efficient Boiler Management; With Notes on the Operation of Reheating Furnaces.** C. F. WADE. 8vo. 266 pp. Price, \$4.50. Longmans, Green & Co., New York.
- Chemical Dictionary: Inorganic Chemical Synonyms and Other Useful Chemical Data.** E. R. DARLING. 12mo. 100 pp. Price, \$1.00. D. Van Nostrand Co., New York.
- Chemical Dictionary: Popular Chemical Dictionary.** C. T. KINGZETT. 8vo. 374 pp. Price, 15s. Baillière, Tindall & Cox, London.
- Chemistry: Elementary Practical Chemistry. Part I. General Chemistry.** F. CLOWES AND J. B. COLEMAN. 7th Ed. 8vo. 241 pp. Price, 6s. J. & A. Churchill, London.
- Chemistry: Elementary Practical Chemistry for Medical and Other Students.** J. E. MYERS AND J. B. FIRTH. 2nd Ed. Revised. 8vo. 194 pp. Price, 4s. 6d. Charles Griffin & Co., London.
- Chemistry: Industrial and Manufacturing Chemistry. Part B. Organic. A Practical Treatise.** G. D. MARTIN. 5th Ed. 8vo. 744 pp. Price, 36s. C. Lockwood & Son, London.
- Chemistry: A Treatise on Qualitative Analysis.** FRANK CLOWES AND J. B. COLEMAN. 9th Ed. 8vo. 400 pp. Price, 12s. 6d. J. & A. Churchill, London.
- Dyeing Industry: Being a Third Edition of "Dyeing in Germany and America."** S. H. HIGGINS. 189 pp. 8s. 6d. University Press, Manchester.
- Graphic Charts: How to Make and Use Graphic Charts.** A. C. HASKELL. 8vo. 550 pp. Price, \$5.00. Codex Book Co., 19 William St., New York City.
- Iron: Le Fer et Ses Dérivés. L'Acier dans la Construction Automobile.** JEAN DELESTRADE. 258 pp. Price, 14fr. 50. Dunod, Paris.
- Iron Bacteria.** DAVID ELLIS. 8vo. 179 pp. Price, \$4.00. F. A. Stokes Co., New York.
- Kinetic Theory of Gases and Liquids.** R. D. KLEEMAN. 12mo. 272 pp. Price, \$3.00. John Wiley & Sons, Inc., New York.
- Lime: Right Use of Lime in Soil Improvements.** ALVA AGHE. 12mo. 89 pp. Price, \$1.25. Orange Judd Co., 315 Fourth Ave., New York.
- Metals: La Taille des Métaux.** F. W. TAYLOR. 295 pp. Price, 16fr. 50. Dunod, Paris.
- Milk: Le Lait. Physiologie, Analyse, Utilisation.** A. MONVOISIN. 2nd Ed. Revised and Enlarged. 540 pp. Price, 20 fr. Asselin et Houzeau, Paris.
- Oil and Gas Prospecting: Prospecting for Oil and Gas.** L. S. PANYITY. 249 pp. Price, \$3.25. John Wiley & Sons, Inc., New York.
- Petroleum Register; An Annual Directory and Statistical Record of the Petroleum Industry in the United States, Canada and Mexico, 1919.** Price, \$10.00. Oil Trade Journal, Inc., New York.
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- White-Lead. Its Use in Paint.** A. H. SABIN. 12mo. 133 pp. Price, \$1.25. John Wiley & Sons, Inc., New York.
- Chemistry the Basis of Industrial Progress. An Appreciation of the Work of the Chemist by the Minister of Trade and Commerce.** GEORGE FOSTER. *Canadian Chemical Journal*, Vol. 4 (1920), No. 2, pp. 51-55.
- Clay: Fire Clay Mixtures for High Temperatures.** R. M. HOWE. *Blast Furnace and Steel Plant*, Vol. 8 (1920), No. 2, pp. 157-160.
- Colloid Chemistry in Textile Industries. Part I.** W. HARRISON. *Color Trade Journal*, Vol. 6 (1920), No. 2, pp. 50-55.
- Cyanide: A New Cyanide. Description of the Process for Converting Calcium Cyanamide to a New Cyanide in a Modern Sodium Chloride Solution.** W. S. LANDIS. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 6, pp. 265-268.
- Dyeing of Human Hair.** E. C. GRAY. *Color Trade Journal*, Vol. 6 (1920), No. 2, pp. 63-65.
- Dyestuffs: Colorimetric Evaluation of Dyestuffs.** H. B. GORDON. *Color Trade Journal*, Vol. 6 (1920), No. 2, pp. 61-63.
- Dyestuffs: Developing the Chinese Dyestuff Business.** L. W. ALWYM-SCHMIDT. *Color Trade Journal*, Vol. 6 (1920), No. 2, pp. 35-38.
- Fertilizers: Problems and Processes in Mixed Fertilizers.** C. H. MACDOWELL. *The American Fertilizer*, Vol. 52 (1920), No. 4, pp. 67-72.
- Glass: Glass Industry of North America.** W. E. S. TURNER. *Journal of the Society of Glass Technology*, Vol. 3 (1919), No. 11, pp. 166-200.
- Glass Furnaces: Some Recent Improvements in the Designs of Glassworks Furnaces and Gas Producers.** J. S. ATKINSON. *Journal of the Society of Glass Technology*, Vol. 3 (1919), No. 11, pp. 148-166.
- Leather: Chemistry in the Leather Industry. The Development of Modifications in methods of Tanning through the Applications of Chemical Principles in This Industry.** T. A. FAUST. *The Canadian Chemical Journal*, Vol. 4 (1920), No. 1, pp. 8-9.
- Leather Manufacture: Industrial Uses for the Shark and Porpoise.** ALLEN ROGERS. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 2, pp. 91-101.
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- Mechanism of the Addition of Hydrogen to Unsaturated Glycerides in the Presence of Finely Divided Nickel.** R. THOMAS. *Journal of the Society of Chemical Industry*, Vol. 39 (1920), No. 2, pp. 101-181.
- Methyl Ethyl Ketone from Normal Butyl Alcohol, Manufacture of.** T. C. NEWMAN. *Canadian Chemical Journal*, Vol. 4 (1920), No. 2, pp. 47-48.
- Nitrate of Soda: L'Industrie du Nitrate de Soude au Chili.** ALEJANDRO BERTRAND. *Chimie et Industrie*, Vol. 3 (1920), No. 1, pp. 19-23.
- Nitrogen: The Demand and Supply of Fixed Inorganic Nitrogen in the United States.** A. H. WHITE. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 8, pp. 369-371.
- Oxygen, Nitrogen and Rare Gases from the Air; Modern Separation Methods.** C. R. HOUSEMAN. *The Chemical Age (London)*, Vol. 2 (1920), No. 36, pp. 198-199.
- Pyridine: Recovery of Pyridine from By-Product Coke Ovens.** F. E. DODGE AND F. H. RHODES. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 6, pp. 274-275.
- Steel: Changes in Steel at Certain Temperatures. Effect on Static and Other Properties of Treatment below the Thermal Critical Range.** ZAY JEFFRIES. *The Iron Age*, Vol. 105 (1920), No. 9, pp. 620-621.
- Steel: Effect of Nitrogen on Steel. A Résumé of the Important Literature on Amount of Nitrogen in Various Classes of Steels.** G. F. COMSTOCK AND W. E. RUDER. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 9, pp. 399-405.
- Steel: Effect of Pickling on Alloy Steels; an Investigation to Determine the Degree of Interference with Quality and Machinability of the Material.** H. L. HESS. *The Iron Age*, Vol. 105 (1920), No. 9, pp. 593-594.
- Sulfate of Ammonia: The Caking of Sulfate of Ammonia.** C. G. ATWATER AND J. F. W. SCHULZE. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 8, pp. 373-374.
- Sulfate of Ammonia: Outlook for Sulfate of Ammonia.** C. G. ATWATER. *The American Fertilizer*, Vol. 52 (1920), No. 4, pp. 74-78.
- Sulfate Recovery: Explosion Process of Sulfate Recovery.** H. K. MOORE. *The Chemical Engineer*, Vol. 28 (1920), No. 1, pp. 8-16.
- Water-Softening: A Study of the Lime-Soda Ash Water-Softening Process.** M. R. HERRLE AND F. M. GLEESON. *Chemical and Metallurgical Engineering*, Vol. 22 (1920), No. 6, pp. 269-272.

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- Acid Phosphate: The Manufacture and Distribution of Acid Phosphate.** S. L. CARTER. *The American Fertilizer*, Vol. 52 (1920), No. 4, pp. 61-66.
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- Brannerite, A New Uranium Mineral.** F. L. HESS AND R. C. WELLS. *Journal of the Franklin Institute*, Vol. 189 (1920), No. 2, pp. 225-238.
- Butyl Alcohol: Manufacture of; A Proposed Theory for the Mechanism of the Weismann Process.** T. C. NEWMAN. *Canadian Chemical Journal*, Vol. 4 (1920), No. 3, pp. 76-77.
- Carbon: The Activation of Carbon.** N. K. CHANEY. *The Chemical Engineer*, Vol. 28 (1920), No. 1, pp. 19-22.
- Charcoal: La Crise du Charbon, Ses Causes, Ses Conséquences, Propositions pour l'Atténuer.** CH. BERTHELOT. *Chimie et Industrie*, Vol. 3 (1920), No. 1, pp. 88-96.
- Charcoal Before the War.** W. D. BANCROFT. *Journal of Physical Chemistry*, Vol. 24 (1920), No. 2, pp. 127-146.





**MARKET REPORT—MARCH, 1920**

FIRST-HAND PRICES FOR GOODS IN ORIGINAL PACKAGES PREVAILING IN THE NEW YORK MARKET

INORGANIC CHEMICALS		Mar. 1	Mar. 15	Acid (Concluded)		Mar. 1	Mar. 15
Acid, Boric, cryst., bbls..... lb.	.16	.17		Citric, crystals, bbls..... lb.	1.05	1.15	
Hydrochloric, com'l, 22°..... lb.	.02	.02		Oxalic, cryst., bbls..... lb.	.43	.45	
Hydriodic..... oz.	.19	.19		Pyrogallic, resublimed..... lb.	2.50	2.50	
Nitric, 42°..... lb.	.08 1/2	.08 1/2		Salicylic, bulk, U. S. P..... lb.	.55	.55	
Phosphoric, 50% tech..... lb.	.25	.25		Tartaric, crystals, U. S. P..... lb.	.72	.75	
Sulfuric, C. P..... lb.	.08	.08		Trichloroacetic, U. S. P..... lb.	4.40	4.40	
Chamber, 66°..... ton	22.00	22.00		Acetone, drums..... lb.	.14	.14	
Oleum..... ton	27.00	27.00		Alcohol, denatured, 190 proof... gal.	.77	.82	
Alum, ammonia, lump..... lb.	.04	.04		Ethyl, 190 proof..... gal.	6.00	6.00	
Aluminum Sulfate (iron-free)..... lb.	.03	.03		Wood, 95%..... gal.	2.50	2.75	
Ammonium Carbonate, pwd..... lb.	.13	.13		Amyl Acetate..... gal.	4.00	4.00	
Ammonium Chloride, gran..... lb.	.15	.15		Camphor..... lb.	3.00	2.60	
Ammonia Water, carboys, 26°..... lb.	.10	.10		Carbon Bisulfide..... lb.	.06	.07	
Arsenic, white..... lb.	.12 1/2	.13		Tetrachloride..... lb.	.11	.11	
Barium Chloride..... ton	160.00	175.00		Chloroform, U. S. P..... lb.	.30	.30	
Nitrate..... lb.	.11	.11		Creosote, U. S. P..... lb.	.75	.75	
Barytes, white..... ton	25.00	25.00		Cresol, U. S. P..... lb.	.18	.18	
Bleaching Powd., 35%, Works, 100 lbs.	3.00	4.00		Dextrine, corn..... lb.	.07	.06	
Borax, cryst., bbls..... lb.	.09	.09 1/2		Imported Potato..... lb.	.12	.12	
Bromine, tech., bulk..... lb.	.75	.80		Ether, U. S. P., conc., 100 lbs..... lb.	.19	.19	
Calcium Chloride, fused..... ton	22.00	22.00		Formaldehyde..... lb.	.75	.75	
Chalk, precipitated..... lb.	.05	.05		Glycerin, dynamite, drums..... lb.	.23	.22	
China Clay, imported..... ton	18.00	18.00		Starch, corn..... 100 lbs.	5.35	5.35	
Copper Sulfate..... 100 lbs.	8.00	8.25		Potato, Jap..... lb.	.08	.08	
Feldspar..... ton	8.00	8.00		Rice..... lb.	.25	.25	
Fuller's Earth..... 100 lbs.	1.50	1.50		Sago..... lb.	.05	.05	
Iodine, resublimed..... lb.	4.10	4.10					
Lead Acetate, white crystals..... lb.	.14	.14		<b>OILS, WAXES, ETC.</b>			
Nitrate..... lb.	.15	.15		Beeswax, pure, white..... lb.	.70	.70	
Red American..... 100 lbs.	.10 1/2	.10 1/2		Black Mineral Oil, 29 gravity... gal.	.22	.22	
White American..... 100 lbs.	.09 1/2	.09 1/2		Castor Oil, No. 3..... lb.	.18	.18	
Lime Acetate..... 100 lbs.	2.00	2.00		Ceresin, yellow..... lb.	.14	.14	
Lithium Carbonate..... lb.	1.50	1.50		Corn Oil, crude..... lb.	.19	.19	
Magnesium Carbonate, powd..... lb.	.19	.19		Cottonseed Oil, crude, f. o. b. mill. lb.	.19 1/2	.19	
Magnesite..... ton	65.00	65.00		Menhaden Oil, crude (southern). gal.	.95	.95	
Mercury flask..... 75 lbs.	87.00	92.00		Neat's-foot Oil, 20°..... gal.	2.25	2.25	
Phosphorus, yellow..... lb.	.35	.35		Paraffin, 128-130 m. p., ref..... lb.	.11 1/2	.11 1/2	
Plaster of Paris..... 100 lbs.	1.50	1.50		Paraffin Oil, high viscosity..... gal.	.45	.45	
Potassium Bichromate..... lb.	.34	.34		Rosin, "F" Grade, 280 lbs..... bbl.	17.85	18.55	
Bromide..... lb.	.90	.90		Rosin Oil, first run..... gal.	.97	.97	
Carbonate, calc., 80-85%..... lb.	.24	.24		Shellac, T. N..... lb.	1.40	1.40	
Chlorate, cryst..... lb.	.15	.15		Spermaceti, cake..... lb.	.30	.30	
Cyanide, bulk, 98-99%..... lb.	.30	.30		Sperm Oil, bleached winter, 38°... gal.	1.95	1.95	
Hydroxide, 88-92%..... lb.	.30	.30		Stearic Acid, double-pressed..... lb.	.27	.27	
Iodide, bulk..... lb.	3.35	3.10		Tallow Oil, acidless..... gal.	1.55	1.55	
Nitrate..... lb.	.14	.14		Tar Oil, distilled..... gal.	.60	.60	
Permanganate, U. S. P..... lb.	.75	.80		Turpentine, spirits of..... gal.	1.94	2.04	
Salt Cake..... ton	17.00	18.00					
Silver Nitrate..... oz.	.81 1/2	.78 1/2		<b>METALS</b>			
Soapstone, in bags..... ton	12.00	12.00		Aluminum, No. 1, ingots..... lb.	.32	.32	
Soda Ash, 58%, bags..... 100 lbs.	2.50	3.25		Antimony, ordinary..... 100 lbs.	11.75	11.75	
Caustic, 76%..... 100 lbs.	5.00	7.00		Bismuth..... lb.	2.52	2.52	
Sodium Acetate..... lb.	.25	.25		Copper, electrolytic..... lb.	.19	.19	
Bicarbonate..... 100 lbs.	2.25	2.25		Lake..... lb.	.19 1/2	.19 1/2	
Bichromate..... lb.	.22	.22		Lead, N. Y..... lb.	.09 1/4	.09 1/4	
Chlorate..... lb.	.12	.12		Nickel, electrolytic..... lb.	.45	.45	
Cyanide..... lb.	.27	.27		Platinum, refined, soft..... oz.	155.00	155.00	
Fluoride, technical..... lb.	.15	.15		Silver..... oz.	1.32	1.27	
Hyposulfite..... 100 lbs.	3.60	3.60		Tin..... lb.	.60	.60	
Nitrate, 95%..... 100 lbs.	3.85	3.85		Tungsten Wolframite..... per unit	7.00	7.00	
Silicate, 40°..... lb.	.02	.02		Zinc, N. Y..... 100 lbs.	9.25	9.25	
Sulfide..... lb.	.05	.05					
Bisulfite, powdered..... lb.	.05	.05		<b>FERTILIZER MATERIALS</b>			
Strontium Nitrate..... lb.	.25	.25		Ammonium Sulfate..... 100 lbs.	7.50	7.25	
Sulfur, flowers..... 100 lbs.	3.75	3.75		Blood, dried, f. o. b. N. Y..... unit	8.00	8.00	
Crude..... long ton	30.00	30.00		Bone, 3 and 50, ground, raw..... ton	48.00	48.00	
Talc, American, white..... ton	20.00	20.00		Calcium Cyanamide, unit of Am- monia.....	4.50	4.50	
Tin Bichloride..... lb.	.21 1/2	.21 1/2		Fish Scrap, domestic, dried, f. o. b. works..... unit	7.25	7.25	
Oxide..... lb.	.60	.60		Phosphate Rock, f. o. b. mine: Florida pebble, 68%..... ton	Nom.	Nom.	
Zinc Chloride, U. S. P..... lb.	.50	.50		Tennessee, 78-80%..... ton	11.00	11.00	
Oxide, bbls..... lb.	.12	.12		Potassium muriate, 80%..... ton	230.00	230.00	
				Pyrites, furnace size, imported... unit	.18	.18	
				Tankage, high-grade, f. o. b: Chicago..... unit	8.00	7.75	
<b>ORGANIC CHEMICALS</b>							
Acetanilid..... lb.	.62	.65					
Acid, Acetic, 28 p. c..... 100 lbs.	3.75	3.75					
Glacial..... lb.	.12 1/4	.12 1/4					
Acetylsalicylic..... lb.	1.00	1.00					
Benzoic, U. S. P., ex-toluol..... lb.	.90	.90					
Carbolic, cryst., U. S. P., drs... lb.	.15	.15					
50- to 110-lb. tins..... lb.	.21	.21					



## COAL-TAR CHEMICALS

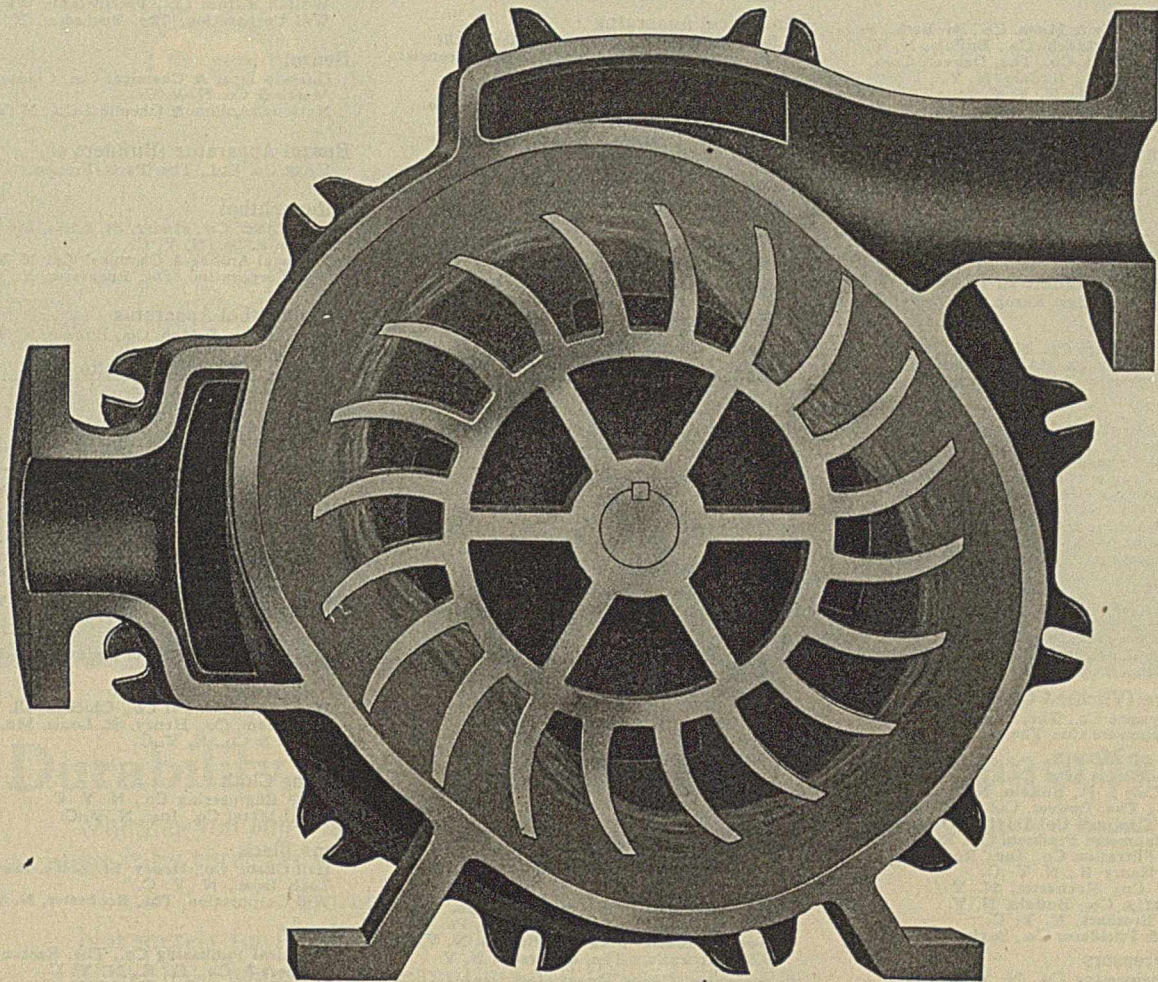
	Mar. 1	Mar. 15		Mar. 1	Mar. 15
<b>Crudes</b>			<b>Acid Colors (Continued)</b>		
Benzol, C. P. .... gal.	.33	.33	Fuchsin .....	2.50	2.50
Cresol, U. S. P. .... lb.	.18	.18	Orange III .....	1.00	1.00
Naphthalene, flake .....	.08 1/2	.08 1/2	Red .....	1.10	1.10
Phenol, drums .....	.15	.15	Violet 10B .....	6.50	6.50
Toluol, 90% .....	.28	.30	Alkali Blue, domestic .....	4.75	4.75
Xylol, water white .....	.40	.40	Imported .....	8.00	8.00
<b>Intermediates</b>			Azo Carmine .....	4.00	4.00
<b>Acids:</b>			Azo Yellow .....	2.00	2.00
Anthranilic .....	4.00	3.00	Erythrosine .....	12.00	12.00
Benzoic .....	.85	.85	Indigotine, conc. .... lb.	3.00	3.00
Broenner's .....	1.75	1.75	Paste .....	1.50	1.50
Cleves .....	2.00	2.00	Naphthol Green .....	1.50	1.50
Cresylic, 97-99% .....	1.00	1.00	Ponceau .....	1.00	1.00
F .....	3.00	3.00	Scarlet 2R .....	1.00	1.00
Gamma .....	2.50	2.50	<b>Direct Colors</b>		
H .....	1.70	1.85	Black .....	.95	.95
Metanilic .....	1.70	1.70	Blue .....	1.10	1.10
Monosulfonic F .....	3.00	3.00	Brown .....	1.55	1.55
Naphthionic, crude .....	.70	.70	Fast Red .....	3.50	3.50
Neville & Winther's .....	1.90	1.90	Yellow .....	2.00	2.00
Phthalic .....	.60	.60	Violet, con't. .... lb.	2.20	2.20
Picric .....	.25	.25	Chrysofenine, domestic .....	2.50	2.50
Sulfanilic .....	.35	.35	Imported .....	3.80	3.80
Tobias .....	2.25	2.25	Congo Red, 4B Type .....	1.60	1.60
Amido Azo Benzol .....	1.15	1.15	Primuline, domestic .....	3.00	3.00
Aniline Oil .....	.35	.35	<b>Oil Colors</b>		
For Red .....	.65	.65	Black .....	.70	.70
Aniline Salt .....	.44	.46	Blue .....	1.65	1.65
Anthracene, 80-85% .....	.75	.75	Orange .....	1.40	1.40
Anthraquinone .....	4.50	4.50	Red III .....	1.65	1.65
Benzaldehyde, tech. .... lb.	.75	.75	Scarlet .....	1.75	1.75
U. S. P. .... lb.	1.25	1.10	Yellow .....	1.70	1.70
Benzidine Base .....	1.40	1.50	Nigrosine Oil, solution .....	.90	.90
Benzidine Sulfate .....	1.10	1.10	<b>Sulfur Colors</b>		
Diamidophenol .....	6.00	6.00	Black .....	.30	.30
Dianisidine .....	10.00	10.00	Blue, domestic .....	.80	.80
p-Dichlorbenzol .....	.10	.10	Brown .....	.35	.35
Diethylaniline .....	1.40	1.40	Green .....	1.00	1.00
Dimethylaniline .....	1.30	2.00	Yellow .....	.90	.90
Dinitrobenzol .....	.33	.36	<b>Chrome Colors</b>		
Dinitrotoluol .....	.40	.43	Alizarin Blue, bright .....	7.75	7.75
Diphenylamine .....	.75	.85	Alizarin Red, W. S. Paste .....	5.00	5.00
G Salt .....	.75	.80	Alizarin Yellow R .....	1.50	1.50
Hydroquinone .....	2.00	2.00	Chrome Black, domestic .....	1.25	1.25
Metol .....	11.50	11.50	Imported .....	2.20	2.20
Monochlorbenzol .....	.10	.12	Chrome Blue .....	2.50	2.50
Monoethylaniline .....	2.15	2.15	Chrome Green, domestic .....	1.50	1.50
a-Naphthylamine .....	.45	.45	Chrome Red .....	2.00	2.00
b-Naphthylamine .....	1.15	1.15	<b>Basic Colors</b>		
b-Naphthol, crude .....	.60	.60	Auramine, O, domestic .....	3.25	3.25
m-Nitraniline .....	1.00	1.00	Auramine, OO .....	4.25	4.25
p-Nitraniline .....	1.35	1.40	Bismarck Brown Y .....	.90	.90
Nitrobenzol, crude .....	.16	.16	Bismarck Brown R .....	1.20	1.20
Rectified (Oil Myrbane) .....	.18	.18	Chrysoidine R .....	1.00	1.00
p-Nitrophenol .....	.80	.80	Chrysoidine Y .....	.90	.90
p-Nitrosodimethylaniline .....	1.90	1.90	Green Crystals, Brilliant .....	6.00	6.00
o-Nitrotoluol .....	.20	.22	Indigo, 20 p. c. paste .....	.75	.75
p-Nitrotoluol .....	1.15	1.15	Fuchsine Crystals, domestic .....	4.00	4.00
m-Phenylenediamine .....	1.25	1.35	Imported .....	12.00	12.00
p-Phenylenediamine .....	2.50	2.50	Magenta Acid, domestic .....	4.25	4.25
Phthalic Anhydride .....	.75	.75	Magenta Crystals, imported .....	10.00	10.00
Primuline Base .....	3.00	3.00	Malachite Green, crystals .....	4.50	4.50
R Salt .....	.75	.75	Methylene Blue, tech. .... lb.	2.25	2.25
Resorcin, tech .....	3.75	4.00	Methyl Violet 3 B .....	2.60	2.60
U. S. P. .... lb.	6.00	6.00	Nigrosine, spts. sol. .... lb.	.85	.85
Schaeffer Salt .....	.65	.75	Water sol., blue .....	.65	.65
Sodium Naphthionate .....	1.10	1.10	Jet .....	.90	.90
Thiocarbanilid .....	.60	.60	Phosphine G., domestic .....	7.00	7.00
Tolidine, Base .....	1.70	1.75	Rhodamine B, ex-con't. .... lb.	35.00	35.00
Toluidine, mixed .....	.44	.44	Victoria Blue, base, domestic .....	6.00	6.00
o-Toluidine .....	.30	.30	Victoria Green .....	6.00	6.00
m-Toluylenediamine .....	1.35	1.35	Victoria Red .....	7.00	7.00
p-Toluidine .....	1.75	1.75	Victoria Yellow .....	7.00	7.00
Xylidene, crude .....	.50	.50	<b>COAL-TAR COLORS</b>		
<b>Acid Colors</b>			<b>Acid Colors</b>		
Black .....	1.15	1.15	Black .....	1.15	1.15
Blue .....	3.00	3.00	Blue .....	3.00	3.00





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## THE SIMPLEST VACUUM PUMP OR COMPRESSOR



**T**HE pump for air, industrial gas, or acid gas. No valves. No gears. No pistons. No piston packing. No sliding vanes. No interior lubrication necessary. Absolutely clean air, delivered without pulsation. Rotor only moving part, cast in one piece, heavily shrouded, and mounted on ball bearings outside of casing. Long life. Constant efficiency. Positive service.

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South Norwalk, Conn., U. S.



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 General Ceramics Co., N. Y. C.  
 Knight, Maurice A., East Akron, Ohio.  
 Lummus Co., The Walter E., Boston, Mass.  
 Thermal Syndicate, Ltd., The, N. Y. C.  
 U. S. Stoneware Co., The, Akron, O.

**Acid and Alkali Resistant Apparatus**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Cleveland Brass Mfg. Co., The, Cleveland, O.  
 Duriron Castings Co., Dayton, O. (Acid Proof)  
 Elyria Enameled Prod. Co., Elyria, O.  
 Jacoby, Henry E., N. Y. C.  
 Pfaudler Co., Rochester, N. Y.

**Acid Distillation Apparatus**

Thermal Syndicate, Ltd., The, N. Y. C.

**Acid Eggs**

Bethlehem Fdy. & Mch. Co., So. Beth., Pa.  
 Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Devine Co., J. P., Buffalo, N. Y.  
 Jacoby, Henry E., N. Y. C.  
 Schutte & Koerting Co., Phila., Pa.  
 U. S. Stoneware Co., The, Akron, O.

**Acid Pitchers**

Guernseyware Co., The, Cambridge, O.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Knight, Maurice A., East Akron, O.  
 Palo Co., N. Y. C.  
 U. S. Stoneware Co., The, Akron, O.  
 Will Corporation, The, Rochester, N. Y.

**Acid Plants (Complete or in Part)**

Knight, M. A., East Akron, O.

**Acid Proof Brick**

General Ceramics Co., N. Y. C.  
 Knight, M. A., East Akron, Ohio.  
 U. S. Stoneware Co., The, Akron, O.

**Acids**

Central Scientific Co., Chicago, Ill.  
 Dow Chemical Co., The, Midland, Mich.  
 Eimer & Amend, N. Y. C.  
 Gazzolo Drug & Chemical Co., Chicago, Ill.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 National Aniline & Chemical Co., N. Y. C.  
 Palo Company, N. Y. C.  
 Will Corporation, The, Rochester, N. Y.

**Acid Valves**

Cleveland Brass Mfg. Co., The, Cleveland, O.  
 Duriron Castings Co., Dayton, O.  
 General Ceramics Co., N. Y. C.  
 Schutte & Koerting Co., Phila., Pa.  
 York Manufacturing Co., York, Pa.

**Acid Ware (Vitrified Clay)**

Guernseyware Co., The, Cambridge, O.  
 U. S. Stoneware Co., The, Akron, O.

**Agitators or Mixers**

Cleveland Brass Mfg. Co., The, Cleveland, O.  
 Devine Co., J. P., Buffalo, N. Y.  
 Dorr Co., The, Denver, Colo., & N. Y. C.  
 Duriron Castings Co., Dayton, O. (Acid Proof)  
 Elyria Enameled Products Co., Elyria, O.  
 General Filtration Co., Inc., Rochester, N. Y.  
 Jacoby, Henry E., N. Y. C.  
 Pfaudler Co., Rochester, N. Y.  
 Sowers Mfg. Co., Buffalo, N. Y.  
 Stevens Brothers, N. Y. C.  
 Werner & Pfeiderer Co., Saginaw, Mich.

**Air Compressors**

Abbé Engineering Co., N. Y. C.  
 Crowell Engineering Co., Brooklyn, N. Y.  
 General Electric Co., Schenectady, N. Y.  
 Hubbard's Sons, Norman, Brooklyn, N. Y.  
 Nash Engineering Co., So. Norwalk, Conn.  
 Schutte & Koerting Co., Phila., Pa.

**Air-Diffusing Plates**

General Filtration Co., Inc., Rochester, N. Y.

**Air Washers**

Carrier Engineering Corp., N. Y. C.

**Alcohol Apparatus (Builders of)**

Egrot Co., Ltd., The, Paris, France.

**Alkalis**

Arnold Hoffman & Co., Inc., N. Y. C.  
 Lithflux Mineral & Chem. Wks., Chicago, Ill.  
 National Aniline & Chemical Co., N. Y. C.

**Alundum Ware**

Denver Fire Clay Co., The, Denver, Colo.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Norton Co., Worcester, Mass.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Ammeters**

Brown Instrument Co., Philadelphia, Pa.  
 General Electric Co., Schenectady, N. Y.

**Ammonia**

Heil Chem. Co., Henry, St. Louis, Mo.

**Ammonia Fittings and Supplies**

York Manufacturing Co., York, Pa.

**Ammonia—Salts of**

Merck & Co., N. Y. C.

**Analysis—Gas**

Sarco Co., Inc., N. Y. C.

**Analysis—Gas Apparatus**

Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Emil Greiner Co., The, N. Y. C.  
 Griebel Instrument Co., Carbondale, Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Sarco Co., Inc., N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Analytical Apparatus**

Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Griebel Instrument Co., Carbondale, Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Analytical Laboratories**

Chemical Laboratories of N. Y., Inc., N. Y. C.  
 Columbus Laboratories, Chicago, Ill.  
 Dearborn Chemical Co., Chicago, Ill.  
 Fort Worth Laboratories, Fort Worth, Texas.  
 General Chemical Laboratories, N. Y. C.  
 Sadtler & Son, Samuel P., Philadelphia, Pa.  
 Westport Mill, Westport, Conn.

**Aniline Oil—Salts of**

Merck & Co., N. Y. C.  
 National Aniline & Chemical Co., N. Y. C.

**Antimony—Salts of**

Merck & Co., N. Y. C.

**Arresters (Dust)**

Sly Mfg. Co., W. W., Cleveland, O.

**Arsenic—Metallic & Salts**

Merck & Co., N. Y. C.

**Arsenic Plants (Acid Proof Stoneware)**

Knight, M. A., East Akron, O.

**Asphalt & Tar Testing Apparatus**

Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thermo-Electric Instrument Co., Newark, N. J.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Autoclaves**

Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Devine Co., J. P., Buffalo, N. Y.  
 Duriron Casting Co., Dayton, O.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Sowers Manufacturing Co., Buffalo, N. Y.  
 Will Corporation, The, Rochester, N. Y.

**Autoclaves (Glass Enameled Steel)**

Pfaudler Co., Rochester, N. Y.

**Autoclaves (High and Low Pressure)**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Devine Co., J. P., Buffalo, N. Y.

**Automatic Pressure Regulators**

Brown Instrument Co., The, Phila., Pa.  
 Taylor Instrument Cos., Rochester, N. Y.

**Automatic Temperature Regulators**

Brown Instrument Co., The, Phila., Pa.  
 Engelhard, Charles, N. Y. C.  
 Taylor Instrument Cos., Rochester, N. Y.

**Bakelite**

General Bakelite Co., N. Y. C.

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Becker Inc., Christian, N. Y. C.  
 Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Griebel Instrument Co., Carbondale, Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Whitall Tatum Co., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Barium—Salts of**

Dow Chemical Co., The, Midland, Mich.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Will Corporation, The, Rochester, N. Y.

**Barometers**

Brown Instrument Co., The, Phila., Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Taylor Instrument Cos., Rochester, N. Y.  
 Will Corporation, The, Rochester, N. Y.

**Beakers**

Braun-Knecht-Heimann Corp., San Francisco  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Guernseyware Co., The, Cambridge, O.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Herold China & Pottery Co., Golden, Col.  
 Palo Company, N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Whitall Tatum Co., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Benzol**

Gazzolo Drug & Chemical Co., Chicago, Ill.  
 Merck & Co., N. Y. C.  
 National Aniline & Chemical Co., N. Y. C.

**Benzol Apparatus (Builders of)**

Egrot Co. Ltd., The, Paris, France.

**Betanaphthol**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 National Aniline & Chemical Co., N. Y. C.  
 Will Corporation, The, Rochester, N. Y.

**Betanaphthol Apparatus**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.

**Bismuth—Metallic & Salts**

Merck & Co., N. Y. C.

**Blowers**

Abbé Engineering Co., N. Y. C.  
 Central Scientific Co., Chicago, Ill.  
 Crowell Mfg. Co., Brooklyn, N. Y.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Duriron Castings Co., Dayton, O.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Schutte & Koerting Co., Phila., Pa.  
 Will Corporation, The, Rochester, N. Y.

**Boiler Coverings**

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**Boiler Water Treatment**

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 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.

**Boiling Cloth**

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 Multi Metal Co., Inc., N. Y. C.

**Bone Black**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Toch Bros., N. Y. C.  
 Will Corporation, The, Rochester, N. Y.

**Books**

Chemical Publishing Co., The, Easton, Pa.  
 Stecher & Co., G. E., N. Y. C.  
 Van Nostrand Co., D., N. Y. C.  
 Wiley & Sons, Inc., John, N. Y. C.

**Bottles (Glass)**

Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Wheaton Co., T. C., Millville, N. J.  
 Whitall Tatum Co., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Brinell Machines**

Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Will Corporation, The, Rochester, N. Y.

**Burner Guards**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Knight, M. A., East Akron, O.  
 Will Corporation, The, Rochester, N. Y.

**Burners—Gas and Oil**

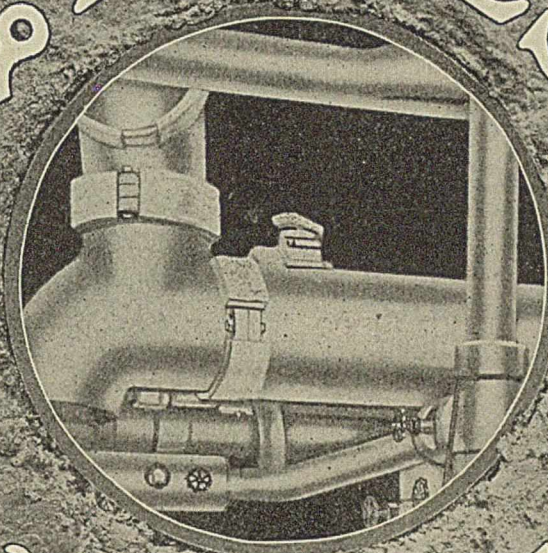
Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
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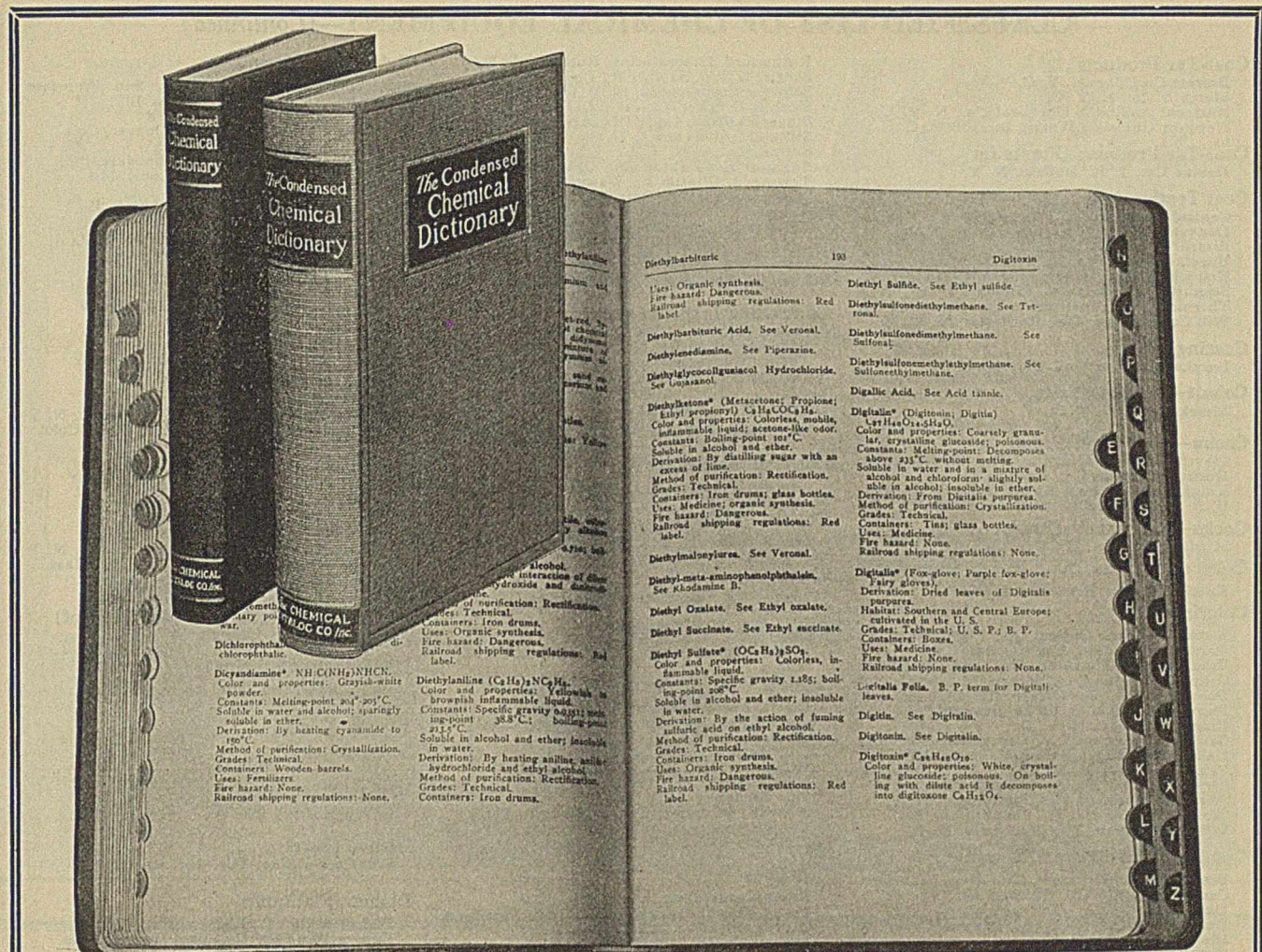
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## CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

- Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Schutte & Koerting Co., Phila., Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.
- Burners—Sulphur**  
Schutte & Koerting Co., Phila., Pa.
- Burnishing Pots and Baskets (Acid Proof)**  
Knight, M. A., East Akron, Ohio.
- By-Product Machinery**  
Devine Co., J. P., Buffalo, N. Y.  
Swenson Evaporator Co., Chicago, Ill.
- Cabinets (Laboratory, Chemical, Sample and Specimen)**  
Schwartz Sectional System, Indianapolis, Ind.
- Cable & Wire**  
General Electric Co., Schenectady, N. Y.
- Cadmium—Salts of**  
Dow Chemical Co., The, Midland, Mich.  
Merck & Co., N. Y. C.
- Calcium—Salts of**  
Dow Chemical Co., The, Midland, Mich.  
Merck & Co., N. Y. C.
- Calorimeters—Fuel and Gas**  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sarco Co., Inc., N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.
- Capsules**  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C. ("Impervite")  
Heil Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.  
Thermal Syndicate, Ltd., The, N. Y. C.  
Will Corporation, The, Rochester, N. Y.
- Carbon Bisulfide**  
Merck & Co., N. Y. C.  
National Aniline & Chemical Co., N. Y. C.
- Carbon Tetrachloride**  
Dow Chemical Co., The, Midland, Mich.  
Gazzolo Drug & Chemical Co., Chicago, Ill.  
Merck & Co., N. Y. C.
- Carboys**  
Whittall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.
- Carboy Inclinator**  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Will Corporation, The, Rochester, N. Y.
- Carboy Stoppers (Acid Proof)**  
Knight, M. A., East Akron, O.  
Whittall Tatum Co., Philadelphia, Pa.
- Casseroles**  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo. (Acid Proof)  
Duriron Castings Co., Dayton, O.  
Eimer & Amend, N. Y. C.  
Guernseyware Co., The, Cambridge, O.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Herold China & Pottery Co., Golden, Colo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.
- Castings (Acid and Heat Resisting)**  
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Jacoby, Henry E., N. Y. C.
- Castings (Chemical, Acid and Caustic)**  
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.  
Buffalo Fdy. & Mach. Co., Buffalo, N. Y.  
Cast Iron Pipe Publicity Bureau, N. Y. C.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Ferguson & Lange Foundry Co., Chicago, Ill.  
Garrigue & Co., William, Chicago, Ill.
- Castings (Chilled)**  
Jacoby, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.
- Castings—Evaporator & Vacuum Pan**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Garrigue & Co., William, Chicago, Ill.  
Sowers Manufacturing Co., Buffalo, N. Y.
- Castings (Machinery)**  
Ferguson & Lange Foundry Co., Chicago, Ill.
- Castings (Semi-steel)**  
Ferguson & Lange Foundry Co., Chicago, Ill.
- Caustic Potash**  
Gazzolo Drug & Chemical Co., Chicago, Ill.  
Merck & Co., N. Y. C.
- Caustic Pots**  
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Caustic Soda**  
Arnold, Hoffman & Co., Inc., N. Y. C.  
General Chem. Co., N. Y. C.  
Merck & Co., N. Y. C.  
National Aniline & Chemical Co., N. Y. C.
- Caustic Soda Dissolvers—Automatic**  
Lummus Co., The, Walter E., Boston, Mass.
- Cement Filler**  
Toch Bros, N. Y. C.
- Cement Refractory**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.
- Cement Testing Apparatus**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.
- Centrifugals**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
DeLaval Separator Co., N. Y. C.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Fletcher Works, Philadelphia, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
International Equipment Co., Boston, Mass.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Sharples Specialty Co., The, West Chester, Pa.  
Tolhurst Machine Works, Troy, N. Y.  
Will Corporation, The, Rochester, N. Y.
- Centrifugal Dryers**  
Fletcher Works, Philadelphia, Pa.
- Centrifugal Engineers**  
Fletcher Works, Philadelphia, Pa.  
Sharples Specialty Co., The, West Chester, Pa.  
Tolhurst Machine Works, Troy, N. Y.
- Cerium Oxalate**  
Merck & Co., N. Y. C.
- Charcoal**  
Heil Chem. Co., Henry, St. Louis, Mo.
- Chemical Apparatus**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Devine Co., J. P., Buffalo, N. Y.  
Eimer & Amend, N. Y. C.  
Fletcher Works, Philadelphia, Pa.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Laboratory Materials Co., Chicago, Ill.  
Schaar & Co., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Sowers Manufacturing Co., Buffalo, N. Y.  
Werner & Pfeiderer Co., Inc., Saginaw, Mich.  
Will Corporation, The, Rochester, N. Y.
- Chemical Glass Tubing**  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Will Corporation, The, Rochester, N. Y.
- Chemical Glassware**  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Wheaton Co., T. C., Millville, N. J.  
Whittall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.
- Chemical Plants (Complete Installation)**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Garrigue & Co., William, Chicago, Ill.  
Hercules Engineering Corp., N. Y. C.  
Knight, M. A., East Akron, O.  
Lummus Co., The, Walter E., Boston, Mass.  
Mantius Engineering Co., N. Y. C.  
Swenson Evaporator Co., Chicago, Ill.  
Technical Products Co., Inc., N. Y. C. (used equipment)
- Chemical Plants (Erected and Supervised)**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Egrot Co. Ltd., The, Paris, France.  
McNamara, M., Everett, Mass.  
Swenson Evaporator Co., Chicago, Ill.
- Chemical Reagents**  
Baker Chemical Co., J. T., Phillipsburg, N. J.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
General Chemical Co., Baker & Adamson Works, Easton, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
National Aniline & Chemical Co., N. Y. C.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.
- Chemicals**  
Baker Chemical Co., J. T., Phillipsburg, N. J.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Dearborn Chemical Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Dow Chemical Co., The, Midland, Mich.  
Eastman Kodak Co., Rochester, N. Y.  
Eimer & Amend, N. Y. C.  
Gazzolo Drug & Chemical Co., Chicago, Ill.  
General Chemical Co., N. Y. C.  
General Chem. Co., B. & A. Wks., Easton, Pa.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Hercules Powder Co., N. Y. C.  
Laboratory Materials Co., Chicago, Ill.  
Merck & Co., N. Y. C.  
Monsanto Chemical Works, St. Louis, Mo.  
National Aniline & Chemical Co., N. Y. C.  
Newport Chemical Works, Inc., N. Y. C.  
Palo Company, N. Y. C.  
Pfeizer & Co., Charles, N. Y. C.  
Roessler & Hasslacher Chemical Co., N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Schaar & Co., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Toch Bros, N. Y. C.  
Will Corporation, The, Rochester, N. Y.
- Chlorine Generators—Acid Proof**  
Knight, M. A., East Akron, O.
- Chlorine—Liquid**  
Arnold, Hoffman & Co., Inc., N. Y. C.  
Electro Bleaching Gas Co., N. Y. C.
- Chromel Metal**  
Hoskins Mfg. Co., Detroit, Mich.
- Chromium (Metal & Salts)**  
Merck & Co., N. Y. C.
- Circuit Breakers and Switches**  
General Electric Co., Schenectady, N. Y.
- Clarifiers**  
Sharples Specialty Co., The, West Chester, Pa.
- Classifiers**  
Dorr Co., The, Denver, Colo., & N. Y. C.
- Classifiers or Washers**  
Dorr Co., The, Denver, Colo., & N. Y. C.





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## CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

## Coal-Tar Products

Barrett Co., The, N. Y. C.  
 Merck & Co., N. Y. C.  
 National Aniline & Chemical Co., N. Y. C.  
 Newport Chemical Works, Inc., N. Y. C.

## Coal-Tar Products (Plants for)

Devine Co., J. P., Buffalo, N. Y.

## Coal Testing Apparatus

Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Engelhard, Charles, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thermo-Electric Instrument Co., Newark, N. J.  
 Will Corporation, The, Rochester, N. Y.

## Coating Sprays

Eureka Machine Co., Cleveland, O.

## Cobalt—Metal &amp; Salts

Merck & Co., N. Y. C.

## Cocks—Plug (Acid Proof)

Cleveland Brass Mfg. Co., The, Cleveland, O.  
 Duriron Castings Co., Dayton, O.  
 Knight, M. A., East Akron, O.  
 U. S. Stoneware Co., The, Akron, O.

## Cocks—Laboratory (Brass)

Mueller Mfg. Co., H., Decatur, Ill.

## Coils or Worms (Acid Proof Stoneware)

Knight, M. A., East Akron, Ohio.

## Colorimeters

Bausch & Lomb Optical Co., Rochester, N. Y.  
 Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Griebel Instrument Co., Carbondale, Pa.  
 Heil Chemical Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

## Combustion Tubes and Boats

Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Engelhard, Charles, N. Y. C. ("Impervite")  
 General Ceramics Co., N. Y. C.  
 Guernseyware Co., The, Cambridge, O.  
 Heil Chemical Co., Henry, St. Louis, Mo.  
 Norton Co., Worcester, Mass.  
 Palo Company, N. Y. C.  
 Sargent & Co., R. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thermal Syndicate, Ltd., The, N. Y. C.  
 Will Corporation, The, Rochester, N. Y.

## Combustion Tubes and Boats (Platinum)

Baker & Co., Inc., Newark, N. J.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Will Corporation, The, Rochester, N. Y.

## Compressors, Rotary

Crowell Mfg. Co., Brooklyn, N. Y.  
 Devine Co., J. P., Buffalo, N. Y.  
 Heil Chem. Co., Henry, St. Louis, Mo.

## Concentrators—Sulphuric Acid

Thermal Syndicate, Ltd., The, N. Y. C.

## Condensers (Barometric and Surface)

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Devine Co., J. P., Buffalo, N. Y.  
 Duriron Castings Co., Dayton, O. (Acid Proof)  
 Jacoby, Henry E., N. Y. C.  
 Lummus Co., The, Walter E., Boston, Mass.  
 Schutte & Koerting Co., Phila., Pa.  
 Stevens Brothers, N. Y. C.

## Condensers—Nitric Acid

Thermal Syndicate, Ltd., The, N. Y. C.

## Conductivity Measuring Apparatus

Central Scientific Co., Chicago, Ill.  
 Leeds & Northrup Co., The, Philadelphia, Pa.

## Consulting Chemists &amp; Chemical Engineers

Abbott, W. G., Jr., Wilton, N. H.  
 Andrews, A. B., Lewiston, Me.  
 Brickenstein, John H., Washington, D. C.  
 Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Carlitz & Co., J. S., Philadelphia, Pa.  
 Columbus Laboratories, Chicago, Ill.  
 Ellis-Foster Co., Montclair, N. J.  
 Fort Worth Laboratories, The, Ft. Worth, Tex.  
 Foster, A. B., Washington, D. C.  
 Gorzo, Julius, Pittsburgh, Pa.  
 Maas, Arthur R., Los Angeles, Cal.  
 McNamara, M., Everett, Mass.  
 Mantius Engineering Co., N. Y. C.  
 Marvin-Davis Laboratories, Inc., N. Y. C.  
 Mount, W. D., Roanoke, Va.  
 Parker, C. L., Washington, D. C.  
 Pease Laboratories, N. Y. C.

Rutherford Laboratories, Rutherford, N. J.  
 Sadtler & Son, Samuel P., Philadelphia, Pa.  
 Schwarz Laboratories, N. Y. C.  
 Sleck & Drucker, Chicago, Ill.  
 Singer-Perlstein Co., Milwaukee, Wis.  
 Stillwell Laboratories, N. Y. C.  
 Sutton, Frank, N. Y. C.  
 Swenson Evaporator Co., Chicago, Ill.  
 Townsend, Clinton P., Washington, D. C.  
 Westport Mill, The (Dorr Co.), Westport, Conn.  
 Wheeler & Woodruff, N. Y. C.  
 Wiley & Co., Inc., Baltimore, Md.

## Contracting Engineers

Swenson Evaporator Co., Chicago, Ill.

## Controlling Instruments

Brown Instrument Co., The, Phila., Pa.  
 Engelhard, Charles, N. Y. C.  
 Taylor Instrument Cos., Rochester, N. Y.  
 Will Corporation, The, Rochester, N. Y.

## Converters—Booster

Westinghouse Elec. & Mfg. Co., E. Pittsburgh, Pa.

## Converters—Rotary

General Electric Co., Schenectady, N. Y.

## Copper—Salts of

Merck & Co., N. Y. C.

## Cores

Norton Co., Worcester, Mass.

## Counters—Revolution

Brown Instrument Co., Philadelphia, Pa.

## Critical Point Determination Apparatus

Brown Instrument Co., Philadelphia, Pa.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Engelhard, Charles, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Hoskins Mfg. Co., Detroit, Mich.  
 Palo Company, N. Y. C.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thwing Instrument Co., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

## Crucibles

Braun Corporation, Los Angeles, Cal.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Duriron Castings Co., Dayton, O. (Acid proof).  
 Eimer & Amend, N. Y. C.  
 Engelhard, Charles, N. Y. C. ("Impervite")  
 Guernseyware Co., The, Cambridge, O.  
 Heil Chemical Co., Henry, St. Louis, Mo.  
 Herold China & Pottery Co., Golden, Col.  
 Norton Co., Worcester, Mass. (Alundum).  
 Palo Company, N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thermal Syndicate, Ltd., The, N. Y. C. (Vitrosil).  
 Will Corporation, The, Rochester, N. Y.

## Crucibles, Platinum

American Platinum Works, Newark, N. J.  
 Baker & Co., Inc., Newark, N. J.  
 Bishop & Co., J. Malvern, Pa.  
 Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Will Corporation, The, Rochester, N. Y.

## Crushers &amp; Grinders (Ind. &amp; Lab'y)

Abbé Engineering Co., N. Y. C.  
 Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chemical Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Raymond Bros. Impact Pulv. Co., Chicago, Ill.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.  
 Williams Patent Crusher & Pulv. Co., Chicago.

## Crystallizers—Ammonia Nitrate

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Devine Co., J. P., Buffalo, N. Y.

## Crystallizing Pans (Acid Proof)

Knight, M. A., East Akron, Ohio.

## Cupels

Braun Corporation, Los Angeles, Cal.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chemical Company, Henry, St. Louis, Mo.  
 Hoskins Mfg. Co., Detroit, Michigan.  
 Norton Company, Worcester, Mass.

## Cylinders (Glass)

Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Griebel Instrument Co., Carbondale, Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Whittall Tatum Co., Philadelphia, Pa.  
 Will Corporation, The, Rochester, N. Y.

## Decanting Pots—Acid Proof

Knight, M. A., East Akron, O.  
 Will Corporation, The, Rochester, N. Y.

## Denitrating Apparatus

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.

## Diaphragms—Acid Proof

General Filtration Co., Inc., Rochester, N. Y.  
 Herold China & Pottery Co., Golden, Colo.

## Dictionary—Chemical

Chemical Catalog Co., The, N. Y. C.

## Diffusion Apparatus

Devine Co., J. P., Buffalo, N. Y.  
 General Filtration Co., Inc., Rochester, N. Y.  
 Lummus Co., The, Walter E., Boston, Mass.  
 Swenson Evaporator Co., Chicago, Ill.

## Dipping Baskets &amp; Jars—(Acid Proof)

Knight, M. A., East Akron, Ohio.

## Dishes (Crystallizing)

Will Corporation, The, Rochester, N. Y.

## Dishes (Glass)

Will Corporation, The, Rochester, N. Y.

## Dishes ("Imperite")

Engelhard, Charles, N. Y. C.

## Dishes (Petri)

Will Corporation, The, Rochester, N. Y.

## Dishes (Platinum)

Bishop & Co., J. Malvern, Pa.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Will Corporation, The, Rochester, N. Y.

## Dissolving Tanks—Automatic

Lummus Co., The, Walter E., Boston, Mass.  
 Werner & Pfeiderer Co., Saginaw, Mich.

## Distilling Machinery and Apparatus

Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Central Scientific Co., Chicago, Ill.  
 Cleveland Brass Mfg. Co., The, Cleveland, O.  
 Daigger & Co., A., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Detroit Heating & Lighting Co., Detroit, Mich.  
 Devine Company, J. P., Buffalo, N. Y.  
 DuPont Chemical Co., Inc., Wilmington, Del.  
 Duriron Castings Co., Dayton, O.  
 Egrot Co., The, Paris, France.  
 Eimer & Amend, N. Y. C.  
 Electric Heating Apparatus Co., N. Y. C.  
 Elyria Enamelled Prod. Co., Elyria, O.  
 Garrigue Co., Wm., Chicago, Ill. (Fatty Acids)  
 General Ceramics Co., N. Y. C.  
 Griebel Instrument Co., Carbondale, Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Jacoby, Henry E., N. Y. C.  
 Lummus Co., The, Walter E., Boston, Mass.  
 Palo Company, N. Y. C.  
 Pfaunder Co., Rochester, N. Y.  
 Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Sowers Mfg. Co., Buffalo, N. Y.  
 Stevens Bros., N. Y. C.  
 Stokes Machine Company, F. J., Phila., Pa.  
 Swenson Evaporator Co., Chicago, Ill.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Wheeler Condenser & Eng. Co., Carteret, N. J.  
 Will Corporation, The, Rochester, N. Y.  
 Zarembo Company, Buffalo, N. Y.

## Draft Gauges

Braun-Knecht-Heimann Co., San Francisco.  
 Brown Instrument Co., Philadelphia, Pa.  
 Central Scientific Co., Chicago, Ill.  
 Denver Fire Clay Co., The, Denver, Colo.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Scientific Materials Co., Pittsburgh, Pa.

## Drum Dryers—Vacuum and Atmospheric

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
 Devine Co., J. P., Buffalo, N. Y.

## Dry Blast Plants

Carrier Engineering Corp., N. Y. C.



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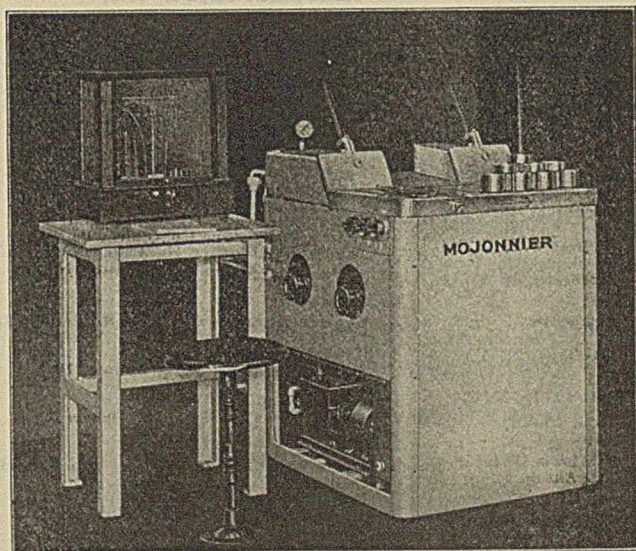
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## CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

**Dryers—Atmospheric**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Gordon Dryer Corp., N. Y. C.  
Philadelphia Textile Machinery Co., Phila., Pa.

**Dryers—Centrifugals**

Fletcher Works, Philadelphia, Pa.  
Tolhurst Machine Works, Troy, N. Y.

**Dryers—Vacuum**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Company, J. P., Buffalo, N. Y.  
Jacoby, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.  
Stokes Machine Co., F. J., Philadelphia, Pa.  
Werner & Pfeiderer Co., Saginaw, Mich.

**Drying Apparatus and Machinery**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Central Scientific Co., Chicago, Ill.  
Devine Co., J. P., Buffalo, N. Y.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Gordon Engineering Corp., N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Jacoby, Henry E., N. Y. C.  
Palo Company, N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.  
Tolhurst Machine Works, Troy, N. Y.  
Werner & Pfeiderer Co., Saginaw, Mich.  
Will Corporation, The, Rochester, N. Y.

**Duriron—Acid-Proof**

Duriron Castings Co., N. Y. C.

**Dyestuffs**

du Pont de Nemours & Co., E. I., Wilmington, Del.  
Gazzolo Drug & Chem. Co., Chicago, Ill.  
Merck & Co., N. Y. C.  
Metz & Co., H. A., N. Y. C.  
National Aniline & Chemical Co., N. Y. C.

**Dynamas and Motors**

General Electric Co., Schenectady, N. Y.

**Efficiency Instruments**

Braun-Knecht-Heimann Co., San Francisco, Cal.  
Brown Instrument Co., Philadelphia, Pa.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Thwing Instrument Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Electric Heating Devices**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Will Corporation, The, Rochester, N. Y.

**Electric Locomotives—Industrial**

General Electric Co., Schenectady, N. Y.

**Electrical Instruments**

Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
General Electric Co., Schenectady, N. Y.  
Engelhard, Charles, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Thwing Instrument Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Electrical Measuring Instruments**

Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
Engelhard, Charles, N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Leeds & Northrup Co., Phila., Pa.  
Palo Company, N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Electrical Supplies**

General Electric Co., Schenectady, N. Y.

**Electrodes—Platinum**

American Platinum Works, Newark, N. J.  
Baker & Co., Inc., Newark, N. J.  
Bishop & Co., J., Platinum Works, Malvern, Pa.  
Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Chas., N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Electrolytic Apparatus—Acid Proof**

Knight, M. A., East Akron, O.

**Enameled Apparatus (Acid Resistant)**

Elyria Enameled Prod. Co., Elyria, O., & N. Y. C.  
Jacoby, Henry E., N. Y. C.  
Pfaudler Co., Rochester, N. Y.

**Equipment—Plant (Used)**

DuPont Chemical Co., Inc., Wilmington, Del.

**Essential Oils**

Gazzolo Drug & Chem. Co., Chicago, Ill.  
Merck & Co., N. Y. C.  
National Aniline & Chemical Co., N. Y. C.

**Ether Apparatus (Builders of)**

Egret Co. Ltd., The, Paris, France.

**Evaporating Machinery**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Company, J. P., Buffalo, N. Y.  
Electric Heating Apparatus Co., N. Y. C.  
Hercules Engineering Co., N. Y. C.  
Jacoby, Henry E., N. Y. C.  
Lummus Co., The, Walter E., Boston, Mass.  
Sowers Mfg. Co., Buffalo, N. Y.  
Sperry & Co., D. R., Batavia, Ill.  
Stokes Mach. Co., F. J., Philadelphia, Pa.  
Swenson Evaporator Co., Chicago, Ill.  
Werner & Pfeiderer Co., Saginaw, Mich.  
Wheeler Condenser & Eng. Co., Carteret, N. J.  
Zaremba Company, Buffalo, N. Y.

**Evaporating Pans and Dishes**

Braun-Knecht-Heimann Co., San Francisco, Cal.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof).  
Eimer & Amend, N. Y. C.  
Elyria Enameled Prod. Co., Elyria, O., & N. Y. C.  
Engelhard, Charles, N. Y. C. ("Impervite")  
General Ceramics Co., N. Y. C.  
Guernseyware Co., The, Cambridge, O.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Herold China & Pottery Co., Golden, Colo.  
Knight, Maurice A., East Akron, Ohio.  
Palo Company, N. Y. C.  
Pfaudler Co., Rochester, N. Y.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Sowers Manufacturing Co., Buffalo, N. Y.  
Thermal Syndicate, Ltd., The, N. Y. C.  
U. S. Stoneware Co., The, Akron, O.  
Will Corporation, The, Rochester, N. Y.

**Evaporators—Crystallizing**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.

**Evaporators—Single and Multiple Effects**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Chemical Equipment Co., Chicago, Ill.  
Devine Co., J. P., Buffalo, N. Y.  
Wheeler Condenser & Eng. Co., Carteret, N. J.

**Exhaust and Ventilating Fans**

General Electric Co., Schenectady, N. Y.

**Exhaust and Ventilating Fans—Acid-Proof**

Duriron Castings Co., Dayton, O.

**Extracting Apparatus**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Devine Co., J. P., Buffalo, N. Y.  
Eimer & Amend, N. Y. C.  
Egrot Co., The, Paris, France.  
Fletcher Works, Philadelphia, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Lummus Co., The, Walter E., Boston, Mass.  
Scientific Materials Co., Pittsburgh, Pa.  
Tolhurst Machine Works, Troy, N. Y.  
Will Corporation, The, Rochester, N. Y.

**Extraction Cylinders**

Lummus Co., The, Walter E., Boston, Mass.

**Extraction Thimbles**

Angel Co., Inc., H. Reeve, N. Y. C.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C. ("Impervite")  
Hell Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.  
Will Corporation, The, Rochester, N. Y.

**Extraction Thimbles (Greens)**

Eimer & Amend, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Extractors—Centrifugal**

Fletcher Works, Philadelphia, Pa.  
International Equipment Co., Boston, Mass.  
Tolhurst Machine Works, Troy, N. Y.

**Fabroil Gears & Pinions**

General Electric Co., Schenectady, N. Y.

**Faucets (Stoneware—Acid Proof)**

General Ceramics Co., N. Y. C.  
Knight, Maurice A., East Akron, Ohio.  
U. S. Stoneware Co., The, Akron, O.

**Filter—Cel**

Celite Products Co., N. Y. C.

**Filter Cloth**

United Filters Corp., N. Y. C.

**Filter Cloth (Metallic)**

Multi-Metal Co., Inc., N. Y. C. (Monel Metal)  
United Filters Corp., N. Y. C.

**Filtering Media**

Celite Products Co., N. Y. C.  
General Filtration Co., Inc., Rochester, N. Y.  
Herold China & Pottery Co., Golden, Colo.

**Filter Paper**

Angel Co., Inc., H. Reeve, N. Y. C.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Gazzolo Drug & Chem. Co., Chicago, Ill.  
Hell Chemical Co., Henry, St. Louis, Mo.

**Palo Company, N. Y. C.**

Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Filter Paper Clippings**

Angel Co., Inc., H. Reeve, N. Y. C.

**Filter Presses (Laboratory & Industrial)**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Johnson Co., Inc., John, Brooklyn, N. Y.  
International Filtration Corp., N. Y. C.  
Jacoby, Henry E., N. Y. C.  
Kelly Filter Press Co., Salt Lake City, Utah.  
Lungwitz, E. E., N. Y. C.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Shriver & Co., T. Harrison, N. J.  
Sperry & Co., D. R., Batavia, Ill.  
Sweetland Filter Press Co., N. Y. City.  
United Filters Corp., N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Filter Pulp**

Angel Co., Inc., H. Reeve, N. Y. C.

**Filters**

Engelhard, Charles, N. Y. C. ("Impervite")  
General Filtration Co., Inc., Rochester, N. Y.  
Norton Co., Worcester, Mass.  
United Filters Corp., N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Filters—(Acid Proof)**

General Filtration Co., Inc., Rochester, N. Y.  
Knight, Maurice A., East Akron, Ohio.  
United Filters Corp., N. Y. C.

**Filters (Bone Black)**

Colwell, Lewis, Chicago, Ill.

**Filters, Folded**

Angel Co., Inc., H. Reeve, N. Y. C.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Filters—Vacuum**

American Continuous Suction Filter  
(See United Filters Corp., N. Y. C.)  
Devine Co., J. P., Buffalo, N. Y.  
General Filtration Co., Inc., Rochester, N. Y.  
United Filters Corp., N. Y. C.

**Filters—Water**

Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Booth Apparatus Co., Syracuse, N. Y.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
General Ceramics Co., N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Schutte & Koerting Co., Phila., Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Filtros**

General Filtration Co., Inc., Rochester, N. Y.

**Filtros—Vacuum**

Devine Company, J. P., Buffalo, N. Y.

**Fire Brick and Clay**

Denver Fire Clay Co., The, Denver, Colo.

**Flasks**

Braun-Knecht-Heimann Co., San Francisco  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Foamite Firefoam**

Foamite Firefoam Co., N. Y. C.

**Fuel Gas Plants**

Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.

**Funnels**

Denver Fire Clay Co., The, Denver, Colo.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Knight, M. A., East Akron, Ohio.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.





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21 Burling Slip, New York



## CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

**Furnaces—Annealing—(Hardening and Enameling)**

Abbé Engineering Co., N. Y. C.  
Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Electric Heating Apparatus Co., N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Furnaces (Case Hardening, Rivet and Bolt)**

Abbé Engineering Co., N. Y. C.  
Denver Fire Clay Co., The, Denver, Colo.

**Furnaces—Electric Laboratory**

Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Electric Heating Apparatus Co., N. Y. C.  
Engelhard, Chas., N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Griebel Instrument Co., Carbondale, Pa.  
Hanovia Chemical & Mfg. Co., Newark, N. J.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Thermo-Electric Instrument Co., Newark, N. J. (Tube)  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Furnaces—(Forge)**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Denver Fire Clay Co., The, Denver, Colo.  
Electric Heating Apparatus Co., N. Y. C.  
Hoskins Mfg. Co., Detroit, Mich.

**Furnaces (Melting, Oil or Gas)**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Electric Heating Apparatus Co., N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Furnaces—Muffle**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Electric Heating Apparatus Co., N. Y. C.  
Engelhard, Charles, N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Galvanizing and Jars—Tanks Acid Proof**

Knight, M. A., East Akron, O.

**Gas Analyzers**

Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Will Corporation, The, Rochester, N. Y.

**Gas Apparatus**

Braun-Knecht-Heimann Co., San Francisco Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Schutte & Koerting Co., Phila., Pa.  
Will Corporation, The, Rochester, N. Y.

**Gas Machines**

Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Gas Masks**

Mine Safety Appliances Co., Pittsburgh, Pa.

**Gas Pipe & Fittings (Chamotte)**

Knight, M. A., East Akron, O.

**Gaskets**

Sarco Co., Inc., N. Y. C.

**Gauges—Recording, Suction & Vacuum**

Brown Instrument Co., Philadelphia, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Will Corporation, The, Rochester, N. Y.

**Gauze—Base Metal**

Hoskins Mfg. Co., Detroit, Mich.  
Will Corporation, The, Rochester, N. Y.

**Generators (Electro-Oxy-Hydrogen)**

Shriver & Co., T. Harrison, N. J.

**Glass Blowing**

Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Greiner Co., Emil, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Glass Enamelled Apparatus**

Elyria Enamelled Prod. Co., Elyria, O., & N. Y. C.  
Pfaudler Co., Rochester, N. Y.

**Graduates—Glass**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Grinders**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Denver Fire Clay Co., The, Denver, Colo.  
Raymond Bros. Impact Pulv. Co., Chicago, Ill.  
Williams Pat. Crusher & Pulv. Co., Chicago, Ill.  
Will Corporation, The, Rochester, N. Y.

**Hardness Testing Apparatus**

Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Heating Apparatus (Laboratory)**

Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Electric Heating Apparatus Co., N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Heating—Industrial (Merrill Process)**

Parks-Cramer Co., Boston, Mass.

**Heating Specialties**

Detroit Heating & Lighting Co., Detroit, Mich.

**Hot Plates (Laboratory Purposes)**

Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Electric Heating Apparatus Co., N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Sowers Manufacturing Co., Buffalo, N. Y.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Hydro Extractors**

Fletcher Works, Philadelphia, Pa.  
Tolhurst Machine Works, Troy, N. Y.

**Hydrochloric Acid Apparatus**

Knight, M. A., East Akron, O.

**Hydrometers**

Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Brown Instrument Co., The, Phila., Pa.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Greiner Co., Emil, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Hell Chemical Co., Henry, St. Louis, Mo.  
New York Thermometer Co., The, N. Y. C.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Scientific Utilities Co., Inc., N. Y. C.  
Taylor Instrument Cos., Rochester, N. Y.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Hydroturbine**

Nash Engineering Co., So. Norwalk, Ct.

**Hygrometers**

Engelhard, Charles, N. Y. C. (Electric)  
Hell Chem. Co., Henry, St. Louis, Mo.  
Taylor Instrument Cos., Rochester, N. Y.  
Will Corporation, The, Rochester, N. Y.

**Ice-Making Machinery**

General Electric Co., Schenectady, N. Y.  
York Manufacturing Co., York, Pa.

**Ichthylol**

Merck & Co., N. Y. C.

**Impregnating & Drying App.—Vacuum**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.

**Incubators—Surgical**

Central Scientific Co., Chicago, Ill.  
Hell Chem. Co., Henry, St. Louis, Mo.  
New York Thermometer Co., The, N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Indicating Instruments**

Brown Instrument Co., The, Phila., Pa.  
Engelhard, Charles, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Taylor Instrument Cos., Rochester, N. Y.  
Will Corporation, The, Rochester, N. Y.

**Indicators—(Speed & Test)**

Brown Instrument Co., Philadelphia, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Industrial Burners & Specialties**

Hell Chem. Co., Henry, St. Louis, Mo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Will Corporation, The, Rochester, N. Y.

**Industrial Motor Control Devices**

General Electric Co., Schenectady, N. Y.

**Injectors for Acids and Gases**

Knight, M. A., East Akron, O.

**Insulation—Fibre (Electrical)**

Diamond State Fibre Co., Bridgeport, Pa.

**Insulation—Heat**

Magnesia Association of America, N. Y. C.

**Iodine—Salts of**

Merck & Co., N. Y. C.

**Iron (Hard)**

Ferguson & Lange Foundry Co., Chicago, Ill.

**Iron—Metal and Salts**

Merck & Co., N. Y. C.

**Jars, Museum & Specimen**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Jars (for Primary & Storage Batteries)**

Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
General Ceramics Co., N. Y. C.  
Guernseyware Co., The, Cambridge, O.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Jars and Jugs (Acid Storage)**

General Ceramics Co., N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Herold China & Pottery Co., Golden, Col.  
Knight, M. A., East Akron, O.  
U. S. Stoneware Co., The, Akron, O.  
Will Corporation, The, Rochester, N. Y.

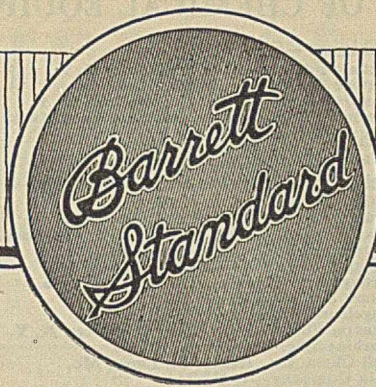
**Kettles—Caustic Soda**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Ferguson & Lange Foundry Co., Chicago, Ill.

**Kettles (Chemical Castings)**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Devine Co., J. P., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Durrion Castings Co., N. Y. C. (Acid Proof)  
Garrigue & Co., William, Chicago, Ill.  
Jacoby, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.





*A guarantee  
of Service*

*As well as  
Quality*

# Refined Coal Tar Products

**BENZOLS  
TOLUOLS  
XYLOLS**

**PHENOL  
CRESOLS  
NAPHTHALENE**

**COAL TAR NAPHTHAS  
TAR ACID OILS  
SHINGLE STAIN OILS  
ALPHA-NAPHTHYLAMINE  
NITRONAPHTHALENE  
TECHNICAL RESORCINOL**

## **DISINFECTANTS**

Our disinfectants are of the emulsifiable coal tar type—made from our own oils—and tested for coefficient in our own bacteriological laboratory. Every precaution is taken to insure high and uniform quality.

COEFFICIENTS RANGE FROM 2 TO 20.

## **LIQUOR CRESOLIS COMPOSITUS U. S. P.**

Both this U. S. P. Cresol Compound and our own commercial Barrett Cresol Compound are most carefully prepared from our own Cresols.

## **ETERNIUM PAINT**

A black paint—impervious to moisture and highly resistant both to acids and alkalis.

The **Barrett** Company

Chemical Department

17 Battery Place



New York, N. Y.



## CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

**Kettles (Lined)**

Devine Co., J. P., Buffalo, N. Y.  
Elyria Enamelled Prod. Co., Elyria, O., N.Y.C.  
Egrot Co., Ltd., The, Paris, France.  
Pfaudler Co., Rochester, N. Y.  
Sowers Mfg. Co., Buffalo, N. Y.

**Kettles (Steam Jacketed)**

Bethlehem Fdy. & Mch. Co., So. Bethlehem, Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Elyria Enamelled Prod. Co., Elyria, O.  
Jacoby, Henry E., N. Y. C.  
Pfaudler Co., Rochester, N. Y.  
Sowers Mfg. Co., Buffalo, N. Y.  
Sperry & Co., D. R., Batavia, Ill.  
Stevens Brothers, N. Y. C.  
Werner & Pfeiderer Co., Saginaw, Mich.

**Kettles (Stoneware)**

General Ceramics Co., N. Y. C.  
Knight, M. A., East Akron, O.  
U. S. Stoneware Co., The, Akron, O.  
Will Corporation, The, Rochester, N. Y.

**Kilns (Bone Black)**

Celwell, Lewis, Chicago, Ill.

**Laboratory Apparatus—Designing & Manufacturing**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C.  
Fletcher Works, Philadelphia, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Laboratory Burners**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Laboratory Gas Machine**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Laboratory Supplies**

Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C.  
Greiner Co., Emil, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Kauffman-Lattimer Co., The, Columbus, Ohio.  
Palo Company, N. Y. C.  
Reynolds, Teschner & Volk Co., N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Schaar & Co., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Scientific Utilities Co., Inc., N. Y. C.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Lacquer**

General Bakelite Co., N. Y. C.

**Lactic Acid**

Hell Chem. Co., Henry, St. Louis, Mo.

**Lamps—Arc and Incandescent**

General Electric Co., Schenectady, N. Y.

**Leaching Cells**

Swenson Evaporator Co., Chicago, Ill.  
Zaremba Co., Buffalo, N. Y.

**Lead Burning**

McNamara, M., Everett, Mass.

**Lead (Red)**

Eagle-Picher Lead Co., The, Chicago, Ill.

**Lead Lined Pipe and Accessories**

Schutte & Koerting Co., Phila., Pa.  
United Lined Tube & Valve Co., Boston, Mass.

**Lightning Arresters**

General Electric Co., Schenectady, N. Y.

**Lime**

Mitchell Lime Co., Mitchell, Ind.

**Linings, Cupola**

Herold China & Pottery Co., Golden, Col.

**Liter (Gas)**

National Liter Co., Chicago, Ill.

**Litharge**

Eagle-Picher Lead Co., The, Chicago, Ill.

**Lubricants**

Dearborn Chemical Co., Chicago, Ill.

**Machinery—Chemical**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Sowers Manufacturing Co., Buffalo, N. Y.  
Werner & Pfeiderer Co., Inc., Saginaw, Mich.

**Machinery—Electrical**

General Electric Co., Schenectady, N. Y.

**Machinery—Mining and Metallurgical**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C.  
General Filtration Co., Inc., Rochester, N. Y.  
Hoskins Mfg. Co., Detroit, Mich.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Stokes Machine Co., F. J., Philadelphia, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Machinery—Mixing and Kneading**

Abbé Engineering Co., N. Y. C.  
Elyria Enamelled Products Co., Elyria, O.  
Jacoby, Henry E., N. Y. C.  
Pfaudler Co., Rochester, N. Y.  
Sowers Mfg. Co., Buffalo, N. Y.  
Werner & Pfeiderer Co., Saginaw, Mich.

**Machinery—Pulverizing**

Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Raymond Bros. Impact Pulv. Co., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Machinery (Special)**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Jacoby, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.  
Stevens Brothers, N. Y. C.  
Werner & Pfeiderer Co., Saginaw, Mich.

**Magnesium—Metal and Salts**

Dow Chemical Co., The, Midland, Mich.  
Lithflux Mineral & Chem. Wks., Chicago, Ill.  
Merck & Co. N. Y. C.

**Mercury—Salts of**

Atlas Powder Co., Wilmington, Del.  
Merck & Co., N. Y. C.

**Metallographic Apparatus**

Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Hommel & Co., Ludwig, Pittsburgh, Pa.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Meters—Flow Air, Gas, Water**

General Electric Co., Schenectady, N. Y.

**Microscopes**

Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Microtomes**

Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Micro Photo. Apparatus Mfrs.**

Bausch & Lomb Optical Co., Rochester, N. Y.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Hommel & Co., Ludwig, Pittsburgh, Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Mills—Ball, Pebble and Tube**

Abbé Engineering Co., N. Y. C.  
Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Will Corporation, The, Rochester, N. Y.

**Mixers**

Elyria Enamelled Products Co., Elyria, O.  
Pfaudler Co., Rochester, N. Y.

Sowers Mfg. Co., Buffalo, N. Y.

Werner & Pfeiderer Co., Saginaw, Mich.

**Mixing Tanks**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Elyria Enamelled Prod. Co., Elyria, O.  
Lummus Co., The, Walter E., Boston, Mass.  
Pfaudler Co., Rochester, N. Y.  
Sowers Mfg. Co., Buffalo, N. Y.  
Stevens Brothers, N. Y. C.  
Werner & Pfeiderer Co., Saginaw, Mich.

**Molybdates**

Gazzolo Drug & Chemical Co., Chicago, Ill.  
Merck & Co., N. Y. C.

**Molybdc Acid**

Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Montejus—Automatic**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
General Ceramics Co., N. Y. C.  
Knight, Maurice A., East Akron, O.  
Schutte & Koerting Co., Phila., Pa.

**Mortars & Pestles**

Central Scientific Co., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.

**Muffles**

Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Engelhard, Charles, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.  
Scientific Materials Co., Pittsburgh, Pa.  
Will Corporation, The, Rochester, N. Y.

**Nickel—Salts of**

Merck & Co., N. Y. C.

**Nitrating Apparatus**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Fletcher Works, Philadelphia, Pa.  
Tolhurst Machine Works, Troy, N. Y.

**Nitrating Pots**

Bethlehem Fdy. & Mch. Co., S. Bethlehem, Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O. (Acid Proof)  
Elyria Enamelled Products Co., Elyria, O.  
General Ceramics Co., N. Y. C.  
Knight, Maurice A., East Akron, O.  
Pfaudler Co., Rochester, N. Y.  
Sowers Mfg. Co., Buffalo, N. Y.  
Stevens Bros., N. Y. C.  
U. S. Stoneware Co., The, Akron, O.  
Werner & Pfeiderer Co., Saginaw, Mich.

**Nitrators—Sulphonators, Etc.**

Bethlehem Fdy. & Mch. Co., S. Bethlehem, Pa.  
Stevens Bros., N. Y. C.

**Nitre Pot Acid Pipes**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Duriron Castings Co., Dayton, O. (Special Alloy)  
Elyria Enamelled Prod. Co., Elyria, O.  
General Ceramics Co., N. Y. C.  
Thermal Syndicate, Ltd., N.Y.C. (Silica Waste.)

**Nitric Acid Apparatus**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Duriron Castings Co., Dayton, O.

**Nitric Acid Plants—Chemical Stoneware**

Knight, Maurice A., East Akron, O.

**Nitric Acid Retorts**

Buffalo Fdy. & Mch. Co., Buffalo, N. Y.

**Nozzles (Acid Proof)**

Duriron Castings Co., Dayton, O.  
Schutte & Koerting Co., Phila., Pa.

**Oil Testing Instruments**

Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Denver Fire Clay Co., The, Denver, Colo.  
Eimer & Amend, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Taylor Instrument Cos., Rochester, N. Y.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Will Corporation, The, Rochester, N. Y.