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## EDITORIALS

### PLATINUM AT WHITE HEAT

During the past month the subject of platinum has leaped to the forefront in the thoughts of the American people. For the first time, and by one of those quick processes of general understanding which have characterized this war period, the nation has learned the important bearing of this element upon the part we are to play in the war.

The chief factor in arousing this interest and understanding has been the widespread publicity given by the press to the stirring address delivered in the House of Representatives on June 7 by Hon. Henry T. Rainey, Congressman from Illinois. In this address he depicted clearly the important function of platinum in explosives manufacture, the inadequate measures taken for its conservation by those charged with this responsible duty, and the factors which had contributed to such inadequacy.

Since the delivery of that address many things have come to light which would prove interesting reading; editorial discussion of these, however, will be postponed. Only one conviction is expressed here—the *immediate place for the platinum in this country is in the vaults of the Treasury Department.*

### THE MODERN MIRACLE

Scarcely two years have elapsed since many lines of our industrial life were threatened with utter demoralization because of the shortage of dyestuffs and medicinals resulting from the blockade of German ports by the British navy. Textile mills faced the imminent possibility of shutting down because of inability to secure dyestuffs for their fabrics. Tanners, lithographers, and wall paper men sought in vain for needed coloring matter, and pharmacists' stocks of many much-used medicinals became depleted. On account of these shortages and the accompanying speculation in the remaining stocks, prices soared to undreamed-of heights.

Yet within this short period of two years, one of which has been devoted to war preparations, a miracle has been wrought. Mills have not closed; all lines of industrial life requiring synthetic colors are operating under normal conditions; the sick have been provided with ample medicinal supplies; prices have been largely reduced and are comparable with prices of all other commercial products; in addition, ships have borne from America to other parts of the world large quantities of dyestuffs.

No more fitting tribute could be paid to the skill and energy of the American chemist than has been done through the medium of two government announcements which have been issued during the past month, the one by the U. S. Tariff Commission and the other by the War Industries Board.

In the statement sent out by the Tariff Commission the results of its census of synthetic dyestuffs and medicinals are given (page 582, this issue). According to these figures the importation of dyestuffs in the

fiscal year 1914 was 45,840,866 pounds, while in 1917 the domestic production amounted to 45,977,246 pounds. The tonnage has been made good and even exceeded. While it is true that there is a difference "in the relative amounts of the various classes of dyes" in the two periods mentioned, it is interesting to note, as the Commission's statement points out, that the lines which have failed of their full share of development have been exactly those to which were given only the *ad valorem* duty of 30 per cent, while in those classes which were given both the 30 per cent *ad valorem* and the 5 cents per pound special duty "the American manufacturers have shown remarkable progress." The statements of manufacturers and consumers at the hearings on the Hill bill are abundantly confirmed by the announcement of the Tariff Commission. By the enactment of tariff and anti-dumping legislation, capital was attracted to the industry, and the chemist has made good.

As to medicinals, the War Industries Board, in an authorized statement in the *Official Bulletin* of June 6, 1918, says:

"Actual or prospective shortages have come to the notice of the Board in but few instances so far as medicine and medicinal chemicals are concerned.\* \*\*"

Congress, capital, and chemists coöperating have accomplished the modern miracle.

### AN ARMY WITHOUT RESERVES

General Foch has aroused the enthusiasm of the allied world by the masterly manner in which he has handled the reserves during the recent mammoth drives of the German army. These struggles have fitly been designated as "the battles of reserves." The morale of all the nations joined in the contest against German aggression has been stimulated by the conviction that an endless flow of reserves is proceeding with all possible haste from American to European shores. In the matter of reserves for the armies in the field all goes well.

The army of American chemists is now in rapid process of complete mobilization. At last the matter of efficient utilization of chemists has been grasped with a firm hand by those in authority. The orders issued by the Secretary of War on May 28, 1918 (page 580, this issue) show that the *may* of previous orders has been changed to *must*, and under these new orders the full strength of the chemistry forces will soon be reached. It appears that every possible contingency as to graduate chemists has been provided for. Congratulations to Secretary Baker and to those who have aided him in the preparation of these comprehensive orders!

With the thus mobilized army of chemists all goes well. But what about the reserves for this army? We are preparing for a long war, how long no one knows, but certainly as long as is necessary to insure the triumph of the principles to which we have dedicated our all. Daily grows the expansion of the con-

ception of the vast forces we must put in the field, on the sea and in the air. Increases in personnel necessitate similar expansions in the program of chemical industries, and these in turn must be manned by chemists. It is full time therefore that serious consideration should be given to the matter of reserves for this army of chemists. The source of reserves is in the universities and colleges, and we beg to urge the most broad-minded and far-sighted consideration of this problem by three groups of men: university administrators, War Department officials, and leaders in the chemical industries.

#### UNIVERSITY CONDITIONS

The universities represent the training camps for the reserves of the chemistry army. These institutions function through their chemistry staffs, their physical equipment, and through the number of undergraduates who present themselves for the chemistry courses.

At the present moment the instructional staffs of most of the universities are in a completely demoralized condition. Many professors and associate professors are on leave of absence in Washington engaged in research on problems vital to the winning of the war and the prevention of needless sacrifice of the lives of our men at the front. No more important work could engage their attention. Others are busy with similar problems in the private laboratories of the universities. The concentrated effort required in such undertakings does not admit of much energy being devoted to effective teaching. On the other hand the university men of lower rank, the instructors and assistants, charged primarily with the instruction of the lower classes, are for the greater part subject to the draft. In certain unusual cases deferred classification has been given such men by Local Boards. These cases, however, are sporadic and form no part of a general policy. Continued low salaries in universities, made necessary by decreased budgets, the high salaries paid in the industries, and the increased cost of living have forced many men from the universities into the industries. The net result is a burning of the candle at both ends, so far as the forces for training reserves are concerned. Bi-terminal combustion is considered a reckless policy in all other matters, why not in this?

In the ranks of the students, present year seniors have already left university campuses and are now either in uniform or in industrial plants. The matter of post-graduate students is evidently a thing of past history until at least the ending of the war. Furthermore, the operation of the draft law (in whose principles we most emphatically believe) and the need of men in the chemical industries have combined to take away from the universities many of the students in the junior classes. Meanwhile preliminary reports for next Fall indicate the largest enrollment of students intending to make chemistry their life work ever known in our educational history. This is only natural in view of the greater recognition given to the importance of chemistry in war work and to its

value in every channel of industrial life. These are the men who constitute the chemistry reserves. They will soon be in the training camps of the universities. Will they find sufficient officers present to give them adequate instruction for their development into efficient reserves?

To add to the complexities of the situation there is no question about the crippled finances of the universities and of their helplessness in preventing the present drain upon their corps of instructors.

These are the conditions, as we see them, in this most critical year of our country's history. Certainly such conditions demand the most earnest consideration on the part of all who can in any way contribute to their amelioration. From what sources may help reasonably be expected?

#### ACADEMIC ASSISTANCE

First, assistance must come from the universities themselves. Such aid cannot be in the nature of increased appropriations, for university finances are already too hard hit. But they can in some instances shake themselves loose from traditions and modify internal organization and the character of courses to meet as far as possible the pressing need of the times. An example of such increased efficiency is shown by the merger of the University and the Sheffield Scientific School departments of chemistry at Yale University, recently announced.

It may be possible in some cases for institutions to combine forces. Many difficulties in such a plan present themselves, though these difficulties may not be as great as at first thought they seem. A certain minimum laboratory space is considered necessary per student. Possibly this space may be more efficiently utilized, at least in the lower classes, by repeated using of the same space by more than one student, in spite of the difficulty which at once suggests itself as to responsibility for equipment. The number of men who can be taught properly by an instructor is limited. This is a real difficulty, and sacrifice may be necessary here. Other courses must be taken besides chemistry and this may present physical difficulties. Hardships may be enforced upon individual students from living conditions under such combined institutions. This is a question of finance for which a remedy might be found. These are but types of the many perplexing academic problems to whose solution the best thought of our university men will be unstintedly given.

#### AID FROM THE WAR DEPARTMENT

Second, no word of exhortation to the War Department is necessary in the matter of instructors for chemical reserves. The provision of such is only the logical following out of the principles now being applied in the creation of our armies. Reserves are of course essential. These are of no value unless adequately trained. For this training instructors are required. In the training camps to-day are many soldiers who could render effective service on the fighting front, yet they are of greater service to the army in the

work of developing the raw recruit. How then can the War Department aid in the provision of instructors for chemistry students? In one of at least two ways: Either by granting class exemption to instructors in chemistry or by detailing men already in the service to instructional work in the university lecture rooms and laboratories. If exemption as a class should be given, then some distinguishing emblem should accrue to such men, for the time is at hand when peculiar glances are cast at young men of draft age who are not attired in khaki. If on the other hand it is deemed best that they should be enlisted and detailed back to universities, then it is fortunate that under the present orders of the Secretary of War the work of detailment would be in the hands of the Chief of the Chemical Service Section, Lieutenant Colonel Bogert, whose long and distinguished career as a teacher in one of our greatest universities preëminently qualifies him for such a duty. Time is an important factor in the matter, however, for every day that lapses after the opening of the universities next Fall is just so much loss in the work these soldier instructors will be expected to perform.

Of course the time may come when it will be advisable to close university doors and stop many other lines of daily effort, in the hope of putting across the one great blow for victory. That time does not seem to have arrived yet. Our enemies are apparently pursuing that policy now, and judging from the results to date it is proving disastrous for them.

#### SUPPORT FROM THE CHEMICAL INDUSTRIES

Third, the future of the industries depends upon no factor more than upon the output of chemists from the universities. Wonderful progress has been made in these industries in the past, particularly during the three preceding years. Much more must be accomplished, if we are not to rest content simply with doing as well as some other country has done. Within university walls next year will be young men who some day will have to take up the reins now so ably held by the present leaders. Already the industries have attracted to their staffs many valuable university teachers. We cannot eat our cake and have it too. Furthermore, the industries have already drained the universities of all available students possessing sufficient training to go into works laboratories. We believe that the heads of industrial laboratories will bear out the statement that the standard of qualifications of student accessions to industrial staffs has within the past two years been decidedly lowered. If this condition becomes worse its deleterious effect will be markedly shown in the industries during the next decade. Foresight is called for.

How can the industries aid? By giving generously of their well earned profits of recent years to strengthen and build up the chemistry departments of the universities. It is not a question of charity or philanthropy, but can well be regarded as an investment. With the aid of funds from this source the pay of professors and instructors can be increased, thereby diminishing the necessity for leaving university ranks because of the increased cost of

living. So, too, equipment of university laboratories can be fully maintained and improved. The account (page 581, this issue) of the anonymous gift to the Massachusetts Institute of Technology for the purpose of further improvement in its equipment for chemistry and physics shows that someone's mind is moving in this channel. Finally, many of the ablest students are in need of financial assistance if they are to get the best training the universities offer. The creation of scholarships and fellowships will do much to alleviate this situation, aside from the stimulation induced by prospective rewards of good work. An important step in this direction has just been taken by the du Pont Company in setting aside a portion of its earnings for this purpose (page 581, this issue). Many other ways of aiding through funds will suggest themselves if once our industrial leaders are convinced that in this matter they have just as important an interest as in the purchase of raw material or in the efficiency of plant operations.

The chemistry army must have its reserves. Good generalship will provide these.

#### A FRENCH LOCAL SECTION

Another tie joins France and America; another offspring of the American Chemical Society takes its place in the family of local sections. On page 575 of this issue is printed the application to the Council for permission "to found in Paris a French section of the Society covering the entire territory of France."

As the usual Spring meeting of the Council was not held, this application for charter is now being voted upon by the Directors. It requires no gift of prophecy to predict that the vote will be of such enthusiastic unanimity as never before characterized a vote of the Directors. The first public announcement of the application, made by Secretary Parsons, on the request of President Nichols, at the recent joint outing of the Philadelphia and Delaware Sections, was greeted with tremendous applause.

The signatures on the application are those of distinguished French chemists intermingled with those of American chemists, known to us all, who are now at the front in the service of our Army, a joint brigading of French and American forces similar to that which has been affected recently between units of the respective armies under the leadership of that great soldier, General Foch. The successful result of the military union has already made itself evident on the battlefields of France; with equal confidence we can look forward to increased strength from the closer coöperation of scientific forces through the medium of the French Section.

As we read the application for charter and note the words "the entire territory of France" we know that these words will carry only one meaning to French and American chemists alike, namely a restoration of the eastern boundary of France, changed from that of August 1914 only in that it shall include Alsace and Lorraine, and to that end the entire resources of this country, men and material, are now dedicated.

All hail to the French Section!

# ORIGINAL PAPERS

## THE MANUFACTURE OF AMYL ACETATE AND SIMILAR SOLVENTS FROM PETROLEUM PENTANE

By BENJAMIN T. BROOKS, DILLON F. SMITH AND HARRY ESSEX

Received March 12, 1918

In a recent paper<sup>1</sup> we noted the effect of very high pressures on the conversion of chlorpentane to alcohol. In the following paper we describe a method for the conversion of chlorpentane to the corresponding acetates under conditions which we believe are capable of realization on a large scale without great difficulty and without very costly or complicated apparatus.

The present prices of amyl alcohol and acetate are, of course, abnormal. Yet it is very doubtful if the prices during several years before the war will be the rule for some time after the war, unless there is a large production from some source not now known or the synthetic article is brought into the market in large quantities. Before the war this country imported annually approximately 6,000,000 lbs. of fusel oil from Russia. The decrease in the manufacture of distilled liquors and the spread of prohibition generally will cause an increased shortage of amyl alcohol and acetate.

As is well known to all familiar with fusel oil and amyl acetate and their uses, these terms are employed to describe the mixture of amyl alcohols, or their acetates, together with more or less butyl and hexyl derivatives. The composition of the natural fusel oils varies considerably with the material fermented and the character of the fermentation. Fernbach's recently developed fermentation, yielding chiefly isobutyl alcohol, is an extreme case, and isobutyl alcohol alone is too volatile for most of the technical uses of fusel oil or amyl acetate. The following analyses from Worden's "Nitrocellulose Industry" illustrate what is commonly found in commercial fusel oils:

### COMPOSITION OF NATURAL FUSEL OILS

FROM POTATO SPIRIT		FROM CORN SPIRIT	
	Per cent by wt.		Per cent by wt.
Normal butyl alcohol...	6.8	Normal propyl alcohol...	3.7
Isobutyl alcohol.....	24.3	Isobutyl alcohol.....	15.7
Amyl alcohol.....	67.8	Amyl alcohol.....	75.8
Fatty acids, etc.....	0.04	Hexyl alcohol.....	0.2
		Fatty acids, etc.....	0.56

The ordinary amyl acetate of commerce contains approximately 70 per cent by weight, distilling within the limits 135 to 140°. The above analyses make clear the fact that it is not necessary to isolate either pure pentane as a raw material for synthetic amyl acetate, or a single monochlorpentane to make an artificial amyl acetate fulfilling all the ordinary requirements of industrial uses.

A fairly large number of patents have been issued which have for their object the manufacture of amyl acetate from light petroleum mixtures, chiefly pentane. The difficulties of most of these processes, so far as we are acquainted with them, appear to be, first, the formation of large proportions of dichlor and trichlor

derivatives when the original hydrocarbon mixture is chlorinated, entailing considerable loss of chlorine, decomposition during distillation with evolution of hydrochloric acid, corrosion of the distilling apparatus, etc. The presence of higher chlorinated pentanes also has the very objectionable result that on decomposition, as during distillation, chloramylenes are formed which are relatively stable and make their presence known in the final synthetic amyl acetate by materially decreasing the solubility of many substances in this solvent. One patentee claims that glacial acetic acid need not be used as a solvent in the chlorpentane-sodium acetate reaction, but acetic acid containing as much as 30 per cent of water may successfully be employed. Our experience is that amyl acetate made with acetic acid containing 10 per cent or more of water, contains considerable unchanged chlorpentane, the presence of which markedly decreases its solvent value. The second serious difficulty, common to all the processes known to us, consists in the formation of relatively large amounts of amylenes.

The first of the difficulties above mentioned has been overcome almost completely and in a very simple manner.

### CHLORINATION OF PENTANE

In the earlier period of our work we lost considerable time by attempting to develop a satisfactory method of chlorination based on the idea that the reaction should take place in the gaseous phase. Accordingly, a large number of chlorinations were made by introducing chlorine into the hydrocarbons at temperatures just sufficient for the complete vaporization of the hydrocarbon. In these experiments we assumed that the chlorine and hydrocarbon gas mixture should be thoroughly mixed before reacting, and this was attempted by introducing the chlorine into the hydrocarbon gases in the dark and then passing this mixture through large illuminated glass tubes. In these experiments it was found that once the chlorinating action had started it would proceed very smoothly in diffused daylight and that ultraviolet light as a catalyzing agent was not necessary; indeed, the reaction often proceeded with considerable violence and the separation of some free carbon. It was difficult to regulate accurately the ratio of hydrocarbon vapors to the chlorine introduced, and the formation of dichlor and trichlor derivatives was unavoidable. The highest yields of monochloride derivatives were obtained when the chlorine-hydrocarbon mixture contained a large excess of hydrocarbons, namely, 2 to 4 times the ratio necessary to form the monochloride derivatives. By employing mixtures containing 3 mols. of hydrocarbon to one of chlorine, a yield of monochloride of 88 per cent of the theoretical was obtained, that is, 88 per cent of the chlorinated material was monochloride. The large volume of hydrogen chloride formed in this reaction carries off large amounts of pentane vapor, this fact necessitating the ab-

<sup>1</sup> *J. Am. Chem. Soc.*, **38** (1916), 1369.

sorption of the hydrogen chloride in cold water in order to recover this pentane. It was easily apparent that the manufacture on a large scale of crude chloropentane by this method would entail great practical difficulties, and would certainly add a great deal to the cost of this comparatively simple operation.

After a number of preliminary experiments, which need not be recorded here, a method was developed which has proved to be quite satisfactory and which has served for the preparation of large quantities of these chlorinated hydrocarbons. The simplicity of the method makes it entirely adaptable to large scale operations, and a fairly large experience with it over more than one year's time shows that the chlorinated product consists of the monochlorides to the extent of 90 to 94 per cent.

The principle of the method simply consists in maintaining always a very large excess of hydrocarbon as compared with chlorine and also, which is very important, a very large ratio of hydrocarbon to chlorinated products. In brief, the method consists in passing chlorine through a large number of small orifices into a large quantity of cold crude pentane, and stopping the chlorination before the concentration of the chlorine derivatives becomes greater than about 20 per cent of the mixture. In practice, escaping hydrogen chloride is absorbed in cold water and the pentane thus recovered is returned to the reaction vessel. The presence of moisture is not objectionable, in fact it seems to be desirable. Illumination of the reaction mixture is not absolutely necessary, although we have employed a high-power tungsten light bulb in the upper part of the reaction vessel, the light catalyzing the reaction at the start. This reaction exhibits a peculiar phenomenon. At first the pentane appears to dissolve the chlorine unchanged, but after an interval of about ten minutes the color of free chlorine quickly fades and disappears and then chlorine may be passed in very rapidly, reacting as fast as dissolved. The liquid should be kept chilled to at least 10° to prevent too great vaporization of pentane with the hydrochloric acid formed.<sup>1</sup>

The control of the process can readily be accomplished by observing the specific gravity of the mixture, since a specific gravity of 0.820 corresponds to about 20 per cent of monochlorides, when a crude pentane fraction boiling at 25 to 45° is taken for chlorination.

In order to illustrate the per cent of suitable crude pentane contained in a high grade of gasoline, 76° Bé., the following table of fractions obtained by slow distillation through suitable column is given:

Boiling Point	Per cent by Volume
28-30°	2.3
30-35°	5.0
35-40°	5.5
40-45°	5.0
45-50°	6.6
50-55°	8.5
55-60°	8.5
60-70°	15.0

} 17.8 per cent suitable for amyl acetate

Casing head or natural gas gasoline naturally contains the largest per cent of pentane and when suitable provision is made to avoid losing the butane, as by

dissolving in a heavier gasoline, this raw material should prove the most economical.

The fractional distillation of the monochlorides furnishes another opportunity to reject material of unsuitable boiling point. A distillation analysis of the monochlorides employed in most of the work here described, the chlorides having been made from a crude pentane fraction boiling point 25 to 45°, is given below:

Boiling Point	Per cent by Weight
90-100°	3.8
100-110°	46.6
110-120°	27.4
120-130°	14.4
130-140°	5.3
Above 140°	2.4

Several results which are typical of those obtained by chlorinating the cold pentane are given below. The per cent yield is calculated on the per cent of monochlorides in the total chlorinated hydrocarbon, not on the amount of pentane originally taken. Thus if the yield is 90 per cent, the balance of 10 per cent is a higher chlorinated product.

TABLE I—CHLORINATION OF CRUDE PENTANE, BOILING POINT 25-45°

Wt. Pentane Grams	Time Hours	Lost with HCl Per cent	Wt. Product Grams	Un-changed Grams	Monochlorides 95-140° Grams	Dichlorides Grams
5500	4.5	13.4	5233	3908	1155	170
5500	5.5	35.0	4108	2555	1371	182
5500	4.0	29.0	4206	3312	884	10
2500	4.0	25.0	2698	1775	611	47
2070	5.5	28.0	1492	975	407	110
3700	6.0	25.0	2781	2309	444	28

Concentration of chlorides when chlorination was stopped

Per cent monochlorides in chlorinated part
21
24
16
24
34
17
87
89
99
93
78
95.5

If desired, a pentane fraction of much smaller boiling point range can be employed as the initial raw material, and this will result naturally in a final acetate of smaller range of boiling points. The fractional distillation to obtain most of the crude pentane used in our work was carried out in a small 50-gallon experimental apparatus. More homogeneous fractions would be obtainable from larger stills provided with suitable columns, such as those employed for the rectifying of crude benzols.

A small amount of hydrochloric acid is formed during the distillation of the crude chloropentane, due to slight decomposition of the chlorides. The amount of decomposition resulting from this cause is so small, however, that no appreciable diminution of the yield of crude chloropentane results. This fact has to be taken account of, however, in the choice of apparatus for distillation, as will be brought out later.

#### CONVERSION OF CHLOROPENTANE INTO AMYL ACETATE

Although the conversion of chloropentane into amyl acetate by heating with anhydrous sodium acetate is, at first sight, merely a direct application of a standard method of organic synthesis, we found it necessary to carry out a large number of experiments in order to determine the optimum conditions, particularly as regards temperature and pressure, nature of solvent, effect of agitating the reaction mixture, yield from

<sup>1</sup> Brooks, Essex and Smith, United States Patent No. 1,191,196.

various gasoline fractions, material of construction for apparatus, effect of catalysts, and possible utilization of the by-product amylene.

Early in the course of the work we determined that somewhat better yields of acetate are obtainable with the lower boiling fractions than from hydrocarbons of higher boiling point. Kerosene chlorides are highly unstable, decomposing slowly at room temperature to such a degree that in several cases the pressure of the HCl developed was sufficient to expel the cork from well stoppered containers. The tendency of the alkyl chlorides to decompose with the formation of olefines increases with those of higher molecular weight, as is shown by the following results.

TABLE II—YIELD OF ACETATE FROM CHLORINATED GASOLINE FRACTIONS<sup>1</sup>

Boiling Point Original Hydrocarbon	Yield Acetate Per cent of Theory	Olefines Per cent of Theory	
28-30°	50	35	Calculated for pentane
30-35°	49	34	Calculated for pentane
35-40°	49	34	Calculated for pentane
45-50°	46	42	Calculated for 50 per cent pentane
50-55°	46	42	Calculated for 50 per cent hexane
55-60°	43	43	Calculated for 50 per cent hexane
60-65°	42	43	Calculated for 50 per cent hexane
65-70°	38	47	Calculated for 50 per cent hexane

<sup>1</sup> Heated 11 hours at 190-195° in autoclave, not stirred.

Owing to the large amount of solid material in the reaction mixture, anhydrous sodium acetate at the beginning and sodium chloride toward the end of the operation, we carried out several comparative experiments to determine the effect of continual agitation of the contents of the autoclave. When the reaction mixture is not agitated, a hard, nearly solid salt cake forms, which prevents further reaction.

TABLE III

METHOD	Reaction Mixture	Time Hours	Pressure Pounds	Acetate Made Grams	Chlorine in Acetate Per cent	Yield Acetate Cor- rected for Con- tent of Chlorides Per cent of Theory
Autoclave rotated	580 g. Chlorides 750 g. Sodium acetate 375 g. Acetic acid 375 g. Amyl acetate	5	230	394	1.2	51.6
Autoclave stationary	580 g. Chlorides 750 g. Sodium acetate 375 g. Acetic acid 375 g. Amyl acetate	5	250	273	7.1	22.1
Autoclave rotated	730 g. Chlorides 800 g. Sodium acetate 472 g. Acetic acid	12	240	458	0.9	50.1
Autoclave stationary	650 g. Chlorides 650 g. Sodium acetate 750 g. Acetic acid	12	240	300	16.5	26.0

The presence of water in the glacial acetic acid retards the reaction very markedly, acetates made in

this way containing relatively large amounts of unchanged chlorides, as illustrated by the following experiments.

TABLE IV—INFLUENCE OF THE WATER IN REACTION MIXTURE

SOLVENT	Time Hours	Temp.	Yield Acetate Per cent of Theory	Chlorine in Product Per cent
Acetic acid, 99 per cent.....	15	220°	42.3	2.0
Acetic acid, 90 per cent.....	15	220°	38.0	5.3

The presence of a solvent is necessary if a product free from chlorides is desired. The result obtained without a solvent is shown in Experiments 11 and 19, Table V, in which it will be noted that the per cent of unchanged chlorides in the product is very high.

A few substances, BaCl<sub>2</sub>, FeCl<sub>3</sub>, and CuCl, were tried for their possible catalytic effect, but no benefit could be noticed in the results.

It is possible that the amylenes resulting in the treatment of the chlorpentane mixture, as herein noted, are derived largely from secondary or even tertiary chlorides or acetates. We tested the stability of acetates of primary alcohols and found that during 8 hours at 205° and about 300 lbs. pressure in a copper lined autoclave, only 6.5 per cent of amylenes were formed. After cooling, the autoclave showed a pressure of 60 lbs., due to gaseous products of the decomposition, probably CO<sub>2</sub> and ethylene.

We have not had the opportunity to examine the constitution of the amyl acetates made from the petroleum fractions, as the primary object in view was the commercial utility and value of the product. We find that the solvent power of synthetic amyl acetate for cellulose esters and gums is such that by these tests alone the natural and synthetic acetates cannot be differentiated from each other. If, however, the acetate contains two or more per cent of chlorine in the form of unchanged chlorides, its solvent power for gums and resins is markedly diminished.

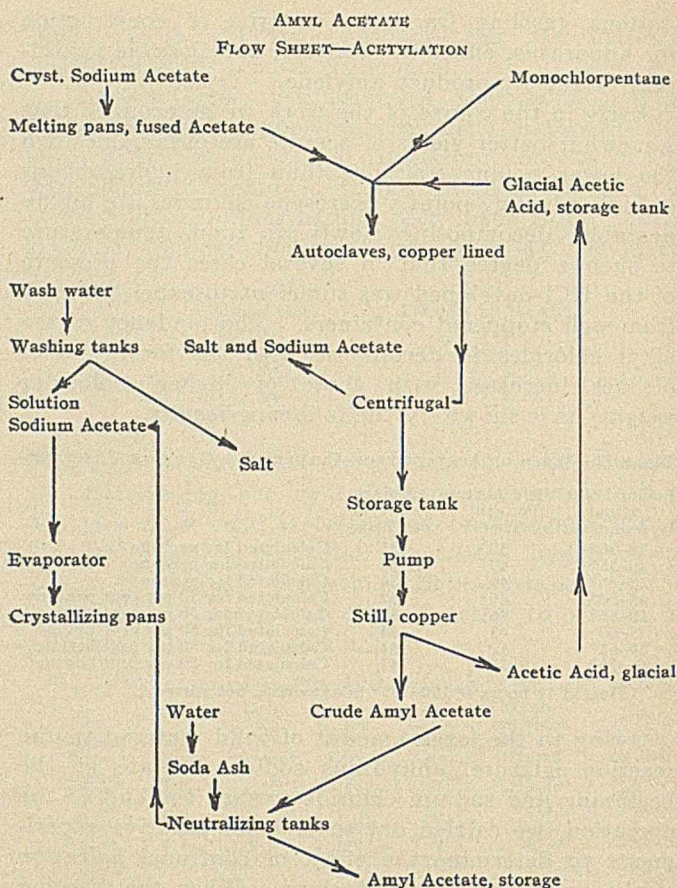
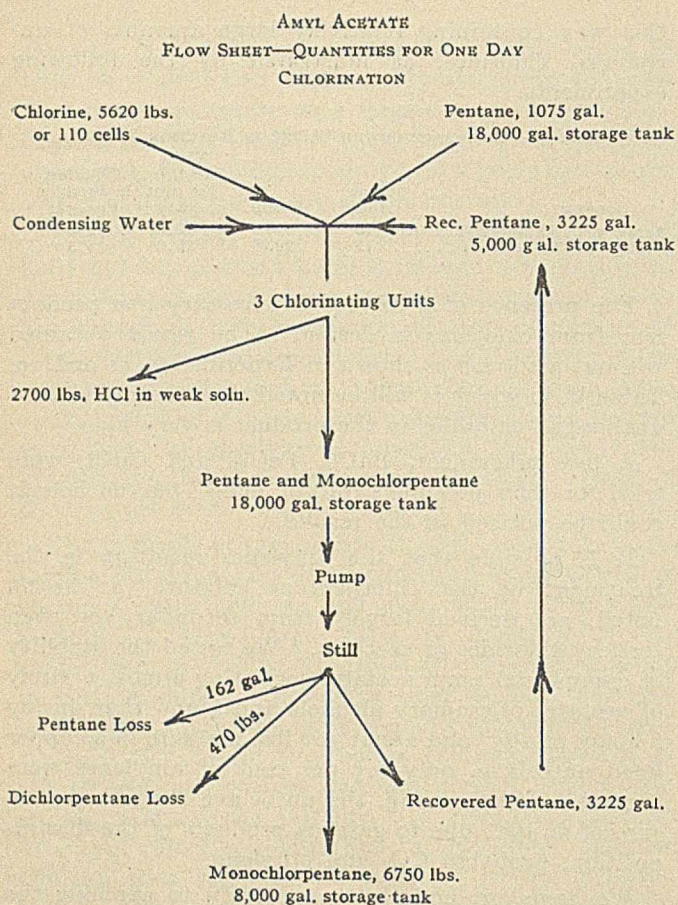
A number of typical results are included in Table V.

## UTILIZATION OF AMYLENES

We believe it is very doubtful if the amylenes, obtained as a by-product in the conversion of the chlorpentanes to acetates, can profitably be converted into alcohols or acetates. So-called hydration of amylenes and other olefines to the corresponding alcohols has been investigated by us and the results

TABLE V

Expt. No.	Chlorides Grams	Sodium Acetate Grams	Solvent Grams	Time Hours	Temp. ° C.	Pressure Pounds	Yield Crude Acetate Per cent of Theoretical	Chlorine in Product Per cent	Yield Acetate Corrected Per cent of Theoretical	REMARKS
94	50	50	100 g. Acetone	8	180-190	...	60	...	...	
89	400	400	500 g. Absolute alcohol	9	190	...	63.3	3.14	53.2	Product contained free amyl alcohol
31	730	900	470 g. Acetic acid	2	220	...	51.5	0.9	49.7	
18	730	945	470 g. Acetic acid	5	195-200	180	55.0	7.0	43.4	
17	580	750	375 g. Acetic acid 375 g. Amyl acetate	5	195-200	230	55.4	1.2	53.4	
19	1100	1422	None	12	195-200	170	47.0	11.0	31.4	
11	950	1215	None	6	205-210	300	45.3	6.4	36.6	
5	580	750	750 g. Acetic acid	4	195-200	400 430	43.0	1.0	41.2	30 g. BaCl <sub>2</sub> in reaction mixture
7	580	750	750 g. Acetic acid	1.5	195-200	200 260	45.0	3.5	31.9	30 g. FeCl <sub>3</sub> added
8	580	750	375 g. Acetic acid 375 g. Amyl acetate	5	195-200	...	50.0	0.9	48.8	



embodied in a separate paper.<sup>1</sup> With sulfuric acid the principal results are polymerization to heavier, more viscous oils (not tars), the formation of alcohols and of acid and neutral esters of sulfuric acid. We have not been able to obtain yields of alcohol as high as noted by Wischnegradsky<sup>2</sup> in the case of amylene made from natural fusel oil. The yields obtained by us are given in the following table, and it will be noted that with 50 per cent acid very little change was effected and with 95 per cent acid at 5.0° mostly polymers were formed.

TABLE VI—ADDITION OF WATER TO AMYLENE-HEXENE MIXTURE BY SULFURIC ACID OF VARYING CONCENTRATIONS

Expt. No.	Conc. Acid Per cent	Reaction Temperature ° C.	Hours Agitated	Alcohol Fraction of Pptd. Oil	Unchanged Hydrocarbons, Per cent	Loss as Sulfuric Esters and Vaporization	Polymers Per cent	
1	50	—	5	2	1.0	90.0	1.0	8.0
2	75	0	0	0	18.0	45.0	16.0	21.0
3	75	—10	0	0	16.0	54.0	17.0	13.0
4	75	0	0	0	10.0	67.0	9.0	14.0
5	75 (2 vols.)	0	2.5	10.0	57.0	15.0	18.0	
6	75	—15	2	10.0	70.0	12.0	8.0	
7	85	—15	2.5	22.0	32.0	14.0	32.0	
8	85 + 1 vol. acetic acid	—10	2.5	13.0	56.0	18.0	15.0	
9	95	—5	2	4.0	5.0	11.0	75.0	

In view of the readiness with which olefines react with many mineral acids, such as  $H_2SO_4$ ,  $HCl$ ,  $HBr$ ,  $HOCl$ , and in some cases even  $HCN$  and  $H_2S$ , we thought it possible that under certain conditions acetic acid might react with the amylenes direct. Accordingly the conditions of the usual acetylation

were tried on amylene itself. A quantity of crude amylene, 125 g., boiling point 30 to 60°, was heated in an autoclave at 200° for 5 hrs. with an equal weight of glacial acetic acid. From the reaction product we isolated only 4.5 g. oil boiling at 100 to 135° and 4.0 g. residue above 135°, these fractions consisting of polymers of the original amylenes. In another experiment 90 g. amylene were heated 20 hrs. at 100° with 90 g. acetic acid and 30 g. zinc chloride and the result was 14.5 g. polymers boiling over 100°. A reaction mixture duplicating the last was let stand 30 hrs. at 20 to 24°, obtaining 8 g. polymers.

It is not the purpose of the present paper to go into the details of producing a given output of synthetic amyl acetate. However, a brief discussion of the character of the apparatus required and a conservative estimate of costs should make clear that the commercial manufacture of synthetic amyl acetate is entirely feasible. In view of the amount of amyl acetate annually consumed in the United States alone and the amount of the synthetic acetate which might conservatively be expected to find a ready place in the market we have, for the sake of definiteness, based estimates on a daily production of 600 gal. of synthetic amyl acetate. The figures used are based upon a yield of monochlorides of 90 per cent, a low average for the method described, and a yield on acetylation of 55 per cent of the theory, a result actually attainable and probably exceeded. The minor by-products, amylene, common salt, and hydrochloric acid, have not been given any value in the estimate. It has been assumed that the crude pentane would be

<sup>1</sup> *J. Am. Chem. Soc.*, 42 (1918), 822.

<sup>2</sup> *Ann.*, 190 (1877), 328.



furnished in tanks or steel drums, being most advantageously manufactured at the source of production of casing head gasoline. The price of such an article in fairly large quantities bears no relation whatever to the prescription doses of pentane sold for photometric work. It has also been assumed that the most advantageous location for such a plant would be in the neighborhood of a chlorine-caustic soda plant, where the chlorine would be available for use without liquefaction. The diagrams indicate roughly the flow of material.

## APPARATUS REQUIRED

## 1—STORAGE FOR

- (a) Crude pentane, 18,000 gal. steel tank.
- (b) Crude chlorinated product, pentane and chlorpentane, 18,000 gal. lead-lined steel tank.
- (c) Distilled chlorpentane, 8,000 gal. lead-lined tank.
- (d) Sodium acetate, crystalline, 100 ton covered wooden bins.
- (e) Glacial acetic acid, 10,000 gal., 5 wooden tanks, 2,000 gal. each.
- (f) Amyl acetate, finished, 15,000 gal., 3 steel tanks, 5,000 gal. each.

## 2—CHLORINATION

- 3 units complete, each consisting of one 500 gal. earthenware vessel<sup>1</sup> fitted with lead coil for cold water or brine, glass Hart condenser of the type used for nitric acid, and earthenware receiver for separating recovered pentane and aqueous HCl.

## 3—STILLS

- 2 Steam jacket Duriron stills, 1000 gal. charge capacity for rectifying chlorpentane.
- 1 Steam jacket copper still for rectifying amyl acetate, acetic acid, and amylene mixture, capacity 900 gal.

## 4—ACETYLATION

- 3 Copper-lined steel autoclaves, jacketed, heated by hot oil circulation, provided with agitators of copper or Duriron, capacity 900 gal. each.

## 5—ACCESSORIES

- 1 Centrifugal extractor, 30 in. copper mesh basket, for separating salt from reaction mixture.
- 1 Agitator, copper, for washing amyl acetate with soda ash solution, capacity 900 gal.
- 1 Wooden tank with chain type agitator for washing excess sodium acetate from salt, capacity 500 gal.
- 2 Crystallizing pans, iron, in brick setting, slow direct fire for recovering sodium acetate, 300 gal. each.
- 3 Melting pans, steel in brick setting, slow direct fire for fusing sodium acetate, capacity 200 lbs. sodium acetate each.
- 1 Motor for agitating autoclave, 8 h. p. steam, 200 boiler h. p. for distillation, heating building, pumping, etc.
- 4 Dump cars and track for conveying salt, sodium acetate, etc.
- 1 Motor, 8 h. p., for operating pumps.
- 3 Low-pressure rotary pumps for pumping pentane, amyl acetate, etc.

## BUILDINGS

- 1 Shed for storing acetate of soda.
- 1 Building for chlorinating pentane and distillation of chlorpentane, fireproof asbestos board.
- 1 Building for acetylating and distillation of amyl acetate.
- 1 Building for boilers and fusing sodium acetate.

<sup>1</sup> Suitable vessels of this size are regularly manufactured.

## ESTIMATED COST OF SYNTHETIC AMYL ACETATE ON THE BASIS OF 600 GALLONS DAILY PRODUCTION

DAILY COSTS	
Gasoline, 990 gal. at 40 cts.	\$ 396.00
Chlorine, 5200 lbs. at 2.5 cts.	130.00
Acetate of soda, 4880 lbs. cryst. acetate at 15 cts.	732.00
Soda ash, 1450 lbs. at 3 cts.	43.50
Interest on \$35,000 at 6 per cent, and depreciation at 20 per cent per annum.	30.30
Labor, one chemist at \$8.00, 10 laborers at \$4.00.	48.00
Fuel and Power.	40.00
<b>TOTAL DAILY COSTS</b>	<b>\$1419.80</b>
VALUE OF PRODUCTS	
Amyl acetate, 600 gal. at \$4.50 per gal.	\$2700.00
Total daily costs.	1419.80
<b>DAILY PROFIT</b>	<b>\$1280.20</b>

MELLON INSTITUTE OF INDUSTRIAL RESEARCH  
PITTSBURGH

THE EFFECT OF ANNEALING ON THE ELECTRICAL RESISTANCE OF HARDENED CARBON STEELS<sup>1</sup>

By I. P. PARKHURST

Received November 24, 1917

The electrical resistance of steels as affected by impurities or by different heat treatments applied over wide ranges of temperature, has attracted the attention of several investigators. A résumé of previous work is here given.

## RÉSUMÉ OF PREVIOUS WORK

W. H. Johnson<sup>2</sup> made a study of the electrical resistance of steels in order to determine the effect of impurities. The resistance of seven samples of different analyses was measured. The impurities were found to increase the resistance, but the data obtained were not sufficient to allow of specific conclusions.

A. Campbell<sup>3</sup> determined the effect of stress on the resistance of iron and iron-nickel wires. Practically no change was observed up to the breaking point.

H. Le Chatelier<sup>4</sup> investigated the effect of high temperatures on the resistance of iron, steel, hard steel, iron-nickel, platinum, etc. His measurements were made in an atmosphere of hydrogen. In a 0.6 per cent carbon steel he observed breaks in the temperature-resistance curves at 710 and 820° C.

H. Le Chatelier<sup>5</sup> studied the effect of hardening and tempering on the resistance of steels. His results showed that the resistance was increased by hardening and decreased by tempering.

L. Compredon<sup>6</sup> made a study similar to that of W. H. Johnson, referred to above, and obtained similar results.

J. De War and J. A. Fleming<sup>7</sup> determined the resistance of iron and nickel at temperatures down to the boiling point of oxygen. They found that the resistance decreased rapidly with the decrease in temperature.

<sup>1</sup> Read in abstract at the Metallurgical Symposium, Boston Meeting, American Chemical Society, September 1917.

<sup>2</sup> "Chemical Composition and Electrical Resistance of Steel Wire," *J. Iron and Steel Inst.*, **19** (1881), No. 2, 605.

<sup>3</sup> "Electrical Properties," *Engineering*, **63** (1887), 468.

<sup>4</sup> "Electrical Resistance of Iron and Its Alloys at High Temperatures," *Compt. rend.*, **110** (1890), 283.

<sup>5</sup> "Effect of Tempering on the Electric Resistance of Steel," *Compt. rend.*, **112** (1891), 40.

<sup>6</sup> "Electro-conductivity of Steel," *Le Genie Civil*, **19** (1891), 309.

<sup>7</sup> "Electrical Resistance of Iron," *Phil. Mag.*, **34** (1892), 326.

F. Osmond<sup>1</sup> investigated the effects of carbon, silicon, nickel and manganese on the resistance of steels. He found that the resistance was increased by all these elements, and especially by silicon, nickel and manganese. The temperature coefficient fell in a parabolic curve with increase in resistance.

H. Le Chatelier<sup>2</sup> studied in detail the effects of carbon, silicon, and manganese on the resistance of steels. Carbon was found to increase the resistance of 7 microhms for 1 per cent, silicon 14 microhms for 1 per cent, and manganese 3 microhms for 1 per cent.

C. Benedicks<sup>3</sup> investigated the effect of impurities on the resistance of iron and steel. He used commercial iron and steel varying from 0.08 to 1.7 per cent. C. and varying in amounts of silicon, manganese, phosphorus, and sulfur. Calculating the percentages of silicon and manganese to equivalent carbon, he proposed the following formula

$$\rho = 7.6 + 26.8 \Sigma C,$$

in which  $\rho$  is the resistance of the steel, 7.6 the resistance of pure iron, and  $\Sigma C$  the sum of the percentages, in terms of carbon, of the impurities in the iron.

M. Portevin<sup>4</sup> determined the resistance of ternary steels, including alloys with nickel, chromium, tungsten, aluminum, molybdenum, vanadium, titanium, boron and thallium. He discussed also the influence of titanium on the arrest points.

A. P. Schleicher and W. Guertler<sup>5</sup> studied the resistance of alloys containing 35.25, 30.6 and 25.2 per cent nickel. It was observed that there was a pronounced discontinuity in the resistance at about 420° C. for the steel containing 35.25 per cent nickel, and at 700° C. for the one containing 30.6 per cent nickel. The alloy containing 25.2 per cent nickel gave, in the original condition, abnormally low values, but the resistance increased with each heating until it became stabilized with a discontinuity at 900° C.

A. Portevin<sup>6</sup> heated steel bars in a salt bath at 750 and 900° C. for varying lengths of time and after quenching, measured the increase in resistance.

K. Honda<sup>7</sup> investigated the influence of high temperatures on the electrical resistance and magnetic properties of iron, steel, and nickel. He concluded that the magnetic transformation was not an allotropic change, but a gradual change of the property of a phase, due to a change in temperature.

G. K. Burgess and I. N. Kellburg<sup>8</sup> measured the electrical resistance of pure iron from 0 to 900° C. The resistance increased with a gradually rising rate

<sup>1</sup> "The Electrical Resistance of Iron and Steel," *La Lumière Électrique*, 46 (1893), 93.

<sup>2</sup> "The Electric Resistance of Steel," *Bulletin de la Société d'Encouragement pour L'Industrie Nationale*, 3, p. 743.

<sup>3</sup> "Electric Resistance of Iron and Steel," *Z. phys. Chem.*, 40, [5], (1903).

<sup>4</sup> "Electric Resistance of Steel," *Revue de Métallurgie, Memoirs*, 6 (1909), 1304.

<sup>5</sup> "Electric Resistance of Iron-Nickel Alloys," *Z. Elektrochem.*, 20 (1914), 273.

<sup>6</sup> "Influence of High Temperatures on Magnetic Properties," *Compt. rend.*, 158 (1914), 51.

<sup>7</sup> "Influence of High Temperatures on Magnetic Properties," *Revue de Métallurgie, Memoirs*, 11 (1914), 383.

<sup>8</sup> "Electric Resistance of Iron at Varying Temperatures," *J. Wash. Acad. Sci.*, 4 (1914), 436.

to A<sub>2</sub>. There was an inflection at A<sub>2</sub> and an abrupt fall in resistance at 894° C. The reverse change took place at approximately the same temperature. The interval was 25° C. The change at A<sub>3</sub> was progressive and thermoelectrically nonreversible. The change at A<sub>2</sub> was reversible.

P. Mahler<sup>1</sup> studied the influence of carbon and manganese on the resistance of steel. The specific resistance increased 7 microhms for 1 per cent of carbon, and 5 microhms for 1 per cent of manganese. Mahler believes that occluded gases increase the resistance, and points out that hydrogen is known to do so.

Edward D. Campbell<sup>2,3</sup> investigated the effect of annealing on the resistance of quenched steels. He measured the resistance of the quenched steels and subjected them in turn to temperatures of 100, 200, 300° C., etc. The annealing at 100° C. was prolonged for 48 hrs. The time of annealing at the other temperatures was from 1 to 2 hrs. He plotted curves showing the resistance against the temperature of annealing.

#### OBJECT OF THE PRESENT INVESTIGATION

The object of the investigation here presented was to determine the rate of softening of quenched steels, under conditions in which the temperature of annealing is kept constant. Changes in the electrical resistance were used as a measure of the changes in hardness.

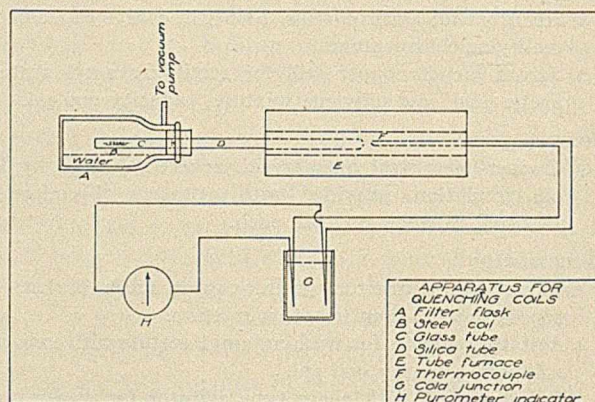


FIG. 1—APPARATUS FOR QUENCHING COILS

The work was carried out by first quenching the steel and measuring the resistance. This was followed by annealing the steel for a definite time at a definite temperature, and again measuring the resistance. This procedure was repeated at the same temperature but with an increasing interval of time, followed by another measurement of the resistance. The total period of annealing for the different specimens varied from 90 to 190 hrs. The results were then plotted as time against resistance.

#### EXPERIMENTAL

The steels were treated in the form of a wire coil. The specimens were prepared by forging steel bars

<sup>1</sup> "Electric Resistance of Steel," *Revue de Métallurgie*, 12 (1915), 345.

<sup>2</sup> "The Influence of Heat Treatment on the Thermoelectric Properties and Specific Resistance of Carbon Steels," *J. Iron and Steel Inst.*, [2], 94 (1916), 286.

<sup>3</sup> "Do Equiatomic Solutions of Iron Possess Equal Resistances?" *Trans. Faraday Soc.*, 12 (1917), 1.

down to  $\frac{1}{8}$  in. round. The pieces of this size, about 4 in. long, were drawn down to wires varying from 0.014 to 0.019 in. in diameter (0.355 to 0.482 mm.). From 20 to 50 cm. of this wire were used as a specimen. The specimens were heated and quenched in a vacuum, being contained in a silica tube which was heated in an electric combustion furnace. Temperatures were measured with a platinum-platinum-rhodium thermocouple.

The arrangement for heating and quenching is shown in Fig. I. The specimen was placed in the glass extension of the silica tube. The tube was then placed in the tube furnace and kept there for 5 min. At the end of this time it was evacuated, withdrawn from the furnace, inverted, and replaced in the furnace. After about 1 min. it was again withdrawn and the specimen quenched. The object of this procedure was to bring the coil to the desired temperature as quickly as possible, in order to prevent oxidation. It was necessary to guard carefully against oxidation, since the coils were long and slender. It will be shown later that the effect of oxidation was small.

After measuring the resistance, the method of which will be explained later, the coil was annealed at a definite temperature. The temperatures chosen were 125, 150, 175 and 250° C. For the first three temperatures the specimens were annealed in a constant temperature paraffin bath, which was heated on an electric hot plate to a temperature which was a few degrees below that desired. The temperature was then further raised and adjusted by means of a resistance coil in the bath. The current in the coil was regulated through a thermostat regulator and a relay. The temperature was constant to about one degree.

For the first 5 min. the specimen was immersed directly in the paraffin. For longer annealing the specimen was placed in a tube immersed in the paraffin. The reason for putting the coil directly in the paraffin was to allow a closer determination of the time of annealing. The specimen would take up the temperature of the liquid much more quickly than that of air, hence the time of annealing could be more accurately measured in the paraffin than in air. For longer annealing, a small difference in time would make much less difference in the results, hence the tube was used. This was desirable whenever possible in order to prevent any chance of carbonization.

For annealing at 250° C. a constant temperature oven was used, in which the temperature varied about 5° C.

For measuring the resistance of the specimens, they were immersed in a tube of alcohol, which was placed in a thermostat regulated to 30° C. The maximum variation of the temperature was 0.05° C.

The resistance was measured with a Kohlrausch bridge used as shown in the diagram in Fig. II. The unknown resistance  $X$  and a known resistance,  $R$ , are connected in series, and in parallel with them is the slide wire of the bridge. The bridge is balanced with each end of each resistance, thus making four readings. If the readings for the known resistance

are  $a$  and  $b$ , and those for the unknown resistance  $c$  and  $d$ , then

$$\frac{X}{R} = \frac{d - c}{b - a}$$

This method was used in order to eliminate the effect of contact resistance. The double-throw switch was used in order to eliminate the effects of all thermo-electromotive forces and generated electromotive forces.

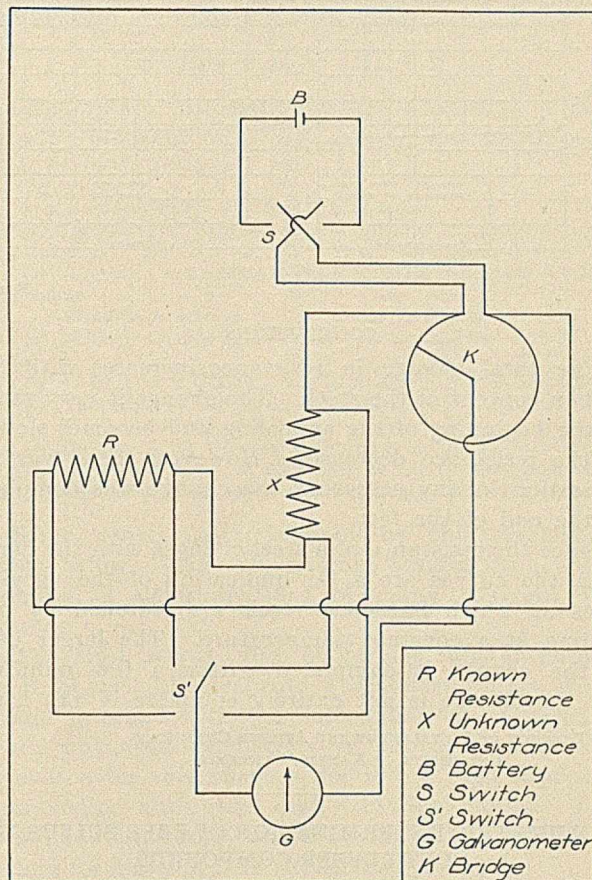


FIG. II—APPARATUS FOR MEASURING RESISTANCE

The resistance measurements themselves were accurate to one part in one thousand. However, the impossibility of placing the contacts at exactly the same point each time increased the error. This added error depended on the length of the coil. For both contacts it was not more than 0.5 mm. This would give a possible error of 0.25 per cent for coil No. 5, which was the shortest, and 0.1 per cent for the longer coils. This makes a total probable error of from 0.2 to 0.35 per cent.

The analyses of the specimens and the dimensions of the coils are given in the following tables:

No.	ANALYSES OF SPECIMENS					DIMENSIONS OF COILS	
	C Per cent	Si Per cent	P Per cent	Mn Per cent	S Per cent	Diameter Mm.	Length Mm.
1.....	0.05	0.006	0.021	0.055	0.025	0.355	514
2.....	0.09	0.059	0.013	0.044	0.019	0.368	500
3.....	0.18	0.025	0.090	0.068	0.092	0.482	486
4.....	0.25	0.058	0.109	0.075	0.036	0.431	503
5.....	0.45	0.122	0.112	0.092	0.106	0.431	196

The results of the resistance measurements are plotted in the following curves, which show the effect of the duration of annealing on the resistance of the steels.

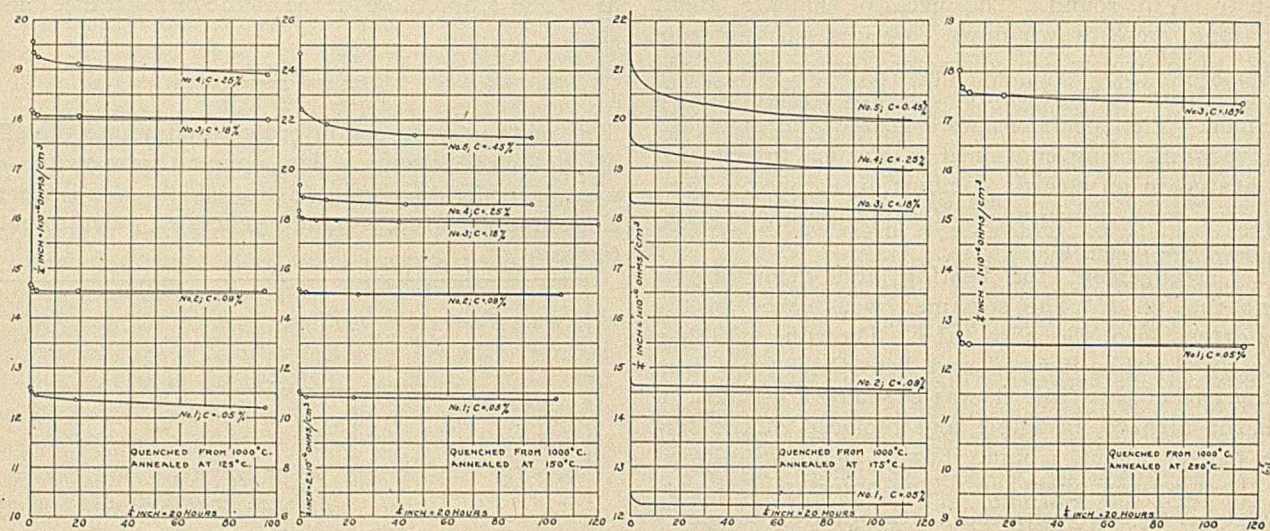


FIG. III

## CONCLUSIONS

The total change in resistance increases with the carbon content of the steel. The change is very rapid at the beginning of the annealing and becomes slower as the resistance decreases. However, there was no indication in any case that the change was complete at the end of the test.

Since the resistance of a steel changes with the hardness, the curves are a fair indication of the varying rates at which hardened steels are softened by annealing at a constant temperature. The larger part of the change is completed within a few minutes, but the change is not entirely complete in 113 hrs.

DIVISION OF METALLURGY AND APPLIED CHEMISTRY  
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VOLUMETRIC DETERMINATION OF FREE SULFUR IN  
SOFT RUBBER COMPOUNDS

By H. S. UPRON

Received October 5, 1917

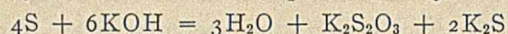
The published and accepted methods of determining free sulfur in a rubber compound are more or less tedious, and, where manufacturing operations are dependent upon them, are the cause many times of serious delays. These delays, the cost of materials required, and the time consumed by the determining process, make these methods expensive. It was with this in mind that the following method was evolved and results obtained both in conservation of time and materials and also in accuracy lead us to believe it may be of interest to others who are using the present-day accepted methods.

## METHOD

The proposed method depends on the fact that sulfur when heated with an alcoholic potash solution is converted into a mixture of potassium sulfide and potassium thiosulfate, both of which may be titrated with standard iodine by usual methods as given in this article. The free sulfur is determined in the acetone extract from the rubber compound. The resinous extract of certain commercial rubbers and the extract of other compounding materials are acted upon by

the alcoholic potash solution and by various standard solutions used in analysis, thereby rendering necessary the application of corrections. The amount of correction or blank used in titrating is determined by the nature and amount of the various ingredients in the extract from the rubber compound. This limits the use of the method with confidence to a sample of rubber, the composition of which is known to the analyst. The proposed method can be used successfully where a large number of tests are being run on a compound made up to a certain formula, for example, insulated wire compounds, boot and shoe compounds, etc.

The equation generally given for the oxidation of sulfur by a water solution of potassium hydroxide is:



This varies with change of conditions. When using an alcoholic potash solution instead of potash in water more thiosulfate is formed than is indicated in the equation and in some cases the conversion to this product is complete. Using this principle, a method has been worked out which is as follows:

## PROCEDURE

Extract a 2-g. sample of rubber compound with acetone into a 300 cc. Erlenmeyer flask until the free sulfur has been removed. A similar compound, which is known to be free of free sulfur, is tested at the same time. This is used to determine the blanks in the subsequent titrations.

Evaporate the acetone gently until little remains, completing the drying in an oven at 100° C. This operation need not take longer than a half hour. The sulfur in the dried extract is oxidized to thiosulfate and sulfide with 25 to 50 cc. of 5 per cent alcoholic potash solution by boiling gently for one hour, using a small glass funnel placed in the neck of the flask for a condenser. Remove from heat. Wash and remove funnel from flask. Add 25 to 50 cc. of ammonium zinc chloride solution<sup>1</sup> and just bring to boiling.

<sup>1</sup> This solution is made as follows: 10 g. of zinc oxide are dissolved in dilute hydrochloric acid. The solution is made alkaline with ammonia (sp. gr. 0.90), adding 50 cc. in excess. The solution is now made up to a liter.

This precipitates the sulfide as its zinc salt, leaving the thiosulfate in solution.

Cool to room temperature in water. Acidify with glacial acetic acid, adding 3 cc. in excess. Dilute to 200 cc. Titrate with *N*/25 iodine and starch. This gives the sulfur present as thiosulfate. The precipitate of zinc sulfide is not acted upon by the acetic acid.

Add ammonium hydroxide to destroy the acidity and a trace of iodine. Add hydrochloric acid to acidity, having 5 cc. in excess. This dissolves the zinc sulfide and liberates hydrogen sulfide. Titrate the liberated sulfide with iodine and calculate to sulfur. The titration in each case is carried to a permanent blue, lasting half a minute. The end-point is generally very distinct.

The determination of thiosulfate is called (*a*) titration, and that of the sulfide, (*b*) titration. The sum of (*a*) and (*b*) titrations gives the total free sulfur. The sulfur-free sample is tested at the same time as the sample containing free sulfur and blanks for (*a*) and (*b*) titrations found. In a straight rubber compound blank (*a*) is about 1 cc. and blank (*b*) is 0.5 cc. of iodine. Often all the sulfur in a pure rubber compound is converted completely to thiosulfate and (*b*) titration may be omitted. When this is the case make the solution acid with hydrochloric, adding 5 cc. in excess, and carry out the determination as for thiosulfate, instead of making acid with acetic.

It is important that the solution should be kept cool during the determination. The different reagents should be measured as their quantity tends to vary the results. Solutions should be kept to approximately the same volume, as this affects the iodine blank. Two hundred cubic centimeters of solution are a convenient volume to work with. In the process of treating sulfur with alcoholic potash a white crystalline precipitate of potassium thiosulfate is sometimes found. This is almost insoluble in 95 per cent alcohol but dissolves readily in the more dilute solution. Potassium sulfhydrate (KSH) may be formed by the action of alcoholic potash, but it is oxidized to potassium sulfide when the potash is present in excess. The strength of the standard iodine solution is determined at least twice a week, as it becomes weaker on standing, due to the volatilization of the iodine.

## FACTORS AND CALCULATIONS

It is very convenient to use factors in calculating the results. The factor for (*a*) titration of sulfur is 0.50536 times the value of 1 cc. of standard iodine solution. This is found from the following equation:



Factor for (*b*) titration for sulfur is 0.12630 times the value of 1 cc. of standard iodine solution and is found from the following equation:



An example of calculation where 0.0328 g. of sulfur or 1.64 per cent on a 2-g. sample was known to be present is as follows:

1 cc. Standard Iodine = 0.005139 g. of Iodine  
 (*a*) Titration: 1 cc. Standard Iodine = 0.005139 × 0.50536 = 0.002597 g. of Sulfur  
 (*b*) Titration: 1 cc. Standard Iodine = 0.005139 × 0.12630 = 0.000649 g. of Sulfur  
 Blank (*a*) = 1.00 cc.                      Blank (*b*) = 0.50 cc.  
 (*a*) Titration required 12.00 cc. — 1.00 cc. (Blank) = 11.00 cc. of Iodine  
 11.00 cc. × 0.002597 ÷ 2 (2 g. sample) = 0.0143 g. of Sulfur  
 (*b*) Titration required 5.80 cc. — 0.50 cc. (Blank) = 5.30 cc. Iodine  
 5.30 cc. × 0.000649 ÷ 2 (2 g. sample) = 0.0017 g. of Sulfur  
 (*a*) Titration + (*b*) Titration = 0.0160 g. or 1.60 per cent of Sulfur  
 By this method 0.1 g. of sulfur may be titrated with good results. There has not been time to experiment with larger amounts of sulfur.

Some actual determinations by this method are given below. They are average results from a large number of determinations of many different compounds.

The most active types of various substances liable to react with the reagents were tested to see if they would interfere with the determination. Linseed oil, while not used extensively in rubber compounding, is chosen for this purpose, for it has high saponification, iodine absorption and oxidation values. These determinations were carried out according to the procedure for free sulfur in rubber compounds.

A much larger quantity of material in each case was tested than would be present in an analysis. Only starch seemed to have any effect which would interfere with the determination. It is well known that correct results are impossible in an iodine titration with much starch present. Starch would not be liable to occur in a rubber mixture and at least would not appear in the acetone extract.

SAMPLE No.	Weight of Sample Grams	Per cent of Acetone Extract Tested	Sulfur Equivalent G. per Cc.		(a) Titration Standard I Used Cc.	(a) Blank Standard I Used Cc.	(b) Titration Standard I Used Cc.	(b) Blank Standard I Used Cc.	Free S by New Method Per cent	Free S by Fuming Nitric Method Per cent
			(a)	(b)						
1.....	2.0000	4.00	0.002598	0.000647	2.20	1.00	0.40	0.50	0.16	0.21
2.....	2.0000	4.00	0.002598	0.000647	1.20	1.00	0.40	0.50	0.03	0.15
3.....	2.0000	4.00	0.002598	0.000647	4.00	1.00	0.40	0.50	0.39	0.37
4.....	2.0000	4.00	0.002598	0.000647	3.80	1.00	0.85	0.50	0.38	0.30
5.....	2.0000	4.00	0.002598	0.000647	3.70	1.00	0.35	0.50	0.35	0.39
6.....	2.0000	4.00	0.002598	0.000647	2.65	1.00	0.25	0.50	0.22	0.22
7.....	2.0000	4.00	0.002598	0.000647	2.50	1.00	0.25	0.50	0.20	0.21
8.....	2.0000	4.00	0.002598	0.000647	2.05	1.00	0.25	0.50	0.14	0.16
9.....	2.0000	4.00	0.002598	0.000637	1.55	1.00	0.25	0.50	0.07	0.12
10.....	2.0000	25.00	0.002598	0.000647	12.95	1.00	6.70	0.50	1.75	1.65
11.....	2.0000	25.00	0.002598	0.000647	12.45	1.00	4.75	0.50	1.61	1.58
12.....	2.0000	28.50	0.002598	0.000647	12.00	1.00	5.80	0.50	1.60	1.64
13.....	2.0000	22.85	0.002598	0.000647	14.40	1.00	2.50	0.50	1.81	2.08
14.....	2.0000	22.85	0.002598	0.000647	15.70	1.00	2.70	0.50	1.98	2.13
15.....	2.0000	24.50	0.002598	0.000647	10.10	1.00	2.40	0.50	1.29	1.40
16.....	2.0000	25.00	0.002598	0.000647	9.30	1.00	4.70	0.50	1.21	1.33
17 <sup>1</sup> .....	1.0000	8.00	0.002586	0.000646	187.0	1.00	0.90	0.50	4.62	4.53
18 <sup>1</sup> .....	1.0000	8.00	0.002586	0.000646	17.70	1.00	2.80	0.50	4.48	4.48
19 <sup>1</sup> .....	1.0000	8.00	0.002586	0.000646	18.70	1.00	0.60	0.50	4.60	4.62
20 <sup>1</sup> .....	1.0000	8.00	0.002586	0.000646	17.00	1.00	0.30	0.50	4.14	4.44

<sup>1</sup> Smoked sheet cured with 5 per cent sulfur

MATERIAL TESTED	Weight of Sample Gram	Per cent of Extract Tested	Standard I Value G. per Cc.	(a) Titration Standard I Used Cc.	(a) Blank Standard I Used Cc.	(b) Titration Standard I Used Cc.	(b) Blank Standard I Used Cc.	Free S by New Method Gram	Free S Present Gram
Blank of Reagents.....	.....	.....	0.005121	.....	0.60	.....	0.30	None	None
Blank of Reagents.....	.....	.....	0.005121	.....	0.50	.....	0.35	None	None
Sulfur.....	0.0200	.....	0.004798	8.10	0.60	5.40	0.40	0.0200	0.0200
Sulfur.....	0.0200	.....	0.004798	8.00	0.60	5.15	0.40	0.0200	0.0200
Sulfur.....	0.0200	.....	0.005121	8.35	0.60	3.80	0.40	0.0211	0.0200
Sulfur.....	0.1000	.....	0.005141	32.60	0.60	24.90	0.40	0.1007	0.1000
Potato Starch.....	0.5000	.....	0.004727	14.55	0.60	0.75	0.40	0.0326	0.0200
Potato Starch.....	0.5000	.....	0.004727	13.65	0.60	0.75	0.40	0.0304	0.0200
Potato Starch.....	0.5000	.....	0.004727	13.20	0.60	1.70	0.40	0.0300	0.0200
Potato Starch.....	0.5000	.....	0.004727	12.55	0.60	1.85	0.40	0.0286	0.0200
Potato Starch.....	0.5000	.....	0.004727	13.50	0.60	0.40	0.40	0.0300	0.0200
Potato Starch.....	0.5000	.....	0.004727	14.25	0.60	0.40	0.40	0.0316	0.0200
Potato Starch.....	0.5000	.....	0.004727	14.20	0.60	0.40	0.40	0.0316	0.0200
Potato Starch.....	0.5000	.....	0.004727	13.40	0.60	0.40	0.40	0.0296	0.0200
Rosin.....	0.0500	.....	0.005121	8.00	0.60	3.60	0.40	0.0201	0.0200
Rosin.....	0.0500	.....	0.005121	8.00	0.60	3.75	0.40	0.0203	0.0200
Linseed Oil.....	0.0500	.....	0.005121	8.70	0.60	3.60	0.40	0.0219	0.0200
Linseed Oil.....	0.0500	.....	0.005121	8.00	0.60	4.40	0.40	0.0207	0.0200
Linseed Oil.....	0.0500	.....	0.005121	8.50	0.60	4.20	0.40	0.0219	0.0200
Acetone Extract of Smoked Sheet Rubber to Determine Blank with Reagents.....	1.0000	4.18	0.004768	.....	0.80	.....	0.35	None	None
	1.0000	4.00	0.004768	.....	0.80	.....	0.30	None	None
	1.0000	3.90	0.004768	.....	1.00	.....	0.30	None	None
	1.0000	4.16	0.004768	.....	1.00	.....	0.30	None	None
	1.0000	3.50	0.005121	.....	0.85	.....	0.55	None	None
	1.0000	3.80	0.005121	.....	0.80	.....	0.30	None	None
	1.0000	4.00	0.005121	.....	0.70	.....	0.50	None	None
	1.0000	4.00	0.005121	.....	0.70	.....	0.40	None	None
Cane Sugar.....	1.0000	.....	0.004727	8.85	0.60	5.50	0.40	0.0221	0.0200
Cane Sugar.....	1.0000	.....	0.004727	8.90	0.60	5.60	0.40	0.0222	0.0200
M. R.....	1.0000	.....	0.004727	10.00	0.60	2.80	0.40	0.0230	0.0200
M. R.....	1.0000	.....	0.004727	10.00	0.60	2.70	0.40	0.0229	0.0200
Ceresine.....	1.0000	.....	0.004727	10.70	0.60	3.10	0.40	0.0249	0.0200
Ceresine.....	1.0000	.....	0.004727	9.15	0.60	3.55	0.40	0.0210	0.0200

The above is a tabulation of results:

The advantages of this method are its accuracy, ease of manipulation, rapidity, and cheapness.

When the dried acetone extract from a rubber compound is obtained, eighteen samples may easily be determined in 2½ hrs. The operator need not spend more than an hour's time in actual manipulation. This method may be used to determine sulfur, and mixtures of soluble sulfides and thiosulfates.

ATLANTIC INSULATED WIRE AND CABLE COMPANY  
STAMFORD, CONNECTICUT

#### RAPID DETERMINATION OF CARBON IN STEEL BY THE BARIUM CARBONATE TITRATION METHOD<sup>1</sup>

By J. R. CAIN AND L. C. MAXWELL

Received May 3, 1918

##### INTRODUCTION

Because of the increased demands now made on the testing and steel works laboratories which analyze steel delivered on government contracts, and because of the reduced number of chemists available for such work, it becomes desirable to increase the efficiency of laboratories in all possible ways. Short-cuts or simplifications which will reduce the time required for determination by standard methods, or reliable new methods which require less time than those now in use are of value in contributing towards increased output of work. With this idea the following modification of the barium carbonate titration method originally described by Cain<sup>2</sup> has been developed. The work was carried on recently at the Bureau of Standards in connection with a military problem where speed was considered important.

The barium carbonate method is much used, especially by testing laboratories which usually have to analyze steels of unknown composition from many sources. It has been recognized that this method as heretofore used is not as rapid as the soda-lime method, but it is also felt by many that it is less subject to disturbing influences and is in most respects

simpler than the latter method. It is believed that the modifications herein recommended put the barium carbonate titration method more nearly on the same basis with the soda-lime method as to time requirement, with but little loss in accuracy, and with added simplicity in manipulation. The time required per determination has been reduced 50 per cent and it is now possible for a chemist during an 8-hr. day to make 50 determinations by the barium carbonate titration method.

The procedure recommended in the cited paper was briefly as follows:

The combustion of the steel was carried out by admitting oxygen at a moderate rate to the combustion tube. No details were given in that paper as to furnace temperatures at the time the boat was inserted in the furnace, nor as to whether the boat was allowed to preheat before admitting oxygen. Actually, the results given in the paper were obtained by placing the cold boat in a furnace kept at 1000 to 1050° and admitting the oxygen immediately; the passage of the oxygen at the moderate rate specified was continued for 20 to 25 min. The oxides thus obtained were frequently not well fused. If an incomplete combustion was suspected, the oxides were crushed and reburned. This method of burning corresponded to practice here and elsewhere at that time. The barium carbonate was filtered under an atmosphere free from carbon dioxide, using a special apparatus illustrated and described in the cited publication. The filter consisted of a carbon tube fitted with a perforated porcelain plate; on this was placed a bed of coarse quartz particles, and on this a layer of asbestos. When filtration was finished the porcelain plate and superimposed layers of quartz and asbestos were transferred to a wide-mouth flask, treated with excess of the standard hydrochloric acid, and the solution titrated against sodium hydroxide, using methyl orange as indicator.

Various precautions in manipulation and filtration were described. These precautions, the necessity for preparing a filter of this type each time, and the slow-

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

<sup>2</sup> Bureau of Standards, *Technologic Paper No. 33*.

ness of the combustion all contributed toward making the barium carbonate method as described longer than others in use. This was compensated by the high degree of accuracy attainable and the insurance against numerous sources of error, as detailed in the paper.

The present method saves time in the following ways:

1—Admission of oxygen at a very rapid rate to the burning sample with automatic provision against too rapid passage at the exit end of the combustion tube. (This would cause loss of carbon.) This rapid admission of oxygen, coupled with the use of a preheated boat and the practice of allowing the sample to pre-heat in the furnace a minute before admitting oxygen, gives complete combustion of a 2-g. sample in 1½ to 2 min. If combustion is allowed to take place gradually, instead of rapidly, as specified, the rapid method herein described cannot be used. The criteria of a successful combustion by this method are:

(a) Well-fused oxides in which no trace of the original grains of steel is apparent.

(b) Much accelerated oxygen inflow during the actual combustion of the sample.

(c) Vivid incandescence while the sample is burning, visible if a quartz combustion tube is used.

Should these signs of a good combustion be lacking, determinations should be rejected.

2—Use of glass plungers to take up dead space in the forward end of the combustion tube so as to decrease the amount of gas that has to be flushed out at each determination.

3—Rapid filtration as described in Section 6.

4—The delivery of all the reagents from reservoirs by air pressure.

The main prerequisite for the use of the modified method is a laboratory atmosphere not contaminated with excessive amounts of carbon dioxide, so that the barium carbonate may be filtered in a simple apparatus not requiring exclusion of the carbon dioxide contained in the laboratory air. This requirement is met by any modern laboratory with even fairly good ventilation and without an excessive number of gas burners operating at one time in a confined space. Practically this point may be tested by comparing blanks run with the filtering apparatus described in the original paper<sup>1</sup> and that herein recommended. If the difference in the blanks is such as would cause an error exceeding 0.005 per cent carbon when a 2-g. sample is used, *i. e.*, 0.0001 g. carbon, either the longer method must be used or steps taken to reduce the carbon dioxide content of the laboratory atmosphere.

## METHOD

### 1—FURNACES AND TEMPERATURE

An electric furnace operating at not less than 1063° C. (the melting point of pure gold) is used.<sup>2</sup> Such a furnace may be obtained upon specification or as a standard article from manufacturers or may be constructed by the operator. The furnace should be equipped with a rheostat so designed with respect

to the line voltage that initially the furnace gives the proper temperature with nearly all the rheostat resistance inserted. As the furnace is used its winding deteriorates and increases in resistance, and this should be compensated by removing resistance on the rheostat so as to maintain the furnace temperature as specified. If the temperature is too low when all the rheostat resistance is removed, the furnace must be supplied with a new winding. Temperatures are checked by the melting point of pure gold. A piece of this metal is flattened out, placed on a clean porcelain or alundum boat and left in the furnace (previously brought to full heat) for 10 min. If the gold is not then melted the temperature is too low.<sup>1</sup> The same piece of gold may be used repeatedly provided care is taken always to place it on a clean surface of either alundum or porcelain.

### 2—BOATS AND LINING MATERIALS

Boats may be of alundum, porcelain, platinum or nickel. Nickel boats may be made very cheaply by cutting a sheet of nickel of proper size at the corners and bending these up. Such boats should be ignited in the furnace in a current of oxygen until free from carbon. To protect the combustion tube from the effects of spattering oxides it is provided with a sleeve of nickel sheet (also ignited until free from carbon). Boats are lined with alundum sand, free from materials causing a blank. The special grade supplied by the Norton Company is quite satisfactory.

### 3—COMBUSTION TUBES AND CATALYZER

Combustion tubes may be standard forms of porcelain or well-glazed quartz, or in fact, any material which has been carefully tested for freedom from porosity. To facilitate fitting of stoppers the cross-section at the end should be circular. In the forward end of the tube a roll of copper gauze 3 in. long and of a diameter sufficient to fill the tube is inserted so that it is heated by thermal conduction from the heated zone of the furnace to a temperature of 200 to 300° C. The copper should not, however, be placed so far in the furnace that there is danger of its melting. The copper is then oxidized by bringing the furnace to a proper temperature while passing oxygen. This copper oxide serves to oxidize any carbon monoxide that might be formed, also any sulfur dioxide, which is oxidized and fixed as copper sulfate. Should too great an accumulation of copper sulfate take place the catalyzer should be renewed.

### 4—RATE OF FLOW OF OXYGEN

The rate at which oxygen is admitted to the forward end of the tube should be not less than 10 to 15 l. per min. At the exit end of the tube the rate should not exceed 225 cc. per min. This latter rate of flow is attained most simply by constricting a glass capillary tube inserted in the forward stopper of the combustion tube so that the desired rate of outflow is obtained with the specified rate of inflow. A plug of glass wool precedes the capillary. The rate of outflow is especially important, for if this is much ex-

<sup>1</sup> *Loc. cit.*  
<sup>2</sup> Lower temperatures may be used with very fine chips; the temperature recommended will burn successfully chips that will just pass a 10-mesh sieve.

<sup>1</sup> The melting point of pure silver (960.5° C.), determined in the same way, is a convenient check on the lower temperature limit.

ceeded, carbon dioxide will be carried past the absorption tube containing the barium hydroxide. The dead space in the combustion tube in front of the copper oxide catalyzer is taken up with a glass tube closed at both ends. This decreases the amount of gas to be flushed out of the tube at each combustion, and materially shortens the time required for a determination.

#### 5—MEYER ABSORPTION TUBES AND BARIUM HYDROXIDE SOLUTIONS

The form of Meyer tube shown in the cited paper has many advantages and may be obtained from dealers on specification. It should be filled with sufficient barium hydroxide solution (25 g.  $\text{Ba}(\text{OH})_2 \cdot 2\text{H}_2\text{O}$  per liter) so that when gas is passing for a determination the liquid fills all the small bulbs and one-half the large bulb at the exit end. The barium hydroxide solution is held in a 10-l. bottle and is delivered by pressure of air free from carbon dioxide.

#### 6—FILTERING AND WASHING

A Büchner funnel fitted to a suction flask and supplied with two superimposed, open-grain, 7-cm. filter papers is used for filtrations. Much time is saved by not having to fold and fit the papers. The large surface exposed insures rapid filtration. The Meyer tube is washed three times, using care to reach all points. The precipitate on the filter is then washed four times, washing the top of the funnel carefully. The wash water is free from carbon dioxide and is delivered from a large glass reservoir by air pressure.

#### 7—STANDARD ACID AND ALKALI

Tenth-normal hydrochloric acid is used. This may be conveniently standardized against weighed portions of chemically pure sodium carbonate which has been fused in a platinum crucible and cooled in a current of pure, dry carbon dioxide. The carbon dioxide is conveniently obtained by heating acid sodium carbonate in a hard glass test tube and passing the liberated gas through a calcium chloride tower. The alkali is standardized sodium hydroxide solution adjusted to be equivalent to the acid. Methyl orange is used as an indicator. Both acid and alkali are delivered from the respective stock bottles to the burettes by air pressure.

#### 8—PROCEDURE

The furnace being at the proper temperature and the filed Meyer tube connected, 2 g. of steel are weighed and transferred to the boat filled with alundum sand. This should have been placed in the hot furnace previous to weighing the sample and then removed so that at the time of placing the sample on it its temperature is just below a visible red. (This will not cause loss of carbon unless the particles of the sample are extremely small—less than 60 mesh.) The boat is then immediately placed in the furnace and allowed to heat for one minute with no oxygen passing. During this time a second sample is being weighed (the balance should be placed in the same room with the furnace for convenience). Oxygen is now admitted at the rate prescribed in Section 4, and at the end of 5 min., if the combustion has been

successful (see Introduction), the Meyer tube may be disconnected and the boat removed from the furnace to cool sufficiently for introducing the second sample. The filtration and washing of the barium carbonate is then carried out as described. By this time the boat is ready for the second sample, which has already been weighed out. The combustion of this sample is then started, using another Meyer tube. The absorption tube used for the previous determination is now washed out by adding to it from the burette the necessary amount of standard acid (this being usually about 5 cc. more than is actually needed to dissolve the carbonate). The acid is transferred from the Meyer tube to a wide-mouth flask, as is also the filter paper carrying the precipitate. The Meyer tube, after washing twice with water, is filled with barium hydroxide solution for the next determination. The flask containing the precipitate is placed on the hot plate until the carbonate is dissolved.<sup>1</sup> Titrations are conveniently made when several flasks are ready. During filtration, washing, etc., of the first determination, the combustion of the second proceeds, but there is still time before its completion for the operator to adjust the acid burette for the second determination, to fit papers to the Büchner funnel, and to weigh out the third sample.

TABLE I—RESULTS OBTAINED BY MODIFIED BARIUM CARBONATE TITRATION METHOD ON BUREAU OF STANDARDS ANALYZED STANDARD SAMPLES

Sample No.	Certificate Value (by Direct Combustion) Per cent	Carbon Found by Method Herein Described Per cent	Number of Determinations	Mean Variation from Certificate Value
35	1.03	1.016 to 1.027	3	-0.01
23	0.805	0.81	3	+0.005(a)
10b	0.373	0.380	2	+0.007(a)
21a	0.617	0.605 to 0.620	12	-0.005

(a) The corresponding differences in the determinations of carbon in Sample 23 given in the cited paper were: 0.000 per cent, 0.000 per cent, 0.000 per cent, and -0.005 per cent; in 10b they were: 0.001 per cent and -0.001 per cent.

BUREAU OF STANDARDS  
WASHINGTON, D. C.

## THE PREPARATION AND TESTING OF PURE ARSENIOS OXIDE<sup>2</sup>

By ROBERT M. CHAPIN  
Received March 12, 1918

### INTRODUCTION

A supply of assuredly pure arsenious oxide is an important matter to the modern analytical chemist. Analytical methods that involve titration with standard iodine, by virtue of their accuracy and convenience, are constantly finding new applications in addition to the already considerable list, and for standardizing such iodine solutions pure arsenious oxide is generally useful and frequently used. Moreover, employment of the substance as an alkalimetric standard has been advocated by Menzies and McCarthy.<sup>3</sup>

From textbooks and various papers dealing with the applications mentioned above, it appears that pure arsenious oxide is very easily obtained. Typical are the statements of Menzies and McCarthy<sup>4</sup>

<sup>1</sup> Avoid long continued heating, which apparently causes some action on the filter paper involving a slight error in the determination.

<sup>2</sup> Published by permission of the Secretary of Agriculture.

<sup>3</sup> *J. Am. Chem. Soc.*, **37** (1915), 2021.

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



that it "may be purchased commercially at a low price already in a high state of purity," and that "it may readily be purified further both by recrystallization, if necessary, and by sublimation." The experience of the writer points to the contrary. Commercial specimens, even of "guaranteed analysis," may be far from pure. Sublimation alone is not a dependable method of purification, and, unless proper conditions prevail, preliminary recrystallization may not be an adequate supplementary measure. In fact it is entirely possible, after recrystallization and sublimation, to end with a "purified" arsenious oxide more impure than the original material. Finally, extant methods for testing are inadequate for detecting the presence of important amounts of certain impurities. Particularly is this true of antimonous oxide, certainly an important impurity and one not unlikely to be present, but for the detection of which, aside from the dubious test of the Pharmacopoeia, there seems to be no practical method extant.

As impurities commonly present in commercial white arsenic, Scott<sup>1</sup> gives "SiO<sub>2</sub>, Sb<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, NiO, CoO, CaO, SO<sub>3</sub>, Cu, Pb, and Zn." The *United States Pharmacopoeia* IX prescribes tests for As<sub>2</sub>S<sub>3</sub>, Sb, Sn, Cd. Thorpe<sup>2</sup> mentions the presence of bismuth, sulfur and carbonaceous matter from the fuel.

#### TESTS FOR PURITY

1. **INSOLUBLE**—To 1 g. of the powdered sample in a wet<sup>3</sup> test tube add 10 cc. of a mixture of 1 vol. concentrated ammonia (sp. gr. 0.90) with 2 vols. water. Heat with shaking to very gentle boiling. The solution should be perfectly clear and colorless with no trace of insoluble residue.

2. **HEAVY METALS**—To the solution obtained under (1) add 10 cc. clear and fresh saturated hydrogen sulfide water, mix, and heat just to boiling. No precipitate, turbidity, or color other than a faint yellow should appear.

3. **ANTIMONOUS OXIDE**—If necessary, filter the hot solution obtained under (2) into another test tube, otherwise cool at once in water and finally place in ice water for 15 min. No turbidity should appear (less than 0.15 per cent Sb<sub>2</sub>O<sub>3</sub>).

4. **SULFUR, SULFIDES**—Dissolve in a test tube, with heat, 1 g. of the sample in 10 cc. normal caustic soda solution, add 1 drop of lead acetate solution, and mix. No coloration should appear (less than 0.005 per cent sulfur).

5. **NONVOLATILE**—Under a hood cautiously heat 1 g. of the sample in a weighed crucible, raising the heat at the end to barely perceptible redness. The material during sublimation should show no darkening (carbonaceous matter) and should leave a nonvolatile residue of less than 0.05 per cent.

Tests 1, 4 and 5 are essentially those of Krauch<sup>4</sup> merely modified in details as experience indicated

<sup>1</sup> "Standard Methods of Chemical Analysis," Wilfred W. Scott. D. Van Nostrand Co., 1917.

<sup>2</sup> *Dictionary of Applied Chemistry*, 1 (1912).

<sup>3</sup> To prevent the substance caking on the glass.

<sup>4</sup> "Chemical Reagents; Their Purity and Tests," E. Merck; Translation by Schenck. D. Van Nostrand Co., 1907.

desirable. Tests 2 and 3, however, are new. Test 2 hardly needs further remark. Test 3 depends on the fact that antimonous sulfide is somewhat soluble in hot ammonia,<sup>1</sup> but separates on cooling, excess of arsenious oxide precluding the presence of ammonium sulfide.

The strength of the ammonia may vary considerably without injury, but the delicacy of the test cannot be increased by using relatively more substance. In fact it is sensibly diminished thereby, probably as the effect of mass action exerted by the arsenious oxide. Merely cooling to about 20° C. will eventually bring out even small amounts of antimony. At this temperature a little over 0.20 per cent is soon plain, but smaller amounts require so much time that the use of ice water is desirable. The first indication of a positive test is an opalescence much like decomposing hydrogen sulfide water. This slowly increases to a yellow turbidity which would tempt the inexperienced to assert positively that the test was a failure and that arsenious sulfide was coming out. Soon, however, examination by transmitted light will reveal a distinct orange tint, and coagulation to characteristic red flocks will ensue after a sufficient time. Pure arsenious oxide with pure reagents will show no trace of turbidity, even on long standing.

The ultimate sensibility of the test is distinctly greater than above indicated, for by letting the tube stand in ice water for 2 hrs. it apparently becomes possible to detect down to 0.10 per cent Sb<sub>2</sub>O<sub>3</sub>. But in this connection it must be remembered that, iodometrically at any rate, small quantities of antimonous oxide will act exactly like arsenious oxide except for the difference in molecular weights. Therefore, the presence of 0.15 per cent of antimonous oxide is equivalent to less than 0.05 per cent of inert matter, a limit sufficient for all purposes except those of such a high degree of accuracy that special purification of commercial material would be imperative in any event.

For experiments on the antimony test, crystallized arsenious oxide was prepared from a mother liquor assuredly freed from antimonous oxide by the removal of an extra and liberal crop of crystals after the regular test on the preceding crop was negative as later described. Antimonous oxide was introduced in the form of a 0.23 per cent solution of tartar emetic, equivalent to a 0.1 per cent solution of the oxide, the desired amount being added to the test tube already containing both the arsenious oxide and the ammonia, but before boiling.

The sulfur test was checked by means of pure arsenious sulfide, dissolved in freshly boiled normal caustic soda.

The matter of tests for pharmaceutical purity being outside the writer's present field of work will be only briefly discussed. Careful sublimation of one gram in a hard glass test tube appears to yield considerable information regarding nonvolatile residue, arsenious sulfide, metallic arsenic, and organic matter. But a test for antimony is certainly desirable. The test in

<sup>1</sup> "Qualitative Chemical Analysis," Prescott & Johnson, 7th Edition, 1916.

the form specified for determining chemical purity is probably too delicate, though it may be made less so by changes of concentration or by cooling at a higher temperature. Apparently the best way of reducing its delicacy is to employ caustic soda solution of definite strength in place of ammonia as a solvent. For example, using 10 cc. of normal caustic soda on a one-gram sample, and cooling in tap water, one per cent of  $\text{Sb}_2\text{O}_3$  in the sample quickly gives a heavy precipitate, while with 0.6 per cent  $\text{Sb}_2\text{O}_3$  the test remains negative for a period much longer than 15 min. Caustic soda might also serve for the detection of insoluble matter in pharmaceutical testing.

The present assay method of the *Pharmacopoeia* must necessarily be inaccurate if antimonous oxide is present, and the only recourse seems to be distillation with hydrochloric acid.<sup>1</sup>

#### PREPARATION OF PURE ARSENIOS OXIDE

The production of pure arsenious oxide on a manufacturing scale is not here considered; merely its preparation in the laboratory. Under such conditions sublimation cannot be depended on to separate volatile impurities. Among these must be included antimonous oxide, as may be easily demonstrated by the specific test for that substance now available. It evidently possesses a sufficient vapor tension at temperatures ordinarily employed, even in careful work, for subliming arsenious oxide to produce appreciable contamination. Since volatile impurities—including carbonaceous matter which will produce elementary arsenic—are apparently quite as important as the nonvolatile, measures to eliminate them ought to precede final sublimation if a reasonably pure product is to be assured.

The classic step preliminary to sublimation is crystallization. The writer has been unable to discover any more practical means for attaining the desired end. But the process of crystallization to be employed is usually very sketchily outlined. Either water or hydrochloric acid of varying strength is specified as the solvent and few details are given. It seems to have escaped notice that arsenious oxide during crystallization strongly adsorbs many impurities—particularly antimonous oxide—present with it in solution. Consequently a "recrystallized" product may be more impure than the original material. Fractional crystallization is the obvious recourse, and properly carried out is very effective and not especially tedious. The absence of antimonous oxide is a rational and convenient index of the success of the operation.

As solvents for arsenious oxide, ammoniacal solutions and strong hydrochloric acid solutions are temptingly effective, but unfortunately both are also effective solvents for accompanying impurities. Water usually dissolves far less of the impurities, so that its disadvantages as a solvent are outweighed by the simplicity of the ensuing fractional crystallization. The final crystals, however, are best deposited from a slightly acid solution.

To 2 liters of boiling distilled<sup>1</sup> water in a 3-liter "boiling flask" are slowly added 150 g. powdered, white arsenic made into a cream with a little water. The mixture is boiled briskly for about 1 $\frac{1}{4}$  hrs. or until the volume is reduced to about 1600 cc., then is removed from the heat and allowed to settle for a few minutes. If the liquid tends to settle fairly clear it may be filtered at once through a fluted paper, but if it remains pronouncedly milky it will filter badly and must be clarified. To this end it is decanted from the sediment into another flask, brought to boiling, treated with milk of lime prepared from 2 g.<sup>2</sup> pure  $\text{CaO}$ , boiled 10 min., then filtered.

The filtrate is boiled down in a stout beaker until 4 or 5 g. of solid have separated. Bumping will not be serious if the flame is lowered sufficiently. It is then decanted, boiling-hot, through a rapid fluted paper into another stout beaker kept hot during filtration, either on the steam bath or over a flame, and the filtrate kept close to the boiling point while the test for antimony is made on the deposited arsenious oxide in the first beaker. For the purpose in view a comparatively coarse and rapid test is adequate. A quantity of the moist substance which is judged to be equivalent to about one gram dry is brought into a test tube and the test made as usual except that cooling for 5 min. under running tap water is sufficient. Unless antimony is very plainly present in the deposit, the mother liquor is certainly antimony-free. Sometimes no antimony will be found even when much was present in the original substance for it appears to be practically wholly adsorbed by solid arsenious oxide if a sufficient quantity of the latter remains undissolved. Conversely, if solid arsenious oxide is absent, antimonous oxide may pass into the filtrate in large amount, evidently being much more soluble in a solution of arsenious oxide than in pure water.

If the test for antimony is positive it will be necessary to take off successive crops of crystals in the same way until a negative test finally results. Fractional crystallization by cooling is not advised for the reason that at temperatures below 85° the formation of crystalline from amorphous arsenious oxide progresses with such rapidity that the process is likely to get out of control.

The antimony-free mother liquor is treated with approximately either one or one and one-half per cent of its volume of concentrated hydrochloric acid, depending on whether lime was employed for clarification, and boiled down in a beaker to evident saturation. After a final filtration, it is cooled rapidly with frequent stirring to prevent the formation of crusts, then let stand overnight. After being well stirred up, the crystals are filtered with suction and washed chloride-free. The yield naturally varies much, but will commonly be around 75 g. The crystals will probably be very nearly pure, but the writer has never been able to secure any which did not show slight darkening during sublimation.

For sublimation the apparatus of Menzies and

<sup>1</sup> Most recently studied by Roark and McDonnell, *THIS JOURNAL*, 8 (1916), 327.

<sup>2</sup> Tap water may introduce undesirable organic impurities.

<sup>3</sup> This quantity has so far proved sufficient.

McCarthy<sup>1</sup> is convenient but of small capacity. On a larger scale excellent results may be obtained with a stout-walled 250 cc. Erlenmeyer flask of rather hard glass, charged with 75 g. arsenious oxide evenly spread over the bottom. The flask is set in a flat sand bath, and the sand brought about one-half inch above the level of the material within. The mouth of the flask is covered with a porcelain crucible cover, and the bath is heated under a hood with a sufficiently powerful burner of the ring or stove type. The sublimation may require 7 hrs. and should not be intermittent for the flask, once cooled, is likely to crack when reheated. It is finished when a line of clear glass appears all around the flask above the sand. When completely cool, the flask is removed from the bath and with cautious handling the bottom is cut off with a hot point just below the line of the sublimate. The latter may then be easily removed with a knife blade. It is best to reject the portion in the neck of the flask, for volatile impurities will tend to concentrate there. The writer prefers to reject also the glassy ring of sublimate just above the sand level. With proper management the yield is excellent, very little being lost by volatilization.

#### CONCLUSION

It must be admitted that arsenious oxide lacks some of the qualities desirable in a standard substance for volumetric analysis. Nevertheless it is capable of affording extremely accurate results and for the purpose of directly standardizing iodine solutions there seems to be no other substance<sup>2</sup> which can fully take its place. Since it must be used, the chemist must possess practical and reliable methods for determining whether commercial samples are sufficiently pure for ordinary analytical work, while in case of deficiency, or when the material must be of the highest purity, dependable methods of preparation must be available. It is believed that these needs are met by the methods of preparation and testing here presented. Especially are methods given for detecting and eliminating an impurity to which far too little attention has been given in the past, *viz.*, antimonous oxide.

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#### THE BISULFATE METHOD OF DETERMINING RADIUM

By HOWARD H. BARKER

Received December 5, 1917

The quantitative determination of the small amounts of radium in radioactive ores and various products obtained in their treatment for the recovery of radium is generally conducted by the emanation method. It is based upon the fact that the gaseous disintegration product of radium—the emanation—can be completely separated from radioactive materials and that the equilibrium quantity is proportional to the radium content. Very small quantities of radium emanation

are capable of exact measurement by electrical methods, for which carefully standardized electroscopes have come into general use.

The care and accuracy with which the emanation is separated from the specimen under examination are very essential in quantitative work and demand skilful manipulation, especially in the case of solids. Since heat alone rarely affects a complete separation of the emanation from the radioactive solids, the recognized methods of determining radium involve chemical treatment that will result in a solution or in decomposition of the material by fusion. Our experiments test the applicability of fusion with bisulfate, for the separation of radium emanation, and afford a comparison of this method with other methods recognized as more or less standard.

Our work deals with substances which are not susceptible of direct solution in water or acids. The emanation is separated by direct fusion with sodium or potassium bisulfate or mixtures of the two. Since solids generally emit, at ordinary temperatures, variable proportions of the emanation continually produced by the radium present, the customary procedure is to first expel the emanation, that is, reduce it to zero, and then seal up the sample, allow it to stand for a definite time—a day or longer—and finally separate the accumulated emanation by fusing again. The equilibrium amount is then conveniently obtained by reference to a table of growth of radium emanation.<sup>1</sup>

Plum<sup>2</sup> made use of potassium bisulfate for radium determination in his study of methods of separating the radioactive constituents from carnotite ores. Schlundt<sup>3</sup> employed potassium bisulfate in a few of his experiments on the quantitative determination of radium mainly by fusion with mixed alkali carbonates. The experiments that follow I hope will serve to establish the reliability of the bisulfate method and also its range of applicability.

#### PLAN OF EXPERIMENTS

Representative carnotite ores and the radium-bearing products obtained therefrom were selected for the determinations of radium which were conducted by at least two other methods for comparison with the bisulfate method. The samples include (1) two carnotite-bearing sandstones from different localities and differing considerably in composition and radium content, (2) a siliceous concentrate obtained from an ore by chemical treatment in which the radium has been concentrated about fifteen-fold, (3) a sample of first sulfate containing about 600 parts of radium per billion of total weight, one-fourth of which is barium sulfate, (4) one sample of tailings, the sandy residue left after the extraction of radium from the ore, (5) a by-product very rich in gypsum, but containing also vanadium, iron and uranium compounds.

The radium was determined in case of these samples by at least three methods.

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

<sup>2</sup> Antimony potassium tartrate, advocated by Metzl (*Z. anorg. Chem.*, 48 (1906), 156), is well known to be efflorescent when crystallized. The present writer has been unable to dry it to strictly constant weight in agreement with the theoretical composition of the anhydrous salt.

<sup>1</sup> Kolowrat, *Le Radium*, 6, 195; also Curie, "Traité de Radioactivité," I, p. 419; *Chem. Kalender*, 2 (1914), 361; Rutherford, "Radioactive Substances and Their Radiations," 1913, 665.

<sup>2</sup> *J. Am. Chem. Soc.*, 37 (1915), 1811.

<sup>3</sup> *Trans. Am. Electrochem. Soc.*, 26 (1914), 163.

## METHODS OF SEPARATING THE EMANATION

1—Preparation of a refined radium-barium sulfate by fusing a weighed sample, to which a small quantity of barium compound has been added, with 3 to 5 times its weight of a mixture of sodium and potassium carbonates. After leaching the melt with water, the insoluble carbonates are dissolved in dilute hydrochloric acid and pure radium-barium sulfate is precipitated by the addition of a few drops of sulfuric acid. The

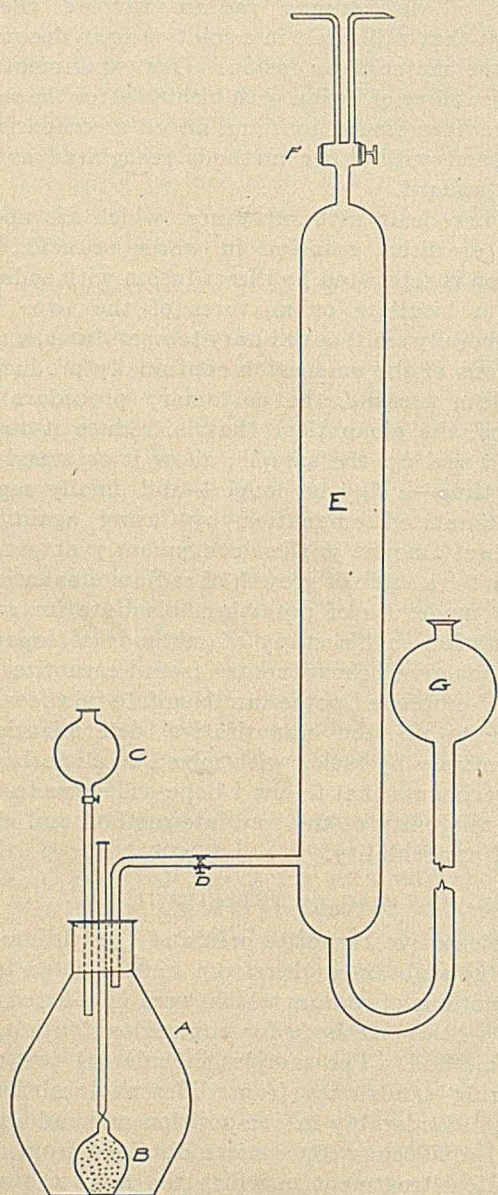


FIG. I.—TYPE OF APPARATUS FOR BOILING OFF EMANATION FROM MIXED CARBONATE FUSION

refined sulfate thus obtained is fused in a platinum boat with a mixture of sodium and potassium carbonates and is then sealed and stored for a certain period after which the accumulated emanation is again separated, collected and transferred to a standardized electro-scope.<sup>1</sup>

2—Direct separation of the emanation from the melt is obtained by fusion of the sample in a platinum crucible with mixed carbonates of sodium and potas-

sium. The melt is poured into a clean iron mortar. The crucible may be rinsed out by fusing a little more of the carbonates in it. The melt is powdered and sealed up in a thin-walled glass bulb, B, shown in Fig. I. After standing for a day or longer the emanation that has accumulated during the period of storage is separated by dissolving the carbonate in nitric or hydrochloric acid. The gas burette E of the apparatus used for this operation (Fig. I) is filled with a boiling hot solution of caustic soda through the levelling reservoir G by closing cock D and having F open. Then, after closing F, the levelling bulb G is lowered to a position below the outlet tube D in order that subsequent boiling will be conducted under slightly diminished pressure. This precaution also enables one to detect small air leaks in the apparatus. The bulb B containing the powdered carbonate is so adjusted in the 3-hole rubber stopper of the wide-mouth flask A, the boiler, that it is cracked by forcing down the stopper. The cock D is then opened and moderately strong acid is admitted gradually from the drop funnel C. When the sample is siliceous and the carbonate fusion exceeds 5 g., the capacity of the boiler should be at least 250 cc., and it is sometimes advantageous to have a little water in it to distribute the carbonate when the bulb is broken and in this way prevent the formation of a coating of silicic acid over the dry powder when acid is admitted. If the volume of air remaining in the boiler still exceeds the capacity of the burette, the air may be partly displaced by carbon dioxide before connecting up with the burette. As the decomposition of the carbonate proceeds the solution is heated and finally boiled until the liquid in the gas burette is forced down by water vapor to the level of D. The gas is then transferred quantitatively to an air-tight electro-scope.

3—The bisulfate method consists in making a fusion of the material whose radium content is to be determined with either sodium or potassium bisulfate alone, or better, a mixture of the two in a hard glass pyrex tube of suitable dimensions. Hard glass pyrex tubes 25 cm. by 2.5 cm. were used in most of the determinations. The material and the bisulfate are thoroughly mixed before introducing into the tube, after which they are fused and boiling continued long enough to expel the last traces of emanation. The sides of the tube are washed down by introducing a little fresh bisulfate into the tube, allowing it to fuse and run down into the melt. The test tube is sealed with a stopper carrying two outlet tubes, one extending to within an inch or two of the melt, and the other just reaching through the stopper. After the emanation has been allowed to accumulate for a given definite period, the tube is connected up with the evacuated ionization chamber of an electro-scope as shown in Fig. II. The micro drying bulb next to the ionization chamber contains concentrated sulfuric acid, whose function is to remove moisture from the air and emanation before entering the electro-scope. The other drying bulb connected to the test tube on the ionization chamber side of the apparatus contains a fairly strong solution of caustic soda to remove any traces of sulfuric acid distilled over in the operation. The drying bulb hitched

<sup>1</sup> Further details are given by Lind, *THIS JOURNAL*, 7 (1915), 1024, and in U. S. Bureau of Mines, *Bull.* 104, 94.

to the other side of the test tube contains concentrated sulfuric acid or water, and is there primarily for noting the inflow of air into the system. It also serves for detecting any leaks in the line of connection. All rubber connections are wired, and the tips of the glass tubes are not broken until all connections have been made. Before breaking the capillary tips, the intermediate system is placed under a slight vacuum. The tips are then broken and by adjusting the stopcocks of the evacuated ionization chamber the flow of air through the chain is regulated. Heat is now gently applied until the mass is molten, after which it is boiled for about 5 min. Sometimes it is a little difficult to get good boiling at this stage, but I find where an equal mixture of the fused bisulfate and crystals is used, that this difficulty seldom appears. The emanation is swept into the chamber by a slow, steady current of air which is maintained during heating until atmospheric pressure is nearly attained in the chamber.

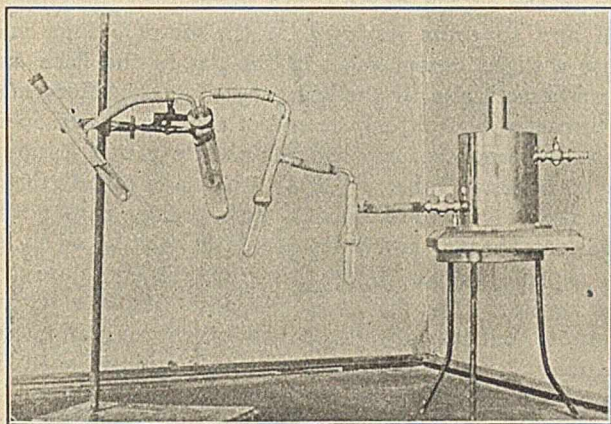


FIG. II

Readings of the rate of discharge are made 3 hrs. after the introduction of the gas, the same as in the other methods.

#### RESULTS AND DISCUSSION

The results obtained are summarized in the following table. The values represent duplicate determinations for the most part, but some of them are triplicate. The values in the table represent grams of radium per gram of material  $\times 10^{-9}$ .

Material	Wt. of Sample	Refined Sulfate	Mixed	
			Carbonate	Bisulfate
Carnotite No. 1.....	1.00	5.15	5.18	5.22
Carnotite No. 2.....	1.00	10.60	10.00	10.40
Concentrate.....	0.50	29.90	30.00	29.40
Tailings.....	3.00	0.90	0.94	1.00
First Sulfate.....	0.05	676.00	680.00	678.00
By-products.....	2.00	1.26	1.24	1.27

The good agreement in the values shows that from the standpoint of accuracy in radium determination, no one method has any advantage for the classes of materials examined. The bisulfate method, however, presents some advantages which deserve recognition.

I—It has been established that the bisulfate method may be used in liberating the emanation from samples of pitchblende, whose radium content is known, used in the standardization of electroscopes. There are two advantages worthy of mention which the bisulfate

method has in this operation over the solution method: (a) It is not necessary to take into consideration the "emanating power" of the pitchblende in the case of the bisulfate method, while it is in the case of the solution method; (b) The same carefully weighed samples of pitchblende may be used for several standardizations by resealing the bisulfate melt after the emanation has been driven off and collected for standardization.

II—Besides simplicity of operation, the bisulfate method is by far the most rapid procedure. Not only is the actual time required for the final separation of the emanation shorter than in the other procedure, but the time in work expended in preparing a sample and getting it sealed up is reduced to a few minutes.

III—The chances for loss due to manipulation are reduced to one operation, when the material is transferred to the test tube. In both of the other methods great care must be exercised in avoiding losses when fusions are in progress. Instead of one, two to three transfers of materials occur in the other methods.

IV—The cost of the operation is reduced. If one is careful the test tubes may be used for several determinations. In the refined sulfate method the small platinum boats, costing at least a dollar each are not good for more than 5 to 6 determinations. The use of larger platinum vessels is essential in the other methods, while in the bisulfate method all fusions are made in hard glass test tubes.

V—After the final separation of emanation by the bisulfate method, the material remains and is ready for another determination in case an accident occurs.

When the material under examination contains thorium, then the gas cannot be transferred directly to the electroscope during fusion, but must be collected in a gas burette to allow the decay of thorium emanation.

I wish to thank Mr. J. C. Simpkins, who assisted me at the outset in this work, and Dr. H. Schlundt for his helpful suggestions.

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#### A RAPID PRESSURE METHOD FOR THE DETERMINATION OF CARBON DIOXIDE IN CARBONATES

By W. H. CHAPIN

Received October 3, 1917

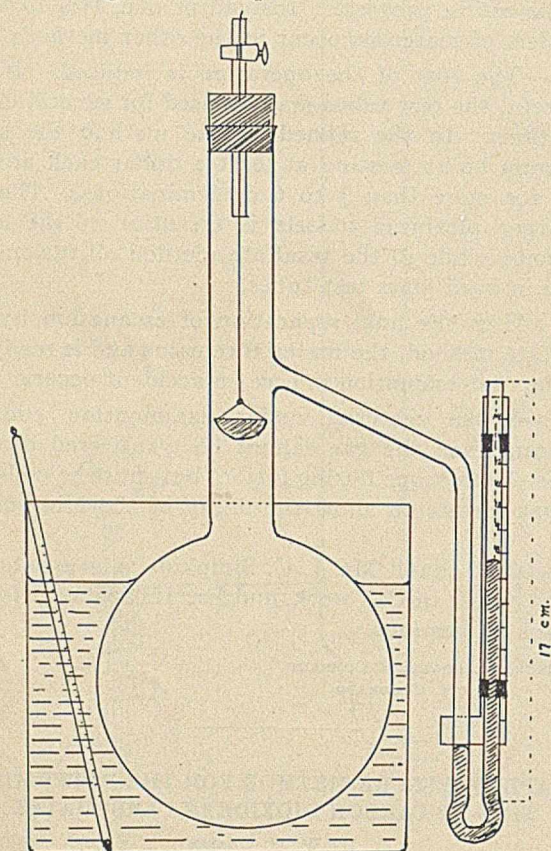
By use of the apparatus sketched below carbon dioxide may be very quickly determined in any carbonate which is soluble in cold hydrochloric acid. The accuracy of the method is equal to that attainable with the absorption method, except possibly when the latter is in the hands of a very skilled manipulator who has had long practice with the method.

The principle is very simple: The carbonate is allowed to dissolve in dilute HCl contained in a flask of known volume to which is attached a small mercury manometer. The change in pressure is read off, and by a simple calculation the weight and percentage of CO<sub>2</sub> are obtained. The necessary details are given in the procedure.

## APPARATUS

The flask is made from a 600 cc. distilling flask by cutting off the side tube and sealing on the manometer tube in its place. The latter should have a bore of 5 mm. If too small, the capillary effect will interfere with accurate reading, and if too large the movement of the mercury changes the volume of the apparatus too much.

Attached to the manometer tube is a sliding scale of celluloid 15 cm. long and graduated in millimeters. This scale is made from a 15 cm. (6 in.) ruler by cutting out one side as seen in the sketch. It is held in place by means of small metal clips which may be slipped out of the way when reading. By use of a small lens it is possible to read the position of the mercury to  $\frac{1}{5}$  mm. The inside of the manometer tube and the mercury used in it must be clean and dry or no accurate readings can be expected.



The capsule used for weighing out the sample is suspended by a thread as seen, the latter being caught and held in the stopcock until it is desired to drop the sample into the acid. This capsule may be made of copper or any metal not displacing hydrogen from the acid.

The stopper should be of rubber, smooth and close fitting. Its tightness may always be insured by wetting slightly at the moment of inserting. It should be adjusted to a mark on the neck of the flask.

To keep the temperature of the apparatus constant and to make it easy to determine, the bulb of the flask is kept immersed in water at room temperature. The thermometer used in reading the temperature is kept standing in this water.

Before using the apparatus, its capacity is determined by filling with water from the bottom of the near arm of the manometer to the stopcock, and weighing. To prevent the water going too far in the manometer tube a rubber connector is placed over the open end and then closed by means of a pinchcock. If this is opened slightly after the flask is filled the water may be adjusted to any position.

## PROCEDURE

First set up the apparatus as seen in the sketch, surrounding the bulb with water at room temperature. Fill the flask with carbon dioxide from a generator or pressure cylinder, and then run in by means of a pipette 10 cc. of 3 *N* HCl which has also been saturated with carbon dioxide.<sup>1</sup> Weigh out a sample of the powdered carbonate into the capsule (0.7 g. where the CO<sub>2</sub> content is about 20 per cent, and 0.4 g. where it is as high as 40 per cent). Suspend the loaded capsule as seen in the sketch, taking care to moisten the stopper well and to press it down to the mark. This may cause a slight compression of the gas in the flask and a consequent rise in the mercury, but the effect may be corrected by holding the end of the thread and opening the stopcock for a moment while gently tapping the manometer tube. When all is ready place a finger over the end of the stopcock tube, taking care not to catch the thread, and then open the cock so as to let the capsule drop. After this immediately close the cock again. The carbonate usually dissolves within a minute, but it is always best to watch the manometer for about 5 min. for further rise, tapping gently in the meantime. Finally, when the reaction is complete, adjust the scale so that the lower end corresponds with the meniscus in the near arm of the tube and then read off the height of the column in the other arm. Great care must be taken to get exact adjustment and to avoid parallax. Read to centimeters and tenths and estimate to hundredths. Also take the temperature accurately.

We now have the volume of the CO<sub>2</sub> at room temperature,  $V_t$  (vol. of flask minus 10 cc. occupied by acid). We also have the pressure  $P$  of the CO<sub>2</sub> (as read on the manometer). Finally, we have the temperature  $t$ . We can get the weight of the CO<sub>2</sub> by calculating down to standard conditions and multiplying into the weight of 1 cc. of CO<sub>2</sub> under standard conditions (0.001965 g.). We then have:

$$\text{Wt. of CO}_2 = \frac{V_t \times P \times 273 \times 0.001965}{760 \times (273 + t)}$$

Since the factors  $V_t$ , 273, 0.001965 and 760 are always the same, we may work out the value of the fraction

<sup>1</sup> If the acid is not first saturated with CO<sub>2</sub> a part of the gas evolved during the reaction will remain in solution. If the flask is not first filled with CO<sub>2</sub> a part of the gas dissolved in the HCl will at first be given off, and later during the determination, when the pressure of the CO<sub>2</sub> rises, gas will again go into solution. Where the pressure of the CO<sub>2</sub> in the flask is one whole atmosphere at the start, no gas will be given off from the acid, and since the change in pressure during the determination will be comparatively slight, very little will then go into solution. It is best to keep the stock of HCl in a gas wash bottle connected with a Kipp generator, where CO<sub>2</sub> may at any time be forced through it. The tube through which the CO<sub>2</sub> is led into the apparatus when filling with this gas is attached to the wash bottle. Thus every time the apparatus is filled just that much CO<sub>2</sub> passes through the acid.

$$\frac{V_t \times 273 \times 0.001965}{760}$$

and call this the constant for the apparatus, K. We then have:

$$\text{Wt. of CO}_2 = \frac{KP}{273 + t}$$

The percentage of CO<sub>2</sub> in the carbonate then follows, thus

$$\text{Percentage of CO}_2 = \frac{100 KP}{(273 + t) \times \text{Wt. of sample}}$$

By use of logarithms the necessary calculation may be made in less than 2 min. The total time consumed in making a determination, including the weighing and calculation, need not be over 15 min.

After completing a determination the capsule may be lifted out of the apparatus by means of a wire hook, and the spent acid may be drawn out with a pipette. The apparatus is then ready for the next determination without even refilling with CO<sub>2</sub>.

RESULTS		
Calcite Per cent CO <sub>2</sub>	Argillaceous Limestone Per cent CO <sub>2</sub>	Na <sub>2</sub> CO <sub>3</sub> aq. Per cent CO <sub>2</sub>
43.53	24.71	34.74
43.67	24.34	34.40
43.40	24.67	By absorption method
43.52	24.36	34.59
	24.59	34.30
Av., 43.51	Av. 24.53	
By ignition	By absorption method	
43.59	24.47(a)	

(a) Average of 100 determinations varying from 24.1 per cent to 24.8 per cent.

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### A PROXIMATE ANALYSIS OF THE SEED OF THE COMMON PIGWEED, AMARANTHUS RETROFLEXUS L

By EVERHART P. HARDING AND WALTER A. EGGE

Received August 3, 1917

It was thought by the authors of this paper that the partially carbonized bracts of the seeds of this common plant might make a good filtering medium for decolorizing sugar and other colored solutions. This suggested other possible uses of the seeds which led to their proximate analysis.

#### DESCRIPTION OF PLANT<sup>1</sup>

This variety of pigweed is commonly called "red root," "rough pigweed," "green amaranthus" and "Chinaman's greens." It is an annual weed which grows from a well-formed and fairly deep-rooted tap root. The root is generally red. The plant grows from 1 to 3 ft. high and is branched, the branches coming obliquely from the stem. The stem and leaves are rough. The plant flowers from July to September. These flowers are very inconspicuous, appearing in the angle formed by the stem and leaf stalk. The seeds are oval, black and shiny, and ripen during August, or before. The weed occurs in all parts of the State of Minnesota and thrives in all kinds of soil, but prefers a rich loam. It is common in gardens and waste places and does most injury by crowding out crop plants.

<sup>1</sup> Minnesota Agricultural Experiment Station, *Bull.* 129, March 1913, p. 37.

#### PREPARATION OF SAMPLE

The seeds were stripped from plants growing in Waseca County in the southern part of Minnesota. They were cleaned by removing foreign matter and chaff (bracts). The separation and removal of the bracts was difficult and tedious. Approximately 75 per cent of the seeds were black and fully matured, the rest were red, showing varying degrees of maturity. The sample was rapidly ground to 20-mesh size and the moisture determined on a portion of this size to represent total moisture in the seeds. The rest was air-dried for 7 days and then ground to a 72-mesh size. Moisture and all other determinations were made on this size sample.

#### ANALYSIS

MOISTURE IN 20- AND 72-MESH SAMPLES—One-gram samples were dried in an electric drying oven at exactly 100° C. Preliminary tests showed that 14 and 4 hrs., respectively, were required to bring the 20- and 72-mesh samples to constant weight.

	20-Mesh		72-Mesh		
	I	II	I	II	III
Moisture content, grams.....	0.1127	0.1129	0.0860	0.0860	0.0860
Average percentage of moisture	11.28		8.60		

ASH—Ash was determined on one-gram samples in an electric muffle at a temperature of 620° C.

	I	II	III
Ash content in grams.....	0.0445	0.0448	0.0446
Average percentage of ash.....	4.46		

It was very difficult to burn the substance completely to an ash over a Bunsen burner.

DETERMINATION OF OIL (ETHER EXTRACT)—About one-gram portions of the material, dried respectively to constant weight in an air oven and in a vacuum sulfuric acid desiccator, were extracted in Soxhlet extractors with anhydrous, alcohol-free ethyl ether to completion, which required 16 hrs.

	I	II	III	IV
Oven dried, grams oil.....	0.0798	0.0793	0.0788	0.0791
Desiccator dried, grams oil.....	0.0814	0.0788	0.0801	....
Oven dried, average percentage of oil...	7.92			
Desiccator dried, average percentage of oil	8.46			

The dried oil dissolved in cold sulfuric ether, but not in cold petroleum ether.

PROTEIN—Protein was determined by Gunning's modification of Kjeldahl's method, using one-gram samples and the nitrogen conversion factor 6.25.

	I	II	III	IV
Protein content, grams.....	0.1880	0.1873	0.1867	0.1880
Average percentage of protein.....	19.13			

STARCH DETERMINATION (ACID CONVERSION METHOD)—Three-gram samples were used. The usual acid conversion<sup>1</sup> of starch into dextrose was made and the amount of dextrose determined by the Munson-Walker method.<sup>2</sup> The seeds occupied a volume of 2.10 cc., for which allowance was made. Aliquot parts of the solution equivalent to 0.30 g. of substance were used in the reductions and the starch conversion factor of 0.90 was used.

The following amounts of reduced cuprous oxide and the corresponding weights of dextrose and starch were found.

<sup>1</sup> Sachse, *Chem. Zentr.*, 1877, 732; Bureau of Chemistry, U. S. Dept. of Agriculture, *Bull.* 107.

<sup>2</sup> Browne, *J. Am. Chem. Soc.*, 1906, 439.

	I		II		III	
	1	2	1	2	1	2
Cu <sub>2</sub> O, mg.....	299.6	298.0	297.5	298.4	300.1	299.4
Dextrose, mg.....	137.0	136.2	135.95	136.40	137.25	136.90
Starch, mg.....	123.3	122.6	122.4	122.7	123.5	123.2
Percentage of starch	41.09		40.91		40.98	
Average percentage of starch.....	40.96					

STARCH DETERMINATION (DIASTASE METHOD)—Three-gram samples were used and the diastase conversion carried out as outlined in Leach's "Food Inspection and Analysis," p. 284. The Munson-Walker method was used for determining the amount of dextrose. The reductions were determined on an aliquot part of a definite volume equivalent to 0.240 g. of material. For the conversion 0.60 g. of commercial diastase with a reduction equivalent of 33.1 mg. of cuprous oxide was used.

	I		II		III	
	1	2	1	2	1	2
Cu <sub>2</sub> O, mg.....	233.9	234.1	232.8	233.3	232.8	232.0
Dextrose, mg.....	89.40	89.50	88.85	89.10	88.85	88.46
Starch, mg.....	80.46	80.55	79.96	80.19	79.96	79.61
Percentage of starch.....	33.53	33.56	33.32	33.41	33.32	33.17
Average percentage of starch.....	33.39					

DETERMINATION OF SUGAR (REDUCING SUGARS)—The determination of sugar was difficult on account of the colloidal condition of the sugar extract. This difficulty was finally overcome by keeping the solution just slightly alkaline, which seemed to settle the colloids. Filtering was avoided as far as possible by increasing the volume of the solution and pipetting an aliquot volume.

Five-gram samples were boiled in 150 cc. of 50 per cent neutral alcohol for an hour on a steam bath with reflux condenser. The solution was cooled to room temperature and the volume made up to 500 cc. with 95 per cent alcohol made just alkaline. After thoroughly mixing and settling over night, 400 cc. were pipetted off with continuous suction and evaporated on a water bath to 20 cc. This volume was made up to 250 cc., using 2 cc. of lead acetate to clarify. After clarifying, 200 cc. were pipetted with continuous suction into a beaker and the excess of lead precipitated with anhydrous sodium carbonate. The solution was filtered and 50 cc. of the filtrate used for determining the sugar by the Munson-Walker method. From an aliquot part of the solution equivalent to 0.80 g. of material only a mere trace of Cu<sub>2</sub>O was formed.

DETERMINATION OF SUGAR (AFTER INVERSION)—Fifty cubic centimeters of the solution in the preceding determination from which the excess lead was precipitated were pipetted into a 100-cc. graduated flask, 5 cc. of concentrated hydrochloric acid added, the volume made up to 100 cc. with distilled water, and allowed to remain over night at about 20° C.

	I		II	
	1	2	1	2
Cu <sub>2</sub> O, mg.....	21.00	21.40	21.90	21.10
Dextrose, mg.....	9.40	9.56	9.76	9.44
Percentage of dextrose.....	2.35	2.39	2.44	2.36
Average percentage of dextrose.....	2.39			
Percentage calculated as cane sugar.....	2.15			

The acid was nearly neutralized and the sugar determined in an aliquot part of the solution, equivalent to 0.40 g. of material, by the Munson-Walker method.

DETERMINATION OF CRUDE FIBER—This determination was made on 2-g. samples containing 8.57 per cent of moisture. Kennedy's modification<sup>1</sup> of Sweeney's method was used and modified by filtering and igniting in alundum crucibles.

	I	II	III	IV
Crude fiber and ash, grams.....	0.2585	0.2650	0.2590	0.2580
Ash, grams.....	0.0358	0.0380	0.0398	0.0382
Crude fiber, grams.....	0.2127	0.2220	0.2192	0.2198
Percentage crude fiber.....	10.63	11.1100	10.96	10.99
Average per cent crude fiber.....	10.92			

TANNIN—Qualitative tests showed tannin.

CONSTITUENTS	SUMMARY			
	20-Mesh as Received	72-Mesh Air-Dried	Oven-Dried	Desiccator-Dried
Moisture.....	11.28	8.60	0.00	....
Ash.....	4.33	4.46	4.88	....
Oil (ether extract).....	7.03	7.24	7.92	8.46
Protein.....	18.57	19.13	20.93	....
Starch (diastase).....	32.40	33.39	36.52	....
Starch (acid conversion).....	39.77	40.98	44.83	....
CONSTITUENTS	PERCENTAGES ON			
	20-Mesh as Received	72-Mesh Air-Dried	Oven-Dried	
Hemicellulose (starch by acid conversion minus starch of diastase).....	7.37	7.59	8.31	
Sugar reducing.....	trace	trace	trace	
Sugar (after inversion).....	2.32	2.39	2.31	
Sugar (after inversion computed to cane sugar).....	2.08	2.15	2.35	
Crude fiber.....	10.59	10.92	11.92	
Tannin and other undetermined substance by difference.....	6.35	6.52	7.17	

The proximate analysis shows that the seeds would make a good component part of a stock food and as seeds of related species have been found to contain considerable amounts of potassium nitrate, the rather high protein content would suggest that they might be valuable as a chicken or bird food.

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### THE DETECTION OF VEGETABLE GUMS IN FOOD PRODUCTS

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Received May 2, 1918

The use of gums in food products is dependent mainly on their physical properties, the most noteworthy of which is their colloidal nature. This property enables the gum substance to hold within itself relatively large quantities of water and still impart a decided "body" to the mixture. Their use is specifically, then, as thickeners and binders in such food products as marshmallow preparations, ice cream, custards, pie fillings, egg substitutes, and flavoring emulsions. The gums ordinarily employed are gum arabic, gum tragacanth, Indian gum, agar-agar, and commercial dextrin. Gelatin, egg albumin, and commercial glucose, as well as starch, are also used for the same purpose.

#### EXISTING METHODS

The methods which have been proposed for the detection of this class of materials are based for the most part on isolated reactions for a particular gum, depending on some color or solubility test of the crude gum itself, and having no reference to the detection of small amounts of the gum in a complex food mixture. Of the few that are more general perhaps the best

<sup>1</sup> THIS JOURNAL, 4 (1912), 600.



known are those proposed by Patrick,<sup>1</sup> and Howard,<sup>2</sup> and the group scheme devised by Congdon.<sup>3</sup> The first two of these were suggested only for ice cream and, while quite simple and easily performed, are unsatisfactory for complicated food mixtures and entirely useless so far as identification of the gum is concerned. The more pretentious scheme of Congdon appeared quite promising and was given a thorough trial on food products and on known mixtures with gums. The results obtained were, however, very disappointing.

Congdon's procedure is evidently based on qualitative tests made on the crude gums, no provision being made for separating the gum from the other components of the complex food mixture, no matter how seriously these interfere with precipitation or color tests. Further, some of the tests included in the scheme were found to be neither specific nor conclusive even with the gum itself.

#### SEPARATION OF THE GUM

As the basis for a workable scheme it was decided at the outset that on account of the complex food mixtures to which gums are added, all tests for the identification of the thickener should be limited to tests made on the relatively pure gum substance, previously separated from the food product. This, of necessity, eliminates many of the tests described in the literature for the individual gums, most of which are dependent on impurities naturally occurring in the raw gums, and limits the available reactions for an orderly scheme largely to the precipitation tests. All such tests that could be found in the literature, and the action of all available solvents were carefully studied on solutions of gum arabic, agar, gum tragacanth, Indian gum, dextrin, gelatin, and egg albumin. Since Indian gum is not so specific a term as "arabic" or "tragacanth" and includes at least two different species, two samples of this gum, of entirely different appearance and obtained from different sources, were used.

After much experimentation, which need not be detailed here, the following systematic procedure was finally adopted for the separation of the gum in a comparatively pure condition from the food product. This procedure consists, in brief, in precipitating the protein of the food mixture by heating with acetic acid and tannin, and then precipitating the gums from the filtrate by acetone. In this way the sugars and other acetone-soluble materials are left in the filtrate. Since milk is a common ingredient of the class of foods in question, soluble phosphates have also to be removed by an extra precipitation with ammonia. Finally, the redissolved gums are precipitated relatively pure by alcohol. The procedure is summarized in Table I.

TABLE I—THE SEPARATION OF GUMS

#### A—ELIMINATION OF PROTEINS

- 1—Dilute sample to suitable concentration with water, add 5 cc. dilute acetic acid and 25 cc. of 10 per cent tannin solution, and heat mixture for 20 to 30 min. Centrifuge and filter. Discard precipitate.

<sup>1</sup> U. S. Dept. of Agr., Bur. of Chem., *Bull.* 116, 24.

<sup>2</sup> *J. Am. Chem. Soc.*, 29 (1907), 1622.

<sup>3</sup> *THIS JOURNAL*, 7 (1915), 606.

*Note*—Casein, coagulable proteins, and some of the gelatin precipitated. Fats and other insoluble substances included in precipitate.

- 2—Add 40 to 50 cc. more tannin solution to filtrate from A1 and heat for short time. Centrifuge and filter. Discard precipitate.

*Note*—Remainder of gelatin and soluble proteins precipitated.

#### B—SEPARATION OF GUMS AND DEXTRIN FROM SUGARS

- 1—Treat clear filtrate from A2 with twice its volume of acetone. Centrifuge and filter. Discard filtrate. Wash precipitate twice with acetone.

*Note*—Precipitate includes gums and dextrin. No precipitate shows absence of gums, dextrin, and milk solids.

- 2—Dissolve precipitate from B1 in 50 cc. of warm water slightly acidified with acetic acid and add 10 cc. of ammonia (sp. gr. 0.90). Centrifuge and filter. Discard precipitate.

*Note*—Calcium phosphate from milk solids precipitated.

#### C—ISOLATION OF PURE GUM SUBSTANCE

Add acetic acid to filtrate from B2 until slightly acid. Add alcohol, one volume at a time, until a well defined precipitate appears.

*Note*—Gums and dextrin precipitated in fairly pure condition. No precipitate with five volumes of alcohol indicates absence of gums and dextrin.

Within certain limitations, which will be discussed later, this procedure is capable of separating gums and dextrin from complex food mixtures. In the numerous experiments on which it was based the amount of gum present varied from 0.1 to over 1.0 g. and the weight of sample from 50 to 200 g. It is certain that amounts of gum as small as 0.1 g. can be separated by the procedure from ordinary food mixtures. It should be remembered in this connection, however, that some gums are more readily detected than others when present in equivalent amounts. Tragacanth, for example, is much easier to detect in small quantities than either arabic or agar. The relation of the amounts of other precipitable matter, especially protein, is also of some importance since the gums tend to be carried down mechanically in the precipitation of protein, hence the ratio of protein to gum may be so great that the procedure will fail to detect the gum through mechanical loss.

TABLE II

	Approximate Volumes of Alcohol Necessary for Precipitation		Characteristic Appearance of Gum Precipitate	Characteristics of Gum Precipitate After Standing for Some Time in Air
	Vols. Alcohol	Vol. Gum Solution		
Agar.....	3-4	1	Finely divided white precipitate; settles very slowly	Usually remains soft and non-coherent
Arabic.....	2	1	White flocculent precipitate; settles quickly; neither sticky nor coherent	Becomes dry and powdery
Indian....	2-3	1	Stringy precipitate; becomes very coherent after settling	Becomes dark colored; tough coherent layer
Tragacanth	2	1	Coherent, jelly-like mass; floats in clots in upper part of solution	Flattens down, becoming a semi-transparent coherent layer
Dextrin...	3	1	White, fine precipitate; settles slowly; very sticky	Tends to become hard on long standing

#### IDENTIFICATION OF THE GUM

Certain of the precipitation tests for the gums, which have been used as the basis for the foregoing method of separation, serve also fairly well for the

identification of the gums. It suffices in general to note the approximate volume of alcohol required for the final precipitation and the nature and appearance of the alcohol precipitate. It is advantageous in this connection to pour off most of the alcohol after the precipitate has settled and allow the moist gum precipitate to stand exposed to the air for a short time, noting its behavior when drying. Table II presents in concise form the characteristic differences which are shown by the common gums.

#### CONFIRMATORY TESTS

While the characteristic differences described in the preceding table have been the chief reliance in identifying the gums, a number of the tests described in the literature have been examined to determine their value as confirmatory tests. Most of these, as previously stated, depend on impurities present in the crude gum and hence, as would be expected, proved of little value when applied to the separated gum precipitate. Several procedures, however, were found even under these conditions to be distinctly helpful. Chief of these was the presence of characteristic diatoms in the agar. The test is a well-known one and consists in identifying under the microscope the peculiar diatoms which are associated with agar, chiefly *Arachnoidiscus Ehrenbergii* and various species of *Cocconeis*, after destroying the organic matter by digestion with acid. The characteristic appearance of these diatoms will be found figured in most standard texts on food analysis.

The procedure consisted in destroying the organic matter of the sample by heating with nitric and sulfuric acids until the solution became colorless, diluting the concentrated acid solution with water, centrifuging to collect the siliceous residue, and examining this under the microscope. The test may be applied to the original material, but much time will be saved by using the tannin precipitate obtained in A<sub>1</sub> of Table I. Since this precipitate is separated by the centrifuge it will obviously contain all the relatively heavy particles, including the diatoms, and its use will eliminate the interference due to soluble carbohydrates, as cane sugar, commercial glucose, etc., which use up time and acid in the digestion. This procedure was tried repeatedly on many samples of agar including the purest bacteriological material, and on food mixtures containing agar, and the presence of the characteristic diatoms noted in every case. Although this test actually depends on the presence of "impurities" in the gum, it was found that the diatoms are so widely distributed in commercial samples and remain so consistently in the tannin precipitate that the test was most useful.

The volatile acidity of Indian gum was also found of value as a confirmatory test. It has been noted by several authors that the species of gum coming under the classification of Indian gum have the characteristic property of developing an acetic odor when exposed to the air, and Emery<sup>1</sup> has made this characteristic the basis of a method for the detection of

Indian gum as an adulterant of gum tragacanth. The method consists, in brief, of accelerating the hydrolysis of the gum by heating with acid, distilling, and titrating the acetic acid produced. Emery gives the following typical figures, expressed as cc. of *N*/10 acid per gram of gum:

Tragacanth.....	3.2- 4.2
Indian gum.....	25.4-28.3

Emery's method was applied to the dried gum precipitates obtained in the systematic procedure of Table I and the following results were obtained:

TABLE III		Volatile Acidity
Gum		
Indian gum, No. 1.....		20.3
Indian gum, No. 3.....		16.4
Indian gum, No. 3 (dried for 4 mos. after precipitating).....		16.1
Indian gum, No. 4.....		9.5
Indian gum, No. 5.....		14.8
Tragacanth, No. 1.....		3.5
Tragacanth, No. 2.....		2.3
Tragacanth, No. 3.....		2.9
Gum arabic.....		0.25
Dextrin.....		1.0

These figures show that the differences found by Emery hold for the precipitated gums, although to a less marked degree. The method, although rather long and tedious, has distinct value as a confirmatory test on gum precipitates when Indian gum is suspected and the characteristics described in Table II are not conclusive.

#### LIMITATIONS OF THE METHOD

The delicacy of the method under favorable conditions has been pointed out in a previous paragraph. There are, however, certain limitations to its usefulness which should be definitely noted.

First, the successful use of the procedure for the identification of the gums depends primarily upon the ability to recognize the different visible characteristics of the gum precipitates. It is, therefore, highly desirable that before using the procedure for analytical purposes the analyst should know the various gum precipitates "by sight." This can be readily accomplished with the aid of a few prepared solutions of the gums concerned.

The identification of the gums where more than one is present in the food mixture is a more difficult matter. With some combinations of gums a partial separation can be accomplished by the fractional precipitation of the procedure, but with such a combination as dextrin and agar-agar, or Indian gum and tragacanth, this would be practically impossible, although there might be indications as to the presence of both gums.

A more serious matter is the possible presence of two substances which interfere distinctly with the separation and identification of the true gums. These are pectin and commercial glucose. The first of these is perhaps less important since, although it is somewhat similar to the gums and would to a certain extent be precipitated with them in the procedure, few of the commercial food products in which gums are used ordinarily would be likely to contain fruit pectins. The most likely combinations would be fruit pie fillings and jellies and jams made from apple stock, food products in which the presence of gums has been noted.

<sup>1</sup> THIS JOURNAL, 4 (1912), 374.

In the few cases where from the source of the sample the presence of pectin might be expected, its removal prior to the alcoholic separation of the gums may be aided by observing the following precautions:

(1) In adding the acetic acid to the ammoniacal solution (C of Table I), it should be added slowly, the mixture stirred, and allowed to stand for some time with occasional shaking. The removal of pectin at this point may be accelerated by adding a few drops of a tannin-iron solution before the ammonia and acetic acid treatment.

(2) Small amounts of alcohol, one-quarter to one-half of a volume, are added with stirring to the ammoniacal solution.

(3) When the amount of iron present is slight, judging from the color of the solution, as well as the precipitates, a few drops of a ferric chloride solution are added to the aqueous solution of the acetone precipitate (B<sub>1</sub> of Table I).

Of greater practical importance is the presence of commercial glucose in products which also contain gum, a combination which is common in commercial products like marshmallow creams. The interfering factor here is of course dextrin and this is precipitated with the final gum precipitate by alcohol, although the scheme of fractional precipitation outlined in Table I should give some indication of its presence.

Numerous tests have shown that in the case of Indian gum and gum tragacanth commercial glucose interferes but little with their detection and identification, even when the ratio between the quantities of glucose and gum is as high as 40 to 1 for Indian gum and 120 to 1 for gum tragacanth. In the case of gum arabic the interference of commercial glucose is distinctly noticeable when the ratio of glucose to gum is 20 to 1, a portion of the dextrin precipitating with 2 volumes of alcohol along with the gum arabic, and by its sticky character masking the flocculent, non-coherent characteristic of the gum arabic. With agar, although no experiments were carried out, the interference would be still greater. Since the amount of commercial glucose present in a marshmallow paste, for example, is likely to exceed the ratio given for gum arabic, in such cases an additional step in the procedure may be needed.

This additional step is based on the fact that dextrin is more readily hydrolyzed by boiling with dilute acid than are the gums. It is carried out on the precipitate obtained with two volumes of alcohol, which will contain the greater part of the gum arabic and a portion of the dextrin. 0.5 g. of the dried gum precipitate is heated for 5 min. with 50 cc. of water and 2.5 cc. of concentrated hydrochloric acid (sp. gr. 1.20). The hydrolysis is conveniently carried out in a large test tube immersed in boiling water.

Experiments on known mixtures have shown that in this way one part of gum arabic may be detected in a mixture with 4 parts of dextrin, a delicacy which allows the detection of the gum in the presence of a considerable proportion of commercial glucose. Further, it must be remembered that the precipitate ob-

tained with 2 volumes of alcohol would not contain the whole of the dextrin involved, for the larger part of this, as has been shown, would come down only with the third volume of alcohol. The precipitate to be hydrolyzed would, therefore, contain the gum with a small amount only of the dextrin present in the original mixture. Without question, then, the procedure is quite delicate and is capable of detecting relatively small quantities of gum arabic in the presence of commercial glucose.

#### SUMMARY

A method is described for the separation of the more common gums from food products based upon elimination of proteins by acetic acid and tannin and precipitation of the gums by acetone and finally alcohol.

The separated gums are identified mainly by their fractional precipitation with alcohol and the characteristic appearance of the precipitated pure gum.

The necessary modification of the method in the presence of such interfering substances as milk solids, pectin, and commercial glucose is described.

The method described is capable of detecting with ordinary commercial products 0.1 g. of gum in 100 g. of a complex food mixture.

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#### UNIFORM NITROGEN DETERMINATION IN COTTON-SEED MEAL

By J. S. MCHARGUE

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Chemists often have trouble in obtaining uniform results in duplicating nitrogen determinations on cottonseed meal. Since cottonseed meal is so extensively used as a source of protein in feeds, it is a matter of considerable importance whether or not all serious errors have been eliminated in a nitrogen determination on this material.

The object of this paper is to call attention to procedures common among chemists which are often the cause of considerable error in the determination of nitrogen in cottonseed meal.

During the past year the writer has been called upon to check a number of cottonseed meal samples in which the amount of nitrogen was in question. On a few of these samples duplicate determinations of nitrogen showed variations ranging from as much as 0.50 to 1.50 per cent of protein, while other samples of cottonseed meal gave almost identical results upon duplication. The samples upon which varying results were obtained naturally suggested further investigation in regard to the cause of the variations.

Self<sup>1</sup> has shown that nitrogen can be lost by volatilization during the digestion if a large excess of potassium sulfate has been added. He attributes this loss to the formation of  $\text{KHSO}_4$  when an excess of  $\text{H}_2\text{SO}_4$  has been boiled off.

The following experiments were made to determine whether or not nitrogen was lost in a cottonseed meal

<sup>1</sup> *Pharm. J.*, **88**, 384-5; *Chem. Abs. (Amer.)*, **6** (1912), 2048.

digestion when there was considerable variation in the amount of  $\text{Na}_2\text{SO}_4$  added.

Six 0.7005 g. portions of cottonseed meal were weighed and transferred into 800 cc. Kjeldahl digestion flasks. 25 cc. of  $\text{H}_2\text{SO}_4$  and the amount of the reagents indicated in the table were added to each flask and the digestion carried on at a brisk boil for 2 hrs. After cooling, the contents of the flasks were diluted, made alkaline, the mercury or copper precipitated with sodium polysulfide<sup>1</sup> and the ammonia distilled in the usual way. The results were as follows:

TABLE I

	Reagents Added		Results	
	Anhyd. $\text{Na}_2\text{SO}_4$ G.	Mercury G.	N Obtained Per cent	Protein Per cent
A.....	2	0.7	6.32	39.50
B.....	4	0.7	6.30	39.37
C.....	12	0.7	6.29	39.31
	CuSO <sub>4</sub> .5H <sub>2</sub> O			
A.....	2	0.5	6.16	38.50
B.....	4	0.5	6.32	39.50
C.....	12	0.5	6.38	39.87

From the foregoing results it is to be observed that there is a gradual diminution in the nitrogen determinations receiving mercury as the amount of  $\text{Na}_2\text{SO}_4$  is increased, whereas in those digested with copper sulfate there is a gradual increase in the nitrogen as the  $\text{Na}_2\text{SO}_4$  increases. The results obtained with mercury agree with the findings of Self.

Robertson<sup>2</sup> has also noted slightly higher results where copper sulfate replaced mercury in the digestion. The loss in nitrogen in the presence of mercury can be assumed to be due either to volatilization of a nitrogen compound during the digestion or to the formation of a mercurammonium compound which is not decomposed during distillation. While the losses of nitrogen in the experiments digested with mercury are noteworthy they are not sufficiently great to account for the variations often experienced in nitrogen determinations on cottonseed meal. Hence it was necessary to continue the search for other sources of error.

Further experiments were made on a sample of cottonseed meal which had baffled the wits of another chemist in regard to its nitrogen content. Six nitrogen determinations were made on this sample for the purpose of showing the variations between determinations and to test the efficiency of  $\text{Na}_2\text{SO}_4$  versus  $\text{K}_2\text{SO}_4$  in cottonseed meal digestions. In three of the determinations 7 g. of  $\text{K}_2\text{SO}_4$  were used in each digestion and 6 g. of  $\text{Na}_2\text{SO}_4$  in each of the remaining determinations. Mercury was used as the catalyst and the amount of acid and time of digestion were the same as in previous experiments. The results obtained were as follows:

TABLE II

	Potassium Sulfate Per cent Nitrogen	Sodium Sulfate Per cent Nitrogen
A.....	6.90	6.71
B.....	6.92	6.75
C.....	6.74	6.64
Average.....	6.85	6.70
	Per cent Protein	Per cent Protein
Average.....	42.81	41.87
Maximum.....	43.25	42.19
Minimum.....	42.13	41.50
Difference.....	1.12	0.69

<sup>1</sup> Sodium polysulfide can now be obtained from Messrs. Charles Cooper & Co., 194 Worth Street, New York.

<sup>2</sup> Brackett and Haskins, "Nitrogen," J. A. O. A. C., 1, No. 3, 395.

In the averages of the above experiments there is a difference of 0.15 per cent nitrogen or 0.94 per cent protein in favor of the  $\text{K}_2\text{SO}_4$  digestions. The difference between the maximum and minimum results in the  $\text{K}_2\text{SO}_4$  digestions is 0.18 per cent nitrogen or 1.12 per cent protein. In the  $\text{Na}_2\text{SO}_4$  digestions these differences are 0.11 per cent nitrogen or 0.69 per cent protein, a little more than one-half the difference in the  $\text{K}_2\text{SO}_4$  digestions. It is also to be noted that the results for "C" in each digestion are decidedly off in comparison with the results for "A" and "B."

In order to locate the cause of the "off" results in the foregoing determinations the following series of experiments was carried out:

One hundred grams of cottonseed meal were transferred onto a series of sieves of 20, 40, and 60 mesh, and after placing a few coins on each sieve the whole was shaken for about 25 min. That portion of the sample remaining on the 20 mesh sieve was 13.58 per cent, on the 40 mesh 30.82 per cent, on the 60 mesh 16.71 per cent, and the portion that passed through the 60 mesh was 38.89 per cent. The parts into which the sample was divided by sieving were bottled separately and six nitrogen determinations made on each sample. The amount of the reagents and the time of digestion were the same as in the previous experiment.

Table III shows the results obtained for nitrogen on each of the samples with  $\text{K}_2\text{SO}_4$  and  $\text{Na}_2\text{SO}_4$ .

TABLE III

	Greater than 20 mesh		Between 20 and 40 mesh		Between 40 mesh and 60 mesh		Less than 60 mesh	
	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N
With $\text{K}_2\text{SO}_4$ .....	A 4.84	4.76	7.01	7.69				
	B 4.72	4.66	7.00	7.65				
	C 4.65	4.66	6.85	7.64				
Average.....	4.74	4.69	6.95	7.66				
Equivalent to protein.....	29.62	29.31	43.44	47.88				
Range of N.....	0.19	0.10	0.16	0.05				
Equivalent to protein.....	1.19	0.62	1.00	0.31				
	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N	Per cent N
With $\text{Na}_2\text{SO}_4$ .....	A 4.72	4.72	6.98	7.65				
	B 4.54	4.67	6.96	7.65				
	C 4.52	4.63	6.93	7.62				
Average.....	4.59	4.67	6.96	7.64				
Equivalent to protein.....	28.69	29.19	43.50	47.75				
Range of N.....	0.20	0.09	0.05	0.03				
Equivalent to protein.....	1.25	0.56	0.31	0.18				
N by $\text{K}_2\text{SO}_4 > \text{N}$ by $\text{Na}_2\text{SO}_4$	0.15	0.02	-0.01	0.02				

From the results presented in the foregoing table on the nitrogen determinations of the different sized particles that were contained in the sample of cottonseed meal, it is to be observed that there is a gradual diminution in the variations of the different nitrogen determinations as we approach the fine ground material. On the 20 mesh sample there is a difference of 0.20 per cent nitrogen, which is equivalent to 1.25 per cent of protein, in the averages of the two different digestions, while on the 40 mesh sample there is a difference in the averages of the two different digestions of 0.02 per cent nitrogen, which is equivalent to a difference of 0.12 per cent of protein. There is also a striking diminution in the difference between the averages of the two methods of digestion on the 60 mesh sample as compared with the difference on the 40 mesh sample. It is also to be noted that there is less variation in the results obtained in the sodium sulfate digestions than in the potassium sulfate digestions.

From the results in Table III it is to be concluded that another and perhaps the greater cause of irregularities in nitrogen determinations in cottonseed meal is due to the lack of regrinding the larger particles to a sufficient degree of fineness to obtain a homogeneous mixture in weighing out portions for digestion.

In order to confirm this conclusion more definitely, about 300 g. of cottonseed meal were sifted with coins on a 40 mesh sieve for about 25 min. The greater part of the coarse particles that remained on the sieve were hulls and were ground to pass through the 40 mesh sieve. The two portions were again thoroughly mixed and quartered down to a 100 g. sample. Upon this sample the following experiments were made for the purpose of testing, first, the uniformity of the results obtained on the material treated in the above manner, and second, the time necessary to complete a digestion on cottonseed meal.

Four series of experiments of twelve nitrogen determinations each were made. The digestions in these series of experiments were carried on for 1, 2, 3 and 4 hrs., respectively. In each series all the digestions were heated gently at first and then to a brisk boil, and after about 45 min. all the solutions were clear. Upon distillation the following results were obtained:

TABLE IV—SHOWING THE EFFECT OF DIFFERENT PERIODS OF DIGESTION WITH MERCURY, COPPER SULFATE, POTASSIUM SULFATE, AND SODIUM SULFATE ON A SAMPLE OF COTTONSEED MEAL

	1 hour Per cent N	2 hours Per cent N	3 hours Per cent N	4 hours Per cent N
7 g. K <sub>2</sub> SO <sub>4</sub> + 0.7 g. Hg + 25 cc. H <sub>2</sub> SO <sub>4</sub>				
A.....	6.10	6.26	6.22	6.12
B.....	6.16	6.23	6.24	6.18
C.....	6.15	6.26	6.27	6.21
Average.....	6.14	6.25	6.24	6.17
Protein.....	38.38	39.06	39.0	38.56
6 g. Na <sub>2</sub> SO <sub>4</sub> + 0.7 g. Hg + 25 cc. H <sub>2</sub> SO <sub>4</sub>				
A.....	6.13	6.26	6.20	6.24
B.....	6.14	6.28	6.22	6.22
C.....	6.18	6.25	6.26	6.12
Average.....	6.15	6.27	6.23	6.19
Protein.....	38.44	39.19	38.94	38.69
7 g. K <sub>2</sub> SO <sub>4</sub> + 0.5 g. CuSO <sub>4</sub> .5H <sub>2</sub> O + 25 cc. H <sub>2</sub> SO <sub>4</sub>				
A.....	6.16	6.24	6.33	6.36
B.....	6.18	6.22	6.25	6.34
C.....	6.19	6.20	6.32	6.26
Average.....	6.18	6.22	6.30	6.32
Protein.....	38.63	38.88	39.38	39.50
6 g. Na <sub>2</sub> SO <sub>4</sub> + 0.5 g. CuSO <sub>4</sub> .5H <sub>2</sub> O + 25 cc. H <sub>2</sub> SO <sub>4</sub>				
A.....	6.18	6.21	6.26	6.36
B.....	6.17	6.29	6.34	6.34
C.....	6.16	6.24	6.28	6.33
Average.....	6.17	6.25	6.29	6.34
Protein.....	38.56	39.06	39.31	39.63

TABLE V—SHOWING THE AVERAGE PERCENTAGE OF PROTEIN FOR EACH DIGESTION PERIOD WITH MERCURY AND WITH COPPER SULFATE

With mercury...	38.41	39.13	38.97	38.62
With copper....	38.60	38.97	39.35	39.57

In comparing the results obtained under the different conditions of digestion, as shown in the foregoing table, the following points are of most interest.

In all of the one-hour digestions the results for nitrogen are low; therefore, a longer period than 1 hr. is necessary for a complete digestion of cottonseed meal. In the experiments in which mercury was used as the catalyst, the 2-hr. digestions gave the maximum results. Longer periods of digestion apparently caused a slight loss of nitrogen, negligible after 3 hrs., but appreciable at the end of 4 hrs.' digestion.

In the experiments in which copper sulfate and 6 g. of sodium sulfate were used, the maximum results

were only obtained after 4 hrs. of digestion. However, the addition of larger amounts of sodium sulfate will reduce the time of digestion, 12 g. being sufficient to bring about complete digestion in 2 hrs.

It was observed that in determinations in which copper was used as the catalyst, the precipitation of the copper as sulfide greatly facilitates the boiling and reduces the time of distillation as compared with determinations in which the copper was not precipitated previously to starting the distillation.

Approximately 300 cc. of liquid should remain in the flask after distillation, since alkali will distil over if the solutions are concentrated to a much less volume. A large excess of alkali should always be avoided. Blanks were run on all of the reagents and deducted from each determination.

#### CONCLUSIONS

From the data presented in this paper the following conclusions may be drawn in regard to obtaining uniform nitrogen determinations on cottonseed meal.

1—The chief source of irregularity in nitrogen determinations on cottonseed meal may be due to a lack of grinding the sample to a sufficient degree of fineness (40 mesh) to obtain a homogeneous mixture of hulls and meal for weighing out a charge.

2—When mercury is used as the catalyst a digestion period of more than 2 hrs. of brisk boiling in an excess of sulfuric acid apparently causes a loss of nitrogen.

3—When copper sulfate is used as the catalyst the digestion period will depend upon the amount of sodium sulfate added, 12 g. being sufficient to bring about a complete digestion in 2 hrs.

4—The precipitation of the copper as sulfide facilitates the boiling and shortens the time of distillation.

5—Sodium sulfate is just as efficient as potassium sulfate in cottonseed meal digestions.

6—The writer suggests the use of the following charge: 0.7005 g. of cottonseed meal, 0.3 g. CuSO<sub>4</sub> or 0.5 g. CuSO<sub>4</sub>.5H<sub>2</sub>O, 12 g. Na<sub>2</sub>SO<sub>4</sub> + 25 cc. H<sub>2</sub>SO<sub>4</sub>, and brisk boiling for 2 hrs.

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#### THE DETECTION AND DETERMINATION OF COUMARIN IN FACTITIOUS VANILLA EXTRACTS

By H. J. WICHMANN

Received March 18, 1918

A quick qualitative test for coumarin is very desirable for separating those extracts that contain it from those that do not. The number of extracts that contain coumarin without a declaration of its presence on the label is so small that the extra work of making the double extraction, as in the modified Hess-Prescott method, seems entirely superfluous and unnecessary. The method of testing for coumarin described in U. S. Department of Agriculture, Bureau of Chemistry, *Circular 95* has, in the hands of the writer, been very efficient for this purpose. The method there outlined requires the distilling of the extract to a low volume, evaporating the distillate containing vanillin and coumarin, if the latter is present, with 1 cc. of

50 per cent potassium hydroxide, followed by fusion. The salicylic and protocatechuic acids formed from the coumarin and vanillin by the fusion are separated by distilling the acidified solutions of these acids or separating them with benzene, the former being volatile with steam and soluble in benzene, properties not possessed by the latter. The writer prefers the benzene method of separation because of its simplicity. The method, however, requires some preliminary experience before very small quantities of coumarin can with certainty be detected. One precaution necessary is not to carry on the distillation so far that yellow decomposition products result, but to stop just short of that point; otherwise it will be difficult to make a clean-cut fusion.

T. R. Dean<sup>1</sup> modified the original method. It was recognized in the original article that certain volatile salicylate-forming compounds like saccharin should not be present. If they are present a qualitative test will indicate coumarin, but a subsequent quantitative determination would give no weighable quantity, because both salicylic acid and saccharin would be removed with the vanillin thus leaving no coumarin where it would be expected. This disagreement between qualitative and quantitative results would indicate salicylate-forming compounds, the identity of which would have to be determined by further work. Dean's modification simplifies the original method in that the fusion is not complicated by the presence of vanillin and other organic matter. Such matter may cause a destruction of the salicylates if sufficient alkali has not been added.

When a vanilla extract is extracted with ether a certain amount of coloring matter always accompanies the vanillin. In the case of an alcoholic extract this is always more than if it had been dealcoholized. However, the amount of coloring matter is greatly reduced in either case if the solution is alkaline. Dean recommends using a dealcoholized extract, preferably the residue from an alcohol distillation. Experiments by the writer have shown, however, that it is not necessary to dealcoholize before testing for coumarin. Certain further simplifications have been introduced and attention is called to the peculiar color changes that coumarin undergoes when heated with concentrated potassium hydroxide. These are interesting and characteristic and furnish the first indication of the presence of coumarin. The method is as follows:

Ten cc. of extract are made alkaline with 10 per cent sodium hydroxide, then diluted with 15 cc. of water to reduce the alcoholic strength and extracted with 20 cc. of ether in a separatory funnel. The ether solution will be slightly colored when the brown lower layer has been drawn off. A few cc. of strong alcoholic potassium hydroxide solution are added, the mixture shaken and then washed with 10 cc. of water. The ether layer will then be found to be white. This procedure removes all organic acids, vanillin, coloring matter or saccharin that might be present. One cc. of 50 per cent potassium hydroxide solution is placed in a test tube and the ether solution of coumarin poured over it. After thoroughly shaking, the ether is hastily evaporated. The tube is then placed over a free flame, the water evaporated and the potassium hydroxide fused. If coumarin is present in any amount a change of color will be

noticed as the evaporation of the water proceeds and fusion begins. Even very small quantities of coumarin in strong, hot potassium hydroxide solution will show a greenish yellow color that suddenly disappears as the heating is continued. The disappearance of the color shows that the coumarin has been converted into the salicylate and heating should be discontinued. The melt is taken up with a few cc. of water, the solution acidified with sulfuric acid and extracted in a small separatory funnel with 5 to 10 cc. of benzene. Benzene is preferred to any other solvent because of its low density, low solvent power for mineral acids, and because it will not dissolve any protocatechuic acid formed from vanillin that might possibly have been carried over with the ether. The acid solution is removed from the separatory funnel and the benzene washed with a few cc. of water. After washing, the benzene is filtered into a test tube and tested for salicylic acid with a cc. or two of water containing a few drops of ferric chloride solution. If no color develops on shaking, one or two drops of *N*/10 sodium hydroxide should be added to neutralize any trace of mineral acid that may be present and which prevents the development of the purple color.

This test can be conducted easily in 15 min., takes only 10 cc. of extract, and does not require dealcoholization or any complicated apparatus. The only evaporation necessary, that of the ether, can be done on a steam bath without appreciable loss of coumarin. The change of color on fusion indicates its own endpoint and gives, together with the purple salicylate color, a double test for coumarin. Coumarin is changed to salts of coumaric acid by hot concentrated potassium hydroxide. The development of the yellow color shows this phase and the sudden disappearance of the color indicates the conversion into a colorless salicylate. The delicacy is unquestionably great since the writer has obtained a very decided purple color with 10 cc. of extract containing only 0.005 per cent of coumarin. This modified method is considered simpler than the original and can be conducted in a shorter time.

This qualitative test has not been made quantitative. The results obtained so far by fusing pure coumarin with potassium hydroxide in test tubes, extracting the salicylic acid and matching the color developed against standard solutions, have been from 2 to 15 per cent too low. This is probably due to a slight volatilization of coumarin before the alkali can attack and hold it. However, results by a gravimetric method, to be described later, have been so satisfactory as to accuracy, speed and ability to determine small quantities that a colorimetric method is hardly necessary.

The Hess-Prescott method directs that the extract be dealcoholized before the vanillin and coumarin are extracted. The vanillin is then removed from the ether by a number of extractions with 2 per cent ammonia, the coumarin remaining in the ether. Unpublished results of the writer indicate that it is possible to titrate vanillin in non-aqueous solutions with alcoholic sodium ethylate or potash. The sodium-vanillin salt can then be removed from the solvent by washing with water. Coumarin is not affected by the vanillin titration. Therefore, it remains in the solvent and, after the vanillin salt has been washed out, can be determined by evaporation,

<sup>1</sup> THIS JOURNAL, 7 (1915), 519.

drying and weighing. Excess alcoholic alkali can be used without affecting the coumarin results if it is not desired to determine the vanillin volumetrically.

It has been found that benzene, chloroform and ether are the best solvents for extracting vanillin and coumarin from either aqueous or alcoholic solutions. Forty cc. of chloroform, 100 cc. of benzene and 60 cc. of ether in four or five extractions will extract quantitatively large amounts of vanillin from water and alcohol solutions up to 25 per cent strength. Since a half aliquot is usually taken for analysis this corresponds to 50 per cent alcohol in the extract which is probably more than the average commercial extract contains. Benzene, chloroform and ether all extract acids and coloring matter from vanillin extracts treated with lead acetate, ether extracting by far the most. Hence ether cannot be used if the vanillin is to be determined by titration, because the acetic acid it extracts at the same time cannot be removed by washing. Benzene or chloroform can be used for such purposes. There is, therefore, a possibility of combining a volumetric vanillin with a gravimetric coumarin determination in such cases where the qualitative test shows the presence of the latter. The details of a volumetric vanillin determination will be published later. However, if results for coumarin only are desired, it is recommended that ether be used as the extracting solvent as it has decided advantages in such cases. It evaporates more quickly and the extractions and washings are speedier because ether and water can be separated faster than water and chloroform or water and benzene. While ether extracts considerably more coloring matter and acid than benzene or chloroform from vanilla solutions, these impurities are neutralized by the alkali and removed with the vanillin in the subsequent washing with water. A water-white solution of coumarin in ether remains. The ether can be evaporated and the coumarin dried and weighed.<sup>1</sup> No dealcoholization is necessary and the results can be obtained speedily. It is not recommended in quantitative work to extract the coumarin from alkaline vanilla solutions as in the qualitative method, because emulsions are liable to form, and coloring matter must be removed anyhow. In many cases where speed is desirable it would be advantageous to determine coumarin in one portion and the vanillin by colorimetric or other methods in another.

To show the results that can be obtained by the above method the following data are submitted:

To 50 cc. of vanilla extract various quantities of coumarin were added, the vanilla solutions then treated with lead acetate without dealcoholization, made up to 100 cc., filtered, and the excess lead precipitated with dry potassium oxalate. The removal of the lead facilitates extraction because it reduces emulsion formation. Fifty cc. of the solutions thus prepared were extracted with ether, benzene or chloroform, as indicated in the table, a few drops of phenolphthalein solution and excess alcoholic alkali added and the vanillin salt removed by washing with several 10 cc. portions of water. The disappearance of the red phenolphthalein color in the wash water is an indication of sufficient washing. The washed solutions containing the cou-

marin were evaporated and the coumarin dried and weighed. The results are given in Table I.

TABLE I

Coumarin added Mg.	Extracting solvent	Quantity of extracting solvent		Coumarin recovered Mg.	Coumarin recovered Per cent
		Cc.	Number of extractions		
25.0	Benzene	100	5	24.5	98.0
24.5	Benzene	100	5	24.0	97.9
24.5	Chloroform	40	4	24.0	97.9
25.0	Chloroform	40	4	25.2	100.4
25.0	Ether	80	4	25.2	100.4
25.0	Ether	80	4	24.8	99.2
5.0	Ether	80	4	4.8	96.0
10.0	Ether	80	4	10.2	102.0
15.0	Ether	80	4	15.0	100.0
25.0	Ether	80	4	24.5	98.0
50.0	Ether	80	4	49.3	98.6

The results shown in the table indicate that an accurate quantitative determination of coumarin can be made by the method outlined above. Quicker methods for lead number and vanillin, to correspond with the coumarin method, are in preparation.

## SUMMARY

A simple and quick modification of the original method for the detection of coumarin in factitious vanilla extracts has been developed. While quantitative results based on the qualitative method are too low, another method has been given which is shown to be both quick and accurate for the determination of coumarin.

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## THE DETERMINATION OF ESSENTIAL OILS IN NON-ALCOHOLIC FLAVORING EXTRACTS

By FRANK M. BOYLES

Received March 28, 1918

There has been appearing for some years on the American market, in increasing numbers, a variety of so-called non-alcoholic flavoring extracts which consist essentially of an emulsion of the respective essential oils in mucilage of acacia, tragacanth, karaya, or other gums. Glycerin is quite often present.

In making a survey of these products, the writer tried first the method suggested by Redfern<sup>1</sup> which was found to be quite unsatisfactory; first, because the procedure of precipitating the gum from 25 cc. of the sample with 25 cc. of 95 per cent alcohol and filtering through a Gooch into a 100 cc. flask and making up to the mark was too tedious and time-consuming; and second, because the writer did not in this case, and never has been able to obtain concordant results by the method of Howard<sup>2</sup> to which Redfern refers. Indeed this method is even less adaptable to the non-alcoholic extracts than to the ordinary alcoholic extracts, for the reason that the former contain, in a number of cases, as much as four times more oil than the strictly standard alcoholic extracts and there is always danger, if not certainty, of losing oil through volatilization when from 10 to 20 per cent is present.

Taking advantage of the fact that many gums are precipitated by lead subacetate, the following procedure was tried: 5 cc. of the emulsion were diluted with 20 cc. water and transferred to a Babcock milk

<sup>1</sup> THIS JOURNAL, 8 (1916), 421.

<sup>2</sup> J. Am. Chem. Soc., 30 (1908), 608.

bottle and the gum precipitated by lead subacetate and centrifuged. The precipitated gum came to the top and the volume of oil could not be read. This was then modified by adding chloroform to absorb the oil after precipitation of the gum. On centrifuging this mixture the gum precipitate settled to the bottom of the bottle with the chloroform, and upon decanting the supernatant liquid, adding ether, and heating to expel the chloroform as recommended by Howard and then diluting with water and centrifuging, the gum precipitate came to the top with the oil and again interfered with the reading of the volume of the oil.

Finally the following method which consists simply of making an alcoholic extract from the emulsion and proceeding according to the official method<sup>1</sup> was found to give satisfactory and concordant results for the lemon and orange extracts.

#### METHOD

Measure 10 cc. of the emulsion into a graduated cylinder, transfer as much as possible to a 50 cc. flask, rinse the cylinder with 10 cc. portions of 95 per cent alcohol, and with the aid of a glass rod transfer all of the emulsion and precipitated gum to the flask, fill to the mark, shake thoroughly, let stand about 30 min. Filter through a folded filter and determine the oil in a 20 cc. portion of the filtrate by the official method.<sup>1</sup> The per cent of oil found in the filtrate is multiplied by 5 to give the per cent of oil in the original emulsion.

The gum is completely precipitated and it is more expeditious to throw down the gum in the volumetric flask and to make up to volume and use an aliquot of the filtrate than to precipitate the gum and attempt to wash the oil from it into the flask. It is possible to pipette the aliquot directly from the flask, but as the precipitated gum gathers at the shoulder of the flask and frequently stops the outlet of the pipette this procedure offers little advantage over filtering, as it is not necessary to avoid transferring the gum to the filter.

In the case of extracts containing less than 5 per cent oil it is necessary to use more sample than specified in the directions given.

Non-alcoholic extracts of lemon and orange containing 10 per cent of the respective oils were prepared according to the formula

Essential oil.....	20 cc.
Tragacanth.....	3 g.
Glycerin.....	40 cc.
Water, q. s.....	200 cc.

The results obtained on these extracts are given in Table I. Results obtained on commercial non-alcoholic extracts are also given.

TABLE I

	Prepared Extracts		Commercial Extracts Oil Found Per cent
	Strength Per cent	Oil Found Per cent	
Orange.....	10	10	7.0
Orange.....	10	10	17.0
Orange.....	10	9.7	15.0
Lemon.....	10	10	6.2
Lemon.....	10	10	14.0
Lemon.....	10	9.5	6.5
Lemon.....	10	10	7.5
Lemon.....	..	..	19.0
Lemon.....	..	..	17.0
Lemon.....	..	..	19.5
Lemon.....	..	..	20.0

Contrary to Redfern's statement it was found that

<sup>1</sup> "Report of Com. on Methods of Analysis," *J. A. O. A. C.*, p. 262.

quite accurate results could be obtained by ordinary steam distillation.

It is necessary first to run blank experiments on pure oils to determine just what percentage of recovery can be accomplished with the particular apparatus at hand.

Using a 200 cc. side-neck distilling flask with outlet tube midway of the neck and a cassia flask as receiver, the writer has consistently recovered 95 per cent of lemon and orange oils when proceeding as follows:

Measure 10 cc. of the extract into a graduated cylinder and transfer it by means of about 35 cc. water to a side-neck distilling flask and distil with steam into a 100 cc. cassia flask. In the case of lemon and orange oils 95 per cent of the oil is recovered so that the amount found must be multiplied by 100 and divided by 95.

Table II gives the results obtained by steam distillation on the 10 per cent extracts described above.

TABLE II

	Strength Per cent	Oil Found Per cent
Lemon.....	10	10.0
Lemon.....	10	9.5
Lemon.....	10	9.7
Orange.....	10	10.0
Orange.....	10	10.0
Orange.....	10	9.5

For cassia, cinnamon, and clove extracts, the following modification of the official method<sup>1</sup> is successful.

Dilute 10 cc. of the sample with 95 per cent alcohol to 50 cc. as in the case of lemon and orange. Filter. Place 10 cc. of the filtrate in a separatory funnel containing 50 cc. water, add 1 cc. HCl (1 : 1), and shake out 4 times with 25 cc. portions of ether. Wash the combined ether extracts twice with water and then shake for a few minutes with about 5 g. granular calcium chloride.

Place a small piece of cotton in the outlet of the separatory funnel and draw the ether into a tared beaker. Evaporate the ether on a boiling water bath, place in desiccator for 3 min. and weigh; divide the weight by the specific gravity of the oil to find the per cent of oil by volume.

Table III gives the results on cassia, cinnamon and clove extracts containing 10 per cent of the respective oils and prepared according to the formula given under lemon and orange. Results on commercial extracts are also given.

TABLE III

	Prepared Extracts		Commercial Extracts Found Per cent
	Strength Per cent	Found Per cent	
Cassia.....	10	9.83	13.5
Cassia.....	10	9.90	12.4
Cassia.....	10	9.94	..
Cinnamon.....	10	9.90	17.5
Cinnamon.....	10	9.96	..
Cinnamon.....	10	9.90	..
Clove.....	10	10.0	..
Clove.....	10	9.89	..
Clove.....	10	9.95	..

Almond, anise, and nutmeg extracts of the non-alcoholic type are converted into alcoholic extracts as in the case of lemon and orange and an aliquot portion analyzed by the official methods.<sup>2</sup>

#### PEPPERMINT EXTRACT

A standard extract was made up containing 10 per cent peppermint oil by the formula given under lemon and orange. 10 cc. of this were made up to

<sup>1</sup> "Report of Com. on Methods of Analysis," *J. A. O. A. C.*, p. 267.

<sup>2</sup> *Ibid.*, pp. 266 and 269.



50 cc. with 95 per cent alcohol, filtered, and an attempt made to assay the filtrate by the official method<sup>1</sup> with the following results. These results are calculated to the original extract.

1—Following the method as given, 2 per cent of oils found.

2—Heating was carried on for only 1 min. (shaking at 15 sec. intervals), 8 per cent oil found.

3—Heating was continued for only 45 sec., 8 per cent oil found.

4—Heating was continued for only 20 sec., 11 per cent oil found.

5—Heating was continued for 25 sec., 8 per cent oil found.

6—Heating was continued for 20 sec. and then the suction was continued for an additional 15 sec. without heat, 9 per cent oil found.

7—Heating was continued for 20 sec. and the suction for an additional 10 sec. without heat, 9 per cent oil found.

The writer's experience with this method has been most unsatisfactory, even moderate agreement in duplicate determinations has never been attained. The method leaves too much to chance. If the flask is not disconnected from the suction the instant the last of the solvent is drawn off, there is a loss of oil, and we have not been able to discover any means whereby we can be assured when this instant is at hand.

Steam distillation gives very good results for peppermint if one determines beforehand what per cent of oil can be recovered with the apparatus in blank experiments using known quantities of pure oil. The writer has found this recovery to be 90 per cent, so that the quantity of oil found is multiplied by 100 and divided by 90 to find the amount present in the extract.

Proceeding according to the directions given for lemon and orange extracts for steam distillation the following results were obtained on a 10 per cent non-alcoholic extract and on commercial extracts.

TABLE IV

	Prepared Extracts Strength Per cent	Extracts Found Per cent	Commercial Extracts Found Per cent
Peppermint.....	10	10	11.0
Peppermint.....	10	9.8	12.8
Peppermint.....	10	10	7.2

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## A CONTRIBUTION TO THE COMPOSITION OF LIME-SULFUR SOLUTIONS<sup>2</sup>

By O. B. WINTER

Received April 19, 1918

The principal constituents of lime-sulfur solutions are calcium polysulfides and calcium thiosulfate. Small amounts of calcium sulfate and possibly of calcium sulfite are also present, and the claim has been made that the solutions may contain other compounds such as hydrogen sulfide, calcium hydrosul-

<sup>1</sup> "Report of Com. on Methods of Analysis," *J. A. O. A. C.*, p. 268.

<sup>2</sup> This work was done in the chemical laboratory of the Michigan Agricultural College Experiment Station and the results are published with the permission of the Director.

fide, calcium hydroxyhydrosulfide, different calcium oxysulfides, free lime, and free sulfur. How much of each of these compounds is liable to be present? It is the purpose of this paper to discuss some of the work done in this laboratory which pertains to the above question.

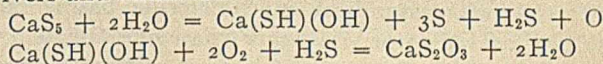
HYDROGEN SULFIDE, H<sub>2</sub>S. CALCIUM HYDROSULFIDE,  
Ca(SH)<sub>2</sub>. CALCIUM HYDROXYHYDROSULFIDE,  
CaSHOH

In reviewing the literature on the composition of lime-sulfur spray, it is interesting to note the discussions on the theoretical composition of the solution, together with the absence (except in a few articles) of even an attempt to prove the existence of certain of the compounds discussed. Divers and Shimidzu<sup>1</sup> give detailed methods for preparing some of the above-mentioned compounds and state some of their properties, but their work has nothing whatever to do with an ordinary lime-sulfur solution. Roark<sup>2</sup> discusses the possible existence of these compounds in a lime-sulfur solution, but does not give experimental proof of their presence. Tartar and Bradley<sup>3</sup> conclude that they are not present in appreciable amounts. Thompson and Whittier<sup>4</sup> claim the presence of hydrosulfide sulfur and give experimental data which they believe justifies their claim. Green<sup>5</sup> says, "We have, however, never been able to detect definitely the presence of hydrosulfide or free sulfuretted hydrogen in lime-sulfur solutions."

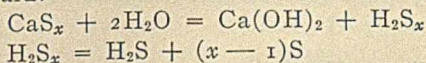
It may be possible to prepare a lime-sulfur solution which contains hydrogen sulfide, calcium hydrosulfide, or calcium hydroxyhydrosulfide, but it has yet to be shown conclusively that any of these compounds are present in appreciable quantities in a "straight"<sup>6</sup> lime-sulfur solution either as a result of the preparation of the solution, or of hydrolysis or other action taking place during storage under normal conditions.

It is also interesting to note that writers seem to have different opinions regarding the chemical actions which take place in lime-sulfur solutions, and for this reason literature is quite confusing in regard to the formation of any of the above-mentioned compounds. For example, the action of water on a polysulfide is represented as follows by different chemists:

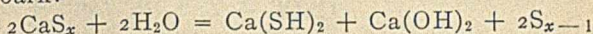
Divers and Shimidzu:<sup>7</sup>



Auld:<sup>8</sup>



Roark:<sup>7</sup>



<sup>1</sup> *J. Chem. Soc.*, 45 (1884), 270-91.

<sup>2</sup> *J. A. O. A. C.*, [1] 1 (1915), 81.

<sup>3</sup> *THIS JOURNAL*, 2 (1910), 271-7.

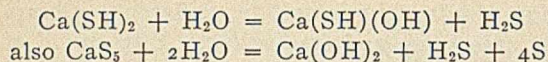
<sup>4</sup> Delaware Agricultural College Experiment Station, *Bull.* 105 (1914), 8.

<sup>5</sup> Union of S. Africa Dept. of Agr., 3rd and 4th Report of the Director of Vet. Research, 1915, p. 179.

<sup>6</sup> By a "straight" lime-sulfur solution is meant a solution prepared from ordinary commercial lime and sulfur to which no foreign substance has been added, and which has stood for several days.

<sup>7</sup> *Loc. cit.*

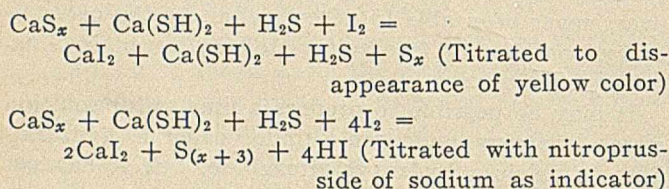
<sup>8</sup> *J. Chem. Soc.*, [1] 107 (1915), 482.



Averitt:<sup>1</sup> Up to a certain dilution no decomposition takes place when freshly boiled; distilled water is added to a lime-sulfur solution.

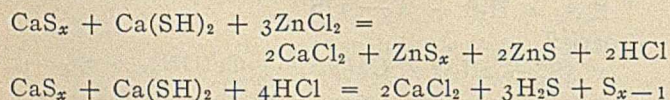
A similar disagreement might be given for many of the other reactions which are supposed to take place in boiling, storing, or diluting a lime-sulfur solution.

Experiments in this laboratory show that any one of the compounds mentioned above (containing the SH radical) may be detected by titrating an aliquot of the solution with standard iodine, determining the end-point by the disappearance of the yellow color, and by titrating an equal aliquot with the same solution determining the end-point with nitroprusside<sup>2</sup> of sodium. When the polysulfide has been entirely decomposed the solution loses the yellow color, while the blue color of the nitroprusside of sodium remains as long as there is any sulfur present either in the form of a sulfide or in the form of any compound containing the (SH) radical. If the two above titrations agree, none of the above-mentioned compounds are present. The following two equations represent the reactions which undoubtedly take place:



One might anticipate that the difference between the two titrations would be a measure of the amount of sulfur present other than sulfides, but we have not been able to prove this since the color end-point in the presence of a considerable quantity of a compound containing the (SH) radical does not appear sharp. However, the method is sufficiently accurate to show the presence or absence of any of these compounds.

It might be well to state here that other experiments have shown that when compounds containing the (SH) radical are present in a solution, an aliquot titrated with standard zinc chloride, using nickel sulfate as indicator, is higher than one titrated with standard hydrochloric acid, using methyl orange as indicator. This may be explained by the following equations:



If any of the compounds referred to above are formed in an ordinary lime-sulfur solution, they must

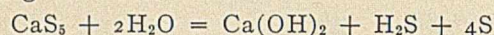
<sup>1</sup> J. A. O. A. C., [1] 1 (1915), 69.

<sup>2</sup> In titrating with nitroprusside of sodium as an indicator for sulfur compounds, it is essential that the indicator should not be added until the end-point is practically reached, since if the blue color is well developed it is almost impossible to change back to a colorless solution. If necessary, a few extra samples should be titrated, and those with the persisting color discarded.

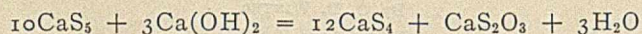
be unstable in the presence of other existing compounds, since the two iodine titrations mentioned in a preceding paragraph agree in every "straight" lime-sulfur solution that has been tested in this laboratory, whether prepared with an excess of lime or sulfur, whether freshly boiled or of long standing, and whether concentrated or dilute. Therefore we believe that none of the above-mentioned compounds exist in appreciable quantities in a "straight" lime-sulfur solution; and *vice versa*, if the spray contains any of these compounds, which can easily be detected, it is not a "straight" lime-sulfur solution.

#### FREE LIME

Roark<sup>1</sup> makes the statement that "While not more than a trace of calcium hydroxide [ $\text{Ca(OH)}_2$ ] may be present in a freshly prepared lime-sulfur solution, it is formed in appreciable amounts upon dilution, according to the reaction



and would be present, therefore, in lime-sulfur solutions which had stood for some time and become partially decomposed." He gives no proof of its actual existence. Thompson and Whittier<sup>2</sup> claim that, "Free calcium hydroxide may occur either from simple solution, where an excess of lime has been used, or it may result from hydrolysis of the polysulfide." They found "free lime present when the ratio of lime to sulfur exceeded a certain definite figure, increasing in amount as this ratio increased until the limit of the solubility of calcium hydroxide was reached." They say nothing about how long the solutions in which they found free lime had stood before making the analyses, and show no data to prove the statement that "free lime may result from the hydrolysis of the polysulfide." Tartar and Bradley<sup>3</sup> reported that no free lime or only a trace was found by them in lime-sulfur solutions. They say, "It appears that if there is hydroxide in the freshly prepared solution it either unites with some of the sulfur already in combination to form more polysulfide, or it unites directly with the polysulfide to form oxysulfides which crystallize out of the more concentrated solutions." Chapin<sup>4</sup> says, "If originally made with an excess of lime or if not boiled long enough, excess of lime is at first present in solution, but if such a preparation be allowed to stand quietly and cool off in the cooking vat, the indications are that the undissolved lime soon settles down, while the small amount of dissolved lime rapidly reacts with polysulfide according to equation



so that in this case also, unless the cooled solution is again stirred up with the sediment, a plus reaction figure can never be present in the end"—in other words, no free lime can be present. Green<sup>5</sup> claims

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

<sup>2</sup> Delaware Agricultural College Expt. Sta., *Bull.* 105 (1914), 11.

<sup>3</sup> *THIS JOURNAL*, 2 (1910) 273.

<sup>4</sup> U. S. Dept. of Agr., *Bull.* 451 (1916), 13.

<sup>5</sup> Union of S. Africa Dept. of Agr., 3rd and 4th Report of the Director of Vet. Research, 1915, p. 180.

that a trace of free lime is usually present but that the amount is very small.

In view of the above apparent differences of opinion, an attempt was made to prove either the presence or absence of free lime in an ordinary lime-sulfur solution.

Two different methods have been used by analysts for determining the amount of free lime in a lime-sulfur solution. The one (Method I)<sup>1</sup> is by titrating with 0.1 *N* hydrochloric acid, using methyl orange as indicator, and with 0.1 *N* ammoniacal zinc chloride solution using nickel sulfate as an outside indicator. In this method, the hydrochloric acid titration indicates the lime combined with sulfur as polysulfides plus the free lime, while the zinc chloride titration indicates only the former. The difference between the two titrations is a measure of the free lime. The other (Method II)<sup>2</sup> is by removing the free lime as calcium sulfate by means of magnesium sulfate solution (the magnesium being precipitated as magnesium hydroxide and filtered out). The solution is titrated with standard acid before and after the removal of the free lime. The difference between the two acid titrations is held by the authors to be a measure of the free lime. The results recorded in Table I were obtained by these two methods on 10 cc. of lime-sulfur concentrates diluted to 200 cc., of which 10 cc. were taken for analysis:

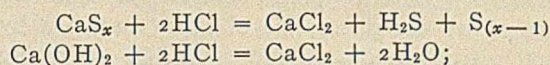
TABLE I

SAMPLE No.	Method I			Method II		CaO G.
	0.1 <i>N</i> HCl Cc.	0.1 <i>N</i> ZnCl <sub>2</sub> Cc.	CaO G.	Before adding 0.1 <i>N</i> HCl Cc.	After adding 0.1 <i>N</i> HCl Cc.	
A.....	8.60	8.65	0.0000	8.60	8.60	0.0000
B.....	8.40	8.40	0.0000	8.40	8.35	0.0001
C.....	40.35	40.25	0.0003	40.35	37.90	0.0069
D.....	12.65	12.65	0.0000	12.65	11.90	0.0021
E.....	18.38	18.40	0.0000	18.38	18.30	0.0002
F.....	17.98	17.96	0.0000	17.98	17.65	0.0009
G.....	17.43	17.48	0.0000	17.43	17.00	0.0012
H.....	12.80	12.82	0.0000	12.80	12.08	0.0020
I.....	23.43	23.00	0.0012	23.43	22.20	0.0034
J.....	23.26	23.20	0.0002	23.26	21.85	0.0039
K.....	10.80	8.70	0.0059	10.80	8.20	0.0092
L.....	8.40	8.40	0.0000	8.40	8.00	0.0011

In explanation of the above table, it may be well to state that Samples A and B were prepared in the laboratory using an excess of sulfur and were analyzed soon after their preparation. Samples C to H, inclusive, were commercial concentrates which had stood in the laboratory from one to ten years, of which some had previously been opened for analysis and a large amount of oxidation products had formed. Sample I was prepared in the laboratory using an excess of lime and analyzed immediately after its preparation. Sample J was the same as Sample I, except that the analysis was made two days after its preparation. Sample K was the same as Sample A, except that 0.0062 g. of calcium oxide had been added in the form of lime water in diluting to 200 cc. Sample L was the same as Sample A after it had been standing, exposed to the air, for several hours, and oxidation had begun. From a study of this table it will be noted: (1) That neither of the two methods show an appreciable amount of free lime in Samples A and B, which

were freshly prepared from materials containing an excess of sulfur. (2) That Method I shows no free lime or only a trace in the commercial concentrates which had stood from one to ten years, some of which had undergone considerable oxidation, whereas Method II shows its presence in all of these samples. (3) That both methods show free lime in Sample I, which had been recently prepared from materials containing an excess of lime, but that Method II shows the larger amount. (4) That Method I shows very little free lime in Sample J (Sample I after standing two days), whereas Method II shows more than when first prepared. (5) That when a given amount of free lime was added to a sample in which neither method indicated its presence, making Sample K, Method I shows nearly all that was added, while Method II shows slightly more than the amount added. (6) That while Method I shows no free lime in Sample L (Sample A after having been exposed to the air for several hours and oxidation had begun), Method II shows its presence.

From the above data it is evident that the two methods do not agree. Which is correct? When we consider the known facts that hydrochloric acid reacts with calcium polysulfides and calcium hydroxide as follows:



that ammoniacal zinc chloride reacts with calcium polysulfide according to the equation



and that in the presence of ammonium chloride, ammoniacal zinc chloride does not react with calcium hydroxide, it seems impossible that titrations made with hydrochloric acid and zinc chloride could agree in the presence of free lime. Further, when free lime was added to a lime-sulfur solution, it was practically all accounted for by the difference between the above-mentioned titrations immediately after the addition. Therefore the author contends that Method I is accurate for determining free lime in a lime-sulfur solution.

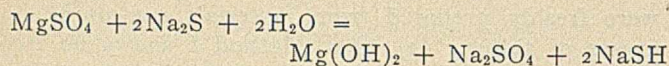
Since Method I, as given above, has been noted in literature and has been shown to be accurate, it seemed worth while to investigate Method II more thoroughly. Concerning this method Thompson and Whittier<sup>1</sup> state that when magnesium sulfate is added to a lime-sulfur solution containing free calcium hydroxide, magnesium hydroxide is precipitated quantitatively and calcium sulfate formed, thus neutralizing the solution and affording a method for determining the free lime. From other sources in literature, it is well known that magnesium hydroxide is precipitated when a solution of calcium hydroxide is treated with magnesium sulfate. However, this reaction is hardly considered sufficiently complete for quantitative determinations, and too, magnesium<sup>2</sup> salts react with sulfides, as follows:

<sup>1</sup> THIS JOURNAL, 2 (1910), 273.

<sup>2</sup> Delaware Agricultural College Expt. Sta., *Bull.* 105 (1914), 11.

<sup>1</sup> Delaware Agricultural College Expt. Sta., *Bull.* 105 (1914), 11.

<sup>2</sup> Prescott and Johnson, "Qual. Chem. Anal.," 6th Ed. (1904), p. 215.



Divers and Shimidzu, in an article on "Magnesium Hydrosulfide Solution and Its Use in Chemico-Legal Cases as a Source of Hydrogen Sulfide,"<sup>1</sup> state that, "The poly(-penta)sulfide that may be in solution is only very slightly decomposed even at boiling heat," thus indicating that the polysulfide of magnesium is a stable compound. Therefore, it appears possible that when a lime-sulfur solution is treated with magnesium sulfate, chemical action may take place even if there is no free lime present.

In order to throw more light on the above question, the precipitate which came down on adding magnesium sulfate was tested for the presence of sulfur, and was found to contain a small amount. This would indicate that the magnesium sulfate acted on some sulfur compound. However, the precipitate was amorphous and it seemed possible that some of the lime-sulfur solution might have been occluded. Therefore the test was not considered conclusive. The samples of concentrates which formed a precipitate with magnesium sulfate were diluted for analyses, with and without the addition of magnesium sulfate. Some of these were titrated for monosulfide and thiosulfate sulfur with standard iodine solution determining the end-point by disappearance of color and also by the use of nitroprusside of sodium. The following results were obtained:

TABLE II

SOLN. No.	I for Monosulfide (With MgSO <sub>4</sub> ) Colorless Solution		I for Monosulfide (Without MgSO <sub>4</sub> ) Colorless Solution		Ppt.	I for Thiosulfate After Titration	
	Titration Cc.	Nitroprusside Cc.	Titration Cc.	Nitroprusside Cc.		um 3 Cc.	um 5 Cc.
1...	8.14	8.32	8.50	8.52	Very little	7.05	7.05
2...	25.60	26.35	27.63	27.63	Much	7.05	Lost

These data show that when magnesium sulfate are added to lime-sulfur solutions, some compound is formed which causes a blue color with nitroprusside of sodium after the polysulfides have been decomposed and the solution has become colorless; and that the thiosulfate content remains the same. Since the solution is colorless but reacts with nitroprusside of sodium, it apparently contains a compound having the (SH) radical, *e. g.*, hydrogen sulfide, calcium hydrosulfide, calcium hydroxyhydrosulfide, or the corresponding salts of magnesium.

Others of the prepared solutions were titrated with hydrochloric acid and with zinc chloride, and the following results were obtained:

TABLE III

SOLN. No.	With MgSO <sub>4</sub>		Without MgSO <sub>4</sub>	
	0.1 N HCl Cc.	0.1 N ZnCl <sub>2</sub> Cc.	0.1 N HCl Cc.	0.1 N ZnCl <sub>2</sub> Cc.
D.....	11.90	12.12	12.65	12.65
E.....	18.30	18.40	18.38	18.40
F.....	17.65	17.70	17.98	17.96
G.....	17.00	17.20	17.43	17.48
H.....	12.08	12.40	12.80	12.82

These data show that the titration of the treated sample with standard hydrochloric acid is lower than that of the untreated. This indicates the loss of some calcium compound. It also shows that the titration of the treated sample with ammoniacal zinc

chloride is slightly higher than with hydrochloric acid. This also is significant. It indicates the formation of some new compound, and this compound must contain the (SH) radical.

The 10 cc. aliquots of a lime-sulfur solution which indicated no free lime by Method II were placed in each of two 100 cc. graduated flasks. One of these (*a*) was diluted to the mark with freshly boiled distilled water, and to the other (*b*) was added a large excess of 10 per cent magnesium sulfate solution and then also diluted to the mark with freshly boiled distilled water. Both solutions were allowed to stand about 12 hrs. The former remained perfectly clear, as was anticipated; the latter also remained clear for several minutes, then gradually became cloudy and by the end of the 12 hrs. a large number of crystals had formed, which, on examination, under a polarizing microscope, were readily identified as calcium sulfate (CaSO<sub>4</sub>.2H<sub>2</sub>O). These solutions were then analyzed and the following results were obtained:

TABLE IV

SAMPLE	Mono-S Gram	M(SH) <sub>2</sub> -S Gram	Thio-S Gram	Sulfide-S Gram	CaO Gram
a.....	0.0302	0.0000	0.0330	0.1505	0.0813
b.....	0.0279	0.0012	0.0333	0.1377	0.0154

In the above table the samples were titrated with iodine to the end-point determined by disappearance of color and also to the end-point determined by nitroprusside of sodium, and the difference between these two titrations was considered as sulfur in the form of compounds containing the (SH) radical and is indicated as M(SH)<sub>2</sub>-S. The results in this table show that there was a slight loss in the monosulfide equivalent and in the polysulfide sulfur; that the thiosulfate sulfur remained practically the same; that a small amount of some compound containing the (SH) radical was formed; and that there was a loss of about 81 per cent of the lime. In other words, they show that a large amount of calcium had disappeared while the loss in the monosulfide equivalent, thiosulfate, and sulfide sulfur were comparatively small. A microscopic examination showed conclusively that calcium sulfate was precipitated from the solutions. Now the following calculations may be made:

$$0.0333 \times \frac{56}{64} = 0.0291 \text{ g. CaO necessary to combine with } 0.0333 \text{ g. S in Thio-S}$$

$$0.0279 \times \frac{56}{32} = 0.0488 \text{ g. CaO necessary to combine with } 0.0279 \text{ g. S in Mono-S.}$$

$$\text{TOTAL... } 0.0779 \text{ g. CaO}$$

From these it will be seen that there should be 0.0779 g. of CaO in the solution, while there are only 0.0154 g. by actual determination. Evidently magnesium has replaced some of the calcium, and the solution now also contains magnesium polysulfide and a small quantity of some compound containing the (SH) radical. These compounds were not present in the original solution and must have been formed by the addition of the magnesium sulfate. It will also be seen that the length of time the solution is allowed to stand and the quantity of magnesium sulfate present are important factors in this reaction. And it appears that the lower polysulfides, or possibly the oxy-

<sup>1</sup> J. Chem. Soc., 45 (1884), 699.

sulfides of calcium are acted on more readily than the higher polysulfides, since solutions prepared by using an excess of lime in which lower polysulfides and possibly oxysulfides are present, react more quickly and with a less concentrated solution of magnesium sulfate than do those in which an excess of sulfur is used and only the higher polysulfides are present. From these data as well as those in Tables I, II and III, we must conclude that magnesium sulfate reacts with some compound in a lime-sulfur solution other than free lime, and in some solutions, at least, this compound is calcium polysulfide. Therefore Method II cannot be considered accurate for determining free lime in a lime-sulfur solution.

Having presented evidence which we believe is sufficient to show that Method I is an accurate measure of the free lime in a lime-sulfur solution, and that Method II cannot be considered accurate for this work, a few more solutions were prepared using different proportions of lime and sulfur, and a few more concentrates were diluted with lime water. These were analyzed by the former method, some immediately and some after standing several days. The results, together with explanatory remarks, are found in Table V:

TABLE V

SAMPLE No.	N/10 HCl Cc.	N/10 ZnCl <sub>2</sub> Cc.	CaO Gram	Remarks
1...	20.17	20.17	0.0000	S : CaO = 3 : 1. Analyzed immediately after boiling
2...	20.15	20.15	0.0000	Same as 1 after standing well stoppered for three days
3...	29.90	29.50	0.0011	S : CaO = 1 : 1. Analyzed immediately after boiling
4...	30.00	30.00	0.0000	Same as 3 after standing well stoppered for three days
5...	10.80	8.70	0.0059	Sample K in Table I
6...	10.10	8.50	0.0045	Same as 5 after standing well stoppered for three days
7...	27.95	24.29	0.0103	Commercial concentrate diluted with lime water (0.0106 g. CaO)
8...	3.97	3.95	0.0000	Same as 7 after standing exposed to air for fourteen days

The results in Table V, similar to those in Table I, are in accord with the contention that free lime is present in a freshly boiled lime-sulfur solution prepared by using an excess of lime, but they are contrary to the contention that free lime will remain in such a solution or that it is formed by hydrolysis. The only conclusions that can be drawn from the two tables are that the small amount of free lime which may be present immediately after preparing a lime-sulfur solution, gradually disappears on standing, and that immediately after adding free lime to a lime-sulfur solution nearly all the lime exists in the free state, but that chemical action takes place slowly, and after some time all the free lime has entered into chemical combination. Therefore, the inference is that the ordinary commercial lime-sulfur solution, either concentrated or dilute, does not contain an appreciable amount of free lime.

## FREE SULFUR

Thompson and Whittier<sup>1</sup> show that the residue which separates from a lime-sulfur solution prepared with an excess of sulfur in an atmosphere of nitrogen, filtered while hot and kept in a completely filled, tightly

stoppered receptacle, is mainly free sulfur. Auld<sup>2</sup> mentions the fact that when a lime-sulfur solution was prepared in an atmosphere of nitrogen and an excess of sulfur was used, a polysulfide was formed which analyzed to be the pentasulfide or slightly higher, and that on standing free sulfur separated out. He believes that sulfur may exist as free sulfur in solution. Harris<sup>3</sup> prepared the pentasulfide by simply using a reflux condenser, and free sulfur crystals were found by the author in some of his solutions after they had stood several years. Tartar and Bradley<sup>3</sup> succeeded in extracting free sulfur from a lime-sulfur solution and found that the quantity extracted gradually decreased with the increase of the time of extraction, but found no definite end-point. Ramsey<sup>4</sup> contends that the so-called polysulfide sulfur is loosely combined and that free sulfur exists in the solution. Green<sup>5</sup> says, "We regard our data as effectually disposing of the contentions both of Auld and Ramsey in regard to loosely attached sulfur, and sulfur in solution." He believes that the polysulfide sulfur is firmly combined and that there is no free sulfur in solution. Thompson and Whittier conclude from their work that no sulfide lower than the pentasulfide is formed and that free sulfur is held in solution.

During the progress of this investigation samples were prepared with and without the use of a reflux condenser, using an excess of sulfur. Those with the condenser approximated the pentasulfide, while those without were lower. In both cases, when the solutions were filtered while hot into flasks which were completely filled and well stoppered and the solutions allowed to cool, crystals which proved to be free sulfur separated out. Even when allowed to cool before filtering and then kept as mentioned above, free sulfur separated out of the more concentrated solutions.

Table VI gives the results of complete analyses on a solution prepared from materials containing an excess of sulfur and kept the lengths of time indicated. The analyses were made on 10 cc. aliquots of the diluted concentrate.

TABLE VI

SOLN. NO.	Time of stand- ing	Sulfur Crys- tals	Mono-S Gram	Thio-S Gram	S-S Gram	Total-S Gram	Total CaO Gram	Ratio. S-S : Mono-S
1 A(a) ..	Still warm	None	0.0441	0.0430	0.2153	0.2620	0.1188	1 : 4.88
1 B.....	12 hrs.	Few	0.0445	0.0492	0.2148	0.2639	0.1212	1 : 4.83
1 C.....	32 days	Many	0.0444	0.0496	0.2110	0.2606	0.1211	1 : 4.76

(a) This solution was less concentrated than the other two because it had not cooled.

This table shows a loss in sulfur from the solution, and the loss lies in the polysulfide sulfur. The fact that before the deposition of the sulfur, the ratio of the sulfide sulfur to the monosulfide sulfur was less than 5, and that as more sulfur was deposited this ratio was lowered, makes it appear that the free sulfur deposited in these solutions probably came from the breaking down of the higher polysulfides and not from free sulfur in solution. However, whether this ex-

<sup>1</sup> *J. Chem. Soc.*, 107 (1915), 484.

<sup>2</sup> *Mich. Agr. Col. Exp. Sta., Tech. Bull.* 6 (1911), 10.

<sup>3</sup> *THIS JOURNAL*, 2 (1910), 274.

<sup>4</sup> *J. Agr. Sci.*, [2] 6 (1914), 194-201.

<sup>5</sup> Union of S. Africa Dept. of Agr., 3rd and 4th Report of the Director of Vet. Research, 1915, p. 192.

planation is satisfactory or not, apparently makes little difference, since free sulfur in such a solution would undoubtedly have properties similar to those of sulfur loosely combined, as in the case of the higher polysulfides.

#### OXYSULFIDES OF CALCIUM

Most lime-sulfur investigators agree that when lime and sulfur are boiled in water, with the lime in excess of the ratio 1 : 2.28, and the solution is allowed to stand in a closed vessel, certain oxysulfides of calcium separate out. However, they do not agree exactly as to the constitution of these compounds. It has been suggested that they are not ordinary oxysulfides<sup>1</sup> as carbon oxysulfide COS, uranium oxysulfide U<sub>2</sub>O<sub>2</sub>S, manganese oxysulfide Mn<sub>2</sub>OS, etc., but that they are compounds made up of a calcium polysulfide and calcium oxide; and that the chief difficulty in the question of their composition is to determine what polysulfide enters into their structure and whether or not this polysulfide is constant.

Undoubtedly the most important evidence concerning these oxysulfides has come from an investigation of the crystals themselves, even though the results of different analyses of these crystals do not agree as well as might be desired. Considering the facts that no solvent has been found by which the crystals can be purified by recrystallization and that they cannot even be washed entirely free from impurities, it is almost surprising to find that different chemists agree as well as they do on their composition and properties. However, since there are the above-mentioned difficulties in dealing with the crystals, it seemed advisable to compare the work done along this line in this laboratory with that of others. In doing this, lime-sulfur solutions were prepared by taking one part of calcium oxide, one part of sulfur and five parts of water, boiling without a reflux condenser until the escaping vapor showed the presence of hydrogen sulfide, filtering while hot, and placing in well-stoppered flasks, some of which were filled completely while others were only partially filled. In all cases orange-red needles separated, even when the flasks were completely filled, showing that they were not dependent on the oxygen of the air for their formation. (In the more dilute solutions crystals formed only in the partially filled flasks.)

Several batches of these crystals were purified by separating them from the mother liquor by filtering through hardened filter paper in a Gooch crucible, washing a few times with small amounts of cold water (10° C.), a few times with small amounts of 95 per cent alcohol, several times with absolute alcohol, ether, carbon disulfide, and then with ether again. They were dried in a vacuum desiccator over calcium chloride.

The crystals purified as given above had an orange-red color. They were found to be insoluble in and not decomposable by ether, petroleum ether, chloroform, carbon disulfide, carbon tetrachloride, pyridine, or absolute alcohol. They were decomposed by hot and cold water, forming a solution similar in appear-

ance and reaction to a dilute lime sulfur solution, and leaving a white, amorphous residue. When treated with 95 per cent alcohol, pyridine containing water, or with a small volume of cold water they were decomposed, forming a solution similar to the above; but some of the crystals retained their original shape, losing only the orange-red color. On heating, the crystals were gradually decomposed, leaving a white substance which did not melt or burn. Under the microscope the smaller crystals appeared like orange-red four-sided prisms with parallel cleavage. Between crossed Nicols, the extinction appeared parallel in one position and at an angle of about 30° in another. The white amorphous residue, as well as the white crystal-shaped masses which remained after treating with cold water, was found to be calcium oxide.

Samples of different batches of these crystals were analyzed by treating with sodium hydroxide solution, oxidizing with sodium peroxide, acidifying with hydrochloric acid, and then determining the total lime and total sulfur in the usual manner. The results are shown in Table VII:

SAMPLE No.	Total S Per cent	Total Ca Per cent	Ratio S/Ca
1.....	20.92	28.28	1.34
2.....	20.75	27.11	1.27
3.....	22.09	28.77	1.28
4.....	21.83	25.47	1.17
5.....	21.08	25.11	1.19
6.....	20.70	27.30	1.31
Average.....	21.23	27.01	1.27

From the above results it is evident that the molecules making up the crystals contain as many atoms of sulfur as of calcium, since the theoretical ratio of sulfur to calcium in such a molecule is 1.25, which is almost identical with the average of the above determined ratios.

On titrating several water solutions of the crystals with standard iodine and comparing the end-point determined by color with that determined with nitroprusside of sodium, it was found that the two end-points were the same, showing the absence of the (SH) radical, and therefore the absence of any of the compounds containing this radical.

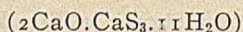
That lime is set free when the crystals are decomposed with cold water is shown by the following: Two samples of crystals were shaken in flasks completely filled with freshly boiled distilled water until decomposition was complete. The solutions were then filtered and aliquots titrated with 0.1 N hydrochloric acid and with 0.1 N zinc chloride. The following results were obtained:

SOLN. No.	HCl Cc.	ZnCl <sub>2</sub> Cc.	CaO Gram
1.....	5.49	2.80	0.0075
2.....	16.04	7.95	0.0226

The general properties of these crystals seem to indicate that they are the Herschell's<sup>1</sup> crystals described in the literature, and a partial analysis shows that their percentage composition agrees with one of the formulas given by Geuther:<sup>2</sup>

<sup>1</sup> *Ann.*, 224 (1884), 181-192.

<sup>2</sup> *Ibid.*



as is shown by the following:

TABLE IX  
Geuther's formula— $2\text{CaO} \cdot \text{CaS}_3 \cdot 11\text{H}_2\text{O}$

	Ca Per cent	S Per cent
Percentage composition—Theory.....	26.9	21.6
Found.....	27.01	21.23

It should be noted, however, that one property is shown by the mother liquor from which the crystals separate which is very difficult to explain if the crystals in solution are considered as being composed of calcium oxide in combination with calcium polysulfide. This is shown by the fact that when aliquots of such a solution are titrated with standard hydrochloric acid, standard zinc chloride, and standard iodine, the three titrations agree. This will be seen in the following table where 10 cc. of lime-sulfur concentrates containing the orange-red needles were diluted to 100 cc. with freshly boiled distilled water and 10 cc. aliquots were titrated.

TABLE X

SAMPLE No.	0.1 N HCl Cc.	0.1 N ZnCl <sub>2</sub> Cc.	0.1 N I Cc.
1.....	19.00	18.95	18.95
2.....	18.65	18.70	18.65

If the mother liquor from which the crystals separate contains in solution a compound which has for its formula  $2\text{CaO} \cdot \text{CaS}_3 \cdot 11\text{H}_2\text{O}$ , it is difficult to explain the above results, since hydrochloric acid, zinc chloride, and iodine could hardly give equal titrations on a solution containing the molecules mentioned. No attempt to explain this will be made at this time, but it should be emphasized that whatever the explanation may be, the fact remains that the three titrations do agree.

#### SUMMARY

I—Compounds containing the (SH) radical, as hydrogen sulfide, calcium hydrosulfide, calcium hydroxyhydrosulfide, and the corresponding salts of other metals, may be detected in a lime-sulfur solution by comparing the titration of the solution with standard iodine to the disappearance of the yellow color with that when the end-point is determined by the use of nitroprusside of sodium.

II—A "straight" lime-sulfur solution does not contain an appreciable amount of any of the above-mentioned compounds.

III—The difference between the titrations of a "straight" lime-sulfur solution with standard hydrochloric acid and standard ammoniacal zinc chloride is a measure of the free lime in the solution.

IV—When an excess of lime is used in the preparation of a lime-sulfur solution and the solution is freshly prepared, or when recently diluted with lime-water, it contains free lime; but on standing, the free lime gradually disappears. Therefore an ordinary lime-sulfur solution cannot contain free lime.

V—When magnesium sulfate is added to a lime-sulfur solution the following may be noted: (1) There is a slight decrease in the monosulfide sulfur and the sulfide sulfur contents. (2) The thiosulfate sulfur content remains practically constant. (3) The magnesium replaces part of the calcium forming magnesium polysulfide and under proper conditions calcium sulfate crystallizes out. (4) A compound containing the (SH) radical is formed.

VI—The magnesium sulfate method for determining free lime in a lime-sulfur solution is inaccurate.

VII—There appears to be no free sulfur in a lime-sulfur solution, and the sulfur that separates out on standing undoubtedly comes from the higher polysulfides.

VIII—When a concentrated lime-sulfur solution is prepared with an excess of lime, orange-red needles separate out. The properties of these crystals indicate that they are the same as those described in the literature as Herschel's crystals, and as being composed of calcium oxide combined with calcium polysulfide. Their analysis agrees most closely with that of Geuther, who gives for their formula the following:  $2\text{CaO} \cdot \text{CaS}_3 \cdot 11\text{H}_2\text{O}$ . However, it seems improbable that they exist in solution in the form indicated by this formula.

#### ACKNOWLEDGMENT

This work was suggested by Prof. A. J. Patten of this laboratory, and was carried out largely under his direction. I wish to express my great appreciation for the continued interest manifested throughout and for his kind advice and criticism.

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## LABORATORY AND PLANT

### A STANDARD APPARATUS FOR THE DETERMINATION OF SULFUR IN IRON AND STEEL BY THE EVOLUTION METHOD

By H. B. PULSIFER

Received April 3, 1918

While investigating the determination of sulfur in iron and steel as briefly described in THIS JOURNAL, 8 (1916), 1115, the author was led to make a few determinations by the evolution method. After the several estimations with dilute acid, both direct and annealed, as recorded in the following columns, it was decided to conclude the work with other series using concentrated hydrochloric acid.

The excellent results obtainable with hot concentrated acid have been reported almost since the inception of the method nearly a hundred years ago; but Williams,<sup>1</sup> in 1892, was apparently the first one to compare results and demonstrate that concentrated acid would furnish far higher and more correct results than dilute acid. During the years following, numerous investigators,<sup>2</sup> both American and European,

<sup>1</sup> *J. Eng. Soc. West. Penn.*, 8 (1892), 328.

<sup>2</sup> Schindler, *Z. angew. Chem.*, 6 (1893), 11; Schneider, *Oesterr. Z. Berg. Hüttenw.*, 41 (1893), 365; Phillips, *J. Am. Chem. Soc.*, 17 (1895), 891; Petřén, *Jern-Kontorets Annaler*, 60 (1905), 187; Schulte, *Stahl u. Eisen*, 26 (1906), 985; Kinder, *Ibid.*, 28 (1908), 249; Orthey, *Z. angew. Chem.*, 21 (1908), 1359 and 1393.

confirmed his conclusions and, going even further, presented exhaustive data to show that quick solution in hot concentrated acid dissolved all ordinary sulfides and evolved the sulfur as hydrogen sulfide from all sorts of the ferrous alloys. In particular the abundant formation of methyl sulfide generated by dilute acid on materials of high combined carbon was entirely overcome by dissolving in the hot concentrated acid. Several designed and used a form of apparatus to return as much as possible of the distilled acid to the flask, thus maintaining the strength

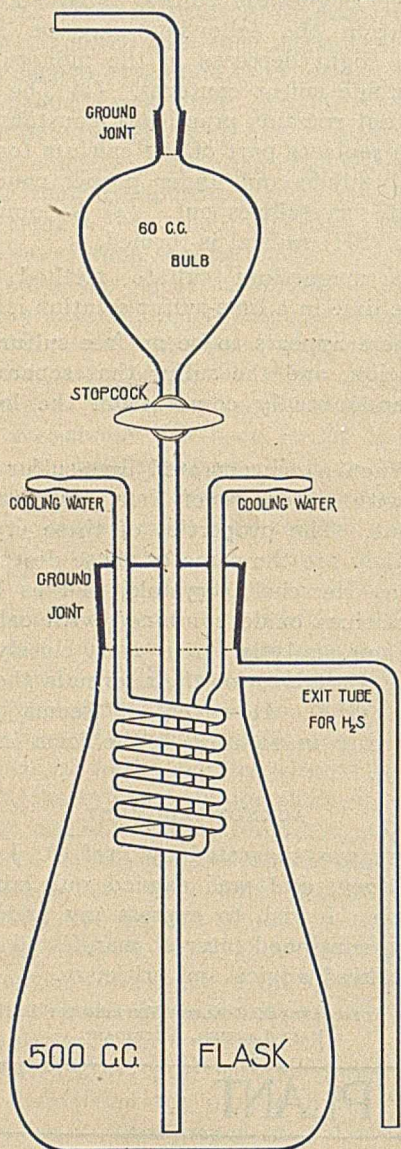


FIG. I

of the solution until the action is complete. The unsuitability of using rubber with the concentrated acid has long been known so that the better types of apparatus have been constructed wholly of glass. Rubber stoppers in contact with hot concentrated hydrochloric acid for 15 min. may easily evolve far more sulfide than most irons contain, 5 g. being taken.

Most of the types of European apparatus appearing rather impracticable and there being none at all available in this country, Messrs. Eimer and Amend, of New York, kindly attempted to construct a special

flask after the design shown in Fig. I. They were unable to make one without using a solid stopper and increasing the ground joints from 3 to 8. The flask, however, serves very well; it is seen set up in operation in Fig. II. Shortly afterwards Schaar & Company, of Chicago, were interested in the matter and were able to procure several excellent units, after the exact original design, from Japan. These flasks have only the three ground joints and are highly satisfactory. After making several hundred determinations with the battery of three, one solid and two with hollow stoppers, as indicated in Fig. III, the author has no changes or alterations to make in the original design. If the large main stopper is kept slightly greased it withstands the internal pressure perfectly yet is easily removed with a slight twisting motion. With a battery of 3 units one operator can continuously make 9 determinations per hour, including weighing out the samples and titrating the results. With enough flasks an operator and assistant ought to make more than 20 determinations an hour.

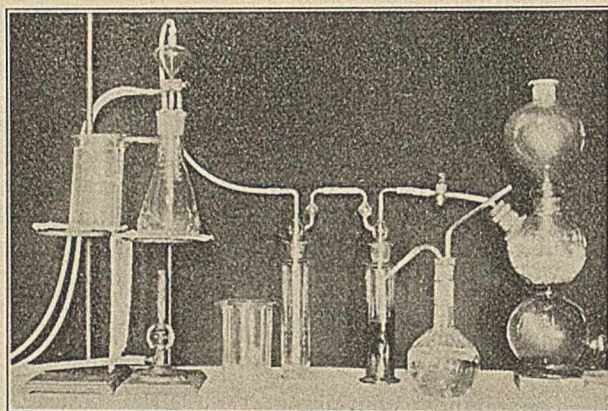


FIG. II

Arrangement of single flask showing hydrogen generator, wash bottles, flask, and beaker of absorbent solution. The cooling water is led into the coil through the tubes at the left.

As this type of apparatus and the results obtainable are apparently slightly recognized in this country it is thought worth while to again record the manner of operation and the sort of results one gets. The samples used were either those checked by other methods or supplied by the Bureau of Standards.

With the reflux apparatus and using 40 cc. of hydrochloric acid (sp. gr. 1.19) 5 g. of iron can be dissolved and the solution held at gentle ebullition for 15 min., leaving the final solution of more than half its original strength. The use of hydrogen to wash out the sulfide obviates vigorous boiling as commonly advocated. Thus at the end of the operation the acid is considerably more concentrated than at the start when 1 : 1 acid is used. Without the reflux only from  $\frac{1}{4}$  to  $\frac{1}{3}$  of the original acid may remain in the flask at the end under the same heat influence. It is naturally presumed that the hot concentrated acid is responsible for the uniformly high results obtained.

The cooling coil in the neck of the flask assists in holding back droplets of the solution which would otherwise carry ferrous chloride over into the absorb-



ing solution; for this reason the catch-bulb frequently used has been omitted. The amount of iron carried over varies greatly with the character of the dissolving and the heat used. Using the hydrogen or carbon dioxide stream to sweep out the hydrogen sulfide there is evidently no advantage in heating to more than the gentlest ebullition. Many measurements showed from a trace to 5 mg. of iron carried over into the ammoniacal cadmium chloride solution. With only 0.01 mg. or less of iron carried over, as is usually the case with this apparatus, the cadmium sulfide precipitate is a clear lemon-yellow. From 0.02 to 0.05 mg. iron makes the precipitate darken to orange, then becoming reddish with over 1 mg. iron. The ferrous hydroxide present in this small amount quickly oxidizes to ferric and takes no part in the subsequent titration with iodine. As iodine is liberated from iodide solution by both ferric salts and hydrochloric acid the solution should not stand unduly after the addition of the iodine and before the back titration with thiosulfate. The author finds 2 min. ample to effect the solution of the cadmium sulfide with the strength of acid used; with longer standing iodine is gradually liberated and the solutions turn blue again.

## PERMANGANATE AND OXALATE

$\text{Na}_2\text{C}_2\text{O}_4$	$\text{KMnO}_4$	To color	$\text{KMnO}_4$ per cc. (0.4717) (a)
0.3500	45.79	0.02	0.003607
0.3500	45.78	0.02	0.003608
0.3500	45.78	0.02	0.003608
0.3500	45.85	0.02	0.003602
0.3500	45.80	0.02	0.003606

Av., 0.003606

(a) Factor for changing weight of oxalate to weight of permanganate.

## PERMANGANATE AND THIOSULFATE

$\text{KMnO}_4$	$\text{Na}_2\text{S}_2\text{O}_3$	Ratio
15.51	49.91	0.3108
15.00	48.22	0.3111
15.04	48.30	0.3114
15.01	48.30	0.3108
15.00	48.21	0.3111
15.02	48.35	0.3107
14.97	48.11	0.3112

Av., 0.3110

## IODINE AND THIOSULFATE

Iodine	$\text{Na}_2\text{S}_2\text{O}_3$	Ratio
49.82	47.10	1.058
49.80	47.21	1.055
49.62	47.08	1.054
49.91	47.22	1.057
49.76	47.11	1.056
49.80	47.22	1.055
49.78	47.19	1.055

Av., 1.056

1 cc.  $\text{KMnO}_4$  is equivalent to 0.001829 g. S (factor = 0.5072).1 cc.  $\text{KMnO}_4$  is equivalent to 3.395 cc. iodine solution.

1 cc. iodine solution on 5 g. sample in percentage equals 0.01077 per cent.

The standardization of the iodine solution is best accomplished through a thiosulfate and permanganate solution against standard sodium oxalate as supplied by the Bureau of Standards. The author prefers large bottles of all the reagents, in bulk from 20 to 50 liters, and made up some months in advance. If preserved in the dark the solutions will alter so slowly that checking once a week is sufficient. As illustrating the precision and uniformity of the standardization work contrasted with the unequal distribution of sulfur in the metals, the above figures may be presented. It will be noticed that the variations between individual measurements are of the order of

1 part in 1,000 in the standardizations, while on the metal samples the sulfur content as measured by repetition under identical conditions will quite likely vary as much as 1 part in 5.

## PROCEDURE

The procedure used on the following samples to obtain the figures in the last column, marked "Conc. HCl," is as follows:

5 g. of the well-mixed sample are weighed on a watch-glass to within 1 mg. and cautiously brushed into the flask so as not to fall against the sides. 40 cc. of hydrochloric acid (sp. gr. 1.19) is poured into the upper bulb. The stopper is placed in position. The absorbent solution is prepared by putting 300 cc. distilled water in a tall form of 500 cc. beaker; to this is added 20 cc. of ammonia (sp. gr. 0.90) and 10 cc. of ammoniacal cadmium chloride solution (300 g.  $\text{CdCl}_2$ , 500 cc. ammonia (sp. gr. 0.90) and 500 cc. distilled water). The exit tube is adjusted in the beaker and a cover glass arranged as well as may be. The cooling water is regulated.

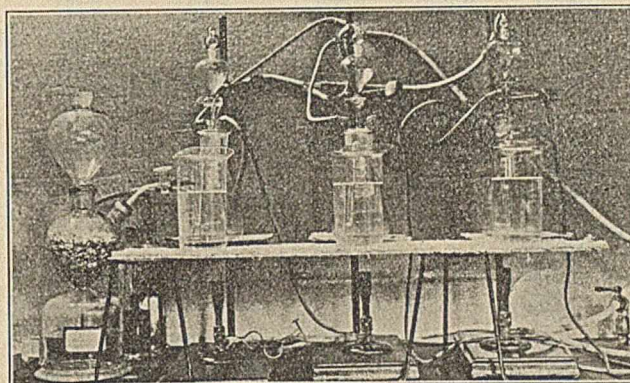


FIG. III

Battery of three flasks with hydrogen generator and water and gas connections. An analyst can maintain a rate of 9 determinations an hour with three flasks.

Hydrogen from HCl on zinc passed through alkaline  $\text{KMnO}_4$  and a safety bottle is then let in to force the acid down on the metal and the burner beneath is lighted. The burner has previously been adjusted so that it will just keep the solution in gentle ebullition. The stream of hydrogen may be passed through continuously to the end or closed off during the peak of the gas evolution. Within 5 or 10 min. the sample will probably completely dissolve. If a series of bottles be substituted in place of the single beaker the results will come no higher nor will the slightest trace of a precipitate be found in the second bottle. Inserting a wisp of glass wool in the beginning of the exit tube will decrease the amount of iron passing over, but will not influence the results.

At the end of 15 min. the flame is removed; the exit tube is withdrawn from the beaker; the hydrogen is shut off; the stopper is loosened and the coil washed down into the flask, after which the stopper is hung on the side support (Fig. III) ready for the next time; the outside of the exit tube is washed off into the beaker and the flask emptied and rinsed ready for the next run.

With the iodine solution in the burette at a known mark and 10 cc. of starch solution added to the solution beneath, 50 cc. of concentrated hydrochloric acid are poured in and iodine added to a strong permanent blue. Two minutes may be allowed for the complete solution of the cadmium sulfide, after which the blue color is discharged with the thiosulfate solution. If carbon dioxide is used as wash, one must add the hydrochloric acid with much care, especially if bicarbonate has separated and fallen to the bottom. This copious gas evolution, de-





is avoided. After learning this, over 300 determinations were made without accident. The breaking of the water connection resulted from the multiplicity of ground joints in the substitute flask; such an accident is hardly possible with the original design.

The cost of a flask is of no consequence if the results are considered worth having. The cost per determination will depend on the number of runs that can be made before it is damaged. If 1,000 determinations should be the average life on an original cost of \$10.00 the cost is 1 cent per determination. The manipulation is simple and only by gross carelessness should an analyst break the apparatus by handling. With the cooling water running and the hydrogen passing through, the flask can be left boiling over the flame indefinitely without damage.

The results which can be obtained with the apparatus are uniformly high, often above those obtainable by any other method. In only two instances with the samples studied did the dilute acid give more sulfur than the concentrated. These were both high sulfur materials and the sulfur from both of these samples easily separates as elemental sulfur; in using the nitric acid oxidation method the globule of sulfur, which may float about if the solution in hot acid is rapid, has to be oxidized by long digestion. The high results which one obtains as in Samples 2 and 3 might keep one in doubt as to the source of all the sulfur, were not the abundant cadmium sulfide precipitates directly in front of one and undeniably of the right proportions. The excellent agreement on some Bureau of Standards samples and the high figures on others emphasizes the advantage of standardization of the iodine solution against sodium oxalate.

A glance at the results shows the fallacy of using 1 : 1 hydrochloric acid, whether the sample has been annealed or not. If the sample is very high in sulfur, dilute acid will liberate more hydrogen sulfide in a 15-min. interval than the concentrated acid, probably because of more elemental sulfur being formed in the latter case. In this case neither will give as high results as an oxidation method but that, too, must be extended to oxidize the separated sulfur. Others have found that a slow digestion carried out for hours will finally bring the evolution sulfur result as high as the oxidation figure, even on these high sulfur materials. For sulfurs not over 0.10 per cent the proposed method is therefore as accurate as any method; for sulfur over 0.10 per cent the analyst must be strictly on his guard.

The advantages of the evolution method are that it gives the true amounts of sulfur present in the sample, the method is exceedingly rapid, the method is the most direct of any yet devised, and only two containers are used. Oxidation methods using nitric acid are all highly liable to loss of sulfur because of the excessive gas formation. The successive filtrations, evaporations, and precipitations in the oxidation and fusion methods (carried to excess in the latter method) render these methods reliable only under the most rigid conditions of laboratory, chemical, and manipulative control. This unfortunate condition is most

vividly presented in the distributed results of the Bureau of Standards showing the *average* result as obtained by the best analysts, all using the same method, with the extreme results usually differing by as much as 1 part in 5. There is certainly something more than the inherent heterogeneity of the samples at fault when the average results differ so widely.

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#### DETERMINATION OF ACETIC ACID BY DISTILLATION WITH PHOSPHORIC ACID

By W. FAITOUTE MUNN  
Received December 12, 1916

On account of the scarcity of literature dealing with methods for the determination of acetic acid in organic mixtures other than the analysis of calcium acetate, it was thought advisable to work along this line and devise, if possible, a new method which would be simple, rapid and accurate, even in the presence of carbon dioxide.

Although the determination as finally carried out bears relation to the regular phosphoric acid distillation method such as is described in the well-known books on technical methods, I think it sufficiently different, due to a number of changes and additions, to describe in detail.

This method has been used by the writer more particularly for determining acetic acid in calcium acetate residues and dry soda and sulfite liquors than for commercial calcium acetate assays and has given in all cases excellent results. Of course, if other volatile organic acids are present they will be estimated with the acetic acid and the analysis will therefore have to be continued so as to further separate these acids before calculating the results as acetic acid. In most cases the amounts of other volatile acids contaminating the acetic acid are very small and for most analyses such as are desired for technical information may be neglected.

The principle of the method is the decomposition of the acetate by means of phosphoric acid, distilling the acid vapors liberated and collecting them in a known amount of a standard barium hydroxide solution which is in excess, finally determining by titrating back with standard acid.

Before describing the method as finally carried out a few remarks relative to the methods now in use and described in books on quantitative analysis will be given.

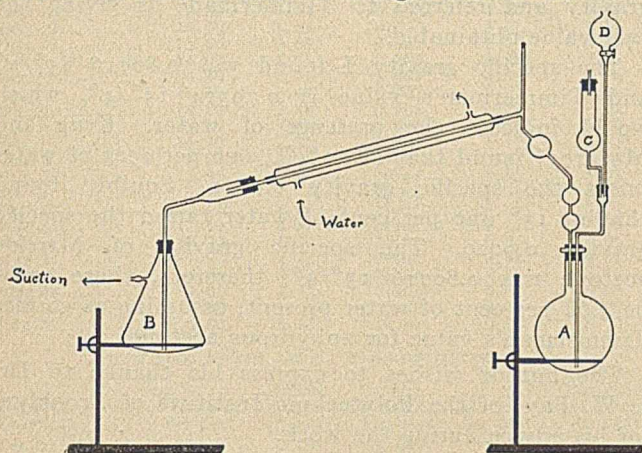
Lunge's "Chemisch-technische Untersuchungs-methoden," dritter Band, Seite 822-823, or the translated edition, Vol. 3, p. 307, directs that the acetate be distilled to dryness two or three times, the resulting distillates being combined and the amount of acid therein determined by titration with alkali.

Allen's "Commercial Organic Analysis," Vol. I, and Treadwell's "Quantitative Analysis" describe methods similar to that of Lunge.

The methods described by A. G. Stillwell<sup>1</sup> and T. S. Gladding<sup>2</sup> differ from Lunge's, mainly in the way of collecting the distillate, *i. e.*, absorbing the acid vapors in standard alkali as they are evolved and collected.

W. M. Grosvenor<sup>3</sup> points out the error liable to be introduced if any appreciable amount of carbon dioxide is present, but does not eliminate this error in his method should such be the case.

The procedures as given above tend to cause a great deal of bumping as the contents of the distilling flask become more concentrated and therefore more syrupy and the distillation often becomes very irregular. This is more noticeable when an organic residue is being acted upon than when acetic acid in acetate of lime is being determined.



There are three improvements in the following method by the writer, *viz.*, (1) making the distillation bulbs and condenser tube in one piece (of  $\frac{1}{4}$  in. glass tubing and of the shape shown in the drawing), thus making one less joint and, as the apparatus is much smaller bore, enabling one to obtain more concordant and accurate results in a shorter time; (2) the use of a slight suction on the latter end of the apparatus, thus preventing bumping to a very great extent, the accumulation of vapors in the flask and reducing the time required to make the determination; (3) the use of a standard solution of barium hydroxide for absorbing the acid vapors. (On account of the use of a current of air drawn through the apparatus it is necessary to provide the inlet of the flask with a soda-lime tube.)

The flask A, of about 500 cc. capacity, is fitted with a two-hole rubber stopper, one hole of which contains the distillation bulbs and the other a thistle tube, bent as shown in the figure. A soda-lime tube, C, and a dropping funnel, D, are connected to the thistle tube. As the dropping funnel contains dilute phosphoric acid, the air drawn through the apparatus during the distillation must enter through the soda-lime tube.

The cooling jacket for the condenser tube is made from  $\frac{3}{4}$  in. glass tubing. After drawing down the

ends and sealing on the water-tube connections, it is slipped on in the usual manner. The remainder of the apparatus needs no explanation as it can be clearly understood by glancing at the figure.

A known amount of a saturated, or nearly so, standard barium hydroxide solution is quickly run into the Erlenmeyer flask B, enough being added so as to still have an excess at the end of the determination.

5 g. of the dry sample, if it is a residue from a dry distillation, or a varying amount depending upon the nature of the substance, are placed in the flask A, and after fitting the corks, a dilute solution of phosphoric acid (20 cc. H<sub>2</sub>O plus 40 cc. 85 per cent phosphoric acid) is added through the funnel D. The stopcock of the funnel is now quickly closed and a slight suction (water pump) started through the apparatus. The flask and contents, which should be on a sand bath, are gradually heated until the liquid boils. After the temperature has reached the boiling point the flame is regulated so as to just keep the contents boiling lightly. The rate of suction is then adjusted so that the bubbles issuing from the tube in flask B are not more than 10 to 15 per minute.

As soon as the distillation and suction have been adjusted, a dilute solution of phosphoric acid is made up (50 cc. H<sub>2</sub>O to 25 cc. 85 per cent phosphoric acid) and poured into the dropping funnel. This dilute acid is allowed to drop into the distilling flask at the same rate as the condensed vapors distil (about 10 to 15 drops per minute). It may be said at this point that the concentrations just given for the phosphoric acid may vary considerably, depending upon the material under examination.

100 cc. Ba(OH) <sub>2</sub> solution used, equivalent to	153.30 cc. N/10 Ba(OH) <sub>2</sub>
N/10 HCl used to neutralize excess.....	90.30 cc.

N/10 Ba(OH) <sub>2</sub> equiv. to acetic acid plus CO <sub>2</sub>	63.00 cc.
N HCl used to dissolve BaCO <sub>3</sub> .....	10.56 cc.
N NaOH used to neutralize excess HCl.....	6.25 cc.

N HCl equivalent to BaCO <sub>3</sub> .....	4.31 cc.
N/10 HCl equivalent to BaCO <sub>3</sub> .....	43.10 cc.
From Ba(OH) <sub>2</sub> .....	63.00 cc.
Subtract cc. HCl equivalent to BaCO <sub>3</sub> .....	43.10 cc.

N/10 Ba(OH) <sub>2</sub> equivalent to acetic acid.....	19.90 cc.
$\frac{19.9 \times 0.006}{5} \times 100 = 2.39$ per cent acetic acid	

At the end of the run, which is generally complete in an hour and a half, phenolphthalein is added to the barium hydroxide solution and the excess of alkali determined by titration with N/10 hydrochloric acid. Normal hydrochloric acid is now added to dissolve the barium carbonate and the excess of acid then present is neutralized by the addition of normal sodium hydroxide and methyl orange for indicator. (As there is often a yellow color present in the liquid before adding the methyl orange for the second titration, the end-point showing when the excess hydrochloric acid has been neutralized is very indistinct. If the end-point is determined by using the methyl orange on a spot

<sup>1</sup> J. Soc. Chem. Ind., 1904, 305.

<sup>2</sup> THIS JOURNAL, 1 (1909), 250.

<sup>3</sup> "Analysis of Commercial Acetate of Lime," J. Soc. Chem. Ind., 1904,

plate, it is very definite.) The acetic acid in 5 g. of sample is estimated on preceding page.

A sample of carbonate of lime (about 40.00 per cent  $\text{CO}_2$ ), part of the residue from the manufacture of acetone by heating calcium acetate, was treated by the above method and the following figures obtained:

<i>N</i> /10 $\text{Ba}(\text{OH})_2$ used.....	97.09 cc.
<i>N</i> /10 $\text{HCl}$ to titrate back.....	67.02 cc.
<hr/>	
<i>N</i> /10 $(\text{BaOH})_2$ equiv. to $\text{CO}_2$ plus acetic acid	30.07 cc.
<i>N</i> $\text{HCl}$ added.....	5.63 cc.
<i>N</i> $\text{NaOH}$ to titrate back.....	2.63 cc.
<hr/>	
<i>N</i> $\text{HCl}$ equiv. to $\text{CO}_2$ .....	3.00 cc. or 30 cc. <i>N</i> /10 $\text{HCl}$
30.07 — 30.00 cc. = 0.07 cc. <i>N</i> /10 $(\text{BaOH})_2$ or 0.008 per cent acetic acid	

As this sample had been re-heated for a long time before this analysis was made, the chance of any acetate still being present was reduced to a minimum. The presence of a large amount of  $\text{CO}_2$  therefore does not seem to interfere with the acetic acid determination.

While the above procedure appears somewhat complicated at first glance, it does not prove to be so, as the distillation after once it is adjusted, needs very little attention, thus allowing the person in charge to attend to other duties.

LEDERLE LABORATORIES  
NEW YORK CITY

## THE DETERMINATION OF ACETONE

By ALLAN J. FIELD

Received June 20, 1917

The purpose of this investigation was to find an accurate method for the determination of acetone in methyl alcohol. All of the published methods were investigated with results as follows:

MESSINGER'S METHOD<sup>1</sup> is a volumetric method depending upon the reaction between iodine and acetone to form iodoform. Messinger claims that accurate results can be obtained by this method. A criticism by Vaubel and Scheuer<sup>2</sup> of this method is that when thiosulfate is used in titrating back, the results are always too low, and, therefore, they recommend the use of arsenious acid instead of the thiosulfate. The writer tested this method, using thiosulfate as well as the arsenious acid, but could not get accurate results. The precautions recommended by Collischon<sup>3</sup> were observed but the results were not satisfactory. I found that if, after the addition of the *N*/5 iodine solution, the mixture is shaken for 5 min., a low result is obtained, while shaking for 20 min. gives a higher result, the percentage varying with the length of time of agitation. These experiments were tried on a pure acetone solution without the presence of alcohol. When methyl alcohol was present the percentage of acetone found was several per cent higher. The increase could not be due to the alcohol furnishing the extra amount of acetone as the quantity it contained was duly considered.

<sup>1</sup> *Ber.*, **21** (1888), 3366.

<sup>2</sup> *Z. angew. Chem.*, **18** (1905), 214.

<sup>3</sup> *Z. anal. Chem.*, **29** (1890), 562.

THE ROBINEAU AND ROLLINS METHOD as modified by L. F. Kebler<sup>1</sup> was found by the author to give the most accurate results. The presence of alcohol and the time of shaking did not influence the percentage of acetone.

The acetone that was used in trying out these methods was Merck's reagent guaranteed by them to be 99 to 100 per cent. The percentages of acetone obtained in four determinations using the Robineau and Rollins method were as follows:

Determination No.	I	II	III	IV
Acetone, per cent. ....	96.37	96.37	95.89	95.74

There remained 4 per cent unaccounted for. The Bureau of Standards at Washington advised checking the purity of the acetone by means of the specific gravity and referred to Timmerman<sup>2</sup> as giving the best value obtainable.

The specific gravity I found was 0.80716  $15^\circ/4^\circ$  and Timmerman's value is 0.79574  $15^\circ/4^\circ$ , which would indicate the presence of water. Krug and McElroy<sup>3</sup> found that at  $20^\circ \text{C}$ ., one per cent of water raises the specific gravity 0.0031. Squibb<sup>4</sup> found that at  $15^\circ$  one per cent of water raised the specific gravity 0.0029. The specific gravity of Merck's acetone was 0.80716  $15^\circ/4^\circ$ , therefore, there could be 3.68 per cent of water present, assuming as correct Timmerman's value for anhydrous acetone.

The author wishes to express his thanks to Dr. I. W. Fay, of the Polytechnic Institute of Brooklyn, for his advice during the work.

ROSEBANK  
STATEN ISLAND, N. Y.

## SOME RESULTS OF ANALYSIS OF AIRS FROM A MINE FIRE

By A. G. BLAKELEY AND H. H. GEIST

Received September 15, 1917

There is no doubt that gas analysis has found considerable use in locating mine fires and especially in following the progress of these underground fires. Not a great deal of data has been published, however. The writers, therefore, considered it proper that they publish some data which may be of interest to those connected with mining work, particularly with anthracite coal mining.

The mine at which the samples were taken is an anthracite coal mine, a mine considered as a rather gaseous mine, or one generating a fairly large quantity of methane or fire damp.

On November 18, 1916, several men were overcome while at work inside the mine. On November 19, a fire was discovered. Batteries were erected for the purpose of smothering out the fire by cutting off all the fresh air supply.

In order to carry out this investigation pipes were extended through the batteries, these pipes being closed by means of valves at the outer ends. From time to

<sup>1</sup> *J. Am. Chem. Soc.*, **19** (1897), 316.

<sup>2</sup> *Bul. soc. chim. belg.*, **24** (1910), 263.

<sup>3</sup> *J. Anal. Appl. Chem.*, **6** (1892), 187.

<sup>4</sup> *J. Am. Chem. Soc.*, **17** (1895), 200.

time these valves were opened and air samples taken. These samples were taken in such a way as to accurately represent the air behind the battery, or in other words, the air from the fire zone.

The carbon monoxide was determined by the iodine pentoxide method, more than one liter of air being passed over the heated iodine pentoxide in each determination.

The results given in Tables 1 and 2 seem to need no further explanation.

The mine fire zone was partially opened in August 1917, and no further air samples were tested.

TABLE 1—7TH LEVEL, GANGWAY BATTERY

DATE OF SAMPLING	Oxygen Per cent	Carbon Dioxide Per cent	Carbon Monoxide Per cent	Methane Per cent	Carbon Dioxide in Black Damp <sup>1</sup> Per cent
Dec. 1, 1916.....	18.00	0.99	0.008	2.40	8.28
Dec. 5.....	16.40	1.16	0.0057	2.33	5.85
Dec. 12.....	15.80	1.34	0.0034	4.27	6.47
Dec. 19.....	16.00	1.61	0.015	3.28	7.80
Dec. 26.....	15.66	1.69	0.01	3.66	7.72
Jan. 5, 1917.....	14.00	1.71	0.006	4.46	5.87
Jan. 11.....	14.30	2.11	0.004	4.51	7.66
Jan. 18.....	13.60	2.48	0.002	5.72	8.36
Jan. 25.....	14.20	2.38	0.002	4.22	8.41
Feb. 9.....	13.00	3.04	0.005	5.16	9.20
Feb. 22.....	14.20	2.31	0.001	4.12	8.13
Mar. 8.....	13.80	2.38	0.0016	3.87	7.78
Mar. 22.....	13.90	1.60	None	3.55	5.23
Apr. 5.....	12.42	2.43	None	4.38	6.62
Apr. 18.....	13.11	2.34	None	3.35	6.79
May 3.....	13.10	2.67	None	4.12	7.93
May 17.....	10.20	3.20	None	6.35	7.06
May 31.....	9.90	2.22	None	4.96	4.59
June 14.....	8.70	3.42	None	7.87	6.70
June 28.....	9.00	3.32	None	6.60	6.53
July 12.....	6.40	4.21	None	8.14	6.82
July 26.....	4.80	4.47	None	10.90	6.71

TABLE 2—7TH LEVEL, MONKEY BATTERY

DATE OF SAMPLING	Oxygen Per cent	Carbon Dioxide Per cent	Carbon Monoxide Per cent	Methane Per cent	Carbon Dioxide in Black Damp <sup>1</sup> Per cent
Dec. 1, 1916.....	13.63	2.09	0.019	5.97	7.13
Dec. 5.....	12.00	2.11	0.009	3.35	5.29
Dec. 12.....	11.51	2.17	0.0044	6.22	5.52
Dec. 19.....	14.10	2.30	0.029	4.73	8.14
Dec. 26.....	13.78	1.98	0.016	5.15	6.73
Jan. 5, 1917.....	9.80	2.12	0.01	5.62	4.40
Jan. 11.....	10.80	3.31	0.0068	6.20	7.77
Jan. 18.....	10.20	3.40	0.006	5.63	7.39
Jan. 25.....	10.73	3.46	0.007	4.76	7.80
Feb. 9.....	9.40	4.10	0.008	6.34	8.35
Feb. 22.....	11.10	3.67	0.0017	5.25	8.72
Mar. 8.....	10.00	3.43	None	5.76	7.32
Mar. 22.....	11.40	3.62	None	4.96	8.85
Apr. 5.....	9.10	3.97	None	6.13	7.82
Apr. 18.....	10.20	3.65	None	3.81	7.63
May 3.....	10.00	3.40	None	6.44	7.36
May 17.....	7.30	4.40	None	7.02	7.52
May 31.....	7.80	4.06	None	6.87	7.21
June 14.....	3.30	4.81	None	9.03	6.36
June 28.....	3.20	4.86	None	10.05	6.47
July 12.....	3.30	4.96	None	11.88	6.81
July 26.....	2.80	4.84	None	10.17	6.29

<sup>1</sup> Foster and Haldane, "The Investigation of Mine Air," p. 124.

are enclosed in a metal case which opens, as indicated, and which may be rotated by means of an arm terminating in a micrometer screw, M. The smallest division on the micrometer corresponds to a difference in index of about 0.00005. The face of each prism is divided into two parts, A and B, by means of a groove so that a drop of the standard liquid may be placed on one face (either A or B) and a drop of the liquid to be tested on the other face. For example, since the temperature coefficient of most solutions is nearly that of water, water may be used as a standard when measuring the index of solutions.

To obtain a dividing line free from color it is necessary to use a monochromatic source of light, such as a sodium flame, or some compensating device. The former is sometimes inconvenient and the latter would considerably increase the cost of the instrument. A monochromatic red glass, G, was used in connection with an electric light, L, and it was found that the dividing line is nearly as sharp as that which obtains when a sodium flame is used. The lamp used was a 7.5 watt, 110 volt, frosted globe tungsten and

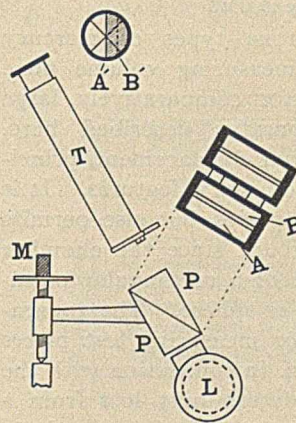


FIG. I

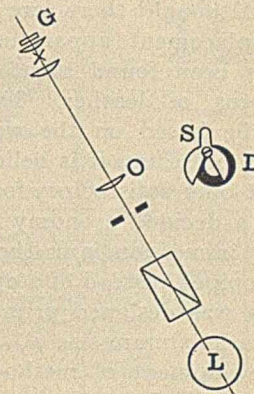


FIG. II

it was made a part of the instrument so that no adjusting was necessary when once in place. It is more convenient than daylight and it also produces a more uniform field.

The refractometer was originally designed to measure the difference in index between hemolyzed and unhemolyzed blood, as it was discovered by Dr. F. H. Howard and the writer, some time ago, that the amount of hemoglobin present in a given sample of blood causes its index to vary markedly and, furthermore, that the difference in index (hemolyzed and unhemolyzed blood) depends only upon the amount of hemoglobin present. Since the absorption bands of blood are in the green and blue parts of the spectrum a red glass is well adapted to this sort of measurement.

After passing through the refracting prisms the light enters a telescope, T, provided with cross-wires as shown. Between the telescope objective, O, and the prisms is a diaphragm, D, provided with a shutter, S, and by adjusting this shutter the light from either A or B may be cut out. For instance, when the light from B is intercepted, the dividing line A', due to the light from A, is seen; and when S intercepts the light from A, then B' is in the field. The distance

THE PHILADELPHIA & READING COAL AND IRON COMPANY  
POTTSTOWN, PENNSYLVANIA

A DIFFERENTIAL REFRACTOMETER

By G. A. SHOOK

Received February 16, 1918

This instrument is the result of an attempt to develop a simple but accurate refractometer for measuring the difference in refractive index between two liquids. It is of the Abbé type but so constructed that two liquids may be examined simultaneously and, therefore, if the index of one is known and if both have the same temperature coefficient, the index of the liquid in question may be accurately determined without knowing its temperature.

The instrument as constructed by the writer is shown diagrammatically in Fig. I, and the optical system in Fig. II. The refracting prisms P, P

between these lines is measured by means of the micrometer M.

When red light is used a difference in index can be measured with an accuracy of about one in the fourth decimal place (*i. e.*,  $\pm 0.0001$ ), but with sodium light the accuracy is about  $\pm 0.00005$ ; that is, if one measures the difference between the two lines by means of the micrometer, cleans the prisms, and makes a second measurement, the two values of the index difference calculated therefrom will agree within  $0.0001$  for red light and within  $0.00005$  for sodium light.

The relation between the micrometer reading and the difference in index is very nearly linear, so that by means of two solutions of known index and a comparison solution, the instrument can be easily calibrated.

WILLIAMS COLLEGE  
WILLIAMSTOWN, MASS.

### A. VOLUMENOMETER

By J. S. ROGERS AND R. W. FREY  
Received February 19, 1918

Although there are numerous types of mercury displacement apparatus for measuring volume none has been found satisfactory for comparatively large pieces of leather. The apparatus described here, while based on the well-known displacement principle, possesses, it is believed, some new features. It is not only satisfactory for large pieces, but also permits of a decided economy in mercury since the chamber for immersion is in the shape of a rectangular parallelepiped instead of a cylinder or sphere. The apparatus was designed primarily for measuring test pieces of a maximum size  $7\frac{1}{4} \times 3$  in. in connection with the development of methods of determining loss from a mechanical wearing test of leather. It has also been found to be very useful in determining the apparent density of leather.

A description of the volumenometer and photographs (Figs. I and II) follow: The immersion vessel consists essentially of the tank A and top B, both of cast iron and having accurately ground surfaces, C, so that the top, when clamped on by means of the bolts, D, make a mercury-tight joint. The tank A has a chamber, E,  $1\frac{1}{8}$  in. wide, 8 in. long, and  $3\frac{1}{2}$  in. deep, and the walls of this chamber are continued in the top B in such a manner that they converge to the small opening F in which is sealed, with shellac, the short thistle tube G. The top has two posts, H, to which the pieces to be measured are fastened.

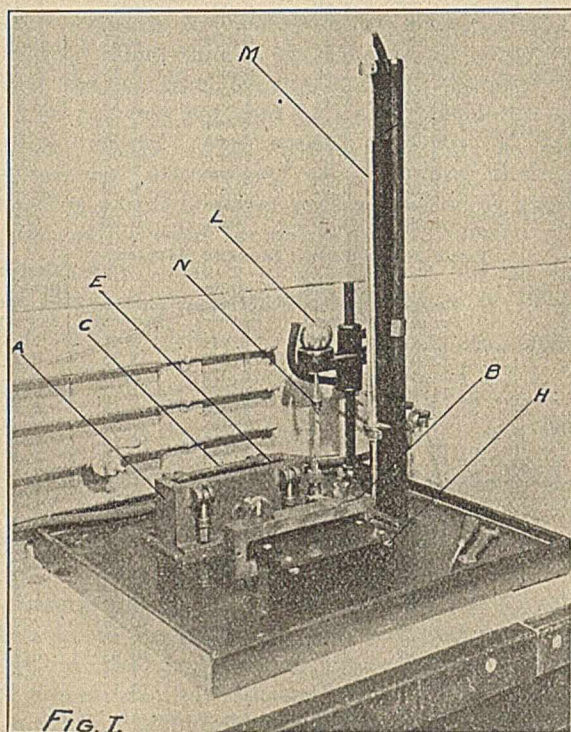
In the metal tube I, which passes through the wall of the tank from the bottom of the chamber E, is sealed a heavy capillary glass Y-tube, J, fitted with the mercury-sealed stopcocks K. One arm of the Y-tube is connected with the bulb L, and the other arm with the burette M. Both the bulb and the burette are connected with the vacuum system.

By means of N and O the zero points on the two glass tubes may be easily adjusted to coincide with the level of the mercury, which may change slightly from time to time, due to temperature variations and me-

chanical loss. This adjustable zero device consists of a small threaded metal sleeve fastened on the glass tube and fitted with a nut. Resting on the nut is a loose-fitting glass sleeve having a fine graduation. By turning the nut the graduation on the glass sleeve may be raised or lowered as desired.

All metal surfaces to which the mercury would have access are treated with bakelite, and the entire apparatus is securely mounted on a large wooden tray. The bulb and burette are also properly supported.

The operation of the apparatus is conducted as follows: With the top removed and the stopcocks open, completely fill the tank with mercury, being careful not to form any air traps in the Y-tube. Then draw the mercury well up into the bulb and burette, and after closing the stopcocks add sufficient mercury to fill that part of the chamber in the top of the tank. Place the top in the tank and fasten it securely by the four bolts, D. By opening the stopcocks let the mer-



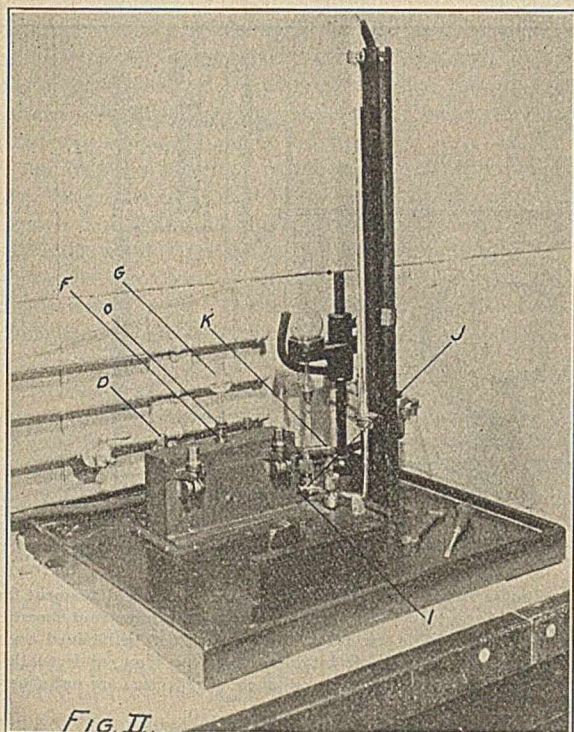
cury down from the bulb and burette until it stands at the same level in all parts of the apparatus. Set the two adjustable zeros, N and O, so that the graduations on the glass sleeves coincide with the menisci of the mercury, and take the zero reading on the burette. Now draw the mercury again well up into the bulb and burette, close the stopcocks and remove the top. Fasten the piece to be measured onto the posts H (Fig. I), replace and secure the top. Open the stopcock communicating with the bulb L and let the mercury run down slowly to the zero mark at N on the stem of the bulb and close the stopcock. Then open the stopcock connecting the burette and allow the mercury to gradually lower until it fills the tank and rises to the zero mark at O on the tube G, close the stopcock and read the burette (Fig. II). The difference in readings will give the volume of the piece.



In making a series of measurements, if care is taken in removing the pieces so as to avoid loss of mercury, the zero reading and adjustments need only be made after about every eight measurements.

The dust and dirt which slowly accumulate on the surface of the mercury in the tank may be very easily swept off with a small camel's hair brush. In fact, a slight layer of dust is somewhat desirable as it forms an envelope around the piece when immersed and helps to prevent the absorption of mercury.

An objection to the apparatus is that it requires two persons to operate it most satisfactorily and expeditiously. Furthermore, unless care is exercised in removing the pieces some mercury is likely to be lost. No doubt a more satisfactory means of fastening the piece could be devised which would overcome the tendency to lose mercury which collects around the screws, and which would decidedly shorten the time required for securing and removing the pieces.



A few of the measurements made with the apparatus are given here. These volumes were obtained on the same series of samples on different days, but always under the same temperature and relative humidity conditions of 70° F. and 65 per cent relative humidity, the apparatus being set up in the constant temperature and humidity room.

Samples	Volume in Cc.	Samples	Volume in Cc.		
S1.....	35.15	35.20	L3.....	40.05	40.05
S2.....	29.35	29.40	L4.....	39.80	39.80
S3.....	39.75	39.80	L5.....	42.95	42.95
S4.....	36.85	36.90	L6.....	47.85	47.90
L1.....	39.45	39.50	L7.....	37.20	37.20
L2.....	41.85	41.90	L8.....	34.40	34.40

A piece of hard rubber has been used as a standard to check the apparatus, and its volume determined at frequent intervals during a period of several months has ranged from 89.10 to 89.15 cc. The volume of this same piece calculated from loss in weight in water at 70° F. gave 89.14 cc.

## CONCLUSIONS

An apparatus has been devised which is satisfactory for measuring the volume of comparatively large pieces of leather. The measurements can be duplicated and are reasonably accurate.

LEATHER AND PAPER LABORATORY  
BUREAU OF CHEMISTRY, DEPARTMENT OF AGRICULTURE  
WASHINGTON, D. C.

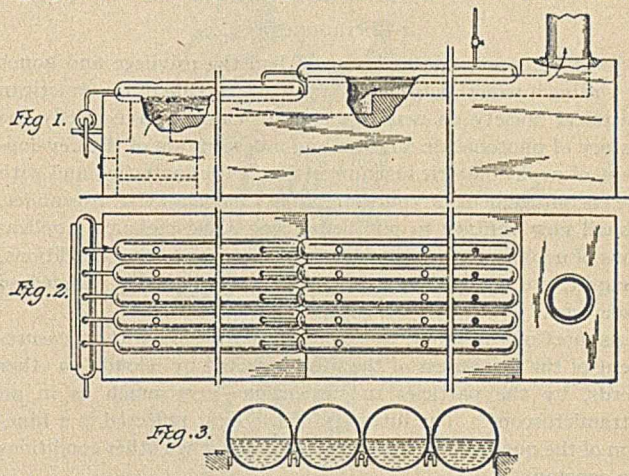
## AN EVAPORATOR FOR ACID LIQUIDS

FOR THE ECONOMICAL EVAPORATION OF ACID LIQUIDS OR OF ANY LIQUID DISCOLORED BY CONTACT WITH METALS

By EDWARD HART

Received February 7, 1918

This system of evaporation for which United States letters patent have been granted but not yet issued (Application No. 202,189) is especially indicated where the solvent is water. The tubes (Fig. 3 is a cross-section) which may be of any number and of any desired size and length (3-in. glass tubes 4 ft. long are recommended for most purposes) are supported on gas pipe covered with asbestos paper as shown in Fig. 3; cross-stirrup supports are not shown. There



are three 1-in. openings in each tube to permit the introduction of liquid and the escape of steam. These openings may, of course, take the form of tubes, and the solvent may be condensed if desired.

The fire grate and course of the fire gases are indicated by the arrows in Fig. 1. Two, three or more sets of tubes may be used (two are shown) and the liquid warmed in the first set boils and evaporates in the second, and is collected and discharged by the end manifold, shown in Fig. 2, into crystallizers. The concentration of the discharged liquid is controlled absolutely and with great accuracy by the feed.

HART LABORATORIES  
EASTON, PENNSYLVANIA

## CONVERSION OF FORMULAS

By WILLIS H. COLE

Received February 8, 1918

In practically all industries in the United States pounds and gallons are still the units of measure, while in the laboratories of these factories the metric system is almost universally used. Thus the industrial chemist is called upon to make many conversions

of formulas as made on a large scale in pounds and gallons, into a size suitable for laboratory batches, in grams and cubic centimeters. I have seen many good chemists waste all the way from minutes to hours doing this, looking up conversion tables and multiplying. By aid of the following simple rule, great saving of time and accuracy is effected. Call the gallons cc., and multiply the pounds by 0.12 (for very accurate work, 0.1198) to get grams. These figures may then be multiplied by any factor which will give a convenient sized batch for laboratory purposes.

EXAMPLE I			
	Varnish Formula Factory Batch	Conversion	Convenient Lab. Batch
Kauri Gum.....	100 lbs.	12 g.	24 g.
China Wood Oil.....	10 gals.	10 cc.	20 cc.
Prepared Linsed Oil.....	4 gals.	4 cc.	8 cc.
Turpentine.....	25 gals.	25 cc.	50 cc.
Petroleum Spirits.....	7 gals.	7 cc.	14 cc.
YIELD.....	52 gals.	52 cc.	104 cc.

In this case you will see that there are more ingredients measured by volume than by weight. Should this condition be the reverse, it is more efficient to change the rule, calling pounds, grams; and multiplying gallons by  $8\frac{1}{3}$  (for very accurate work, 8.3455) to get cc. Here follows a formula to illustrate this method.

EXAMPLE II			
	Flat White Enamel Factory Batch	Conversion	Convenient Lab. Batch
Zinc Oxide.....	90 lbs.	90 g.	45 g.
Lithopone.....	90 lbs.	90 g.	45 g.
Whiting.....	25 lbs.	25 g.	12.5 g.
Silica.....	25 lbs.	25 g.	12.5 g.
Varnish.....	12 gals.	100 cc.	50 cc.
Benzine.....	10 gals.	83.3 cc.	41.6 cc.
YIELD.....	28 gals.	233.3 cc.	116.6 cc.

The factors used hold for U. S. Standard gallons only and not for Imperial gallons.

RESEARCH LABORATORY  
MOLLER & SCHUMANN CO., BROOKLYN, N. Y.

## ADDRESSES

### TECHNICAL APPLICATIONS OF NEPHELOMETRY<sup>1</sup>

By PHILIP ADOLPH KOBER

#### I—INTRODUCTION

Something over two years ago I had the privilege and honor of reading a paper<sup>2</sup> before the New York Section of the American Chemical Society on nephelometry in which I gave briefly the history of photometric analysis and a description of the development of nephelometric instruments. At the invitation and with the encouragement of your honorable Chairman, Dr. Alexander, I shall now venture to put before you some technical applications of nephelometry, which, owing to war and other conditions, I regret, are not nearly as complete as I originally planned to have them.

As most of you know, the method is based upon the measurement of the brightness of the light reflected by cloud—in other words, by the particles in suspension—very much as in an ultramicroscope. The intensity of the light reflected is a function of the quantity of suspended particles, when other conditions are constant.

The principle of the nephelometer can best be shown by the diagrammatic sketch, shown in Fig. I.

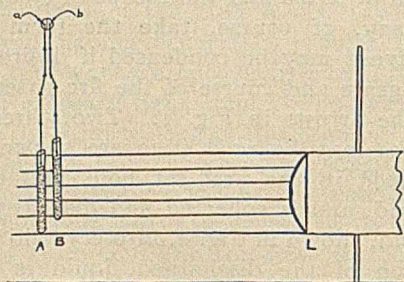


FIG. I

The path of light in nephelometer.

Let A and B represent tubes containing a precipitate in the form of a suspension, and L represent a strong light which throws its uniform beam on tubes A and B at right angles; then *a* and *b* will be the light in the eyepiece due to the reflections from the two suspensions. If tube A, for example, contained distilled water and the instrument were perfect, no light at *a* would be

<sup>1</sup> Lecture and demonstration given before the N. Y. Sections of the Society of Chemical Industry, American Chemical Society, and American Electrochemical Society, Chemists' Club, October 19, 1917.

<sup>2</sup> Kober and Graves, *THIS JOURNAL*, 7 (1915), 843.

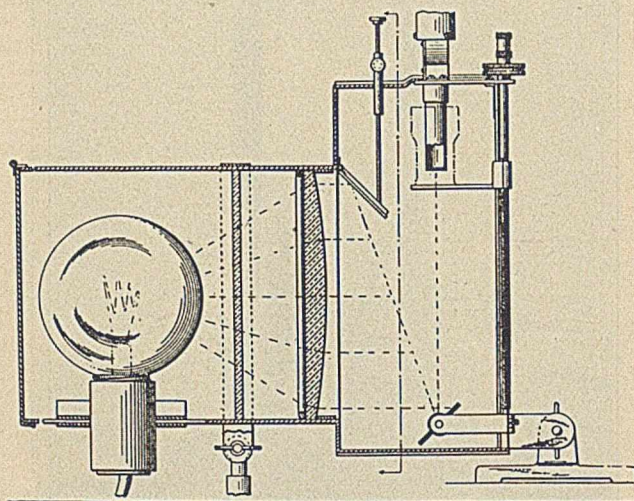


FIG. II

Lamp house and instrument, showing the concentrated filament lamp, air space, condenser, and lamp house. When the doors (not shown) are closed, no light is visible except in the eyepiece. The inclined angle of the instrument, which allows air bubbles to escape from underneath the plungers, and the exact position and angle of reflectors are not shown in the sketch.

visible. As soon as the smallest amount of suspension is produced in the tube A, light is obtained in *a* in approximate proportion to the amount of suspended matter. This light *a* is never measured absolutely, but is always matched at *b*, which is that reflected by the precipitate of a standard solution—a known amount of the substance to be determined dissolved in a known volume.

The matching of the two lights could be done by changing the standard solution step by step until it would be exactly that of the unknown. In practice this would be tedious and, therefore, instruments were designed to eliminate this in whole or in part.

Fig. II shows a diagrammatic sketch of such instrument and lamp house, and is known as a nephelometer-colorimeter,<sup>1</sup> because it is both a nephelometer and colorimeter.

Fig. III shows the instrument without the lamp house, particularly the scales, verniers, and the screw arrangement for raising and lowering the cups, which is used to match the reflected light from the suspensions.

<sup>1</sup> *J. Biol. Chem.*, 29 (1917), 155.

Fig. IV is a photograph of the instrument, with solutions, or, better said, suspensions, viewed from the front.

The cups and plungers for this instrument are shown in Fig. V. The nephelometric cups have transparent sides and opaque bottoms, while the colorimetric cups have opaque sides and transparent bottoms.

In Fig. VI is shown the photograph of the instrument used as a colorimeter, where the adjustable reflectors are in position. The lamp switch may be seen in front, as well as the doors of the lamp house, which when closed cut off any glare from the source of light.

Before going over to the details of the applications, it may be well to point out briefly the correct use of the nephelometer and a few points on the production of nephelometric suspensions.

A large number of workers do not have favorable conditions when they first apply the nephelometer. The work should be conducted in the dark; glares from windows, doorways, and the like, as well as artificial lights, being just as bad for photometric work as for the moving picture show. Some laboratories have permanent special dark rooms, others portable dark rooms, while still others darken the regular laboratory with opaque shades. A few workers forget that the eye is a sensitive instrument, and by using it too much or too continuously, and not allowing a period of rest, like the standard electric cell, it can easily become polarized, or fatigued. When these precautions are taken photometry or nephelometry can be practiced all day without any special fatigue or eyestrain.

A few forget or overlook many of the following obvious details: They ignore the dust in the various parts of the optical equipment and fail to fill the cups, so that the cups are clean on the outside, which thus allows the light to be absorbed. They overlook the fact that any air bubbles and dust underneath the plungers reflect light. The zero point of the instrument, too, as illustrated in Fig. VII, is not adjusted to represent zero light, and other oversights are often present.

Probably the greatest source of error connected with nephelometry or colorimetry is practically an instrumental one, pointed out to me by Mr. Klett, head of the company<sup>1</sup> manufacturing this instrument. He found that most diaphragms or openings at the top of the eyepieces, even in the best of instruments, are too large. He found that by putting a small diaphragm of the size of a pinhole over the eyepiece, he could make one or the other side of the field of almost any intensity.

Only by putting the aperture over the optical center could he obtain equal illumination of both sides of the field. When he moved the pinhole 0.1 to 0.2 mm. to any side, as illustrated in Fig. VIII, he obtained black spots and sides in the field. What errors would be introduced by a nervous individual, who would probably never have the eye twice in the same position, or by a beginner doing the same, can easily be imagined. By the use of such a diaphragm, not only will many discrepancies in readings, such as an occasional off-reading, disappear, but the field will become more flat; and only by its use it seems can the instrument be accurately adjusted and used.

Time does not permit us to dwell on the production of colored solutions suitable for colorimetry, however interesting and important from both theoretical and practical standpoints. As we have pointed out elsewhere,<sup>2</sup> the chief requisite for making nephelometric clouds or colloidal suspensions, and for keeping them as such for a definite time, is that the substance be in a dilute solution, usually not stronger than 100 mg. per liter. Therefore, to apply the method to large amounts of substance it is only necessary to dilute suitably. Clouds, produced by one part in 500,000 of liquid, may be determined quantitatively. In some cases one part in 2,000,000 can be easily determined.

<sup>1</sup> Klett Manufacturing Co., Inc., 202 E. 46th St., New York City.

<sup>2</sup> P. A. Kober and Sara S. Graves, "International Clinics," 24th Series, 2 (1914), 121.

Since the amount of substance may vary greatly it is important to know whether the nature of the precipitate imposes any limitations on the method. It is necessary to consider, therefore:

1. COLOR—If the precipitate is highly colored and remains in suspension, it is best determined colorimetrically; if slightly colored, it is best determined nephelometrically.

2. FORM OF PRECIPITATE—It must be colloidal in the form of a suspension. A large number of precipitates found in practical work are colloids, a number are partly so, while some are so entirely crystalloidal, such as barium sulfate, that they settle immediately. According to previous work published, certain solutions of protective colloids can be used, such as egg albumin and soluble starch, which cause crystalloids like barium sulfate and other partly colloidal precipitates to remain in suspension long enough for the application of this method.

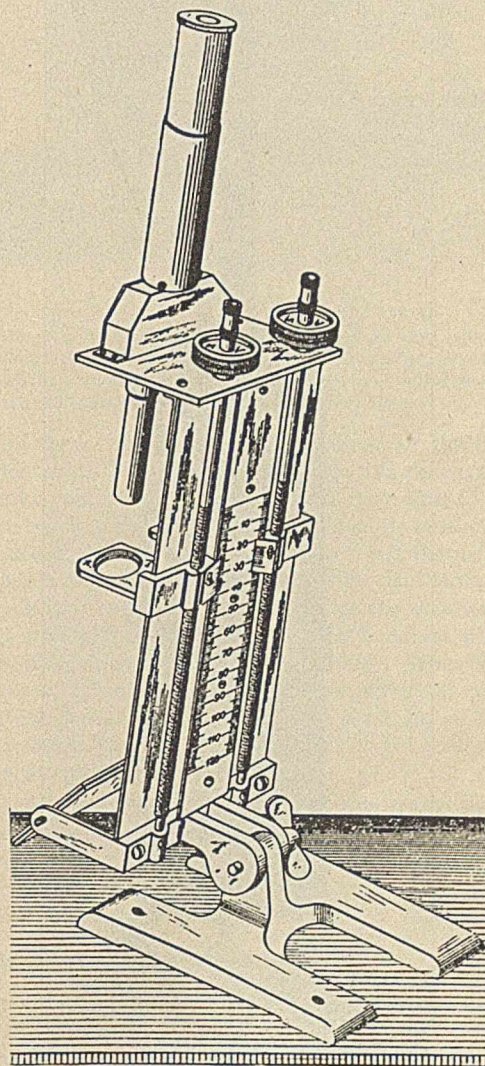


FIG. III

The new nephelometer-colorimeter, showing screw arrangement with adjustable verniers, also the double milled head. There are no open spaces through which dust may enter and light escape when fitted to the lamp house.

While the application of nephelometry is comparatively simple, the correct condition for producing and keeping the colloidal suspension, the fundamental condition of nephelometry, is by no means a simple matter, in spite of Harry Jones' statement<sup>1</sup> that "the colloidal solution or at most the colloidal suspension is the natural condition of solid matter when first formed as the result of a reaction."

<sup>1</sup> "New Era of Chemistry," p. 248. Published by D. Van Nostrand Co., New York City.

Although time does not permit us to consider the details of producing suspensions and the theory of nephelometry, which I hope to present in a special future communication, a few words and an illustration will help to make clear and emphasize the difference between theory and practice, and give an example of the unexpected difficulties encountered in developing nephelometric methods.

Silver chloride has been estimated as a suspension for a correction in atomic weight work since Mulder's time in 1859, yet the production of nephelometric suspensions of silver chloride is as yet far from satisfactory.

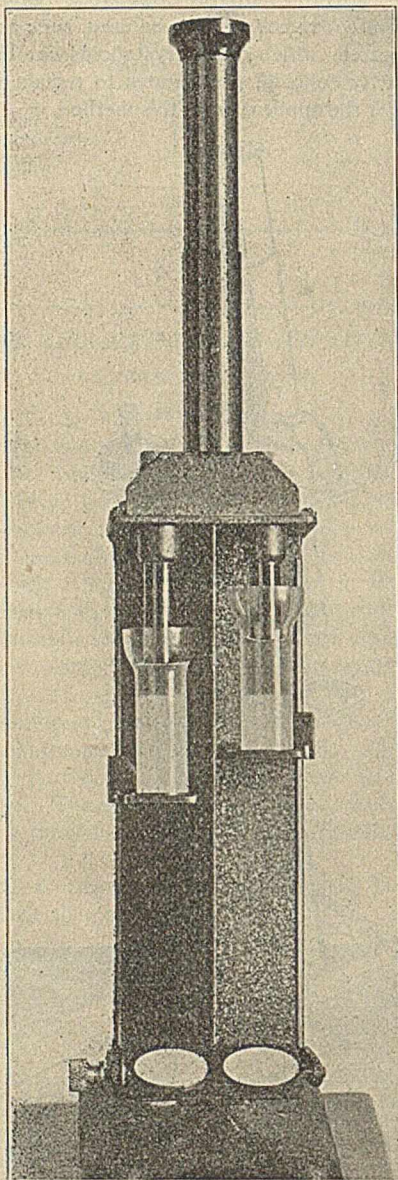


FIG. IV

The instrument used as a nephelometer, viewed from in front, in the direction of light; showing standard solution on the right, unknown ready to be matched on the left.

It has been assumed by the few adverse critics of nephelometric work, that when chlorides and an excess of silver nitrate are present in dilute solution, silver chloride must be formed, and, being one of the most insoluble substances, it must be in the form of a precipitate, and, therefore, impart reflected light. If the amount of light was changed or was not up to what was expected from previous experiments, the critics of nephelometry assumed that the light-reflecting properties of silver chloride changed. On this assumption they have made the sweeping conclusion

that unless both the standard and the unknown were treated exactly the same and in the same medium, a difficult condition to realize, the nephelometric results would be inaccurate, if not valueless. The simpler assumption that a change in the amount of light indicated a change in the amount of precipitate seems not to have been considered at all.

But the following experiment will show that the nephelometric production of silver chloride is not as simple as the formula would indicate it to be. If we take a dilute solution of sodium chloride (0.0005  $N = 0.030$  mg./L) and add silver nitrate in graduated amounts, keeping the volume constant, as shown in Fig. IX, we get silver chloride precipitate in all experiments.

But, as may be seen, the maximum cloud is obtained with one equivalent and the amount of precipitate decreases as the excess of silver nitrate is increased. Our experiments indicate that silver chloride in solution, before it precipitates, forms with silver nitrate an unstable, soluble complex, which slowly hydrolyzes, or decomposes, with the production of silver chloride. By adding the same excess of silver nitrate as in the weakest one, slowly, with stirring, the silver chloride is precipitated before it can form the soluble complex with silver nitrate, and, therefore, the maximum suspension is obtained even though the medium is chemically the same in the end as in the weakest suspension. In diluter solutions this phenomenon is still more marked.

It is interesting to note that Professor Richards, in his atomic weight work, avoided this source of error intuitively by allowing his silver chloride suspensions to come to an equilibrium by standing from 24 to 48 hrs. If our explanation of this phenomenon is maintained by further work, as these experiments would indicate, the adverse critics of nephelometry are without much, if any, experimental basis for their criticism. This experiment, like all heretofore, seems to show that the amount of light reflected by colloidal suspensions within moderate limits, *i. e.*, from the time the particles are formed until they are almost visible to the unaided eye, is a function of the amount of precipitate in suspension.

#### II—APPLICATION

The number of possible applications of nephelometry is too large for consideration here, and, therefore, a few representative estimations have been chosen, *i. e.*, three inorganic and three organic determinations: the estimation, respectively, of ammonia, phosphorus, calcium, acetone, oils and fats, and, finally, proteins. This choice was made not only to cover as broad a field as possible, but also to bring out the nephelometric peculiarities as well, especially the use of protective colloids.

While the "colloidal solution or at most the colloidal suspension is the natural condition of solid matter when first formed as the result of a reaction," as Harry Jones gives it in his book, yet it rarely, if ever, stays in that condition. In most cases it quickly agglutinates and settles, a condition unfavorable to accurate nephelometry. By adding a protective colloid we can delay the agglutination, or, to be more exact, decrease its speed sufficiently to make uniform and constant nephelometric conditions. The protective colloids vary considerably, not only in nature, but also in amount, as will be pointed out in detail in the rest of the paper.

Not all the details of making the reagents and solutions will be given in this paper, as recourse can be had to the original articles on the subject; but for these six analyses, all the steps, more or less, will be mentioned, so that from the six different analyses a clearer judgment may be formed of the value of nephelometric estimations.

(a) AMMONIA ESTIMATIONS—Nessler's reagent for ammonia, developed in the early part of the last century, was applied to water analyses in 1867. It has stood the test of time and has come to be used extensively; but with the development of color-

metry its disadvantages as well as its value have become apparent and innumerable modifications of the reagent have resulted. Its instability and tendency to produce a cloud in dilute solutions are the chief difficulties.

Recently efforts have been made to apply the reagent in micro-Kjeldahl work, without previous distillation, with varying degrees of success; the precipitate due to salts makes the matching of colors, however, extremely difficult.

A probable explanation of why the colored solution produced by Nessler's reagent becomes cloudy, especially in the presence of salts, may be found in the following considerations:

1—Only the iodide complex of mercury and ammonia is highly colored.

2—The other complexes of mercury and ammonia, like the chloride described in this paper, are colorless insoluble compounds.

Therefore, in the presence of the other salts the colored iodide complex is probably partially changed to and in equilibrium with the colorless complexes such as the chloride or sulfate.

On this basis Sara S. Graves<sup>1</sup> developed a sensitive nephelometric reagent for ammonia as follows:

- 80.0 g. Sodium Chloride
- 130.0 g. Water
- 100.0 cc. Saturated Solution of Mercuric Chloride
- Shaking until NaCl is dissolved, then add
- 70.0 cc. Saturated Solution of Lithium Carbonate (1.0 per cent)
- Filter

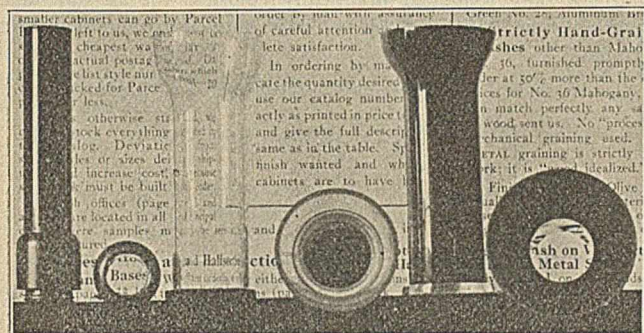


FIG. V

Showing construction and optical clearness of plungers (on the left), of the nephelometric cups (in the center), and colorimetric cups (on the right). The bottoms of all are fused on, though of glass having different coefficients of expansion.

To show the sensitiveness some reagent was added to a liter of water containing 0.006 mg. of ammonia. The cloud which was obtained means that we can detect one part of ammonia in 160 million parts of water.

For the purpose of illustrating how the instrument is used and how the results are calculated, suppose some precipitated standard solutions of ammonia were placed in the instrument. If we had simply added reagent to the known solution of ammonium sulfate, the precipitate would have become agglutinated before the observations were finished. Therefore, just before adding reagent a protective colloid is added. In this case for every 10 cc. of ammonium sulfate solution, 15 cc. of a 0.003 per cent solution of starch will keep the suspension from agglutinating and settling for about an hour. The standard solution contains 9.43 mg. ammonium sulfate, which is equivalent to 2.43 mg. ammonia, or 2.00 mg. nitrogen, per liter. When precipitated with 5 cc. of reagent the resulting suspension is put into both cups of the instrument.

If we put the left cup at any convenient height, say 20 or 25 mm., and move the other cup up or down until the light in the eyepiece, coming from both tubes, is equal, we find that the

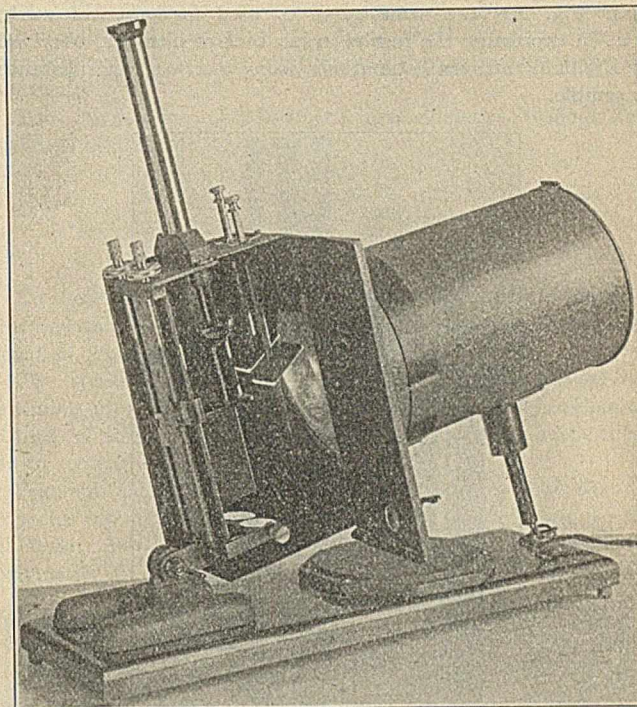


FIG. VI

Showing the instrument with lamp house used as a colorimeter; the adjustable reflectors are reflecting the light to the round instrument reflectors, which in turn transmit the light to the cups and instrument. The light switch can barely be seen on the front of the base.

height on the right is rarely, if ever, equal to the height set on the left side. It is in this respect like the zero point of the analytical balance; it must be determined from time to time, and is seldom constant for a long time. The reading on the right side we will denote as the standard reading,  $S$ , but the actual value on the left is of no consequence and may be considered as a tare, so long as it is constant. If in place of the standard on the right we now put another solution of ammonium sulfate, say 0.900 as strong as  $S$ , we find a higher reading. If we then put in 0.800, 0.700, 0.600 and 0.500 standard strength, we obtain, respectively, a series of corresponding readings. All of these readings except that from the standard  $S$  are indicated as  $Y$  in the curve and formula.

If we plot these readings on cross-section paper we obtain a curve which will be very useful in practical work. In Fig. X we have plotted the readings obtained from such a series of gradationed known solutions of ammonium sulfate solutions and drawn a curve through these points. Algebraically the curve is expressed by the formula  $Y = \frac{S}{x} - \frac{(1-x)Sk}{x^2}$ , where  $k = 0.052$  and  $S = 20.0$ . The lower curve, shown here, is the colorimetric curve where the readings are inversely proportional to the concentrations.

When the instrument changes so that a restandardization is necessary, the nephelometric formula obviates considerable work, especially the readings for and the drawing of a new curve.

Since the formula is complicated and many prefer to do without mathematical calculations, the following scheme can be used: The instrument is standardized as before, but the curve is used alone in getting the amount of substance equivalent to the readings. When the value of the standard readings changes, due to any change in the instrument, the height of the solution on the left side, the one used as a tare, is adjusted so that the original reading for  $S$  is obtained and, therefore, the original curve is applicable. It is equivalent to changing the zero point of a balance by adjustment so as to avoid calculation.

<sup>1</sup> *J. Am. Chem. Soc.*, 37 (1915), 1171.

In Fig. XI, the same curve is used for practical work, *i. e.*, in expressing the results in per cent of nitrogen, obtained in Kjeldahl nitrogen estimations, using 0.1000 g. of substance or sample.

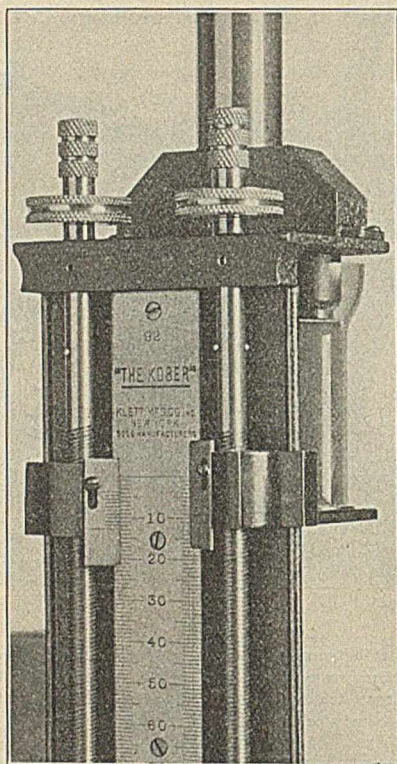


FIG. VII—SHOWING SCALE AND ADJUSTABLE VERNIERS

The one on the right shows the plunger touching bottom of cup, thereby giving more zero or color, with vernier in right position, *i. e.*, exactly on zero. The verniers can be put on zero by loosening the screw of vernier and moving vernier to right position after plunger touches the bottom of the cup, or its equivalent.

Suppose we had a Kjeldahl flask containing 20 cc. concentrated sulfuric acid, a little mercury or mercuric oxide as catalyzer and 0.1000 g. ammonium sulfate. As far as the nephelometric method is concerned, this could as well be the result of digesting an equal weight of almost any nitrogenous substance, such as a sample of fertilizer, leather, rubber, food, beer; any synthetic or natural organic substance, as aniline, dyes, drugs, etc. The melt is now dissolved in water and made up to 500 cc. Although the heat of dilution of sulfuric acid is sufficient to warm the solution considerably, we do not need to bring it to room temperature, provided, of course, we pipette an aliquot portion off at once at the same temperature. Any error due to high temperature in the flask will then be compensated by the same error in pipette and thereby automatically eliminated.

To 5 cc. of this solution are then added 5 cc. of *N* NaOH and then drop by drop of the same solution until it is neutral to litmus paper. The solution is then diluted to 200 cc. with ammonia-free water, and a 10 cc. portion is treated with 15 cc. of 0.003 per cent starch solution, precipitated with Graves' reagent (5 cc.), and matched against 10 cc. of standard solution (containing 2.0 mg. of nitrogen per liter) similarly treated. From the reading and the curve we can find out at once the exact per cent of nitrogen. In the first step we diluted to 500 cc.; and in the second step, when we took 5 cc. of a 200 cc. final volume, we diluted 40 times, thus giving a total volume of 20,000 cc., or 20 liters. If the standard reading *S* were 20.0 mm. we would get for the unknown 39.10 mm., which would give us 21.36 per cent nitrogen<sup>1</sup> in ammonium sulfate.

<sup>1</sup> If the reagents contain ammonia a control estimation, using about  $\frac{1}{20}$  the dilution of above, will give a correction, *i. e.*, a certain fraction of per cent to be deducted from the per cent of total nitrogen.

Not only will this method save considerable time, but it will eliminate the expense, attention and errors connected with a bank of Kjeldahl stills.

(b) PHOSPHORUS—The estimation of phosphorus in biological and industrial fields, especially of small amounts, is becoming of increasing importance. A large number of volumetric and colorimetric methods have been proposed but thus far none have been satisfactory for micro-quantitative work. While looking for a suitable nephelometric precipitant for phosphorus, Kober and Egerer's attention was called to the reagent developed by Pouget<sup>1</sup> and Chouchak. This reagent consists of a nitric acid solution of strychnine and molybdic acid and produces a very sensitive reaction with phosphates. Pouget and Chouchak found that it will detect one part of phosphorus in 20 million parts of water. As the precipitate is slightly yellow and remains in suspension for a long time, the authors have recommended it for a colorimetric reagent, but really the estimations were turbidimetric, *i. e.*, they measured the absorbed light of the suspension.

On studying the reaction carefully, Egerer and Kober found that (1) it was not constant and quantitative, and (2) the reagent gradually became yellow and deteriorated, probably because of the action of nitric acid. Pouget and Chouchak realized this since their directions state that the mixing of constituents must be made only just before using. After making many variations of all constituents, no marked improvement was obtained, but on substituting hydrochloric for nitric acid, the solution not only remained practically colorless for an indefinite length of time, but was stable and gave quantitative and constant results.

#### VARIATIONS IN DIAPHRAGMS OF COLORIMETERS AND NEPHELOMETERS

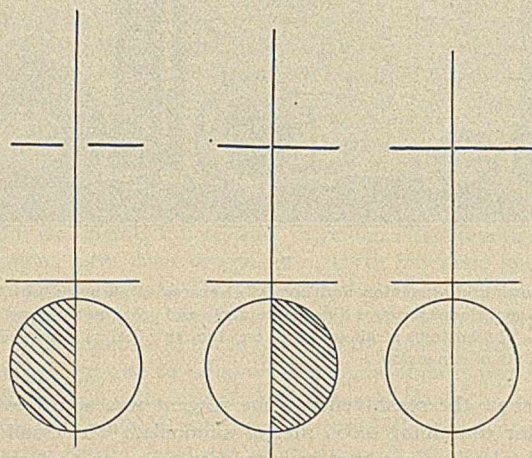


FIG. VIII

Showing a wide aperture as found in most instruments (on the left), next a pinhole aperture off optical center. The pinhole aperture (to the right) in correct position.

The directions for making Kober and Egerer's nephelometric reagent for phosphorus<sup>2</sup> are as follows:

150.0 g. Sodium Molybdate  
250.0 cc. Water  
100.0 cc. Hydrochloric Acid (1-1)  
Add slowly with shaking  
150.0 cc. Strychnine Sulfate Solution (2.0 per cent)  
Filter

Protective action in addition to any produced by the strychnine does not seem to be necessary. The reagent is so sensitive that ordinary filter paper cannot be used for filtering the reagent, as it extracts from it a substance which seems to be a phosphorus compound, gradually giving a very marked phosphorus-reaction.

An idea of its sensitiveness may be obtained by the following:

<sup>1</sup> Bull. soc. chim., 5 (1909), 104; 9 (1911), 649.

<sup>2</sup> J. Am. Chem. Soc., 37 (1915), 2375.

experiment: A solution of potassium phosphate, containing 0.0125 mg. of phosphates, 0.003 mg. phosphorus per liter, still shows the reaction very plainly which means one part in 333 million parts.

As an example of its usefulness for technical purposes the determination of phosphorus in a sample of cast iron will be described briefly. The weighing of the sample, its solution in nitric acid, and the elimination of the nitric acid by boiling a few minutes with an equal volume of sulfuric acid require but a few minutes for completion.

The actual details are as follows:

Two (2.00) g. of cast iron borings were dissolved in 100 cc. of  $\frac{1}{3}$  nitric acid by boiling. After cooling under the tap the solution was brought up to 100 cc. and 5 cc. taken and boiled for 2 min. with 5 cc. of concentrated sulfuric acid. This mixture is then diluted to 100 cc. and filtered through a dry, acid-washed filter paper and 25 cc. are again diluted to 100 cc. 10 cc. are now treated with 35 cc. of 0.5 *N* HCl and precipitated with 5 cc. of reagent. On matching in the instrument with a standard containing 5 mg. of  $\text{KH}_2\text{PO}_4$  per liter the percentage<sup>1</sup> of phosphorus can be obtained from a curve made similarly to one shown with ammonia.

(c) CALCIUM—The estimation of calcium, owing to its wide distribution, is of considerable importance, and, therefore, simple and rapid methods of estimation would be very valuable. Lyman first sought to solve this problem by the nephelometric estimation of calcium oxalate, but was not able to obtain satisfactory results. He then tried, after a preliminary separation of the magnesium, to precipitate the calcium as a calcium soap. This method gave excellent results. It reduced the time required in certain calcium estimations from 3 days to 2 hours for a set of four estimations.

Lyman's nephelometric reagent for calcium<sup>2</sup> is made up as follows:

4.0 g. Stearic Acid  
0.5 cc. Oleic Acid  
400.0 cc. Alcohol (95 per cent)  
Boil, add  
20.0 g. Ammonium Carbonate in  
100.0 cc. Hot Water  
Boil, cool, add  
400.0 cc. Alcohol (95 per cent)  
100.0 cc. Water  
2.0 cc. Ammonium Hydroxide (sp. gr. 0.90)  
Filter

To show its sensitiveness the following experiment is made:

A solution of calcium oxalate in nitric acid containing 0.2 mg. of calcium per liter still gives a decided reaction, which is one part in 5 million.

For the estimation of calcium in milk, 10 cc. are diluted to 100 cc. with distilled water. Five cc. of this mixture are then treated with 15.0 cc. of 6.5 per cent trichloroacetic acid, which precipitates the proteins, and filtered. The calcium is now precipitated as oxalate by McCrudden's method,<sup>3</sup> redissolved, and the calcium determined after precipitation as soap, nephelometrically.

Twenty cc. of standard calcium oxalate solution (dissolved in nitric acid) containing 0.4 mg. of calcium are poured into 50 cc. of Lyman's reagent and gently shaken. This cloud is used for matching an unknown treated in a similar manner. Here, as in the other estimations, only a few circumventions are necessary to eliminate interfering substances.

In this precipitation oleic acid acts as a protective colloid, a small amount delays the agglutination for hours.

(d) ACETONE—Marriott has applied the extremely sensitive Scott-Wilson reagent for the nephelometric estimation of ace-

tone. The acetone in each case may be distilled from the original solution into the reagent or may be aerated<sup>1</sup> at room temperature into sodium bisulfite solution, and then estimated nephelometrically.

The composition of Marriott's nephelometric reagent for acetone<sup>2</sup> is given as:

10.0 g. Mercuric Cyanide  
180.0 g. Sodium Hydroxide  
1200.0 cc. Water, shaking, slowly add  
400.0 cc. Silver Nitrate (0.73 per cent)  
Filter

To show the sensitiveness of the reagent, a solution of freshly distilled acetone containing 0.010 mg. per liter still gives a marked reaction, which is one part in 100 million.

The standard and unknown solutions are precipitated by distilling into 50 cc. water and 15 cc. of Marriott's reagent, 0.5 mg. of acetone and finally making the solution or suspension up to 100 cc.

Standard solution if made with  $N/4$  sulfuric acid so that it contains 0.5 mg. of acetone in 10 cc. will keep for a few weeks at least. Without  $N/4$  acid the acetone solutions quickly polymerize.

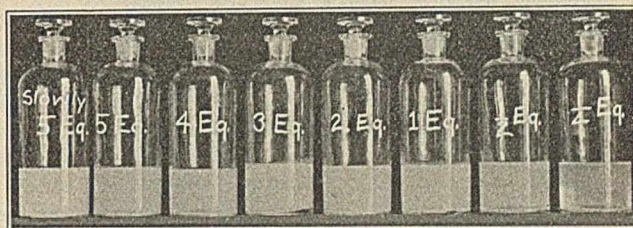


FIG. IX—SILVER CHLORIDE SUSPENSIONS

The one on the left made by slowly adding 5 Eq.  $\text{AgNO}_3$  solution with shaking or stirring. All the other equivalents were added at once. Unfortunately, the large volumes photographed, which are more or less opaque, make it difficult to see the differences, except where it shows the curvature of the bottoms.

As may be observed no additional protective colloid is added, as the organic nature of the complex is sufficient protection or, to be exact, makes the speed of agglutination low.

(e) FATS AND OILS—In quantitative work where the precipitate is easily thrown down, easily washed, and easily dried and weighed, gravimetric analysis imposes no difficulty, although it may fail to estimate small amounts. Fats and oils are difficult to filter and to free from solvent and, therefore, Bloor devised a nephelometric method which overcomes these obstacles. The fat or oil is extracted with an alcohol-ether mixture and then poured into water, when the fats or the oil separate out in fine globules or suspensions. Bloor's nephelometric reagent for fats<sup>3</sup> is made up as follows:

250 cc. Redistilled Ether  
750 cc. Redistilled Alcohol  
As accessory  
500 cc. Normal Sodium Ethylate

Five-hundredths of a milligram (0.05 mg.) of fat can be easily determined quantitatively, and a marked cloud is produced by one part in a million.

The usual standard and unknown are precipitated by running 5 cc. alcohol-ether solution containing 2.0 mg. of fat or oleic acid into 100 cc. of water and, after adding 10 cc. of 1 : 4 HCl and gently stirring or shaking and allowing to stand for 5 min., are read in the nephelometer.

Murlin and Riche<sup>4</sup> have modified this method by pouring the fat solution into 0.05 per cent gelatin solution.<sup>5</sup> These authors

<sup>1</sup> Folin, *J. Biol. Chem.*, **18** (1914), 263.

<sup>2</sup> *Ibid.*, **16** (1913), 289.

<sup>3</sup> *J. Am. Chem. Soc.*, **36** (1914), 1300; *J. Biol. Chem.*, **17** (1914), 377.

<sup>4</sup> Private communication.

<sup>5</sup> 1 g. gelatin in 2 liters of water with 5 cc. glacial acetic acid.

<sup>1</sup> A check on the purity of reagents should also be made by a blank estimation and corrections made if necessary.

<sup>2</sup> *J. Biol. Chem.*, **29** (1917), 172.

<sup>3</sup> *Ibid.*, **10** (1911), 187.

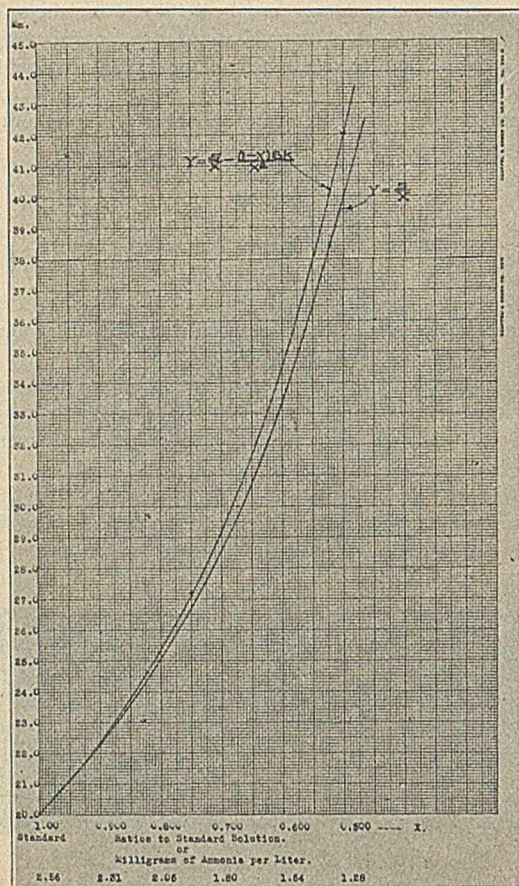


FIG. X

Showing a nephelometric curve (upper) and a colorimetric curve (lower). The abscissas give the concentrations of substance, in this case of ammonia, coordinates give the readings, *i. e.*, the heights of solutions. Greater accuracy can be obtained by drawing curve to a larger scale.

found that the addition of this small amount of protein as protective colloid delayed the coalescence of the fat globules and thereby enabled them to keep the suspensions photometrically constant for hours.

Woodman, Gookin and Heath<sup>1</sup> have worked out a similar method for the essential oils.

The solvent and extracting medium for essential oils is alcohol alone and 5 cc. of the standard solution—containing 100 mg. in 100 cc. 95 per cent (redistilled) alcohol—are precipitated by pouring into 25 cc. of water, or better 25 cc. of 1 : 4 HCl.

By adding acid as used in the Bloor method, I found that one could use a much weaker standard than the authors recommended and still get suitable nephelometric clouds. They were able to estimate the oils of roses, peppermint, anise, and nutmeg nephelometrically with ease and accuracy.

(f) PROTEINS—Proteins like other colloidal substances are extremely difficult to filter, and when filtered are rarely free enough from adsorbed substances to be useful for gravimetric work. Also they are dried only with difficulty. The application of nephelometry to protein estimations has been found to be fortunate for two reasons: (1) It enables us to estimate small amounts of proteins easily since nephelometry fits protein suspensions as a key does a lock; (2) because protein suspensions were so easily produced and maintained that the possibility of nephelometry as an accurate method of analysis became apparent, just as the difficulties mentioned with silver chloride prevented, without much doubt, the adoption of nephelometry for twenty or more years. The former give a true picture and the latter a false picture of its possible accuracy and general

usefulness. Kober's nephelometric reagent for coagulable proteins<sup>1</sup> consists of:

2,000.0 cc. Sulfosalicylic Acid  
For Casein, 0.3 per cent  
For Globulins, 0.6 per cent

Time does not permit me to go into all the details of protein estimations, so only a test of its sensitiveness will be given.

A protein solution containing 1.0 mg. of protein per liter still gives a marked test which is equivalent to 1 part in a million.

In milk the fat is first removed, by adding to the diluted milk (5 cc. of milk in about 200 cc. of water) 10 cc. *N*/10 sodium hydroxide, making it up to 250 cc. and shaking with ether. As further details will be found in the original communication,<sup>2</sup> nothing more will be said except that the nephelometric method reduced the time for the estimation of casein, globulin, and albumin in milk. It requires from 2 to 3 days, if it is done according to the usual technic, whereas with the nephelometric method it can be done in 20 to 30 min.

### III—GENERAL DISCUSSION

There are many more applications which have not been touched upon, but their development and their application to analyses do not differ essentially from those just given.

It will be observed that practically all of them are of the nature of an organic complex, *i. e.*, the precipitate has an organic constituent in its composition. In gravimetric analysis this would not be an advantage, since it is necessary as far as possible to have the precipitate in relatively large masses for filtering and in a condition easy to dry. In many cases the occlusion and adsorption in gravimetric precipitates is very appreciable.

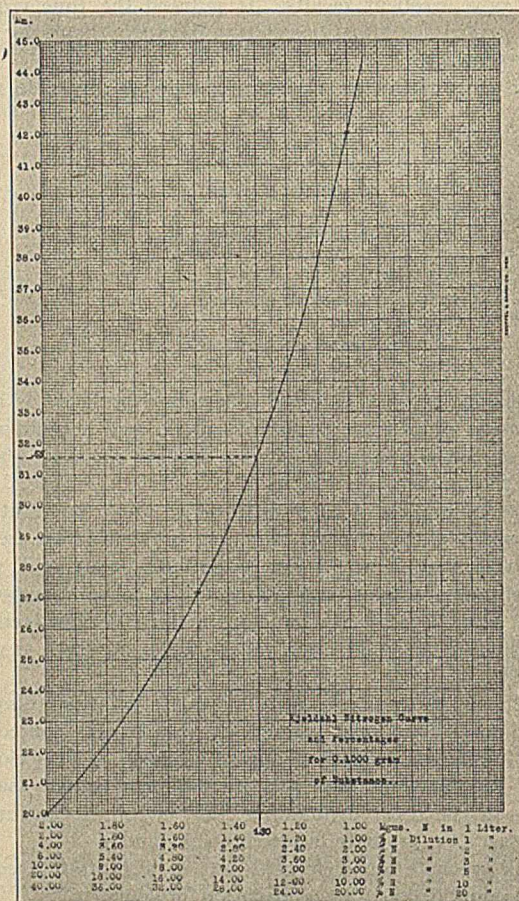


FIG. XI

Showing a similar nephelometric curve but with the abscissas giving the per cent of nitrogen per 0.1000 g. of sample. Greater accuracy can be obtained by drawing curve to a larger scale.

<sup>1</sup> *J. Am. Chem. Soc.*, **35** (1913), 290.

<sup>2</sup> *Ibid.*, **35** (1913), 1589.



In nephelometry, however, occlusions are of no consequence as the standard, having practically the same occlusion or adsorption, would cancel the error. Of course, it does not follow that occlusion or adsorption in nephelometric precipitates causes any difference in the amount of reflected light, as the occlusion may be in a dissolved phase, and, therefore, would not reflect light. Furthermore, as organic reactions as a rule are more specific, and, therefore, more accurate, nephelometry and colorimetry enable us to make use of organic substances, which means that our sources of reagents and possible methods are unlimited.

The great advantage of nephelometric analysis over gravimetric, outside of the enormous saving in time and labor as well as in material, is in its more complete control over the media of precipitation. By the great dilution necessary ordinarily in nephelometry, color and other interfering substances are so reduced that they seldom play a rôle.

The accuracy obtainable in nephelometry, as the instruments and methods steadily improve in efficiency, is growing rapidly. With the instrument, as it now is, when properly adjusted and used, it is not difficult to obtain an accuracy of 0.25-0.50 per cent in a single reading, and with two to three readings 0.1-0.2 per cent. Not all nephelometric suspensions have been developed, however, so that the maximum accuracy of the instrument is utilized. As in gravimetric work, the accuracy obtainable in final results depends on the particular method and precipitant, as well as on the balance. As we have become so accustomed to gravimetric work, to agglutinate or crystallize our precipitates, it is difficult for most of us to consider doing our work in an exactly reverse way. Like all other methods it takes time to get accustomed to a new way of doing things, no matter how good or convenient it is. Much more progress is made when the new way is the only way an experiment can be done at all. We all know when once an old method has been entrenched, as it were, by custom, it takes considerable time for the new to find its way into favor, irrespective of its merits.

Much more work remains to be done, especially the study, from as many angles as possible of the quantitative production of suspensions.

Almost all colloidal chemistry, heretofore, the theoretical or practical study, especially that of colloidal suspensions, has been qualitative and not quantitative. We need a great deal more information about quantitative colloidal chemistry for nephelometry to find its greatest usefulness.

How many more applications nephelometry will find in applied and quantitative chemistry the future alone will tell. This much we are sure of: the nephelometric method must be used for colorless colloidal suspension and for the accurate determination of amounts of material which give no delicate color reaction and are too minute to filter, but which are daily commanding our interest and attention. The use of the method has already been extended to all classes of substances and since, by careful work, considerable accuracy can be obtained, its application promises to be general in the different branches of chemistry.

#### SUMMARY

To sum up, attention has been called to the following:

I—A few erroneous conceptions about nephelometry and the pitfalls of many beginners in nephelometry.

II—The great sensitivity of:

(a) Graves' nephelometric reagent for ammonia, which is able to detect 1.0 part of ammonia in 160 million of water and its usefulness in various tests and Kjeldahl estimations.

(b) Kober and Egerer's nephelometric reagent for phosphorus which will detect 1.0 part in 333 million of water and its usefulness in various tests and in phosphorus estimations applied to iron and steel as illustrated by an experiment.

(c) Lyman's nephelometric reagent for calcium which will detect 1.0 part in 5 million of water and its usefulness in various tests and calcium estimations applied to water and milk.

(d) Marriott's nephelometric reagent for acetone which will detect 1.0 part in 100 million of water and its usefulness in determining acetone quantitatively applied to various distillates.

(e) Bloor's method for estimating fats and oils with which 0.05 mg. of fat can be estimated quantitatively and the presence shown of 1.0 part of fat in a million of water, its usefulness in nephelometric fat estimation in milks, and its application in Woodman, Gookin, and Heath's nephelometric estimation of essential oils.

(f) Kober's nephelometric method of estimating proteins which will show the presence of 1.0 part of protein in a million parts of water.

III—The advantage of nephelometry, its possible accuracy, its enormous field, and its possible future.

DIVISION OF LABORATORIES AND RESEARCH  
STATE DEPARTMENT OF HEALTH  
ALBANY, NEW YORK

### MUNICIPAL CONTRIBUTION TO CONSERVATION THROUGH GARBAGE UTILIZATION<sup>1</sup>

By EDWARD D. VERY

The word "garbage" is commonly used indiscriminately to define any kind of refuse and is generally considered as a synonym for "offal," but for the purpose of this paper it is used to define the waste of both animal and vegetable matter which results from the preparation of food for human consumption. It is further limited to that portion of this class of waste which is collected and disposed of as a municipal function.

When we take into consideration the fact that this material results from action governed by no set rule or regulation and is, in fact, subject only to the whim of the individual, there would seem to be no general statement possible as to the constituents of garbage, and yet it is found that this class of material, as it is collected in different parts of the country, does not vary widely in quantity per capita, weight per unit of volume, or in mechanical or chemical analysis, and it is possible, within reasonable limits, to make a statement of the average content of garbage.

The average quantity of garbage produced per capita per day is one-half pound.

The average weight of garbage is 1100 lbs. per cu. yd.

The average sample of garbage contains:

16 per cent animal matter  
79 per cent vegetable matter  
5 per cent rubbish

and it analyzes, approximately:

70 per cent moisture  
20 per cent tannage  
3.5 per cent grease  
1.5 per cent bones  
5 per cent rubbish

Published analyses from the cities of New York, Cleveland, and Washington show this comparison as follows:

	NEW YORK	CLEVELAND	WASHINGTON
Pounds per capita per annum....	181	193	286
Pounds per cu. ft.....	41	49	47
Moisture, per cent.....	67	76	74

#### LABORATORY ANALYSES

	Per cent	Per cent	Per cent
Ether extract.....	4.85	4.00	5.00
Phosphoric acid as P <sub>2</sub> O <sub>5</sub> .....	0.59	0.24	0.39
Nitrogen, Kjeldahl method....	0.94	0.64	0.71
Potash as K <sub>2</sub> O.....	0.33	0.30	0.28

The figure here given for the pounds per capita per annum for Washington seems at variance with the others, but it must be remembered that this city has an enormous floating population which produces garbage but is not considered in the census. Then, too, in Washington practically all of the hotels and res-

<sup>1</sup> Read before New York Section, American Chemical Society, May 10, 1918.

taurants have their garbage collected by the municipality, whereas in some cities the garbage is not so collected, but is taken by private collectors, under an arrangement with the proprietors of these establishments.

The quantity of garbage produced varies by seasons very materially, the maximum daily amount being collected during the summer months when vegetables and fruits constitute the principal diet, and the minimum amount being collected during the winter months. Usually August is the highest month, with about 11 per cent of the total annual amount, and February the lowest month, with about 6 per cent. The moisture content is high in percentage in the summer months and low in winter, whereas the grease content and the chemical plant food values are low in the summer and high in winter, for obvious reasons.

Fresh garbage, as it reaches the can, will remain, in ordinary weather, at a temperature of about 70° F. for from 12 to 14 hrs. before any change takes place. From that time alcoholic fermentation sets in and this will continue for another period of, approximately, 12 to 14 hrs. If the can is loosely covered, acetic acid fermentation develops, but if cans are fairly well closed, the alcoholic fermentation continues for about 36 hrs., when there is practically no further action. There is no decomposition of the animal matter, as that is inhibited by the alcoholic fermentation. By test it has been found that in garbage which has remained in the can under ordinary temperatures for from 3 to 4 days, and even as long as 21 days, the free fatty acids of the grease are not more than from 1.5 to 7 per cent, whereas where matter of a like nature is subjected to putrefactive action, the grease analyzes from 30 to 40 per cent of free fatty acids, which indicates the absence of decomposition in ordinary garbage as it is contained in the can.

The fermentation noted develops small amounts of alcohol and acetic acid, with slight changes in the vegetable oils, but none in the animal oils.

The sour odor of garbage is the result of this fermentation developing acetic acid, together with certain fruit esters, aldehydes, and alcohol.

We have then a material made up of a combination of moisture, fat, starch, sugar, albuminoid and nitrogenous bodies, cellulose, and ash. The problem is to recover from this mass whatever may be of value, in the most economical manner, having at the same time due regard for the sanitary requirements.

Disregarding the method of final disposition of municipal wastes which attempt no reclamation of by-products, we find the methods which have been adopted for the treatment of garbage, as herein defined, are pig feeding, or some mechanical treatment commonly known as reduction.

According to the latest statistics, there are in the United States, 75 cities of 90,000 population and upwards; of these, 13 resort to dumping or burial, 21 have incinerators, 11 feed to pigs, 6 sell or give to farmers, probably to be used for pig feeding, and 24 use a reduction process.

Those who have reduction plants are:

Boston, Mass.	Baltimore, Md.
New Bedford, Mass.	Washington, D. C.
Bridgeport, Conn.	Akron, Ohio
Buffalo, N. Y.	Cincinnati, Ohio
New York, N. Y.	Cleveland, Ohio
Rochester, N. Y.	Columbus, Ohio
Syracuse, N. Y.	Dayton, Ohio
Utica, N. Y.	Toledo, Ohio
Philadelphia, Pa.	Indianapolis, Ind.
Pittsburgh, Pa.	Chicago, Ill.
Reading, Pa.	Detroit, Mich.
Wilmington, Del.	Los Angeles, Cal.

The feeding of garbage to pigs is a matter which caused considerable discussion, both as to its economical phase and also as to its propriety from the standpoint of sanitation.

Tersely, the advantages of pig feeding have been stated as "using the pig as a middle-man, we find that 25 per cent of the

protein and 45 per cent of the total energy is returned to the consumer as pork. The pig charges 55 per cent brokerage for converting vegetable protein and fat into animal protein and fat."

Of course, we must remember that there is a considerable portion of the garbage that the pig will not consume, such as fish scrap, fruit skins, coffee grounds, and miscellaneous rubbish. Then, too, the garbage must come to the piggery fresh, especially in the summer time.

Again, there is the menace of hog cholera, or the foot and mouth disease, which may cause the total loss of "plant." To be sure, modern science is ready with methods of prophylaxis, which may prove a safeguard, but there is always the peril of slipshod methods, which result in a partial or complete loss.

The United States Food Administration, in a booklet entitled "Garbage Utilization," issued in February 1918, says:

"We have indicated that the reduction process is hardly suitable for cities of under 100,000 population. A proper question would be, is pig feeding more applicable to cities now reducing than the reduction process?"

"In so far as the monetary return applies, the two methods are practically identical. The reduction process possibly has the advantage of improvement to a greater extent than pig feeding. By improving the breed of the hog, gains might be made more economically, but the reduction process requires only simplified machinery, or additional recovery to make a ton of garbage more valuable.

"It also seems that the larger the city the less adapted its garbage to pig feeding. One might say that smaller cities were better managed, but it is obvious that the difficulties of controlling materials placed in the garbage increase more rapidly than does the population. Although a pig is blessed with a digestive system capable of assimilating almost anything, its efficiency cannot be compared with the mechanical digestors of the reduction plants.

"While from a purely conservation standpoint, pork production may seem more important than the production of grease and fertilizer tankage, the use of the grease recovered releases an equivalent amount of edible oils, while our stock of agricultural fertilizers is so depleted, at the present time, that fertilizer tankage is a national resource not to be overlooked.

"The test of the practicability of the feeding method of disposal is the selling possibilities of the pork produced. There is no benefit in feeding if the pork is unfit for food, or if a popular prejudice will prohibit it from selling freely.

"We have not been able to find any market where garbage-fed hogs are being sold at a lower price than grain-fed animals.

"There is at present a mistaken idea throughout the country as to the value of garbage. In a large number of cases it is a question whether the value will be sufficient to pay the cost of collection and transportation. Where collection and disposal both are to be made by the contractor, we doubt if the work will be done without cost to the city, unless the quality of the garbage is exceptional, and there is keen competition for the material.

"The ratio of 1 lb. of marketable pork to 50 lbs. of garbage has already been established. With pork on the hoof at 15½ cents, this would give a gross feed value of \$6.20 to a ton of garbage. In a general way, it is safe to assume that the cost of disposal, after the farm is reached, including overhead charges at the farm, would not exceed \$3.00 per ton."

Reduction processes now in use in this country are of three types, the drying method, the cooking method, and the Cobwell process.

In the drying method the green garbage is first deposited on a floor or belt to facilitate the picking out of the bottles, tin cans, and similar refuse. The material is then fed into a disintegrator, either of the chopping or the crushing type, wherein the garbage

is ground to a fairly uniform size throughout, to allow of efficient drying and percolating action. From the disintegrator the material passes through a direct heat dryer so that the moisture content may be reduced. It is then fed into a percolator, in which, by the action of a solvent, grease is taken out and reclaimed, leaving a resultant tankage fairly free from both grease and moisture.

The dryer consists of a revolving cylindrical steel shell, fitted with lifting vanes, which keep the material moving toward the discharge end and at the same time draw it to the top of the cylinder and drop it in a film through the hot gases, which are passing through the dryer from a furnace to the stack.

The percolator may be either of two styles, a stationary vertical or a horizontal rotary.

The former consists of a cylindrical tank, into which the garbage is fed through an opening at the top. When filled sufficiently, a heated solvent is introduced through perforated pipes at the top, and this solvent percolates through the mass and is withdrawn at the bottom, taking with it the grease which it has absorbed in its passage. This introduction of solvent is repeated until the outgoing liquid appears to carry too little grease to warrant further attempt at recovery. Live steam is then introduced into the percolator at the bottom, in order to drive off, so far as possible, the solvent which has been taken up by the tankage. The tankage is then taken out of the percolator, through a door near the bottom, by means of a hoe, rake, or some similar tool.

Results from the stationary vertical percolator are unsatisfactory, principally because the solvent jets usually form channels through the material by which the remaining solvent finds its way to the bottom without having been in contact with a considerable portion of the material being treated, thus resulting in an incomplete recovery of the grease. Similar channels are formed by the steam, thus giving an equally incomplete result in the driving off of the retained solvent.

The horizontal rotary percolator consists of a cylinder, having three openings on one side for receiving and discharging the tankage. There are three pipe sections running the full length of the cylinder inside, perforated for the spraying of the heated solvent on to the material being treated. There is also a burlap-covered strainer-plate, made of brass cloth, extending the full length, and so supported as to form a chamber beneath, into which the saturated solvent, after passing the strainer, passes to an outlet at one end of the cylinder. There are vapor lines for the introduction of the steam.

The operation of this percolator is as follows:

The percolator is charged by feeding the material to be treated through the three charging openings. The covers of these openings are then put in place and securely bolted. The cylinder is then revolved a one-quarter turn, thus bringing the solvent pipes to the upper side and the strainer chamber to the lower side. Heated solvent is then pumped into the cylinder in sufficient quantity to fill all the voids and to completely submerge the material. This solvent is allowed to stand for about 10 min. and is then withdrawn by pumps, after passing through the material, becoming thoroughly saturated, and passing through the strainer. This operation is repeated until the outflowing saturated solvent appears to carry insufficient grease to warrant further attempt at reclamation. After the final washing and after all solvent possible has been withdrawn by the pumps, live steam is introduced into the cylinder under a pressure of from 15 to 20 lbs. per sq. in. This pressure forces out the major portion of the remaining solvent through the strainer. The cylinder is then revolved so that the three openings are at the bottom and the tankage is emptied through them onto the floor or into a conveyer.

The saturated solvent from the percolator, in either case, is piped to a still, wherein, by the application of heat, the solvent is driven off as a gas, leaving the grease to be withdrawn, separated from any water present by gravity, and then barreled for shipment. The vaporized solvent is then condensed and re-used.

The last two washings are usually used for primary washing in succeeding treatments because they are imperfectly saturated.

The tankage from the percolator is passed through a dryer and thence to a screen and that portion which fails to pass through the screen is taken to a grinder and pulverized and again sent to the screen.

This method is practically the first used in this country and is still in use in Chicago, Allegheny City, and Buffalo. It is the cheapest when first cost is considered, but the results are in proportion to its cost of installation, and so, while as an investment it may prove satisfactory, it still does not give the full possible recovery of grease. In the drying of the material, carbonization takes place, which affects the quality of the grease and tankage adversely, and also in this part of the process there is a very considerable loss of ammonia.

This method is operated with a considerable resultant nuisance from odors because of the great volume of gases passing off from the dryers, which cannot be economically deodorized.

According to test made in Chicago, which is probably representative, by this process, 46 lbs. of grease per ton were recovered.

In the cooking method the garbage is picked over for the removal of the glass, tin cans, etc., and is then fed into large vertical tanks, known as digestors or autoclaves.

These tanks, when filled, are sealed by a bolted cap, a small quantity of water is added, live steam is introduced and the mass is cooked, under pressure, until the whole has become a pulpy mass, with the grease sacs ruptured and the entrained grease released. The material is then pressed to express what moisture can be mechanically released, the material then passes to the dryer, thence to the percolator, to the redryer, the screen, and then to storage.

The expressed moisture is piped to a settling tank or basin, wherein the grease separates by gravity, is skimmed or pumped off and placed in tanks to allow the final separation of the grease from whatever slight amount of water is present, and then the grease is drawn off and barreled.

These types vary usually and only in the manner of pressing.

In one type the pressing is done in the tank, there being a perforated plate fitted just above the bottom, and pressure is obtained by the introduction of live steam at the top, resulting in the material being compressed and the expressed liquid being forced through the perforations to the pipes leading to the settling tanks or basin.

Another uses a roller press, having an endless perforated metal belt, which, after receiving the material, passes between rollers, causing the fluid to pass through the perforations to the pipes leading to the settling device.

Another uses the old cider press, wherein the mass is fed into forms made up of slats covered with burlap, which are built upon a car and are then placed under a hydraulic ram, causing the expression of the liquid through the burlap.

Then there is the curb press, wherein the material is fed into a latticed steel basket, which later is run under a hydraulic ram.

The steam cone press is also used. This press is in the form of two bottomless cones, lying horizontally, with their bases meeting, and fitted with a bottom of perforated plates. After the material has been fed into this form, steam is applied and the liquid is forced through the perforations.

The operations of this method, which follow the pressing, are similar to those described for the drying method after the disintegration, except that some operators attempt the reclamation of valuable portions of the tankage which are carried away in suspension by the expressed liquid. This reclamation is made up by the use of triple-effect evaporators and results in a gelatinous material, known as "stick," which is added to the tankage before drying and adds something to the fertilizing value.

In the cooking method, the material being subjected to pressure and high temperatures gives a resultant grease wherein the free fatty acids and glycerin are split to a great extent, causing a considerable loss of glycerin, and unsaponifiable bodies are

formed to an undesirable extent. Then, too, the starches and sugars are changed to glucose and dextrin and are more or less caramelized and even carbonized, leaving them indigestible and of small nutritive value. The albuminoids are broken up and rendered difficult of digestion. The final product is made up principally of cellulose fiber with small amounts of nitrogen and bone phosphates. This material is under suspicion as a fertilizer as to its availability because of the length of time necessary for the chemicals to be released in the soil.

This method is in use in all of the cities of the country using reduction methods, except those noted above and New York, Los Angeles, and New Bedford.

The operation of this method is usually the cause of considerable complaint because of the odors which arise from the emission of gases, fumes, and liquids consequent to the different processes, but there are reasonable means for the elimination of those noxious features which can be used, although their use entails a consequent reduction of the value of the reclamations.

Reclamation of grease by this method varies from 50 to 80 lbs. per ton, depending upon the efficiency of the individual plant.

We now come to the Cobwell process, and while I desire to avoid any appearance of taking advantage of your courtesy by introducing a trade argument, I am forced to use this trade name as there is no other means of differentiating this method from the others, because in theory and practice it differs materially from them. In order to carry out my intention to present facts to you without any prejudice, I take the liberty of quoting verbatim from a paper read before Section D, Engineering, of the American Association for the Advancement of Science, in December 1916, by C. R. Tuska, consulting engineer and also lecturer on municipal waste disposal at Columbia University, who is in no way connected with the company which controls this process.

After describing the other methods and their results, Mr. Tuska said:

The operation of this process is as follows:

The raw garbage is placed in a closed tank which is sealed air-tight. This tank or reducer is constructed with jacketed walls and jacketed bottom. Into these jackets the steam, which is used in the reduction of the garbage, is delivered, these jackets being so designed that it is impossible, under proper operation, for the steam to enter the tank or come in contact with the garbage. In the interior of this tank there is an agitating device operated by power from the exterior. When the proper charge of garbage has been placed in the reducer and the covers placed thereon, the tanks are sealed and the solvent is pumped into the reducer and steam admitted to the jacketed walls. The heat from the steam which is transmitted to the garbage through the walls of the reducer, causes the evaporation of the solvent and the water in the garbage.

Garbage is usually composed of over 75 per cent by weight of water. The steam heat vaporizes the solvent and the water from the garbage and these mixed vapors are drawn off from the reducer to the condenser. The economy in this method of evaporation rests in the fact that water is vaporized at a lower temperature when evaporated with a solvent having a low boiling point than when evaporated without such solvent.

The mixed vapors of the solvent and water, while in the condenser together, are conveyed to a closed tank. Owing to the solvent being of lighter specific gravity than the water, the solvent and the water are separated by gravity, the solvent rising to the top from which it is drawn back to the storage tanks from which it is pumped back to the reducers and used over and over again. The condensed water which has been largely diluted, owing to the jet condensers used, is discharged into sewers or waterways.

When the garbage has been thoroughly dried by this method, the solvent is pumped into the reducer and dissolves the grease. The solvent with the grease is drawn off into a closed tank or evaporator where the same is heated by steam pipes, where the steam is kept separated from the grease. The solvent therein is vaporized and carried to a condenser where the same is again liquefied and carried to storage tanks to be used again.

After the grease has been extracted from the garbage in the reducer, the garbage is further dried by means of the steam in

the jacketed walls and is now in the form of degreased garbage tankage, which is used for fertilizer purposes, after being ground and screened.

It will be seen from the above description of the process that if there are any leakages or vents in any of the tanks or piping where the solvent is handled, more or less solvent is lost, and thereby a substantial additional expense is imposed upon the operation of the system. It is evident, therefore, that it is to the financial interest of the owner of the plant to see that the same is properly operated.

Furthermore, under this system, the garbage is at no time brought in contact with the atmosphere, from the time of its original entrance into the reducer until, after 12 hrs. of cooking, it is finally discharged therefrom as finished products, dried, sterile, and practically odorless. These finished products are grease and the tankage above referred to.

It will be seen by the description that the process is one of straight dehydration, and from the time the material is at the boiling point, no further chemical action takes place. No process of "digestion" occurs, and, therefore, the odors and gases incidental to such process are not created. Only the volume of gas contained in the raw material is driven out and only the essential oils of an extremely volatile nature are carried over in the current of steam and solvent vapor evolved. That little or no conversion takes place in the operation is shown by the fact that in the dehydrated material, at the end of the operation, there exists practically the same amount of unconverted starchy bodies as existed in the garbage at the time of its entrance into the reducer.

The water condensed contains all the gases evolved and has, when fresh, a slight odor of the mixed essential oils. Some traces of alcohol are detected in the effluent and a very small quantity of fixed oils is carried over. Any ammonia evolved, if it has escaped the acid in the garbage, is neutralized by acid carried over in the vapor. Whatever albuminoid ammonia exists in the effluent is carried over by mechanical entrainment, as dust particles, during the steaming out of the solvent.

The effluent from this process consists of almost pure water, this water being the condensed moisture drawn from the reducer while the garbage is being treated and from which the solvent has been extracted as completely as possible. The effluent is cold and gives forth no steam or vapor and is practically odorless, and, as a result, can have no effect when run into a large body of water.

Mr. Tuska's paper continues to give some very excellent data on this subject, but I will refrain from quoting further because my time is limited.

The grease, which is the principal by-product from garbage treatment, is a combination of glycerol and fatty acids, the principal fatty acids being palmitic, stearic, and oleic. At the present time this is sold as recovered and the purchaser refines it and obtains glycerin, stearin, stearic acid, red oil, candle tar, and soap fats.

The glycerin content of grease from the reduction methods, other than Cobwell, runs from 5 to 6 per cent, the free fatty acids from 18 to 40 per cent. In Cobwell the glycerin runs from 7 to 8 per cent and the free fatty acids not to exceed 10 per cent. The purchaser demands that saponifiable shall run about 97 per cent.

While there are many solvents which might prove available in this work, economy has reduced the number to gasoline or kerosene distillate, and engine distillate has been used. In using an economical solvent due regard must be given to the practicability of freeing the grease and tankage from any residue from it at the end of the process.

As to the cost of a reduction plant, it is impossible to make a general statement, as so much depends upon local conditions, and also the cost of machinery varies as the market price of

steel fluctuates. However, it is usually estimated that a cooking method plant will cost, approximately, \$1,500 per rated ton with steel at 3 cents per lb., while the cost of a Cobwell plant will be about one-third more than this. In net recovery values it is generally stated that for the cooking method \$2.00 per ton is a fair average, while Cobwell gives, approximately, \$1.00 more per ton. The only safe method of estimating is to analyze the material in each case. I have no definite figures as to the drying method.

While in most of the cities the disposal of garbage is done by contract, and in the majority of cases a bonus is paid to the contractor, the following cities receive payment from the contractor:

New York.....At the rate of about 45 cents per ton  
Los Angeles.....At 51 cents per ton

Minneapolis has just closed a contract for pig feeding at a revenue of \$1.26 per ton, and Richmond, Va., under the same system, expects to receive \$2.00 per ton. Denver was the first to adopt pig feeding under contract, without the payment, and is doing so at no cost to the city nor is anything paid to it.

These figures appear very attractive, but it is well to be conservative as to the expected revenues from this source, as this system is, as yet, under trial and the result of the experiment will only be assured when a renewal of such contracts show that the contractor has been successful in operating under such a method of payment. An inspection of successive reports from the cities which operate municipal reduction plants gives a very good idea of the uncertainty of the revenue derived from year to year.

I am very much impressed with the patriotic purpose evidenced by this association in giving so much of its valuable time to the study of the subject of municipal waste disposal, with the intention of performing a civic duty in endeavoring to promote more efficient methods and better financial results, if possible, through the employment of the high professional skill of its members, and I, therefore, take the liberty of calling your attention to the fact that the United States Food Administration recommends that at this time such energy be used in obtaining more efficient operation of existing methods rather than the evolution of new methods.

COBWELL CORPORATION  
50 CHURCH STREET  
NEW YORK CITY

## AMERICAN GARBAGE DISPOSAL INDUSTRY AND ITS CHEMICAL RELATION<sup>1</sup>

By RAYMOND WELLS

It is undoubtedly true that before the world war there were chemists in this country and that they were doing most wonderful and useful work, but we were so bewitched by the work done by German copyists and grinds, that when the awakening came, we were astonished to find that the animals at home were likewise endowed with horns and of no inconsiderable magnitude.

At once every industry that had ever thought of the possibility of chemical assistance obtained three of this rare species, and those that had never thought of it at all clamored for at least one. Every business, industry, and manufacture wished for this strange new thing—"scientific control," obtained it as best it might, and found it good. Of course there may be some things the chemist cannot do, but they do not exist in the popular mind.

An industry at this date without a chemical advisor, director, or, at least, a plain ordinary "lab. man" is in the same class with the great auk, and yet there have been explorers who have claimed to have seen specimens of this rare bird, even at the present time. It does not take an explorer to locate the

"garbage industry," but it might prove a stiff task for anyone without the natural instincts of the lowly ferret to find more than one or two even "lab. men" in the business, leaving out of consideration a "regular chemist."

An industry has grown up in this country, taking care of the household table and kitchen waste of 17,000,000 people, serving twenty-nine large cities and returning an annual revenue from the by-products of this service amounting to \$11,500,000. At the same time disposition is made of 1,200,000 tons of raw garbage, from which, disregarding the monetary return, the nation is the richer by producing from its own so-called waste 70,000,000 lbs. of grease and 175,000 tons of valuable fertilizer. And such an industry is practically without a chemist or without even being given recognition by chemists as being one of the few unexplored fields, for their efforts, at the present day.

The disposition of garbage with recovery of the by-products is essentially a chemical problem and its neglect has been due to several causes. The attitude of all or of practically all men or corporations engaged in the business, has been one of antagonism toward chemists, either as individuals or as representatives of science. They were not very unreasonable in this, since all of those chemists attempting to work or to improve the garbage business, did so by long range treatment of garbage as it ought to be from a theoretical standpoint, and from their knowledge of some other business in their mind similar to it. It is not like any other business under the sun, so they fell down most lamentably and the so-called "practical swill man" stood off and scoffed. The "swill man," satisfied with partial success, with sometimes profit and sometimes loss, but with the profit just enough in the right pan of the balance to keep the attraction always there, did not like the idea of disturbing things which had been done, and was thus prevented from asking anything of science. His whole attitude has been one of secrecy about the simplest of operations and one of horror at the idea of starting anything which might or might not turn out to their pecuniary advantage.

Don't blame the swill man for his attitude. He set out to make something out of nothing, out of something which everyone turned away from, about which no one knew anything, and in approximately thirty years built up a real industry, without assistance from anyone. It was all "try it and see," and after a period of several years something was arrived at, which was moderately satisfactory and sometimes made a little money. After that, why change? Changes meant increased investment, possible big financial failure—better to jog along and keep quiet, taking all that could be obtained and when not obtained, slip it over quietly to the next chap.

Seeing this attitude and not looking into what promised only to be a dirty, disgusting hot, sweaty business, where things were accomplished by rather circuitous methods at times, viewed from political and engineering standpoints, the chemist withdrew to more savory and apparently more promising fields. As a matter of fact he chose, as usual, the line of least resistance.

In spite of this neglect from the chemist, the business has prospered and is doing very excellent work for the city and for the nation. A short time ago the city and the nation woke up to the garbage situation and were surprised to find that a real industry existed. Immediately propaganda started and continue to start, which is well, for out of them may grow an interest both popular and scientific, valuable to the industry and to the nation.

Before venturing too far in denouncing our nation as a nation of wasters and as a nation neglectful of its waste materials, it is well to consider all of the facts in the case.

For instance, the largest city in the United States has the largest garbage plant in the world and the most up-to-date one from the chemical and engineering standpoints, representing the effort and financial hazard of many men. This plant possesses the *sine qua non* of the swill business, it is sanitary and does not

<sup>1</sup> Remarks following Mr. Very's paper, New York Section, American Chemical Society, May 10, 1918.

commit a nuisance either in the legal sense or actually. It takes care of the waste food material from 6,000,000 people, amounting even at the present time of conservation, to an average of 800 tons daily or 320,000 tons per annum. Not one particle of this material is wasted. Only the water is eliminated and that as distilled water. In one operation the garbage is transformed or separated into 19,200,000 lbs. of grease of a value of \$2,200,000 and at the same time 64,000 tons of tankage of a value of \$1,000,000 are produced. Several other items, as rags, bones, etc., give an additional value of several hundred thousand dollars. Aside from the money value, this grease recovery means to the nation 1,344,000 lbs. of "dynamite glycerin" and over 150,000,000 cakes of soap. The fertilizer value of 64,000 tons of tankage makes fertile many acres, at a time when the nation is starving for fertilizer. This grease all enters the soap, candle, and glycerin industry and constitutes no small source of supply, and the fertilizer manufacturer regards garbage tankage as a most valuable "base goods." These materials are in no sense low grade and they find a ready market. This one plant employs several hundred men and consumes daily 200 to 250 tons of coal and 3000 to 4000 gal. of kerosene. Kerosene is used for percolation on a huge scale, an innovation in the percolating line which no other industry conceived of or dared to try on a large scale. When it is noted that in the regular course of operation 300,000 to 400,000 gallons of solvent are in constant use, and that still capacity for solvent recovery of 500,000 gal. actual operating capacity per 24 hrs. is required, then one can realize what the industry in one plant means. This is the largest of 29 garbage reduction plants in the country. All of our largest cities have them, only a few of the medium-sized cities still being unenlightened and sticking to the prehistoric and very European method of expensively burning valuable material. These twenty-nine cities produce the quantities of material mentioned in a preceding paragraph. Not all of them have perfect plants, not all of them utilize all of the values in the garbage, but they are performing a public duty and doing it efficiently, as far as their equipment makes it possible.

It is hoped that the preceding statements may lead to the realization that a big industry of great value, not only from the conservation standpoint, but from that of public service and health, has been built up quietly and without attracting, till this time, any public notice or any notice from the scientific world.

The industry is doing very well, it has improved much after a sleep of twenty-five years, it has at last a real method, now operating four years, which is the best yet and promises to be better in the near future, but even now the whole business is nothing more than a healthy embryo, which will require many years of patient work on the part of many patient chemists to incubate to a real live animal and then it will have to grow.

ANALYSIS OF SAMPLE OF AVERAGE HOUSEHOLD GARBAGE

	Per cent
Moisture.....	71.00
Grease.....	4.54
Protein.....	4.24
Ash.....	2.17(a)
Fiber.....	3.21
Cane Sugar.....	0.77
Invert Sugar.....	0.50
Starch, Dextrin, etc.....	10.46
Alcohol.....	0.23
Acid as Acetic.....	0.17
Essential Oils.....	..
Ethers, etc.....	0.11

(a) Potash, bone phosphate, lime, silica, etc.

That it is a chemist's problem the foregoing analysis will demonstrate. Garbage is a conglomerate of all the odds and ends of all the things which men eat, a mixture of every naturally occurring organic material. Think of it in terms of

this typical analysis and see if it does not suggest opportunities.

This analysis does not total 100 per cent, since none of the determinations were made "by difference."

The opportunities suggested by such a mixture offer for research almost virgin soil. And this field has never been touched by our erstwhile German rivals. A nation which is lucky to get garbage to eat, has no garbage problem to solve. Many urge that we too should have no garbage cans. It may be so some day, but not for several generations and it is a very nice question, whether with proper methods of utilization, it may not be as economically profitable to so utilize it and not attempt to force on the human anatomy that which is unattractive and unpalatable. It may be better to let a mechanical digestive tract turn less easily digested materials into substances of greater value for other purposes than for food. Maybe it is better to throw away rancid fat, tough sinews, and potato parings and have the same come back as soap, fertilizer, and alcohol and eat the vegetable oils, animal fats, and the grain released by such an exchange. Nothing is ever destroyed. It can be badly mixed up and out of place, that's garbage. It is the duty of science to put it back into place, and chemistry is the one science most urgently called upon.

HOMER, N. Y.

THE POTTERIES AT SHEK WAAN, NEAR CANTON, CHINA

By CLINTON N. LAIRD

Received March 12, 1918

The prevalence of white ants in South China restricts the use of wood as a building material to a minimum, and therefore all but the most temporary structures are built of brick with tile roofs. The bricks used range from sun-dried, in the poorest villages, to well-burned gray or red of various dimensions. They are made at many different places. The tile are of two kinds, a pan tile, 9 in. square, curved like a shallow trough, and a round tile, approximately half of a truncated cone. The latter are laid in rows over the joint between the vertical rows of overlapping pan tile. These round tile are often glazed but the pan tile very rarely. Most of the common unglazed tile, of different grades, used in and near Canton, are made in Fa Uen district, about 30 miles north of Canton. The only place where any glazed roof or fancy tile or other glazed earthenware articles are made in South China is at the village of Shek Waan, about 20 miles west of Canton.

The pottery industry there is said to be over 700 years old. When the process for glazing earthenware was developed is not recorded, but as one of the temples in Canton has a glazed tile roof (never relaid and still in good condition) known to have been laid 400 years ago, the process is not one of recent origin. At this village are made earthenware jars and dishes of all kinds, clay idols and figures of men and animals, and, in recent years, tile pipe, as well as the glazed ware of many kinds, in color chiefly brown, green, blue and yellow.

For some unknown reason or reasons, possibly through consideration of both beauty and expense, the use of colored glazed roof tile was restricted long ago to temples and imperial buildings. Other blue and green articles made of the same materials in the same way have had a wide use in private buildings, and after the First Revolution (1911) the restriction on the use of glazed roof tiles was removed. General use, except by foreigners, will probably not become common for many years, however, because of the strong association between the tiles and temples. I have been told by one who has travelled widely in the interior of China, that the only place he has seen the green roof tile used on buildings other than temples is at Peking where they are used on the tombs of the imperial concubines. The imperial buildings in

Peking and the imperial temples throughout China were the only buildings having yellow tiled roofs.

The original deposits of clay at Shek Waan were exhausted long ago, and now all the materials are imported. The clay,

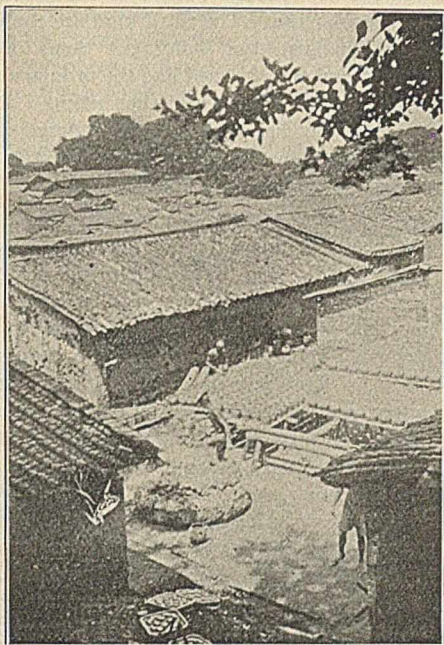


FIG. 1—THE MIXING MACHINE<sup>1</sup>

which varies in color from a cream, streaked with red, to a dark gray, comes from two places, the more plastic from Tung Koon district, about 40 miles east of Canton, and the stiffer from Fa Uen district where the ordinary tiles are made. It is all bought at Shek Waan of the people who bring it there in boats from the districts named. The fuel used is wood, which comes down the river in rafts from the province to the west. It is cut to size and dried across the river from the village of Shek Waan. The potteries are low, one story structures, cheaply built and poorly lighted, but the workers are protected from the heat and glare of the tropical sun.

The article to be made will determine the relative proportions of the two kinds of clay used in any batch. Sand is mixed with the clay in the proportions of one part sand to four parts clay to lessen the danger of cracking in the firing. Nearly 2300 pounds of clay and sand are mixed at a time by one man who mixes it with his feet, by tramping on it, adding a little water from time to time to make it work easier. This is said to be the original method, and the workmen claim that it can be done more thoroughly this way than by machinery. Two batches are mixed by each man in a day, working 4 hrs. on each batch. Large lumps of clay, when mixed, are stuck up against a wall to dry for a day or two, depending on the weather, before being ready for use.

Three methods are used at these potteries for making the various articles: some are formed on the wheel, others in a mold, and some are modeled by hand. They are glazed in practically the same way and all are burned in the same kind of kiln. The chief articles made on the wheel are earthenware dishes, pots used in cooking, and covers for certain kinds of jars. The clay comes to the potter wet. His wheel, nearly 2 ft. in diameter, is mounted on a pin which rotates in a block set in the ground so that the upper surface of the wheel is only a few inches from the ground. The potter sits on a low stool. At his left, close to the wheel,

<sup>1</sup> Because of the Chinese superstition that a man will never be able to do a kind of work different from that which he was doing when his picture was taken, it is very difficult to get satisfactory pictures of the men at work.

is a flat dish about 10 in. in diameter, partly filled with rice straw ashes, and at his right, also close to the wheel, is a vessel containing water. With his left hand he picks up a ball of clay from the dish and with his right hand dips up a little water to moisten the clay. The wet clay is then put on the wheel which is turned by his assistant. Working from the center out he shapes the vessel, holding his thumbs in and his fingers out. The assistant, generally a boy, propels the wheel by striking its upper surface with his right foot as he swings it back, a running motion. Before giving the first impulse to the wheel he picks up a lump of clay which he rolls into a ball while kicking the wheel. He then throws the ball into the dish at the potter's left on to the gritty ashes which make the bottom of the vessel, when finished, a little rough so that it will not stick to the wheel. The boy then takes a piece of flexible bamboo which he holds between his hands in the shape of a U. This acts as a spring to help keep his hands far enough apart so that he will not deform the dish as he picks it up from the wheel and sets it on a board. When the wheel is empty the potter puts on another lump of clay and begins working it. As soon as the boy has put down one dish he picks up another lump of clay and begins kicking the wheel again. The performance is repeated until the board is full, when work stops while the boy puts the board up into a rack over head where the dishes dry until the next day. They are then put out into the sun. I have seen the simplest form of dish, about 1½ in. deep and 6 in. in diameter made in 8 sec., though this would be under the average time. A day's work for the pair would be 1500 or more of these dishes. Larger vessels take more time, and more kicking by the boy who has no chance to kick with his left leg occasionally for a change.

These earthenware dishes, used chiefly in the household, are glazed only on the inside. The glazing material is wood or rice straw ashes, mud from the river bottom (the fouler, the better, according to the workmen), and water, forming a muddy cream. Some of this is poured into the sun-dried dish, and, with a short turning motion, most of the inside of the dish is covered with the cream. The rest is poured out. The dish is allowed to dry before being taken to the kiln to be fired. The final color, after burning, is a dark brown.

All the jars, roof and window tiles, tile pipe, etc., are formed in molds. A wooden model is first made and from it the clay mold. This is burned from 4 to 6 hrs. in a moderate fire; too hot a fire will ruin the mold. The clay is worked stiff and shaped into a

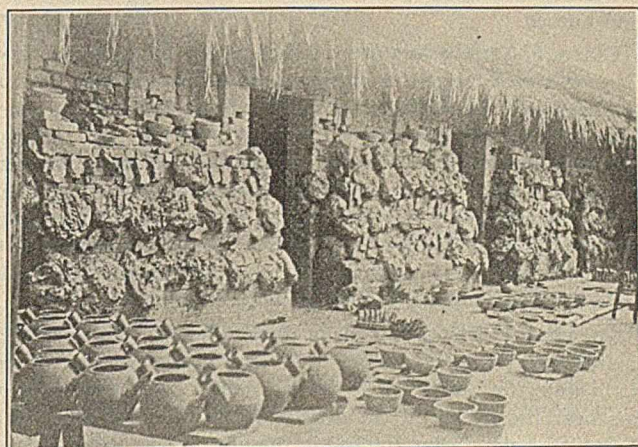


FIG. 2—THE DRYING ROOM

large lump with straight sides of such a shape that a slice cut transversely through the lump for use in the mold will be of the right shape and dimensions for the particular article to be made. This lump is placed on a smooth table and cut into slices from ¼ in. to ⅜ in. thick. The workman then selects two flat bamboo sticks, notched on the sides at the proper intervals, and,

holding in those notches a wire kept tight by a bow, pulls the whole towards him through the lump of clay, the bottom ends of the guides sliding along the table. He then drops the wire down a notch and cuts another slice, thus working from top to bottom through the lump.

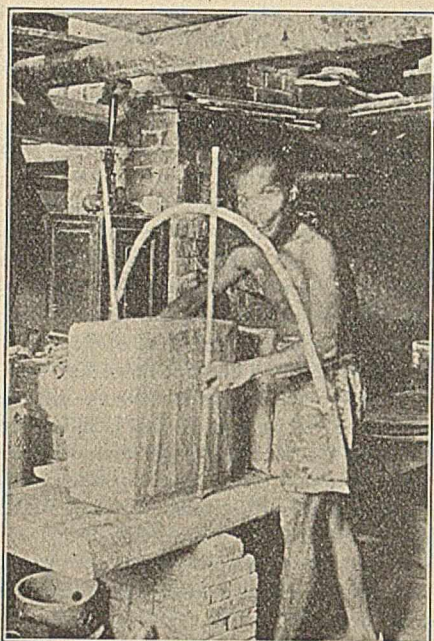


FIG. 3—CUTTING SLICES OF CLAY FOR USE IN THE MOLDS

The slice of clay is placed in the mold and pressed into position with the fingers. Then it is taken from the mold and sun-dried for 2 hrs. If the article is, say, a lattice work tile to be used in windows instead of glass, the two complementary pieces are then put carefully together and all joints made smooth and tight with a little clay put on with the fingers or a bamboo tool. Jars, smaller at the top and bottom than in the middle are made of five pieces of clay. The lower half is made in one mold of a bottom and two side pieces, the edges being pressed together with the fingers; the upper half is formed in another mold. The two sections, after partially drying in the sun, are put together and the joint made tight. These articles are then ready to be glazed and burned.

Glazes of three colors are common: green, blue, and yellow. Copper is used to get the green and cobalt for the blue. One of the leading tile manufacturers in the village told me that they add lead to get the yellow. The same man informed me that they had always made their own copper oxide by roasting copper, but I have never been permitted to see how they do it. The cobalt is imported under the name "English green" and costs more than the copper, but the yellow is the most expensive color of all, which probably explains why the only yellow articles made are roof tile for imperial buildings and burial urns.

The glazing mixture is made of powdered glass, ashes of either mulberry bushes or rice straw, and the coloring matter, all stirred up in water to form a thick, black cream. This is kept in large jars near the kilns and the articles to be glazed are dipped a short time before being stacked in the kilns. The coating is not uniform in thickness, as the glaze runs somewhat, giving a difference of shade, if the color is green, in the final product. This is not noticeable in the roof tile when laid, but is objectionable in other articles. Some of the green tile will show spots or streaks of blue if the firing is not done properly, but generally the color is nearly uniform in the green and quite satisfactory in both the blue and the yellow. Salt is not used in any of the glazes.

The articles modeled by hand are of three classes. Small

birds, animals, figures of people, teapots, etc., are modeled by hand, some of solid lumps of clay, and are burned at the bottom of the kiln used for all the other articles. The fancy glazed tile of unusual shape or size are made by hand without a mold but often forms for curves, etc., are used. These are then treated the same as the regular tile. The third kind are the figures and grotesque decorations used on buildings. Some of these are sun-dried, painted, and then burned like the ordinary tile; others are burned without being glazed and then painted or otherwise decorated. They do not have the life of the glazed articles, but the purpose for which they are intended, or the style of decoration, prevents firing after the decoration has been put on.

The most novel feature of the whole process is the kiln. There are seventy of these in the village. They are long tube-like structures, up to 200 ft. long, built on the sides of the hills in the village. As the hills slope at an angle of from  $15^{\circ}$  to  $20^{\circ}$ , (the angle may not be the same all the way up), the kilns are really long inclined chimneys. At any place a cross section will be an inverted U. Each kiln is protected from the sun and weather by a low roof, built in a series of steps to allow of better ventilation, supported on pillars without walls. Thatched palm leaf awnings are hung at the sides in summer time for further protection from the sun.

One long kiln was only 3 ft. 4 in. wide and 3 ft. 4 in. high, inside measurements, at the bottom. The dimensions gradually increase towards the top where the kiln was 6 ft. wide and 7 ft. high in the center, also inside measurements. The walls are 8 in. thick, made of vitrified brick locally burned for the purpose. Access to the kiln is secured through openings in the sides, 20 to 24 in. wide by 3 ft. high at the lowest opening, gradually increasing in height to 5 ft. at the opening nearest the upper end. The openings are bricked up when the kiln is being fired. The articles to be burned are stacked on the sloping floor, boys doing the work where the kiln is small, each pile having some old broken burned pieces on top. At every 30 in. up the top of the kiln are transverse rows of holes, each not over 2 in. in diameter, through the top wall into the kiln. There are 3 holes in each row near the bottom and 5 holes in each row at the top. The holes are generally closed with pieces of brick set in loosely.

The fuel used is wood. A fire is built at the bottom and the smoke goes up through the kiln, warming the ware therein.

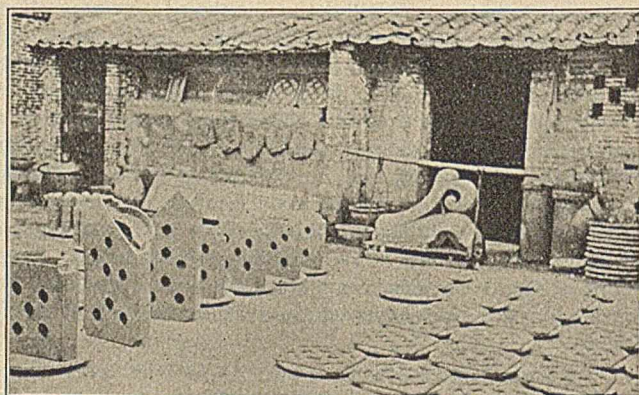


FIG. 4—THE YARD OUTSIDE A POTTERY. NOTE LUMPS OF CLAY DRYING ON THE WALL, AND MOLDS FOR LATTICE WORK TILE ABOVE THEM; ALSO DRYING IN THE SUN, HALVES OF THE LATTICE WORK TILE, LARGE TILE FOR A RIDGE POLE, AND SMALL IMITATION BAMBOO TILE

When the fire is hot enough and sufficient draft has been created, no more fuel is added to the fire at the bottom, but the burners take their places at the first row of transverse holes and feed the fire by dropping in pieces of wood about 15 in. long. When the master burner thinks the fire is hot enough the burners move to the next row and add more fuel. The fire is thus fed through



each row of holes all the way to the top. It takes 2700 lbs. of wood and 12 hrs. work to fire a long kiln. As the fire at the bottom goes out before long, the cheaper articles which do not require much heat, or these that would be spoiled by too much heat, are packed near the bottom. The top of the kiln heats more slowly, becomes much hotter, and cools more slowly so the better grades, like the colored glazed ware, are burned in the upper part of the kiln, and only in a long kiln.

Following the burners and their helpers, the woodcarriers, come another set of men who put the articles for the next run on top of and beside the kiln to be dried out thoroughly before being fired. (When the accompanying picture (Fig. 6) was taken the packers, working from one side, had taken off half of the articles to put them in the kiln.) A kiln is packed on the first day, fired on the second, allowed to cool over the third, and is unpacked on the fourth day; but ordinarily the packers for the next run are following close on the heels of those who are unpacking the previous run, both crews beginning at the bottom and working up the kiln. A large kiln will hold up to 10,000 articles at a time. If 80 per

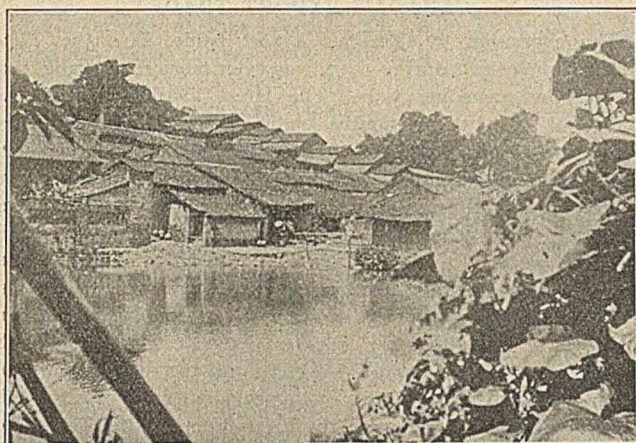


FIG. 5—ROOF OVER A KILN SHOWING OVER THE HOUSETOPS

cent of the articles fired are marketable, the yield is said to be good. The kilns are not owned by the shops which employ the labor and make the goods, but must be rented at the rate of \$5.00 for each run, renter to furnish fuel and labor.

The population of the village is said to be 15,000. As is usual at industrial centers, most of the men have left their families back in the country villages, so the statement that two-thirds of the people there are men and boys is not an exaggeration. The only industry in the village is the potteries, with the necessary shops that supply food, clothing, etc. There are a few women working in the potteries, chiefly at tasks like luting together the halves of a fancy tile. They also make most of the small figures, but this modeling work is done in their homes.

The workmen are well organized, there being at least sixteen guilds or local unions of the men. The men doing the same kind of work belong to the same guild—there are four guilds of men working on the wheel, the members of each guild making a different article. It is difficult to get accurate general information as no one can speak with authority about conditions in other guilds, and he does not care to tell much about his own. A man must serve an apprenticeship of six years, during which time he is said not to receive any wages (this may mean no cash, though he may get both food and lodging), before he is admitted to the guild. A fee of \$75 must be paid the guild at the time of joining, but this is generally paid by the shop or employing company. The wages run up to 40 cents a day for skilled potters (a good carpenter in Canton will get only 25 to 30 cents a day), most of whom are paid by piece work. A semi-skilled workman gets \$4.00 a month and his food, and the boys about a dollar a month and food. Any of the workmen can rise to be an employer if he

can get a little capital. The employers, too, have their guild house, said to be a very fine one.

These potteries are typical of most of the native industries of China in state of development, ingenuity and skill of her artisans, and labor conditions. This description should also suggest the opportunity in China for American goods. At the present time those interested in reaching this market will probably do best to deal through the foreign (chiefly European) firms established in the port cities, who may, or may not, be interested in pushing American lines.

The future growth of the industries of China may be along one of two lines: either the native industries may be developed along modern scientific lines, or the effort may be made to completely ignore the native industry and establish a foreign industry using a foreign process, foreign machinery, and foreign methods. The latter method will almost certainly fail when applied to most industries at most places because of cheap labor, the close relationship between the guilds of both workmen and merchants, and the fact that the market is conservative and

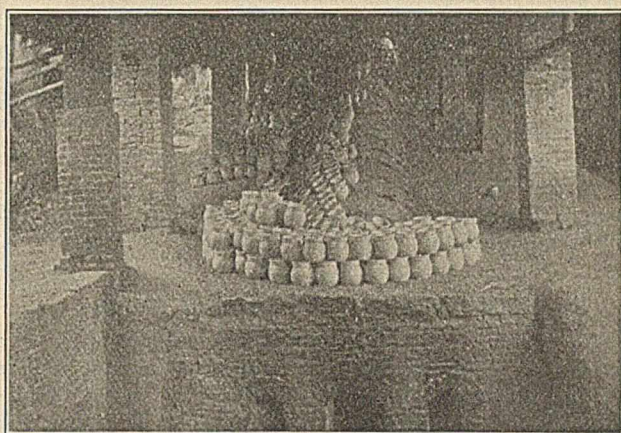


FIG. 6—LOWER END OF A KILN, SHOWING FIRE PIT, HOLES IN TOP THROUGH WHICH FUEL IS FED, AND VESSELS DRYING OUT FOR THE NEXT RUN

demand, in general, a low-priced article. In lines where there has been no native industry it will be necessary to introduce foreign methods, but full recognition must be paid to economic, social, and labor conditions in adapting the foreign process to the local situation.

Those Chinese who have been educated either in the United States, Europe, or the few modern schools in China will be the ones best fitted to develop China's industries. Being conversant with both local conditions and western science and practice they will be able to so remodel the industries that the good points will be conserved, no prejudices aroused, and scientific results obtained. British firms have equipped the engineering laboratories at the University of Hongkong with British machinery, thus training the students at that institution to prefer British goods in future years. So far-sighted Americans, looking for future business in China, should see that American institutions in China lack no facilities for advancing American methods and products, and that at least those Chinese who are studying chemistry in the United States are persuaded to join the AMERICAN CHEMICAL SOCIETY. Those who join will be kept in later years conversant with scientific developments in America and be able, through the advertisements, to know where to get the supplies they need in their own work. The advantages to both countries are unlimited in having an enthusiastic body of Chinese who admire the United States and turn first to Americans when in need.

# CURRENT INDUSTRIAL NEWS

By A. McMILLAN, 24 Westend Park St., Glasgow, Scotland

## NITER CAKE

An interesting discussion on niter cake is reported in the *Journal of Chemical Industry* for December 15, 1917. Various uses for the substance were given, among which the following may be quoted: (1) The utilization of the niter cake as a substitute for sulfuric acid in the manufacture of hydrochloric acid and the salt cake from salt; (2) for obtaining ferric sulfate for sewage precipitation by furnacing burnt pyrites with niter cake, grinding and leaching the product with water; (3) as a diluent for sulfuric acid in the manufacture of superphosphate. Dr. Terlinck stated that he had used niter cake as a substitute for sulfuric acid in the recovery of fats from wool waters and he further proposed to use it in the purification of ammonium salts. In the Nottingham district niter cake was used for lace bleaching, grease extraction from wool, pickling of metals, and mineral water manufacture. Dr. E. Naef proposed utilizing the sodium sulfate which, on reduction, gives sodium sulfide by grinding with anthracite, charcoal or boiler coal and heating at 500 to 600° C., the yield obtained being 95 to 98 per cent. The free acid may be neutralized by adding soda during the grinding. No less than 50,000 tons of sodium sulfide are used for the preparation of sulfur dyes alone per annum. By treating niter cake at 300 to 350° C. with superheated steam, 90 per cent of the free acid is driven off but the product is too dilute to concentrate.

## PETROLEUM IN THE BRITISH EMPIRE

The *Bulletin of the Imperial Institute* states: "In 1903, at the request of the Admiralty, the Imperial Institute prepared a memorandum describing the known and prospective sources of supply of petroleum within the Empire. Since that time continuous attention has been given to this subject and a large number of samples of crude petroleum, oil shales, asphalt, etc., have been reported on from British Guiana, Trinidad, Barbados, New Brunswick, Gold Coast, Newfoundland, Somaliland, Nigeria, Australia, Papua, etc. In certain of these cases, important developments have since taken place, notably in Trinidad, while in others, investigations are still in progress, in some instances with considerable promise of success. It cannot be claimed that any source of supply of petroleum of first-class importance has yet been found within the empire, but sufficient has been done to show that including deposits of oil-shale, there is a considerable possibility of further oil production within the Empire.

## POTASH LYE

The manufacture of potash lye from vegetable ashes and its application for boiling straw in the paper industry is the subject of an article by Mr. S. Tanaka in a Japanese technical journal. It shows that ashes from vegetable materials have been investigated from the point of view of the manufacture of caustic potash for the digestion of straw for paper making. Ashes from soy-bean pods contained 16.19 per cent potassium carbonate, and from chestnut, 13.96 per cent. For the manufacture of caustic potash it is necessary that the ashes should contain more than 10 per cent potassium carbonate. Lime may be added directly to the solution of ash in water without separating the insoluble residue. The yield of caustic potash depends very largely on the perfection of the filtration and lixiviation processes. The potash obtained is quite efficient for the manufacture of straw pulp, and its substitution for caustic soda is a question of cost. A constant and sufficient supply of potash from these sources is hardly to be expected and the difficulties of the filtration process increase the cost.

## OIL CLARIFIER

According to a German patent, a cylindrical vessel fitted with a removable cover is provided with a false bottom, perforated, and covered with corrugated wire gauze which, in turn, is covered with a layer of felt secured to the false bottom by a metal ring and bolts so as to make a tight joint all round. This arrangement also keeps the felt from pressing tightly against the false bottom and by means of the corrugations in the gauze increases the filtering surface. Above the filter bed, the vessel is charged about  $\frac{2}{3}$  full with shavings to remove coarse impurities and the oil is kept fluid by a heating coil embedded in the shavings. Taps are provided at different levels to draw off the oil and any separated waters.

## CANADA'S EXPORT TRADE

The expansion of Canada's export trade, says the *Times Trade Supplement*, during 1917, was even more remarkable than during the preceding year. The latest available figures are those for the twelve months ending November last. These show that the total value of the trade for that period in merchandise alone was \$1,575,233,006, which was an increase over 1916 of 46 per cent and over 1915 of 160 per cent. The increase extended to all general classifications except forest products in which there was a slight decrease. The most remarkable increase was in manufactured goods. The total under this classification was \$703,147,168, which was an increase over 1916 and 1915 of 72 and 410 per cent, respectively. The value of the manufactured goods exported becomes all the more remarkable when the fact is taken into account that it exceeds by the substantial sum of \$104,405,262 the total export trade of all kinds of merchandise in 1915. As a result of this remarkable development in the export trade, there was a favorable balance over merchandise imported of \$563,832,904, whereas the year before the war broke out, there was an adverse balance of \$300,000,000. The total trade balance for three years ending November amounts to the sum of \$1,056,538,845.

## IRRIGATION PLANT

According to the *Board of Trade Journal*, there are very promising prospects for business to be done in irrigation plants in Yunnan Province, South China. At present, four irrigation sets, owned by a private company, are in operation within a few hours' journey of the city of Yunnanfu, and it would be comparatively easy to install 100 such sets if the business were followed up and cared for, as there are immense tracts within easy reach of water, but at an elevation of some 15 ft. above the water level. There are also great opportunities for trade in machinery at the tin mines. Innumerable small pumps are needed and mining machinery in general would find a ready market.

## RECOVERY OF SOLVENT NAPHTHA

According to a recent German patent, the resinous mass left behind in the still after refining solvent naphtha and benzol with sulfuric acid is subjected to dry distillation and decomposes at 330° to 385° C., leaving a pitchy residue. On redistilling the distillate, two fractions are obtained, the bulk consisting of heavy naphtha with boiling point 160 to 220° C. The second fraction is a heavy oil of mineral character which does not gum even in warm air and does not corrode metals or give any deposit in the cold. It may, therefore, be used as a lubricant and as a rosin oil substitute.

## PURE BISMUTH

M. Mylins and E. Groschuff describe a satisfactory method for preparing pure bismuth suitable for electric and magnetic measuring instruments. Bismuth of 99 per cent purity may be obtained fairly easily but the 1 per cent of impurities is difficult to remove. By heating the nitrate of bismuth, the oxide is formed and this can be reduced by hydrogen. The residue is melted and crystallized. The method of purifying bismuth by distillation does not work out in practice as most of the impurities remaining after the ordinary refining process boil at a temperature of less than 1500° C. which is the temperature of boiling bismuth. The method given above is stated to yield a metal which does not contain more than 0.01 per cent impurities and which melts at 271° C. Wire of 1 mm. diameter has an electric resistance of 1.20 ohms at 22° C. If the percentage of impurity amounts to 0.1 per cent, the resistance value is 3 ohms.

## VARIOUS CLASSES OF ENGINES

The following average weights per brake horse power for various classes of engines were given in a paper recently published by Mr. P. N. Everett: Triple expansion steam engines for cargo-boats (no boilers or auxiliaries), 130; triple expansion including boilers and auxiliaries, 450; Diesel engines for cargo-boats (no auxiliaries), 250; Diesel engines with all auxiliaries, 400; turbines for cross channel boats with boilers and auxiliaries, 200; Diesel engines for submarines, 50; steam reciprocating engines for destroyers, 35; turbines for destroyers with boilers, etc., 30; petrol engines for motor cars, 15; petrol engines for racing boats, 7<sup>1</sup>/<sub>2</sub>; aero engines, 2<sup>1</sup>/<sub>2</sub>.

## REGISTER OF OVERSEAS BUYERS

The first edition of this register, which measures 10 in. × 7 in. and contains close on to 400 pages, has been compiled with the assistance of chambers of commerce and the consuls abroad and is issued at the price of \$5 by Messrs. Bemrose and Sons, London. Besides containing a list of the principal imports in allied and neutral countries, arranged geographically under the class of goods they import, the volume includes articles on overseas trade, written under the authority of the chamber of commerce abroad. The requirements of the colonies and foreign countries and how to extend trade with them are dealt with thoroughly, as also are the causes which have hitherto impeded British trade development. A large amount of information is also given on the coinage of various countries, lighthouse, quay and other duties, Government officials, chambers of commerce, banking facilities, newspapers, local trading conditions, etc., all of which will prove useful for the development of trade.

## INDUSTRIAL USES OF BISMUTH

The most important use of bismuth at present is as a component of fusible alloys. An alloy of bismuth, lead, tin and cadmium melts below the boiling point of water. It may also be used as a component of the alloy used for silvering mirrors. Safety plugs for boilers are made of an alloy containing bismuth which fuses at a temperature just above the boiling point of water. Automatic sprinklers or fire extinguishers placed in the ceilings of buildings are also sealed with an alloy containing bismuth, the rise of temperature caused by a fire fuses the plugs and jets of water fall over the fire. Bolivia produces the largest quantity of bismuth, the output in 1915 being 559 tons valued at \$1,115,755. In Queensland the product in 1915 was valued at \$67,445, including some wolfram. In New South Wales, the output in 1916 was 29<sup>1</sup>/<sub>2</sub> tons valued at \$27,365. Smaller quantities are produced in Tasmania, South and West Australia.

## RUSSIAN MONAZITE SAND DEPOSITS

It is stated that in Nizhi Tagil district there are deposits of monazite sand with a large cerium content up to 23 per cent. Hitherto little interest has been shown in Russia in the production of cerium which certainly does not exist in large quantities in the ground in any of its combinations, in fact, only monazite and orthite have been found in the country. The latter is sold in small quantities from the mines of the Transbaikalian territory, while the former is found in many parts of the Urals where it was known long ago by the natives. It has also been found in the Cabinet lands of Transbaikalia. It is proposed to send the monazite sand to Petrograd for the extraction of the rare metals.

## MINERAL OUTPUT OF GREAT BRITAIN

The report of the Inspector of Mines for the year 1916 has just been issued. The total output of coal for the year was 256,375,366 tons which is an increase of 3,169,285 tons over the year 1915. The value of the output was \$1,000,073,130, being no less than \$210,919,780 more than 1915. The total value of the output of minerals in 1916 was \$1,070,072,620, an increase of \$217,881,330 over 1915. The quantity of coal exported was 55,001,113 tons against 59,951,925 tons in 1915. The coal was used as follows:

	Tons
Exported.....	55,001,113
Used for gas coke.....	39,384,819
Manufacture of pig iron.....	19,780,690
Domestic purposes.....	142,208,684

The other principal minerals of value are:

	Tons	Value
Chalk.....	12,786,321	\$ 727,520
Clay and shale.....	6,500,388	6,236,690
Gold ore.....	1,338	3,250
Copper ore.....	787	31,170
Iron ore.....	13,494,658	27,725,360
Lead ore.....	17,107	1,695,845
Limestone.....	11,115,909	6,979,150
Oil shale.....	3,009,232	5,161,470
Salt.....	1,960,448	4,520,665
Tin ore.....	7,892	3,560,710
Tungsten.....	394	248,495
Uranium ore.....	51	5,005
Zinc ore.....	8,476	326,520

Some of the figures show an increase and some a decrease over the figures for the preceding year.

The following table gives the amount and value of the metals obtained by smelting from the ores given above:

Antimony.....	4 tons	\$ 1,700
Copper.....	278 tons	188,970
Gold.....	273 oz.	4,420
Iron.....	4,319,096 tons	175,226,055
Lead.....	12,573 tons	1,947,245
Silver.....	86,483 oz.	56,420
Tin.....	4,697 tons	4,278,280
Zinc.....	3,000 tons	1,026,750

The total value was \$182,729,840, an increase of nearly \$45,000,000 over the year 1915.

## EFFECT OF INSULATION ON STEAM DRUMS

The *Electrical World* describes an interesting test for determining the loss of heat by radiation from boiler settings and steam drums. The 560 h. p. Babcock and Wilcox boiler tested had steam drums covered with one course of common brick. A rectangular can containing a measured amount of water was placed on the top of one drum and the boiler was run at its rated capacity for three days, the rise in temperature of the water being noted. A course of Armstrong nonpareil insulating brick 2<sup>1</sup>/<sub>2</sub> in. thick was then placed on the top of the common brick covering and the readings repeated. The result showed that the saving in heat radiated, if converted into the equivalent consumption of fuel per year, would be more than enough to pay for the cost of the insulation brick and the labor required for its insulation.

### HARDENING CARBON STEEL

An automatic detector fitted by Automatic and Electric Furnaces, Ltd., Westminster, London, to their electric furnace for use in connection with the hardening of carbon steel depends on the principle that carbon steel at its non-magnetic point is at the best temperature for quenching. The furnace chamber consists of an inner pot, which contains a special mixture of salts having a comparatively low melting point but a high vaporizing point. The pot is wound with a heating coil, and is provided with a special heat-resisting lagging. Round the outside case of the furnace is wound an insulated copper coil, the ends of which are connected to a special galvanometer. A current of electricity passed through the heating coil quickly heats the furnace and renders the salt mixture molten and also magnetizes any steel article that is placed in the pot. When the steel has been heated to the non-magnetic point a small current is induced in the outer winding and the consequent deflection of the galvanometer needle informs the attendant that the best temperature for quenching has been reached. A 4 in. furnace will, it is stated, harden 10 lbs. of tools, gauges, or other articles in an hour.

### TUNGSTEN FILAMENTS

A new German process for making tungsten filaments is based upon the idea of forming the lamp filament out of one long crystal. Tungsten crystals can be made to form gradually out of a mixture of tungsten powder and thorium oxide. The mixture is squirted through diamond dies into a filament of 0.02 mm. to 1 mm. in diameter. This filament is then drawn through a chamber in which it is rapidly heated to a temperature of 2400° to 2600° C. and with a velocity of 2.5 miles per hour, which is rather slower than the crystallization velocity of tungsten, so that a single crystal of indefinite length is formed. The chamber is filled with a neutral gas and the heating is performed in two stages by a pair of electrically heated coils, through the center of which the filament is passed. The filament in this state is ready for use in a lamp without further treatment. It is softer than drawn tungsten wire at low temperatures and hard at high temperatures, making it particularly suitable for use in lamps. According to the *Electrician*, filaments consisting of a single crystal of 25 in. length have been produced in this manner.

### TAR-STILL CORROSION BY CHLORINE

Some particulars have been communicated to us, says the *Chemical Trade Journal*, 62 (1918), 360, of a useful investigation carried out by Mr. L. Crawford, late of Liffburn Colliery Tar Works, Durham, with reference to the chlorine content of tars and the consequent corrosion of tar stills and particularly of the domes. In seven different tars from varying types of by-product coke ovens and from vertical and horizontal gas retorts, Mr. Crawford found the chlorine percentage varying from 0.0053 to 0.148 and, in one case, though the bulk showed only 0.111 per cent, as much as 0.226 per cent was found in a sample from the coolers. The investigator states that his experience with the different tars does not enable him to draw conclusions but, in regard to one showing chlorine content of 0.111 per cent, he has been concerned in the distillation of large quantities for a year and he thinks that a chlorine content above 0.1 per cent is above the limit compatible with longevity in still domes. The results are put forward, he states, in the hope that others with experience in individual tars will contribute theirs, so that by correlation of chlorine content with corrosion effects a maximum permissible percentage might be established. It is recognized that other factors may have an influence on still corrosion, but Mr. Crawford believes chlorine to be the predominant factor.

### COTTON-SAMPLING MACHINE

In the course of a paper on some instances of applied science in the cotton trade, read before the Royal Society of Arts, London, Dr. W. Lawrence Balls described a machine for sorting cotton hairs according to length. It consists primarily of a pair of rollers which, as they revolve, are transversed or translated bodily along a path at right angles to their axis of rotation. The cotton to be examined is first run through ordinary drafting mechanism so as to cause the hairs to lie parallel and straight and this "sliver" is presented to the rollers at the beginning of a traverse until they have seized a millimeter or so of the front ends of the hairs. The sliver is then drawn away, leaving in the nip of the rollers a tuft of hairs which are all held by their front ends. The rollers continue to revolve and obviously the first hairs to be delivered from them on the other side will be the shortest ones, while the longest ones (since all started with their front ends level) will be the last to escape. But since this feeding action of the rollers is combined with and positively geared to the motion which causes the traverse, it follows that the short hairs will escape on a suitable collecting device at the beginning of the traverse, the long ones at its completion and intermediate lengths at intermediate points. Thus, the cotton is fractionated by a continuous cycle of repeated operations, as many times as is convenient not merely into separate parcels of hairs but into a graduated series which may be subdivided to any degree desired. The device provides a technique by which it is possible to take a sample of raw cotton, make it into a sliver, treat that sliver for 2 min. only in an automatic machine, weigh the graduated produce of the machine's activity, and, at the end of half an hour, plot frequency curves of a reasonable and measurable degree of precision, showing the variation of length of staple within a sample.

### THE LONG-RANGE GUN

In the issue of *Le Génie Civil* for April 20, Mr. Nicolas Flamel, a French authority, continues the discussion of the German long-range gun. Interesting information is given regarding the type of gun, powder, shell, etc. It appears that the Germans have taken one of their 15-in. naval guns and, by means of the technical process known as relining, reduced the caliber to 8.2 in. The powder is probably an ordinary slow-burning powder, the weight of the charge being increased to give the desired muzzle velocity to the gun. The shell is in two parts: the special fine-pointed head and the body. The shell has special driving bands turned on projecting portions of the body, in addition to the usual copper bands. The burster is either T. N. T. or trinitroanisole (an explosive similar to T. N. T. but having a lower melting point). The writer of the article does not incline to the theory of a special propellant shell, but the gun has been produced in accordance with the usual practice, with necessary modifications in charge, shape of shell, and other minor details.

### BRITISH BOARD OF TRADE

During the month of April the British Board of Trade received inquiries from firms in the United Kingdom and abroad regarding sources of supply for the following articles. Firms which may be able to supply information regarding the things are requested to communicate with the Director of the Commercial Intelligence Branch, Board of Trade, 73 Basinghall St., London, E. C.

Buckles, belt, paper covered	MACHINERY AND PLANT FOR:
Cider vinegar	Manufacture of fasteners for
Eyelets, small, colored, black and brown	cardboard boxes
Ink powder (writing and printing)	Production of fish products (canned)
Turf cutting machines	
Vegetable ivory discs (for export)	Making electric blankets
Watch keys (manufacturers only)	

# SCIENTIFIC SOCIETIES

## FRENCH SECTION, AMERICAN CHEMICAL SOCIETY

PARIS, LE 15 MAI

MAJOR HAMOR

Gas Service, A. E. F.

DEAR MAJOR HAMOR:

Please find enclosed copy of the letter sent to Dr. Nichols, requesting the foundation of a French section of the AMERICAN CHEMICAL SOCIETY. This has been greatly delayed in order to obtain the signature of Mrs. Curie, who is the first French honorary member and of whom it was necessary to ask her compliance.

It took me nearly a month to find the occasion of getting in touch with her, mainly due to my lack of time.

Very sincerely yours,

RENÉ ENGEL

.....

DR. W. H. NICHOLS

President American Chemical Society

Washington, D. C.

DEAR DR. NICHOLS:

According to the constitution of the American Chemical Society and the privileges granted by Article IX for the formation of local sections, the following undersigned members have the honor of requesting the permission of the Council to found in Paris a French section of the Society covering the entire territory of France.

A more propitious time could not be chosen for such a movement, as it would contribute to the formation of an American chemical home for our members belonging to the Expeditionary Forces and bring into closer fellowship the chemists of the two sister Republics.

With our best wishes, we are

Very sincerely yours,

V. Grignard	Raymond F. Bacon
Edward Bartow	Ben H. Nicolet, Capt., C. S. S., U. S. N. A.
Reston Stevenson	G. N. Lewis, Major, C. S. S., U. S. N. A.
George Scatchard	Joel H. Hildebrand, Capt., O. R. C.
W. F. Durand	A. R. Norton, 1st Lt., C. S. S., U. S. N. A.
L. Lapiequy	A. R. Olson, 2nd Lt., C. S. S., U. S. N. A.
Elienne Meens	D. H. McMurtree, 2nd Lt., C. S. S., U. S. N. A.
Y. Guyot	Leonard H. Cretcher, 1st Lt., C. S. S., U. S. N. A.
Baum	G. S. Skinner, 2nd Lt., C. S. S., U. S. N. A.
M. Curie	P. R. Parmelee, 1st Lt., C. S. S., U. S. N. A.
René Engel	Jos. W. MacNaugher, 2nd Lt., C. S. S., U. S. N. A.
J. E. Zanetti	Louis C. Whiton, 1st Lt., San. Cps., U. S. N. A.
W. A. Hamor, Major, C. S. S., U. S. N. A.	C. W. Crowell, Sgt., C. S. S., U. S. N. A.
L. V. Walker, 1st Lt., C. S. S., U. S. N. A.	E. B. Peck, 2nd Lt., C. S. S., U. S. N. A.

May 14, 1918

## THE GERMAN UNION OF TECHNICAL AND SCIENTIFIC SOCIETIES<sup>1</sup>

A short account of this important movement is as follows:

On March 4, 1917, at a meeting of the Verein Deutscher Eisenhüttenleute (German Iron and Steel Institute), held at Düsseldorf, Dr. Fr. Springorum said the war had intensified

<sup>1</sup> The following statement and translations have been prepared by Sir Robert Hadfield.

the need for closer coöperation of the German Technical Societies. Preliminary negotiations had therefore led to a combination of such Societies, and the Verein Deutscher Eisenhüttenleute had gladly joined such Union and promised their support.

On April 19, in an article published in a German newspaper, it was stated that the Managing Committee of the Union has decided to create an Intermediary Agency between the technical world and scientific institutions for the carrying out of scientific and technical research work, so that industry not equipped for experimental work, specially smaller concerns, might be afforded an opportunity of having problems solved through the aid of the Union.

In November the Union held its first General Meeting at the premises of the Association of German Engineers in Sommerstrasse under the Chairmanship of Privy Councillor Busley, at which the Imperial Government Offices, Federal Council, and Legislative Bodies were represented. The purposes and aims of the Union were explained. Herr Busley said their object was to establish a balance between science and practice, and that the technical world ought to be represented more than was hitherto the case in the Legislative Bodies.

Professor Dr. Wiedenfeld, of Halle, speaking on "Economics and Technics During and After the War," stated the blockade of the sea had necessitated the remodelling of the foundations of German economic life, the production from her own resources of raw materials and food, the utilization of waste materials and the production of substitutes. Technical science could only meet these new requirements by disregarding the question of cost price and all considerations as to the possibilities of markets and the risks involved.

The above meetings and the objects of this important German Union of Technical and Scientific Societies are described more fully in the accompanying statements:

### Statement I

Meeting of the German Iron and Steel Institute held at Düsseldorf, March 4, 1917.

#### GERMAN UNION OF TECHNICAL-SCIENTIFIC SOCIETIES

Dr. Fr. Springorum, during the course of his address, stated: The war has intensified the need, already felt before, of closer coöperation of the German Technical Societies, and preliminary negotiations on this question have led to a combination of the Technical Societies into a German Union of Technical-Scientific Associations. We have gladly joined this Union and promised our coöperation, feeling sure that the purposes and aims of the Union are the right ones. The Union leaves to its individual members complete liberty in the special domain which each Association has hitherto been dealing with, but wishes to ensure joint action of the Associations (whose number has now risen to eleven) on all important questions.

### Statement II

This is a précis of the full Statement III, which describes the objects of the German Union of Technical and Scientific Societies acting as Intermediary for scientific technical research work.

#### INTERMEDIARY AGENCY FOR SCIENTIFIC-TECHNICAL RESEARCH WORK

1—Managing Committee of German Union of Technical-Scientific Associations decided to create department to act as intermediary between technical world and scientific societies of universities and technical academies for carrying out scientific-technical research work.

2—Knowledge of departments of work so highly specialized, and so many problems at present, that sometimes few operators available for dealing with a certain question in scientific institutes.

3—It can direct all such problems to suitable operators, might gain very material advantage with minimum of labor.

4—Great intellectual and material resources of scientific institutions of universities and technical academies, and knowledge and experience of their heads, might be rendered serviceable to German industry more than hitherto.

5—Industry, where not equipped for carrying out the task by means of its own arrangements and staff (particularly smaller concerns less equipped for experimental research), will be afforded opportunity of having questions otherwise left unsolved conducted into proper channels for solution through aid of Union.

6—Sometimes even not undesirable for large industrial establishments to come into touch in this way with academicians to judge on complicated questions from scientific standpoint, yet in cohesion with technics.

7—Large number of heads of institutions in departments of (a) applied and physical chemistry, (b) physics, (c) electro-technics, (d) engineering science willing to undertake such work through Intermediary Agency.

8—Further, men of special experience in each of above-named departments have placed themselves at disposal of Agency to assist in selecting suitable operators.

9—German Union and heads of scientific institutions hope Agency will be valuable and useful not only during war but in economic life afterwards.

10—Union asks industrial works engaged in departments of (a) applied physics, (b) electro-technics, (c) machinery construction, (d) engineering science in general to address inquiries to them.

### Statement III

The following statement describes the objects of the German Union of Technical and Scientific Societies, acting as Intermediary for scientific-technical research work, April 19, 1917.

This is shown as a précis in Statement II:

#### GERMAN UNION OF TECHNICAL-SCIENTIFIC SOCIETIES

##### INTERMEDIARY AGENCY FOR TECHNICAL-SCIENTIFIC RESEARCH WORK

The Managing Committee of the German Union of Technical Scientific Associations has decided to create, at its offices, a department which is to act as an intermediary between the technical world and the scientific institutions of the universities and the technical academies for the carrying out of scientific-technical research work.

Very many problems, and likewise the special knowledge of the departments of work, are nowadays so highly specialized that sometimes there are but few suitable operators available for dealing with a certain question in the scientific institutes. If now it were possible to direct all such problems to suitable operators in each instance, a very material advantage might be gained with a minimum expenditure of labor.

On the one hand the great intellectual and material resources which are abundant in the equipment of the scientific institutions of universities and technical academies and in the knowledge and experience of their heads, might be rendered serviceable to German industry to a greater extent than hitherto. On the other hand, industry—as far as it is not itself equipped for carrying out the task by means of its own arrangements and staff or other connections, hence in particular medium sized and small concerns are less amply equipped with experimental research arrangements—will be afforded the possibility of having questions which would otherwise have to be left unsolved, conducted into proper channels for effecting their solution, through the aid of Union. Even to large industrial establishments it might sometimes be not undesirable to come in this way into touch with academicians who are willing to judge as to complicated questions from the scientific standpoint, yet in cohesion with technics.

A large number of heads of institutions in the departments of applied and physical chemistry, physics, electro-technics and engineering science have declared themselves willing to undertake such work introduced to them through the Intermediary Agency. Further, those of special experience in each of the departments named have placed themselves at the disposal of this Agency with a view to assisting it in the selection of suitable operators for the purpose in question.

The German Union and the heads of the scientific institutions hope that this Agency will be of value and prove very useful not only for the duration of the war but also in the subsequent economic life of peace time.

The Union therefore requests industrial works engaged in the departments of (a) chemistry, (b) applied physics, (c) electro-technics, (d) machinery construction, and (e) engineering

science in general, to address inquiries to "Vermittlungsstelle des Deutschen Verbandes, 4<sup>a</sup> Sommerstr., Berlin, N. W.," care the Secretary (member of the committee in charge of the secretarial office).

### Statement IV

This is a précis of the full Statement V, showing the latest information with regard to the work being done by the German Union of Technical and Scientific Societies.

#### TECHNICS DURING AND AFTER THE WAR

1—German Union of some 13 technical scientific societies recently held its first General Meeting in Berlin.

2—Those present included Privy Councillor Busley (Chairman), Representatives of Imperial Government Offices, Federal Council, and Legislative Bodies.

3—Object—to establish balance between science and practice.

4—Many technical tasks could not be carried through without collaboration of several branches of science; metallurgist required cooperation of technician, architect that of engineer, etc.

5—Influence to be exerted on technical education towards admitting academically trained technicians to all administrative departments of Federal States.

6—Technical world should be more represented in Legislative Bodies.

7—Union should be consulted in preparatory work of drafting regulations or enactments. Imperial Treasury alone had availed itself of their advice in preparatory work, namely, taxation of coal and sources of energy.

8—Desire expressed for an Austrian and Hungarian Section of Union.

9—Professor Wiedenfeld said Germany managed with her own production formerly, then more dependent on foreign countries owing to her increased population; afterwards owing to sea blockade, having to produce her own war materials and food.

10—Technical science could only meet new requirements by disregarding question of cost price, which formerly was one of chief consideration in competing with other countries.

11—All considerations relating to market and risk involved disregarded, and substitutes produced by new methods.

12—Though cannot carry over all new conditions and products into peace times, cannot revert to old economic conditions. "What has been will never return."

13—Technical science had tried to help economic life in three ways:

(a) Procuring raw materials formerly obtained from abroad partly by re-establishing unremunerative industries, e. g., manganese production, increasing iron production, sulfur production, agricultural intensification.

(b) Increasing use of waste products (term "non-utilizable substance" eliminated by war) such as obtaining lubricants from coal tar, supply of enough clothing by using waste material.

(c) Producing substitutes, e. g., nitrogen from the air, substances by synthetic processes where natural way not available, such as cattle food from straw.

14—The speaker recalled the dictum of His Excellency Professor Fischer: "I cannot imagine any substance for which a substitute could not be found." Too much regard paid to quality during early part of the war. This impossible as war continues.

15—Deprecated multiplicity of Government authorities controlling construction. This had prejudiced German production in peace time.

16—Ought to aim, even after war, at reduced use of certain raw materials. Owing to high expense for industrial war installations, production would have an unfavorable aspect in certain departments.

17—Amortization necessary in this respect during war of war installation expenses.

18—Monopolies (not necessarily State ones) for materials produced wholesale.

19—Could only establish steady movement of prices by strictly regulated syndicates, then obtain sure remunerativeness, favorable to technical science, and consequent brisk investment.

### Statement V

The following statement shows the latest information with regard to the work being done by the German Union of Technical and Scientific Societies, November 1917.

This is shown as a précis in Statement IV.

#### TECHNICS DURING AND AFTER THE WAR

The "German Union of Technical Scientific Societies," which has recently been formed by the combining of thirteen associa-

tions and unions, held its first General Meeting this morning on the premises of the Association of German Engineers in Sommerstrasse, where the Chairman, Privy Councillor Busley, after welcoming those present—among whom the Imperial Government Offices, the Federal Council and Legislative Bodies were represented by members—explained the purposes and aims of the Union.

Herr Busley said that their object was to establish a balance between science and practice. Many a technical task could not be carried through without participation and collaboration of several branches of science: the metallurgist required the coöperation of the technician, the architect that of the engineer, etc. An influence was also to be exerted on technical instruction and education, and towards ensuring that the academically trained technician should be admitted to all administrative departments of the Federal States. The technical world ought to be represented, more than was hitherto the case, in the Legislative Bodies. The Union had also applied to the authorities with a view to being consulted in the preparatory work of drafting regulations or enactments. Unfortunately, hitherto, the Imperial Treasury alone had availed itself of their advice, in the preparatory work for the taxation of coal and sources of energy. Finally, the speaker stated that a desire had been expressed that an Austrian and Hungarian Section should be attached to the Union, as to which resolutions were still to be passed.

Professor Dr. Wiedenfeld (Halle) then spoke on "Economics and Technics During and After the War." The speaker showed, in a very exhaustive manner, how, in former days, Germany could manage well with her own production, how subsequently she became more and more dependent on foreign countries owing to the increase of her population, and was then subjected, by the blockade of the sea, to the necessity of 'remodelling all the foundations of her economic life, of producing from her own resources, raw materials and food. Technical science could only meet these new requirements by fundamentally disregarding the question of cost price, which formerly, in competing with other countries, was necessarily one of the foremost considerations. Disregarding all considerations as to the possibilities of the market and the risk involved, substitutes were produced by calling in the aid of new modes of production and devoting thereto all human powers. Although not all of these new conditions and products can be carried over into times of peace, nevertheless the old economic conditions cannot be reverted to. "What has been will never return," nor would this be even desirable. He said that technical science had been endeavoring to come to the aid of economic life in a threefold manner.

1—By procuring the raw materials formerly obtained from abroad partly by the re-establishment of industries which had become unremunerative (production of manganese, increase of the production of iron, production of sulfur, intensification of agriculture).

2—By promoting the technical tendency, already existing in pre-war times, towards increased utilization of waste products. The term "non-utilizable substance" has been eliminated by the war. The speaker emphasized in this respect obtaining lubricants from coal tar and supplying clothing requirements by utilization of waste material.

3—By producing substitutes, such as for instance nitrogen from the air, and the production of substances by synthetic processes, where the natural way is no longer available, as for instance the cattle food produced from straw.

The speaker recalled a dictum of His Excellency Professor Fischer: "I cannot imagine any substance for which a substitute could not somehow be found." In the speaker's opinion too much regard had been paid during the early part of the war, to the quality of the production, which however became impossible with the continued duration of the war. The speaker also found fault with the multiplicity of Government authorities controlling construction, which had already manifested itself in peace times to the prejudice of German production. With a view to the projects, the speaker demanded that even after the war we ought to aim at a reduced utilization of certain raw materials. Production would assume an unfavorable aspect in certain departments owing to the high expense for industrial war installations. In this respect amortization during the war of these expenses for war installations would be necessary. Further, wherever materials produced wholesale are in question, the speaker would be in favor of monopolies, though not necessarily state monopolies. He held that it would only be by strictly regulated syndicates that steadiness in the movement of prices could be established, and an assurance of remunerativeness, favorable to the display of technical science, and consequent brisk investment of capital, obtained. The speaker concluded by attempting to lay down guiding principles for the

collaboration of technical science and enterprise, which cannot do without each other.

Finally Dr. Taaks, Dr. Eng., spoke on "Technical Academic Study After the War."

#### RESEARCH WORK ON IRON AND STEEL IN GERMANY<sup>1</sup>

The following is an account of an important movement now going on in Germany relative to research work upon iron and steel. This does not appear to have yet been noticed in this country.

The German proposition is to found and establish a special institution and research laboratory to be entirely devoted to researches on iron and steel. Surely we in this country will not allow this action of the enemy to go unchallenged. While Great Britain has several important laboratories devoted to research on iron and steel, there is certainly required a general building and common meeting place for the following important Institutions.

- (A) Iron and Steel Institute
- (B) Institute of Metals
- (C) Institution of Mining and Metallurgy
- (D) Institution of Mining Engineers
- (E) Faraday Society
- (F) Society of Chemical Industry
- (G) And others

On March 4, 1917, at a General Meeting of the Verein Deutscher Eisenhüttenleute (German Iron and Steel Institute) held at Düsseldorf (see the accompanying Statement I), Dr. Fr. Springorum, during the course of his address, said the committee appointed by the Board of that Institute had recently discussed the subject and recognized the necessity of promoting progress in metallurgy by the establishment of a special research institute probably to be attached to the Kaiser Wilhelm Society.

This was followed on June 19, 1917, by a further meeting of the same Institution (see Statement II) at which a resolution was unanimously passed with regard to the establishment of such an institution and research laboratory to be devoted to research on iron and to be attached to or affiliated with the Kaiser Wilhelm Society, an important new German association.

On July 6, 1917, at a meeting of the Senate of the Kaiser Wilhelm Society, held under the presidency of Professor Von Harnack (see Statement IV) the Senate declared itself in agreement with the proposal of the Verein Deutscher Eisenhüttenleute to establish this institute and laboratory for research on iron and steel.

On July 28, 1917 (see Statement V), reference was made to the meeting of the Verein Deutscher Eisenhüttenleute held on June 19, and after discussing the foundation and site of the building the writer stated that "the means for building and maintaining the Institute, except a small contribution from the Kaiser Wilhelm Society, will be raised by the iron and steel industry of Germany."

Finally, from the latest information in the possession of the compiler of this present statement there was held on November 13, 1917, the first meeting of the "Curatorium" (Trustees Committee) of the Kaiser Wilhelm Institute with regard to the establishment of the research institute and laboratory for research on iron and steel in the "Stahl und Eisen" Building in Düsseldorf, when Dr. Springorum was elected Chairman. (See Statement VII.)

It may be added that the Kaiser Wilhelm Society was founded by the initiative of Emperor William II in January 1911 for establishing and maintaining in a scientific manner independent institutes for research in the sphere of physical science. It has assisted in the foundation of the Institute for Chemistry; Institute for Experimental Therapy; Institute for Coal Research; Institute for Labor Physiology; and now the Institute for Research on Iron and Steel; also (1914) projected the Biological Institute and the Institute for Aerodynamics and Hydrodynamics.

The president is Dr. Harnack and the first vice president is Dr. Krupp von Bohlen und Halbach. Half the members are elected, the other half nominated by the Emperor and the Committee of Management. The election by the Senate and confirmation by the Emperor carries with it the obligation of a contribution of £1,000 with an annual contribution of £50.

#### Statement I

Meeting of the German Iron and Steel Institute, held at Düsseldorf, March 4, 1917.

#### INSTITUTION FOR METALLURGICAL RESEARCH

Dr. Fr. Springorum, during the course of his address, stated:

<sup>1</sup> Statement and translations prepared by Sir Robert Hadfield.

"The titanic struggle of nations has confronted our iron industry in every direction with particularly great difficulties, and I hope that some day, when the war-time records of our works and our associations may be more freely disclosed, not only the full tribute of recognition will be paid for the great things which have been achieved, but that it will also be possible, immediately, to find ways and means of further pursuing, in the interest of the Fatherland, the thousandfold problems which, arising from the emergencies of war, could perhaps be solved only in part during the war. We shall, after the war, far more than hitherto, have to depend on ourselves and to rely on our own strength. Accordingly, the demands on us will be enormous. Industry will only be able to meet them by strenuous work, and will, above all, have to study better utilization of fuels and the further perfecting of metallurgical processes. Coördination between metallurgical practice and metallurgical research, which has always been insisted on and promoted by us, will in future be imperatively needed.

"The weightiness of these facts has revived among our Board an old idea which, to our gratification, has once more been brought forward, of late, by Prof. Oscar Simmersbach, *viz.*, to prepare the soil in order to call into life as quickly as possible, and to promote by financial assistance, an increased activity of research in the domain of the metallurgy of iron and its alloys. For the study of this question the Board has appointed a committee which has recently held an exhaustive discussion on this subject. They were unanimous in recognizing the necessity of promoting with all energy, by scientific research, the progress of metallurgy, with an eye to the exceedingly keen competition in the world's markets, to be anticipated after the war. The only divergency of opinions still existing is as to how such promotion can be effected in the best and most effectual manner; whether by the establishment of a special research institute, possibly attached to the Kaiser Wilhelm Society, or by the expansion of an already existing similar institution, or by studying the problems of research, as they arise, in one or the other scientific laboratory, but always while maintaining the requisite relations with the practical working establishment.

"We may leave the decision as to the course to be adopted to be quietly matured, but the Board recognized that it seemed appropriate just now, in view of the present General Meeting, to acquaint the members with this far-reaching and weighty project, and to gain for it adherents in the widest circles of our works and members. No doubt, funds to a considerable amount will be required for carrying it through; the Board has therefore approved the proposal of its committee that steps should be taken for collecting a nucleus of funds, which should for the present remain at the disposal of the Board of the German Iron and Steel Institute. This fund is to serve in the first place to assist towards turning to account, in practical working, the results of the hitherto purely theoretical labors of the special branch committees of the Institute in the manner appearing most suitable in each case, if a research committee, to be specially appointed for this purpose, endorses the proposal of the respective special branch committee, and if the Board, on its part, considers the granting of funds for the respective purpose appropriate. In this way it would be possible, by enlisting suitable experts or institutions, to take in hand, with the least possible delay, the study of scientific questions important to the iron industry, and at the same time we should, in this way, collect reliable data to serve as a basis for the establishment of a special institution.

"In thus submitting, for the first time in public, the idea of the creation of a fund for an 'Institution for Metallurgical Research,' I feel convinced that, when hereafter the works are called upon to contribute towards the creation of the fund, we may also rely on general and liberal support in the shape of contributions. The German iron industry would thereby erect to itself a worthy war memorial, a cradle for the solution of the manifold problems, still confronting us, and which are of great importance, not only as regards the iron industry, but also as regards the common weal of our Fatherland."

#### Statement II

Meeting of the German Iron and Steel Institute, held at Düsseldorf, June 19, 1917.

#### ESTABLISHMENT OF A GERMAN INSTITUTION FOR RESEARCH ON IRON AND STEEL

In the midst of the war, the German iron and steel industry has laid the foundation stone of an important work of peace. At a meeting of leading men of the German iron and steel industry from all parts of the Empire, which was held on June 19, 1917, at Düsseldorf, the establishment of an institution for research on iron and steel was discussed. The invitations to the

meeting were issued by the Verein Deutscher Eisenhüttenleute (German Iron and Steel Institute), whose president, A. Vogler, General Manager of Dortmund, took the chair at the meeting.

Dr. O. Petersen, Dr. Eng., secretary of the association, gave an exhaustive exposition of what had hitherto been done in the department of scientific advancement of the metallurgy of iron in the various countries. He pointed out the necessity of rendering foundry technical research more and more profound, in order that we may be well armed in every respect in the inevitable economical struggle of the after-war period. He could only refer briefly to the great and important tasks devolving on a research institution, especially as they are shortly to be dealt with more explicitly in an exhaustive commemorative publication. The speaker pointed out, however, that only a research institution free from any one-sided direction or delimitation of its purpose would be capable of taking these in hand with any prospect of success. The speaker further explained how such an institution should be arranged, developed, and maintained, in detail, thus sketching to the meeting the main outlines of the new research institution itself as well as of the prerequisites of its fruitful working.

The subsequent expression of opinions on the part of those present led, we are glad to say, to the momentous resolution, passed unanimously, that the German iron and steel industry was willing to establish a special scientific institution for iron and steel research, to be attached to (or affiliated with) the Kaiser Wilhelm Society. The preparatory work will be set on foot at once by the Verein Deutscher Eisenhüttenleute, and the association will also be subsequently afforded the opportunity of ensuring, in conjunction with the administrative committee of the institution and a scientific advisory council, the indispensable cohesion between the iron industry and the new research institute.

The locality of the new foundation has not yet been definitely decided upon; the decision hereon has been left to the Verein Deutscher Eisenhüttenleute. But, according to the views expressed in this respect at the meeting, the research institution will be located in the Rhenish-Westphalian industrial region. The Southwestern and Silesian iron industries have unselfishly renounced from the outset, in the interest of the great cause, all claims in this direction.

The considerable funds required for the construction and maintenance of its research institution will be provided by the iron and steel industry alone, apart from a small contribution from the Kaiser Wilhelm Society, while the town in which the institution will be established will have to undertake to provide, in addition to a subsidy towards the building expenses, the requisite ground for its site, and its connection with the railway by a siding, etc. The possibility is not precluded that, later on, other industries engaged in the further elaboration of iron and steel, may themselves take a share in the new research institution.

Thus it seems that all prerequisites are provided for to enable this new creation to exercise a highly momentous and beneficent influence on the further technical development of German metallurgy of iron. It must be considered a high merit of the German iron and steel industry that the project submitted by Dr. F. Springorum, Doc. Eng., Councillor of Commerce, of Dortmund, at the last General Meeting of the Verein Deutscher Eisenhüttenleute, has so speedily been realized.

#### Statement III

This is a précis of the full Statement IV, report of meeting of the Senate of the Kaiser Wilhelm Society agreeing to proposal to establish an institute for research on iron and steel.

#### KAISER WILHELM INSTITUTE FOR RESEARCH ON IRON

#### KAISER WILHELM INSTITUTE FOR PHYSICAL SCIENCE

1—Meeting of Senate of Kaiser Wilhelm Society (founded 1911) held on July 6, 1917, under presidency of Professor von Harnack.

2—Also present, Dr. Schmidt, Ministerial Director, and Richter, Privy Councillor, on behalf of Ministry of Public Health and Education.

Senators:

Dr. Fischer  
von Gwinner  
Koppel  
Dr. Krupp von Bohlen und  
Halbach  
Franz von Mendelssohn  
von Moller  
von Passavant-Gontard

Dr. Planck von Rath  
Dr. von Schjerning  
Dr. von Schnitzler  
Dr. von Siemens  
Count Tiele-Winckler  
von Waldhausen  
Dr. Trendelenburg, District  
Judge, Gen. Sec.



3—Senate agreed with proposal of Verein Deutscher Eisenhüttenleute to establish the "Research Institute for Steel and Iron," in form of a "Kaiser Wilhelm Institute."

4—Intend to establish jointly with "Leopold Koppel Foundation" an "Institute for Physical Research."

5—To promote investigations of promising problems of theoretical and experimental physics by monetary grants to individual investigators.

6—Professor Einstein, Member of Academy of Sciences, proposed for appointment as Director of Institute.

7—Scientific management to be entrusted to Directorate of eminent physicists; administration to a body of Curators.

#### Statement IV

The following statement is a report of the meeting of the Kaiser Wilhelm Society held on July 6, 1917, at which they agreed to the proposal of the German Iron and Steel Institute to establish an institute for research on iron and steel.

This is shown as a précis in Statement III.

#### KAISER WILHELM INSTITUTE FOR RESEARCH ON IRON

#### KAISER WILHELM INSTITUTE FOR PHYSICAL SCIENCE

On the 6th inst. a meeting of the Senate of the Kaiser Wilhelm Society (founded 1911) was held under the presidency of Professor von Harnack.

Dr. Schmidt, Ministerial Director, and Richter, Privy Councillor, had appeared on behalf of the Ministry of Public Worship and Education.

There were present the Senators Dr. Fischer, von Gwinner, Koppel, Dr. Krupp von Bohlen und Halbach, Franz von Mendelssohn, von Moller, von Passavant-Gontard, Dr. Planck von Rath, Dr. von Schjerning, Dr. von Schnitzler, Dr. von Siemens, Count Tiele-Winckler, von Waldhausen, and Dr. Trendelenburg, District Judge, General Secretary.

The Senate declared itself in agreement with the proposal of the Verein Deutscher Eisenhüttenleute to establish the "Research Institute for Steel and Iron," projected by them in the form of a "Kaiser Wilhelm Institute."

It is intended to establish, jointly with the "Leopold Koppel Foundation" an "Institute for Physical Research." The Institute is to promote the investigation of promising problems of theoretical and experimental physics by monetary grants to individual investigators.

Professor Einstein, Member of the Academy of Sciences, has been proposed for appointment as Director of the Institute.

The scientific management is to be entrusted to a Directorate of eminent physicists, and the administration of the Institute to a body of Curators.

#### Statement V

Verein Deutscher Eisenhüttenleute (German Iron and Steel Institute), July 28, 1917.

#### FOUNDATION OF A BUILDING FOR IRON AND STEEL RESEARCH

In the midst of the War, the German iron and steel industry has laid the foundation of a significant peace work. At a meeting of leading men of the industry from all parts of the Empire, which took place at Düsseldorf on June 19, 1917, the foundation of a building for research was discussed. The meeting was convened by the German Iron and Steel Institute, the president of which, General-Director A. Vogler, Dortmund, gave an introductory address.

The business manager of the Institute, Dr. Engineer O. Petersen, gave a detailed account of what has already been done in the way of scientific research for the iron and steel industry in various countries. He showed the necessity which will be increasingly greater for technical research in all directions in order to arm for the business struggle which will follow the War. He could only deal shortly with the great and important tasks which will fall on a research institute, especially as a detailed memoir will shortly be published on the subject. The speaker called attention, however, to the fact that the one aim of such a technical research institute should be to attain success in every task coming before it. How such an establishment is founded, built, and maintained, was the subject of the speaker's further discussion, who unfolded to the meeting in outline a picture of the research building and the requirements for its profitable working.

The concluding speeches of the meeting produced a satisfactory and unanimous resolution that the German iron and steel industry is willing to erect, in conjunction with the Kaiser

Wilhelm Society, a special Technical Institute for Ferrous Research. The preliminary work will be put in hand by the German Iron and Steel Union, as then the Union will be able later, in conjunction with the Governing Board of the Institute and a technical assistant counsel, to guarantee proper connection between the steel industry and the research institute.

The site of the new buildings has not yet been settled. The decision has been left to the Committee of the German Iron and Steel Institute. But it is apparent from the views expressed at the meeting that the research building will be established in the Rhenish-Westphalian locality. The Southwest and Silesian industry have renounced their claims in this direction.

The means for building and maintaining the Institute, excepting a small contribution from the Kaiser Wilhelm Society, will be raised by the iron and steel industry, while the town in which it will be situated will be responsible, in addition to a building subsidy, for the necessary land and its connection to the railway, etc. It is not impossible that later other industries will participate in the new research institute.

All appearances show that the new institution will exert a significant and valuable influence on the technical development of the German steel industry. That the plan has matured must be largely attributed to the speech of Dr. Engineer F. Springorum before the last General Meeting of the Iron and Steel Institute (see "Stahl und Eisen," March 15 and April 19, 1917).

#### Statement VI

This is a précis of the full Statement VII, report of first meeting of Curatorium of Kaiser Wilhelm Institute for Research on Iron.

#### KAISER WILHELM INSTITUTE FOR RESEARCH ON IRON

1—On June 19, 1917, German iron and steel industry adopted a resolution to found an institution for free scientific research in the domain of iron.

2—On November 13, 1917, a first meeting of "Curatorium" (Trustee Committee) was held in "Stahl und Eisen" Building, Düsseldorf.

3—This research institution will carry on its activity under the title of "Kaiser Wilhelm Institute for Research on Iron."

4—Curatorium consists of:

NAME	QUALIFICATIONS	REPRESENTATIVE OF
Dr. Schmidt (Substitute: Prof. Kruss)	Minister of Education and Public Worship	Ministry of Education and Public Worship
Prof. D. A. von Harnack (Sub.: Dr. Trendelenburg, Sec.-Gen.)	Royal Privy Councillor, President of the Kaiser Wilhelm Society	Kaiser Wilhelm Society
Prof. Emil Fischer Dr. G. Krupp von Bohlen und Halbach	Royal Privy Councillor	
Dr. F. Springorum	Dr. Eng. General Manager Councillor of Commerce Member Prussian Upper Chamber (Dortmund)	German Iron and Steel Institute
Dr. O. Niedt	Dr. Eng. General Manager Councillor of Commerce (Gleiwitz)	
A. Vogler	Gen. Manager (Dortmund)	

5—The meeting, at which all above were present, except Prof. Fischer, was also attended by the Secretary of the German Iron and Steel Institute, Dr. O. Petersen, Dr. Eng.

6—Dr. Springorum was elected Chairman of the Curatorium.

7—On November 24, 1917, a second meeting of the Curatorium is to be held.

#### Statement VII

The following statement is a report of the first meeting of the Curatorium of the Kaiser Wilhelm Institute for Research on Iron, November 13, 1917. This is shown as a précis in Statement VI.

Verein Deutscher Eisenhüttenleute (German Iron and Steel Institute.)

#### KAISER WILHELM INSTITUTE FOR RESEARCH ON IRON

Since the German iron and steel industry, on June 19 last, adopted the momentous resolution to found an institution for free scientific research in the domain of iron, the preparatory work intended to afford to this new centre of research an assured organizational and financial basis has been advanced by the Verein Deutscher Eisenhüttenleute sufficiently far to admit of the first meeting of the Curatorium (Trustee Committee)

being held on the 13th inst. in the "Stahl und Eisen" Building in Düsseldorf. The Curatorium of the research institution which will carry on its activity under the style and title of "Kaiser Wilhelm Institute for Research on Iron" consists of His Exc. Dr. Schmidt, Minister of Education and Public Worship (Substitute: Prof. Krüss) as representative of the Ministry of Education and Public Worship; His Exc. Prof. D. A. v. Harnack, Royal Privy Councillor, President of the Kaiser Wilhelm Society (Substitute: Dr. Trendelenburg, Secretary General); His Exc. Prof. Emil Fischer, Royal Privy Councillor, and Dr. Gustav Krupp von Bohlen und Halbach, as representatives of the Kaiser Wilhelm Society; and Dr. F. Springorum, Dr. Eng., General Manager, Councillor of Commerce, Member of the Prussian Upper Chamber (Dortmund), Dr. O. Niedt, Dr. Eng., General Manager, Councillor of Commerce (Gleiwitz), and A. Vögler, General Manager (Dortmund), as representatives of the Verein Deutscher Eisenhüttenleute. The meeting, at which all the above-mentioned gentlemen except His Exc. Prof. Fischer were present, was also attended by the Secretary of the Verein Deutscher Eisenhüttenleute, Dr. O. Petersen, Dr. Eng.; Dr. Springorum, Councillor of Commerce, was elected Chairman of the Curatorium. The subject to be dealt with at the meeting was the rules and regulations of the new research institution. A second meeting of the Curatorium, for the discussion of further questions concerning the organization is to be held on November 24, 1917, in Berlin.

#### AMERICAN PHARMACEUTICAL ASSOCIATION

The Annual Convention of the American Pharmaceutical Association will be held in Chicago, August 12 to 17, 1918. Following is a brief outline of the program:

AUGUST 12: Meeting of the National Association Boards of Pharmacy and American Conference of Pharmaceutical Faculties.

- AUGUST 13: Second Session N. A. B. P. and A. C. Ph. F.  
Trip to Municipal Pier and Luncheon.  
Card Party for Ladies.  
Address of President of A. Ph. A.  
Nomination of Officers.
- AUGUST 14: Session of A. Ph. A.  
Section Sessions.  
Dinner of Alumni Organizations.  
Theatre Party for Ladies.  
President's Reception and Ball.
- AUGUST 15: Sessions of A. Ph. A.  
Section Sessions.  
Automobile Ride.  
Banquet.
- AUGUST 16: Sessions of A. Ph. A.  
Section Sessions.  
Election of Officers.
- AUGUST 17: Closing Sessions.

#### CALENDAR OF MEETINGS

National Fertilizer Association—Annual Convention, Atlantic City, N. J., week of July 15, 1918.

American Pharmaceutical Association—Annual Convention, Chicago, Ill., August 12 to 17, 1918.

American Chemical Society—Fifty-sixth Annual Meeting, Cleveland, Ohio, September 10 to 13, 1918.

National Exposition of Chemical Industries (Fourth)—Grand Central Palace, New York City, September 23 to 28, 1918.

## NOTES AND CORRESPONDENCE

#### IMPORTANCE OF CHEMISTS RECOGNIZED BY SECRETARY OF WAR

WAR DEPARTMENT

THE ADJUTANT GENERAL'S OFFICE

WASHINGTON

May 28, 1918

From: The Adjutant General of the Army.

To: Department Commanders, Commanding Officers of Replacement Training Camps, Depot Brigades and Recruiting Depots, and to the heads of Bureaus and Staff Corps.

Subject: Chemists.

1—Owing to the needs of the military service for a great many men trained in chemistry, it is considered most important that all enlisted men who are graduate chemists should be assigned to duty where their special knowledge and training can be fully utilized.

2—Enlisted chemists now in divisions serving in this country have been ordered transferred to the nearest depot brigade.

3—You will make careful inquiry into the number of graduate chemists now on duty in your command and report their names to this office. The report will include a statement as to their special qualifications for a particular class of chemical work, and whether they are now employed on chemical duties.

4—Enlisted graduate chemists now in depot brigades, or hereafter received by them, will be assigned to organizations or service by instructions issued from this office. The report called for in paragraph 3 herein will be submitted whenever men having qualifications for chemical duties are received by depot brigades, or replacement training camps, or by the training camps organized by the various staff corps.

5—Enlisted men who are graduate chemists will not be sent overseas unless they are to be employed on chemical duties.

Prior to the departure of their organization for overseas duties, they will be transferred to the nearest detachment or organization of their particular corps.

6—The Chief of the Chemical Service Section will be charged with the duty of listing all American graduate chemists, including those in the Army and those in civil life.

7—Whenever chemists are needed by one of the bureaus or staff corps, requests will be made on the Chief of the Chemical Service Section for recommendation of a man having the qualifications necessary for the particular class of work for which he is desired. If men having chemical qualifications are wanted for only a short period of duty, they will be temporarily attached to the bureau or staff corps; where the duty is of a permanent nature, instructions covering their transfer will be issued. Whenever the chemists thus attached or transferred are no longer needed for purely chemical duties a report will be made to the Chief of the Chemical Service Section in order that they may be assigned to chemical duties at other places.

By order of the Secretary of War.

Roy A. HILL

Adjutant General

War Department, A. G. O., May 29, 1918. To the Chief, Chemical Service Section, 7th and B Streets, N. W., Washington, D. C., for his information and guidance. When reports showing the number of chemists now at recruit depots, depot brigades, and other places are received, they will be furnished you, in order that proper notation may be made in your register of chemists and for consideration in connection with recommendations for assignment of chemists which you may, from time to time, be called upon to make.

By order of the Secretary of War.

Roy A. HILL

Adjutant General

### COÖPERATION OF AMERICAN CHEMICAL SOCIETY WITH THE CHEMICAL SERVICE SECTION

The following letter to Secretary Parsons, of the AMERICAN CHEMICAL SOCIETY, has been placed in our hands by Lt. Col. Bogert, of the Chemical Service Section, N. A., with a request for its publication in the belief that it will be of interest to the members of the AMERICAN CHEMICAL SOCIETY.—[Editor]

WAR DEPARTMENT  
CHEMICAL SERVICE SECTION, N. A.  
Unit F, Corridor 3, Floor 3,  
7th and B Streets, N. W.  
Washington, D. C.

May 24, 1918

Dr. Charles L. Parsons, Secretary  
American Chemical Society  
Washington, D. C.

DEAR PARSONS:

There has just been placed on my desk a memorandum of a conference between you, representing the American Chemical Society, and Major Victor Lenher and Captain Frederick E. Breithut, representing the Chemical Service Section of the National Army. From this memorandum I note that a routine mode of procedure has been agreed upon whereby the American Chemical Society and the Chemical Service Section will coöperate in order to keep in touch with every available chemist in the country.

May I take this opportunity to express to you my sincerest appreciation of all that you have done to aid the Chemical Service Section. When, in February 1917, before the United States entered the war, you, with rare foresight, sensed the future course of events, and began the taking of a census of our chemists, few of us realized how speedily such a census would be needed nor how dependent we would be on your records for the successful prosecution of our work.

The experiences and mistakes of France and England were known to us all. The sending of chemists to the line in a war which can be most accurately described as a chemical war, was a suicidal blunder which we all hoped would not be repeated in this country. But this feeling on our part could have meant but little when translated into action, had we not had the necessary facts regarding the chemical man power of our country. These facts you gathered, card-catalogued, and indexed in a manner which made immediately available precisely the data which were needed.

In thanking you for this latest offer of cordial coöperation, I desire also to express my great pleasure in being able to be associated with you in an undertaking which must mean much, not only for the winning of the war but also for the elevation of the chemical profession to its true status.

One of our aims in the Chemical Service Section is the organization and maximum utilization of the chemical man power of the country for the general good. In endeavoring to realize this, your work is going to count more than any other single factor.

Cordially yours,

MARSTON T. BOGERT  
Lt. Col., Chem. Serv. Sect., N. A.

### DU PONT FELLOWSHIPS

The situation in regard to the supply of chemists and chemical engineers both for the present and for the future has been giving many industrial concerns a great deal of uneasiness. It is a well-known fact that the number of men studying chemistry and engineering in the advanced classes of all the colleges and universities is on the decline, due to the inroads made by the draft and by enlistments. The du Pont Powder Company,

one of the largest employers of chemists in this country, has been finding very great difficulty in obtaining a sufficient number of experienced chemists to meet the requirements involved in the enormous expansion required to meet the situation brought about by the war. As one means of inducing young men to continue the study of chemistry, the du Pont Company has recently set aside a sum of money to establish fellowships and scholarships in a number of colleges and universities throughout the United States. The fellowships are intended for graduate students and have been offered to a number of the larger universities which have strong and well-developed graduate schools of chemistry. The scholarships may be granted either to seniors or to graduate students and have been offered to a number of the best of the smaller schools which have the reputation of doing high-grade undergraduate work. The fellowships amount to \$750 each and the scholarships from \$300 to \$350. The money appropriated for the scholastic year 1918-19 has been offered to a list of forty-eight of the representative institutions of higher learning.

The object of this plan is to promote the study of chemistry and to assist deserving students who have shown special aptitude for chemistry to pursue further work. The scholarships and fellowships have been granted entirely without restriction except that they are to be awarded by the college authorities to advanced students of chemistry and it is hoped that the plan may prove an initial step toward the true spirit of coöperation between American educational institutions and industrial concerns. The du Pont Company feels that the accomplishment of these objects should materially assist in placing the manufacturing and commercial interests of the United States on a firmer basis.

### FOUR HUNDRED THOUSAND DOLLAR GIFT TO THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY

At a recent meeting of the Corporation of the Institute, President Maclaurin announced a gift of \$400,000 from an anonymous benefactor. The income of this fund is to be available for the general purposes of the Institute during the war and thereafter is to be applied to the development of courses in chemistry and physics.

It is a matter of the first importance in a school of applied science to develop the fundamental sciences of physics and chemistry as thoroughly as possible. The Institute has already a strong staff in these departments.

A great group of the graduates of the chemistry department are now serving the country in the development of its chemical industries and in the prosecution of research with reference to war problems, among this number being ten of the professors of chemistry who have been wholly relieved from regular academic duties to devote themselves to the national cause.

The building up of strong departments of physics and chemistry at a school like the Institute of Technology which draws men in large numbers from all parts of the country is a matter of national importance. There is not only a great need for well-trained chemists and physicists to solve the vital problems of the war, but there will be a similar need in the industrial struggle that will come when peace is declared. The opportunities presented by the war are being seized upon by alert Americans, and great chemical industries are being built up which will need the support of the most highly trained experts to carry them on successfully under the conditions that will later prevail. It is interesting to note that the rising generation also recognizes the opportunity as evidenced among other things by the fact that out of 620 freshmen at the Institute of Technology this year, 160 are being trained for the profession of the chemist.

### COAL-TAR PRODUCTS FOR 1917

The United States Tariff Commission announces the completion of its census of coal-tar products for 1917. This group of products includes not only the coal-tar dyes and the crude and intermediate materials required for their manufacture, but also all of the medicinal and photographic chemicals, explosives, synthetic resins, synthetic perfume materials, and flavors which are in any way derived from coal-tar products. There were produced in the United States (not inclusive of explosives and synthetic phenolic resins) 54,367,994 lbs. of dyes and other finished products, which have a total value of \$68,711,228. The production of the materials known as intermediates amounted to 322,650,531 lbs., with a value of \$106,942,918.

The annual production was reported for the following groups of products made in whole or in part from materials derived from coal tar: 45,977,246 lbs. of dyes valued at \$57,796,027; 5,092,558 lbs. of color lakes valued at \$2,764,064; 2,236,161 lbs. of medicinal chemicals valued at \$5,560,237; 779,416 lbs. of flavors valued at \$1,862,456; 263,068 lbs. of photographic chemicals valued at \$602,281; and 19,545 lbs. of perfume materials valued at \$125,960.

There were 81 establishments engaged in the manufacture of coal-tar dyes in 1917 and their production during that year was practically identical with the amounts annually imported before the war. The imports for the fiscal year 1914 amounted to 45,840,866 lbs. and the production in the United States in 1917 was 45,977,246 lbs. However, an analysis of this total reveals that the domestic production, though equal in quantity to the preceding imports, differs in the relative amounts of the various classes of dyes. Only a small production was reported for indigo, and the alizarin and vat dyes derived from anthracene and carbazol—classes of dyes which include some of the best and fastest colors known to the textile trade. The United States produced only 2,166,887 lbs. of these dyes in 1917; and

the elimination of 1,876,787 lbs. of extract made from imported indigo, reduces the output of these dyes to less than 3 per cent of the pre-war imports. Dyes of this class are dutiable at 30 per cent in the Tariff Act of 1916. The lack of development in the manufacture of these particular dyes promises to be remedied to a considerable extent in 1918, for a number of firms have begun their manufacture and a large increase in production can clearly be foreseen.

In the classes of dyes which if imported would be dutiable at 30 per cent plus 5 cents per lb., the American manufacturers have shown remarkable progress, producing 43,810,359 lbs. at a total value of \$57,639,990. That this represents something of an excess over the American needs is evidenced by the fact that during the fiscal year 1917, American-made dyes to the value of \$11,109,287 were exported to other countries. Thus the exports exceeded the pre-war imports in total value although not in tonnage nor in the variety of the dyes.

The development of the manufacture of intermediates is equally marked, for before the war almost all of these necessary materials were imported from Germany. The Tariff Commission finds that intermediates were manufactured by 117 firms in 1917 and that the production amounted to 322,650,531 lbs. valued at \$106,942,918. These figures, however, are somewhat misleading as there is inevitable duplication in the totals. It is well known that many of the intermediates are derived from other products of the same class. Thus starting with benzol the following succession of products is obtained: nitrobenzol, anilin, acetanilid, nitroacetanilid, and nitranilin. Each of these products had to be reported by the manufacturer and hence there has been some cumulative counting.

The totals for all of the coal-tar products will be published in the final report which may well be expected to offer accurate evidence on the progress of the American dyestuff industry.

## WASHINGTON LETTER

By PAUL WOOTON, Union Trust Building, Washington, D. C.

Revenue legislation, industrial curtailment, restriction of imports, stimulations of domestic production and freight rates have vied with each other during the past month in their bids for public attention. Representatives of the chemical industries have been prominently associated with these activities. Edgar Gilbert, the general manager of the Lyster Chemical Works of New York, brought to the attention of the Ways and Means Committee some interesting facts with regard to the taxation of capital invested in secret processes. While Mr. Gilbert appeared primarily in his own behalf, he stated that the matter was one of peculiar importance to the chemical industry. In part, Mr. Gilbert said:

"The process patent is of such a nature very frequently that when it is disclosed it makes it very difficult to protect the inventor. I might cite an illustration that is old and familiar to all of us, that of the nitration of cotton.

"We will go back to the time when nitrocellulose was first introduced; it is an unknown thing; and we will assume it was to be used in this marketed product, because it had no use as nitrocellulose, but in a solvent form it was used as a nitrocellulate. Now, the inventor, if he discloses it to the world, discloses at once the nature of his patent and of his process. But he maintains his secret and patents it and puts his nitrocellulose in another product, such as celluloid. Now if he patents that, he has no means of protecting himself against infringements, because nitrocellulose appears on the market and someone is producing it, but it would be difficult to know that it had not been produced by some other patent. In that case the inventor would naturally keep secret the nature of his invention and find a market for it in the finished form of some other product. There is an illustration of that recently, in the act of Congress in recognizing the inventor of this substance we have known as Garabed, and recognizing in the inventor the right of protection. The nature of the thing may be such that protection can be afforded neither by a patent nor by a secret process, because the disclosure would cut short the presentation of the product. So I think the right of protection is so well established that it is only a step to say that the patent and the secret process are identical from the inventor's point of view. He takes whatever course seems to give him the greatest security.

"Now, about his assignee. The inventor discovers a process for doing a certain work. In one case he patents it and in another case he keeps it secret. Now, he sells to a corporation for a certain consideration and then that becomes an item of invested capital for the corporation. Now, in the last bill secret processes were not mentioned at all, and my point simply is that in the redraft of the new bill secret processes should be classified as such, because they form, in the chemical industry especially, a considerable item in the assets of the corporation.

"I had this matter up with Mr. Roper's committee for rulings, and they felt that it was proper, they were identical, but they had no power to reach it, they thought, to reach a secret process, to classify it; it had to go into the patent processes."

Industrial curtailment is still in an uncertain stage. To relieve this undesirable condition, official promise has been given that announcement will be made of the amount of curtailment it is necessary to make just as soon as the matter can be weighed sufficiently to permit of an intelligent ruling. Careful surveys have been made by the War Industries Board and the Fuel and Railroad Administrations. It is shown conclusively that the fuel supply is not adequate for all purposes. At the same time, steel and other materials cannot be manufactured in sufficient volume to meet all requirements. In deciding which industries should be curtailed, great difficulties have been experienced. The Fuel Administration has a list of some 360 industries which are classed as non-war activities. The list has not been made public and the amount of curtailment that will be expected from each has not been determined, with the exception of a few cases. The manufacture of pleasure cars has been reduced to 25 per cent of the volume of last year. Other restrictions are expected to follow promptly.

By heavier loading this year, the fertilizer manufacturers of the country have saved space equivalent to 87,000 cars. The average loading this year was 30.02 tons, as compared with 21.31 tons in 1917. This shows an increased loading efficiency of 40.8 per cent.

Hearings on the War Minerals bill continued throughout the month of April, but delay in the consideration of the bill by the committee was occasioned by the enforced absence of Senator Henderson, the chairman of the Committee on Mines and Mining. Great uncertainty exists among members of the committee as to this legislation. Some of the most capable men in the country appeared before the committee and urged that no legislation be enacted which would permit governmental interference with delicately poised industries which handle the war minerals. The power to fix prices and to regulate the industry otherwise might have such far-reaching consequences that some of the members of the committee, at least, are of the opinion that such measure of control as is needed can be exerted under existing law by the War Industries Board.

Announcement was made by the War Industries Board of a schedule of manganese prices, which ran from 86 cents per unit for 35 per cent metallic manganese to \$1.30 per unit for 54 per cent metallic manganese. While the schedule did not cover the chemical ores, it has direct bearing on them.

In the trade agreement recently consummated with Norway, the War Trade Board was particularly liberal in allowing the exportation to that country of chemicals. Among the chemical substances which may be exported under this agreement are: linseed oil; turpentine; palmitic acid; stearic acid; paraffin wax; varnishes; rapeseed oil; ceresine and carnauba wax; rosin; animal and vegetable oils; fats and fatty acids; Chinese wood oil; small quantities of starch, chalk, pyrites, copper, borax, nitrate of soda, bleaching powder, sulfuric acid, and silicate of soda; alum; sulfur; lead; and Solvay soda. Most of the chemicals are to be shipped into Norway consigned to the Oil and Color Merchants' Association and to the Norwegian Papermakers' Association.

Additional restriction on imports have been announced by the War Trade Board. They include castor beans, castor oil, caffeine, and gypsum. All outstanding licenses allowing ocean shipment of castor oil and castor beans have been revoked. Imports may be made only on the approval of the Bureau of Aircrafts Production.

Imports of caffeine, theine, and trimethylxanthine have been cut off entirely.

Importation of gypsum from overseas has been stopped, with the exception of that which can be carried on sailing vessels or barges, which may be designated for that purpose.

Daniel C. Jackling, director of the Explosive Plants Division of the War Department, announced on June 11 the completion, two months ahead of schedule, of the big plants at Charleston, W. Va., and at Nashville, Tenn. Sulfuric and nitric acid production at these plants began early in June.

Nitrate plant No. 3 is to be constructed and operated by the Air Nitrate Corporation. The plant is to consist of two units, one to be built near Cincinnati and one near Toledo. In each instance, construction work has started. Each of the plants will require the services of 1700 operators.

Exports of chemicals during April fell off decidedly, as compared with forwardings abroad during the corresponding month of last year. According to preliminary figures, based on returns to the Department of Commerce, chemical exports in April of this year were valued at \$12,646,505. This compares with \$16,159,506, the value of chemical exports in April of last year.

F. J. Goodfellow, Secretary of the National Wood Chemical Association, has been appointed charcoal representative of the Fuel Administration.

## PERSONAL NOTES

John Harper Long, dean of the Northwestern University School of Pharmacy and a member for many years of the AMERICAN CHEMICAL SOCIETY, died at his home in Evanston, Ill., June 14, 1918, after an illness of nine months. He had been making definite progress toward recovery and was looking forward to complete restoration of his health when a sudden attack of the heart trouble from which he had been suffering caused his death.

Funeral services were held on June 17. Professor Long's sons and son-in-law, Dr. Holgate, president of Northwestern University, and Dr. Ira Remsen, past president of the AMERICAN CHEMICAL SOCIETY and a close friend of Dr. Long, were the active pallbearers. The colleagues of Professor Long on the University staff and Dr. Julius Stieglitz, past president of the AMERICAN CHEMICAL SOCIETY, who had been closely associated with Dr. Long in many scientific undertakings, were the honorary pallbearers.

John Harper Long was born near Steubenville, Ohio, in December 1856. In 1877 he received the degree of B.S. at the University of Kansas; during the years 1877-1880 he studied at Tübingen, Würzburg, and Breslau, receiving the degree of Sc.D. at Tübingen in 1879. In 1881 he became professor of chemistry at Northwestern University Medical School and in 1913 was made dean of the School of Pharmacy at the same institution.

Professor Long was a member of the referee board of consulting scientific experts for the U. S. Department of Agriculture, the revision committee for the U. S. Pharmacopoeia, the council on pharmacy and chemistry of the American Medical Association. He belonged to the Deutsche chemische Gesellschaft, the Washington Academy of Sciences, the Society of Biological Chemists, the American Association for the Advancement of Science of which he was a Fellow, and the American Chemical Society of which he was a past president.

Charles Christopher Trowbridge, assistant professor of physics in Columbia University and a noted ornithologist, died suddenly on June 2 in Roosevelt Hospital, New York City, of blood poisoning. Professor Trowbridge had been a member of the teaching staff of Columbia since 1892. He was in his forty-ninth year.

Mr. Lester F. Weeks, assistant professor of chemistry in the University of Maine, has been appointed assistant professor of chemistry at Colby College to succeed Dr. Robert G. Caswell, who has resigned.

Mr. S. H. Diehl was killed in the explosion at the Aetna Chemical Works in Oakdale, Pa., on May 18, 1918.

At the annual meeting of the American Academy of Arts and Sciences held on May 8, 1918, acting on the recommendation of the Rumford Committee, it was unanimously voted to award the Rumford Premium to Theodore Lyman for his researches on light of very short wave-length.

Mr. Frank Maltauer, formerly employed as bacteriologist in charge of research work on biological chemistry problems at the New York State Laboratories and Research Division of the Health Department, is at present serving as a private at General Hospital No. 14, Fort Oglethorpe, Ga.

The name of Dr. A. B. Lamb, who has given generously of his time and energy to the work of the Bureau of Mines Experiment Station in Washington, was inadvertently omitted from the list of representative leaders in that work mentioned in the editorial "America in Safe Hands" in the June issue of THIS JOURNAL.

Mr. John H. Card, teacher of chemistry at the High School, Brockton, Mass., has joined the Chemical Service Section, N. A. He has been assigned to the Offensive Research Investigations at the American University Experiment Station, Washington, D. C.

Dr. Geo. R. Bancroft has resigned the professorship of chemistry and physics in Transylvania College, Lexington, Ky., to accept a position in the University of Kentucky as assistant professor of organic and physical chemistry.

The honorary degree of Doctor of Chemistry was conferred upon Mr. Arthur D. Little, of Cambridge, Mass., by the University of Pittsburgh at the commencement exercises on May 31, 1918.

Mr. A. Gordon Spencer, consulting chemist and metallurgist, 619 Transportation Building, Montreal, P. Q., is giving up his consulting practice to devote all his time to the munition and other work of the Peter Lyall and Sons Construction Co., Montreal, as their consulting metallurgist.

Mr. C. A. Clemens, formerly instructor of chemistry at Rensselaer Polytechnic Institute, Troy, New York, who is now with the Gas Defense Service, Sanitary Corps, has been sent to the Lakehurst Experimental Grounds, Lakehurst, N. J.

Mr. Chas. E. Waller, formerly with the Pennsylvania Trojan Company at Allentown, Pa., is now with the Non-Freezing High Explosive Corporation at Kingston, N. Y.

Mr. William A. Waldie, formerly chemist for the Glidden Varnish Company, but more recently with the Thibaut and Walker Company, has again returned to the Glidden Company as chief chemist.

In *Science* for May 10 Associate Director Edward R. Weidlein of the Mellon Institute, who is Acting Director in the absence of Director Bacon, gives the following summary of the industrial fellowships in operation at the Institute on March 1, 1918.

NAMES OF INDUSTRIAL FELLOWSHIPS IN OPERATION		INDUSTRIAL FELLOWS, NAMES AND DEGREES	FOUNDATION SUMS AND DATES OF EXPIRATION
92	Leather Belting	E. D. Wilson (Ph.D., University of Chicago)	\$3,800 a year. April 1, 1918
95	Magnesia	G. D. Bagley (E.E., University of Illinois)	\$4,750 a year. November 1, 1918
99	Glyceryl Phosphates	F. F. Rupert (Ph.D., Massachusetts Institute of Technology)	\$1,500 a year. Bonus, 10 per cent of profits. October 1, 1918
102	Fruit Juice	R. R. Shively (Ph.D., University of Pittsburgh)	\$5,000 a year. April 1, 1918
114	Enameling	R. D. Cooke (M.S., University of Wisconsin)	\$2,200 a year. April 1, 1918
115	Bread	H. A. Kohman (Ph.D., University of Kansas), Senior Fellow	\$7,500 a year. Bonus, \$10,000. March 1, 1919
116	Refractories	R. R. Irvin (M.S., University of Kansas) (Vacancy) R. M. Howe (M.S., University of Pittsburgh), Senior Fellow (Vacancy)	\$6,000 a year. May 1, 1918. Bonus, \$500
117	Window Glass	A. C. Nothstine (B.S., Ohio State University)	\$3,000 a year. Bonus, \$2,000. June 1, 1918
118	Leather Soling	C. B. Carter (Ph.D., University of North Carolina)	\$3,500 a year. June 4, 1918
119	Iron Ore	F. M. McClenahan (M.A., Yale University)	\$3,000 a year. June 15, 1918
120	Dental Products	C. C. Vogt (Ph.D., Ohio State University)	\$2,400 a year. Bonus, royalty on sales. July 1, 1918
121	Copper	C. L. Perkins (B.S., New Hampshire College)	\$5,400 a year. July 1, 1918
122	Soda	J. W. Schwab (B.S., University of Kansas)	\$3,500 a year. September 1, 1918
123	Oil	C. W. Clark (Ph.D., University of Pittsburgh) Harry Essex (Ph.D., University of Göttingen)	\$10,000 a year. Bonus, \$10,000. September 1, 1918
124	Cement	I. W. Humphrey (M.S., University of Kansas) (Vacancy) E. O. Rhodes (M.S., University of Kansas)	\$4,000 a year. Bonus, \$3,500. August 1, 1918
125	Hair	B. A. Stagner (Ph.D., University of Chicago)	\$3,000 a year. October 1, 1918
127	Collar	H. D. Clayton (B.A., Ohio State University)	\$2,800 a year. October 1, 1918
128	Coffee	C. W. Trigg (B.S., University of Pittsburgh)	\$1,800 a year. Bonus, 2 per cent of gross receipts. October 1, 1918
129	Illuminating Glass	A. H. Stewart (A.B., Washington and Jefferson College) (On leave of absence)	\$900 a year. October 1, 1919
130	Food Container	F. W. Stockton (A.B., University of Kansas)	\$5,000 a year. October 16, 1918
131	Gas	J. B. Garner (Ph.D., University of Chicago), Senior Fellow (Vacancy)	\$7,500 a year. September 15, 1918
132	Yeast	F. A. McDermott (M.S., University of Pittsburgh) Ruth Glasgow (M.S., University of Illinois) T. A. Frazier (B. Chem., University of Pittsburgh) P. H. Brattain	\$12,700 a year. Bonus, November 1, 1918
133	Glass	I. S. Hocker (B.S., University of Pennsylvania)	\$3,000 a year. November 1, 1918
134	Glycerin	E. E. Bartlett (Pet.E., University of Pittsburgh) J. E. Schott (M.A., University of Nebraska)	\$3,000 a year. November 15, 1918
135	Fiber	J. D. Malcolmson (B.S., University of Kansas)	\$2,500 a year. November 15, 1918
136	Copper	G. A. Bragg (B.S., University of Kansas), Senior Fellow of all Copper Fellowships (Vacancy)	\$5,000 a year. November 1, 1918
137	Toilet Articles	L. M. Liddle (Ph.D., Yale University)	\$3,500 a year. December 1, 1918
138	Silicate	M. G. Babcock (M.S., Iowa State College)	\$2,500 a year. December 1, 1918
139-A	Organic Synthesis	G. O. Curme, Jr. (Ph.D., University of Chicago), Senior Fellow J. N. Compton (M.S., Columbia University) H. R. Curme (B.S., Northwestern University) E. W. Reid (M.S., University of Pittsburgh)	\$10,000 a year. Bonus, \$5,000. January 1, 1919
139-B	Organic Synthesis	H. A. Morton (Ph.D., University of Pittsburgh), Senior Fellow C. J. Herrly (B.S., Pennsylvania State College)	\$5,000 a year. Bonus, \$5,000. January 1, 1919
140	Silverware	H. E. Peck (B.S., Clarkson Memorial College of Technology)	\$2,500 a year. December 11, 1918
141	Insecticides	O. F. Hedenburg (Ph.D., University of Chicago)	\$3,000 a year. January 1, 1919
142	By-products Recovery	Walther Riddle (Ph.D., University of Heidelberg)	\$3,000 a year. January 1, 1919
143	Coke	F. W. Sperr, Jr. (B.A., Ohio State University), Advisory Fellow Marc Darrin (M.S., University of Washington) O. O. Malleis, (M. S., University of Kansas) L. R. Office (B.S., Ohio State University)	\$7,000 a year. January 1, 1919
144	Fertilizer	H. H. Meyers (B.S., University of Pennsylvania)	\$3,000 a year. Bonus, \$5,000. January 5, 1919
145	Soap	(Fellow to be appointed)	\$2,000 a year. January 5, 1919
146	Glue	R. H. Bogue (M.S., Massachusetts Agricultural College)	\$2,500 a year. January 5, 1919
147	Distillation	David Drogin (B.A., College of the City of New York) H. F. Perkins	\$5,300 a year. January 18, 1919
148	Tobacco	W. B. Pattison (M.A., University of Nebraska)	\$2,100 a year. Bonus, \$2,000. February 1, 1919
149	Laundry	H. G. Elledge (M.S., University of Pittsburgh), Senior Fellow K. R. Beach (A.B., Southwestern College)	\$5,000 a year. February 15, 1919

Kenneth D. Kahn, research chemist and metallurgist for the Cleveland Brass Manufacturing Company, Cleveland, Ohio, has been appointed assistant chemical engineer at the Bureau of Mines Experiment Station, American University, Washington, D. C., engaged in the manufacture of war chemicals.

Mr. William Rhafferty, who was formerly chief chemist at the Camden Works, General Chemical Company, and was later transferred to the Bayonne Works, has resigned his position to take up work as assistant to the superintendent of Chas. Lennig and Company, located at Bridesburgh, Pa.

Mr. M. E. Jennings, formerly with the Central Pharmacal Co., Seymour, Indiana, is now a Sergeant in Company E, 30th Engineers, and is located at Fort Meyer, Va.

Mr. F. A. Fererer has left Eli Lilly and Company and is in the Medical Department of the Army, located at Newport News.

Mr. C. M. Sharp, formerly an instructor at Shortridge High School, is in the chemical service of the Medical Corps, located at St. Louis.

Mr. V. P. McManus, well known in drug and chemical circles and for many years associated with McKesson and Robbins, has severed his connection with that house and is now with H. W. Henning & Son, 80 Maiden Lane, New York City.

Henry Coit MacLean, for two years assistant manager of the Foreign Trade Bureau of the Merchants' Association of New York, has assumed his new duties as manager of the New York Office of the United States Bureau of Foreign and Domestic Commerce. Mr. MacLean is thoroughly familiar with the work of the Bureau of Foreign and Domestic Commerce and has been in close touch with questions confronting exporters and importers arising from war conditions.

Mr. Howard A. Winn, formerly of H. H. Hay Sons, manufacturing pharmacists, Portland, Me., is now with the United Drug Co. in the pharmaceutical department.

Dr. A. S. Eastman has given up his position as professor of chemistry at the University of the South, Sewanee, Tenn., and is now connected with the research laboratory of the Hercules Powder Co., Kenil, N. J., in charge of the research work on T. N. T.

Dr. Robert G. Caswell has resigned as assistant professor of chemistry at Colby College, to accept a position as one of the research chemists for E. I. du Pont de Nemours & Co., Wilmington, Del.

Prof. Herman I. Schlesinger has been promoted to assistant professor of chemistry at the University of Chicago.

## INDUSTRIAL NOTES

Statistics compiled by the Bureau of Foreign and Domestic Commerce show that nearly twice as much sulfuric acid was produced in the United States in 1917 as 1913, which is taken as a normal before-the-war-period.

Contracts have been awarded by the Ordnance Department for the establishment of two large picric acid plants at Little Rock, Ark., and Brunswick, Ga. It is expected that the Little Rock plant will be in operation in September 1918. These will be the first Government-controlled picric acid plants to be established in this country.

How 2,000,000 gallons a year of cymene which is now going to waste in the manufacture of paper pulp can be used to produce a line of dye colors very close in their properties to aniline dyes has been worked out in the Color Investigations Laboratory of the Bureau of Chemistry, United States Department of Agriculture. This is the first and very important result of extensive research, investigation, and experimentation conducted in this laboratory under the direction of Dr. H. D. Gibbs.

Paul Wenger and Co., 35 Nassau Street, New York City, well known in the metal trade, have opened a chemical branch in which they will act as buyers, sellers, exporters, and importers. They intend eventually to enter into the manufacture of chemicals and drugs. Charles W. Buck, who up to now has been manager of the Coöperative Drug Company, at South Norwalk, Conn., has been appointed manager of the new plant.

The Cobwell Corporation will have completed in Cleveland within the next 35 days a plant of 25 to 35 tons' daily capacity handling garbage, butcher's offal, and dead animals. This will be of most modern construction and will incorporate all of the newest features covered by the patents of Raymond Wells including a complete equipment for the manufacture of alcohol as an additional product. This plant has been undertaken as a large scale demonstration and experimental plant and will be open at any time to those interested in the business or its development.

On June 2 the J. K. Mosser Tanning Company's plant at Noxen, twenty-five miles from Wilkes-Barre, Pa., was destroyed by fire. The loss is estimated at \$3,000,000. The cause of the fire is believed to have been due to crossed wires in the hair-drying room. The plant was controlled by Armour and Co.

For the first quarter of 1918 the metalliferous production of the Province of Ontario was \$17,909,000, a gain of \$2,315,000 over the same period last year. Gold production amounted to 113,387 ounces, and of that amount the Hollinger mines contributed 68,804 ounces, or a little more than half of the total. The increase was principally silver, owing to the higher price obtained. Gold, on the other hand, in the aggregate fell off, owing to labor difficulties. The three months' silver total was \$3,740,000, against \$2,831,000 in 1917. Total gold production fell from \$2,601,000 to \$2,265,000. Nickel and copper show a small decrease. Hollinger is now producing gold at the rate of approximately \$6,000,000 annually.

H. Koppers Company announce that they have been awarded a contract by the Jones and Laughlin Steel Company for the construction of a by-product coke plant of 300 ovens. This plant will have a carbonizing capacity of approximately 2,000,000 tons per year, and will replace beehive coking capacity to that amount. The plant will be complete in every respect, and will be equipped for the recovery of ammonia in the form of ammonium sulfate, of tar, and of benzol and toluol as pure products. The ammonium sulfate and pure toluol from this plant will be sold to the Government for war purposes. The steel company proposes to use the gas in its steel plant operations. It has also been announced recently that H. Koppers Company are to build two more batteries of by-product ovens for the plant of the Steel Corporation at Clairton, Pa. This will give the Steel Corporation a plant of 748 ovens which, when completed, will be the largest by-product coke plant in the world. The plant of the Illinois Steel Co., Gary, Ind., which has recently added 140 Koppers ovens to its original installation of 560 ovens, is at present the largest by-product coke oven plant in the world.

The War Industries Board has announced that a commodity section on medicines and medical supplies has been created, with Lieut. Col. F. F. Simpson as its chief. The work of this section will be closely coordinated with that of the Chemical Division. This new section will deal incidentally with chemicals as they enter into medical compounds, preparations, etc., and will work in conjunction with the section of the Chemical Division dealing with fine chemicals and bulk medicinal chemicals.

Any doubt about the status of the steel industry in relation to the Government was cleared away Friday, May 24, at the annual meeting and banquet of the American Iron and Steel Institute. A good index to the situation was furnished by Judge E. H. Gary, representing the steel interests; Charles M. Schwab, representing the Emergency Fleet Corporation; and J. Leonard Replogle, director of steel supplies for the Government. The steel manufacturers pledged their support to the Government to the extent of 100 per cent of their respective outputs. They also agreed that steel would be allotted to consumers in order of the importance of its use for national purposes, the judge being the director of the steel supply. Preference is now given to shipbuilding steel, following which in importance comes shell steel, and then steel rails. Mr. Replogle has announced that to fill the Government's demands will require the entire capacity of the steel mills for at least a year. The prospects of manufacturers engaged upon other than Government work keep growing dimmer, though producers are anxious to aid them so far as possible. Steel for Japan has begun to go forward. Already 20,000 tons have been shipped from the Pacific Coast, and the balance, 155,000 tons still due, will follow later.

The Independent Filter Press Company has removed from 47 West 34th Street, New York City, to 418 Third Avenue, Brooklyn, N. Y.

Like most other countries Japan has suffered from the shortage of dyes and chemicals due to the European War. Prior to the war, Japan imported annually dyes valued at \$3,500,000, nearly all of which came from Germany. As most of these dyes were used in the important textile industries of Japan and prices had been advancing by leaps and bounds, the Government passed a law in 1915 providing for the grant of subsidies to companies engaged in the manufacture of dyes (including aniline salt, aniline dyes, alizarine dyes, and synthetic indigo) and chemicals in Japan, and requiring that more than half of the capital of any such company be subscribed by Japanese subjects. The amount of the subsidy to be granted is sufficient to enable the companies to pay a dividend of 8 per cent per annum on their paid-up capital. The subsidies are for a period of ten years from the date of the promulgation of the law. Medicines or perfumery specified by Imperial Ordinance, manufactured from by-products of coal tar, are regarded as manufactured dyes and chemicals. The manufacture of the materials of gunpowder and explosives and of certain medicines, to be determined by Imperial Ordinance, are also regarded as the manufacture of dyes and chemicals. One of the results which attended the efforts made by the Japanese Government to solve the dyestuff problem was the formation of the Japan Dyestuff Manufacturing Company, Ltd., with a capital of 8,000,000 yen (about \$4,900,000), subsidized by the Government.

The War Department authorizes the statement that operations in the Government's new powder plants near Charleston, W. Va., and Nashville, Tenn., have begun two months ahead of schedule. The Nashville plant started June 5, and the Charleston plant started June 12. These plants will produce sulfuric and nitric acids. The capacity of these two plants is expected to equal that of all the other American smokeless powder plants combined.

At a meeting of the Iron and Steel Institute on May 3, awards of \$500 from the Carnegie Research Fund were made to Mr. George Patchin, of London, an associate of the Royal School of Mines, and formerly head of the metallurgical department of Birkbeck College, to enable him to pursue research on "Semi-steel and its heat treatment;" to Mr. Samuel L. Hoyt, U. S. A., to enable him to study "The foreign inclusions in steel, their occurrence and identification;" and to Professor J. A. Van den Broek, of the University of Michigan, for research work on "The elastic properties of steel and alloys."

During May nine new companies were organized for the manufacture of drugs, chemicals, and dyestuffs. The aggregate for the entire war period now stands at \$378,987,000. The figures for May compare with eighteen concerns formed in April for an aggregate capitalization of \$3,980,000. The average incorporation per company in May was \$133,333. This figure compares with \$221,111 in April and \$439,838 in March. Two war companies were created during May. They were the Swift Aircraft Manufacturing Company with an authorized capital of \$50,000, and the United States Ammunition Company, with a capitalization of \$2,500,000. The addition of these two concerns brings the total of investments in this industry up to \$269,625,000.

# GOVERNMENT PUBLICATIONS

By R. S. McBRIDE, Bureau of Standards, Washington

**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.

## COUNCIL OF NATIONAL DEFENSE

**List of Staple Surgical and Medical Supplies.** Reprint with revisions of the lists of medicines, antiseptics, disinfectants, chemicals, etc., selected to meet war conditions by the Committee on Standardization. 27 pp.

**Laboratory Supplies.** Reprint of list selected by the Committee on Standardization. 26 pp.

## PAN AMERICAN UNION

**Some Andean Sulfur Deposits.** B. L. MILLER AND J. T. SINGEWALD, JR. Reprint from January 1918 number of Bull. of the Pan American Union. 16 pp.

## PUBLIC HEALTH SERVICE

**A New Disinfectant Testing Machine.** A. M. STIMSON AND M. H. NEILL. Public Health Reports, 33, 529-39 (April 12).

**Arsphenamine (Salvarsan) and Neo-Arsphenamine (Neo-Salvarsan).** Public Health Reports, 33, 540-2 (April 12). Licenses ordered and rules and standards prescribed for their manufacture.

**The Dietary Deficiency of Cereal Foods with Reference to Their Content in "Antineuritic Vitamine."** G. VÖEGTLIN, G. C. LAKE AND C. N. MYERS. Public Health Reports, 33, 647-66 (May 3).

**The Present Status of Our Knowledge of Fatigue Products.** E. L. SCOTT. Public Health Reports, 33, 605-610 (April 26).

1—Substances carrying hydrogen ions, as lactic and  $\beta$ -oxybutyric acids, potassium dihydrogen phosphate, and carbon dioxide stand as causal agents of fatigue.

2—Certain products of protein disintegration, as indol, skatol, and phenol may produce fatigue symptoms and may be active agents in producing normal fatigue.

3—There is some evidence that the negative ion of lactic and  $\beta$ -oxybutyric acids and that certain positive ions, especially that of potassium, are capable of producing certain fatigue phenomena.

4—There is no evidence that the negative ions of carbonic, phosphoric, or sulfuric acids are fatigue substances.

5—There is no evidence at present for the existence of specific fatigue substances as proposed by Weichardt.

6—There is very little probability that creatin or creatinine have any relation to fatigue or to muscle work in general.

7—There are no doubt numerous bodies, as purine bases, uric acid, etc., which may be increased by work, but which have no causal bearing on fatigue.

## GEOLOGICAL SURVEY

**The Lake Clark-Central Kuskokwim Region, Alaska.** P. S. SMITH. Bulletin 655. 147 pp. and 2 maps. With special attention to mineral resources.

**Cannel Coal in the United States.** G. H. ASHLEY. Bulletin 659. 116 pp. Paper, 15 cents. The present paper is not intended as an original contribution to the subject, though the writer has drawn on his own notes in describing many of the deposits mentioned, particularly those in Pennsylvania, Indiana, and parts of West Virginia and Kentucky. It consists of a preliminary review of well-known facts about the character, uses, and value of cannel coal and brief descriptions of workable deposits of cannel coal, including cross sections of the beds, and it gives such analyses of the coal as are available.

† **The Structural and Ornamental Stones of Minnesota.** O. BOWLES. Bulletin 663. 199 pp. Prepared in coöperation with the Minnesota State Geological Survey.

**Manganese at Butte, Montana.** J. T. PARDEE. Bulletin 690-E. From Contributions to Economic Geology, 1918, Part I. 20 pp. Published April 9. A search through the published reports describing the ore deposits of Butte, supplemented by a brief field examination in August 1917, revealed the fact, perhaps not generally appreciated heretofore, that the amount of material in the lodes that is sufficiently rich in manganese to be considered a possible source of that metal is very large. The smaller part of this material, which is found in the outcrops and upper parts of the lodes, consists of manganese oxides associated with more or less quartz.

By far the most of the manganiferous material below the oxidized zone at Butte consists of rhodochrosite and rhodonite, the carbonate and silicate of manganese, respectively, associated in different proportions with quartz and sulfides. The most interesting and promising feature concerning the occurrence of manganese at Butte is the fact that portions of this unoxidized material consist of fairly pure rhodochrosite and are, therefore, very valuable as a source of the metal. Reported analyses of material of this character in the Emma mine run from 34 to 41 per cent manganese and as low as 1 per cent silica. According to reports this material when lightly roasted gives off its carbon dioxide, and as a result the percentage of manganese is increased in the product. The known workable bodies of this ore aggregate several thousand tons, and there is reason to expect that future developments will disclose large additional amounts.

Whether the general run of the unoxidized manganiferous material can be considered under any conditions as a possible source of manganese is a question for metallurgists to answer. The amount of material that contains 15 per cent or more manganese and occurs within the depths ordinarily reached in mining is indicated, by the evidence available, to be millions of tons. Because they lie at the surface the oxide ores can be mined as rapidly as desired, and the extensiveness of the underground workings, of which many that are temporarily abandoned could probably be made usable in a short time, will permit the carbonate and silicate ore also to be rapidly extracted. Therefore, whether Butte can be counted upon without delay for a considerable production of manganese depends on the solution of problems concerning the metallurgy rather than the mining of its manganiferous deposits.

**The Structure of Parts of the Central Great Plains.** N. H. DARTON. Bulletin 691-A. From Contributions to Economic Geology, 1918, Part II. 26 pp. Published April 2. Mainly of geologic interest.

**Gold, Silver, Copper, Lead, and Zinc in Idaho and Washington in 1916.** C. N. GERRY. From Mineral Resources of the United States, 1916, Part I. 52 pp. Published March 14. Mines report; general report later.



# NEW PUBLICATIONS

By IRÈNE DEMATTY, Librarian, Mellon Institute of Industrial Research, Pittsburgh

- Agriculture:** The Teaching of Agriculture. A. W. NOLAN. 12mo. 277 pp. Price, \$1.30. Houghton, Mifflin Co., Boston.
- Blast Furnace:** Principles, Operation and Products of the Blast Furnace. J. E. JOHNSON, JR. 8vo. 551 pp. Price, \$5.00. McGraw-Hill Co., New York.
- Carbon and Its Allies.** R. M. CRAVEN. 8vo. 468 pp. Price, \$5.00. J. B. Lippincott Co., Philadelphia.
- Chemical French.** M. L. DOLT. 8vo. 398 pp. Price, \$3.00. Chemical Publishing Co., Easton, Pa.
- Chemistry:** What Industry Owes to Chemical Science. R. B. PILCHER. 8vo. 164 pp. Price, 3s. Archibald Constable & Co., London.
- Chemistry of Food and Nutrition.** H. C. SHERMAN. 2nd Ed. 12mo. 545 pp. Price, \$2.00. The Macmillan Co., New York.
- Concrete and Cement; a List of Books.** 12 pp. Gratis. The Public Library, Brooklyn, N. Y.
- Enamels.** R. D. LANDRUM. 8vo. 106 pp. Gratis. The Harshaw, Fuller & Goodwin Co., Cleveland.
- Fuel Oil: Elements of Fuel Oil and Steam Engineering.** ROBERT SIBLEY AND C. H. DELANY. 8vo. 320 pp. Price, \$3.00. Technical Pub. Co., San Francisco.
- Gear Cutting, In Theory and Practice.** J. G. HORNER. 8vo. 404 pp. Price, 8s. 6d. Emmott & Co., London.
- Kiln Drying of Lumber.** H. D. TIEMANN. 8vo. Price, 18s. J. B. Lippincott Co., Philadelphia.
- Lubricants: American Lubricants.** L. B. LOCKHART. 8vo. 236 pp. Price, \$2.00. Chemical Pub. Co., Easton, Pa.
- Measurement of Gas by Orifice Meter.** H. P. WESTCOTT. 12mo. 408 pp. Price, \$3.50. Electric Metal Works, Erie, Pa.
- Mechanics: Cours de mecanique professé à l'ecole polytechnique.** LEON LECORNU. 8vo. 676 pp. Price, 25 fr. Gauthier-Villars, et Cie, Paris.
- Metallurgical Study of the Steel Base as Related to Galvanizing.** G. A. WHITE. 4to. 58 pp. Price, \$2.00. Matthews-Northrup Works, Buffalo, N. Y.
- Metallurgy of the Non-Ferrous Metals.** WM. GOWLAND. 2nd Ed. 8vo. 619 pp. Price, 25s. Chas. Griffin & Co., London.
- Oil: A Short Hand-book of Oil Analysis.** A. H. GILL. 8th Ed. 12mo. Price, \$2.00. J. B. Lippincott Co., Philadelphia.
- Plant Products: An Introduction to the Chemistry of Plant Products.** PAUL HAAS AND T. G. HILL. 2nd Ed. 8vo. 411 pp. Price, \$3.50. Longmans, Green & Co., New York.
- Rubber: Its Production, Chemistry and Synthesis.** (Amer. Ed.). A. DUBOSE AND A. LUTTRINGER. 8vo. 383 pp. Price, \$6.50. J. B. Lippincott Co., Philadelphia.
- Steel and Its Treatment.** E. F. HOUGHTON & Co. 3rd Ed. 12mo. 165 pp. Price, \$1.50. The Author, Philadelphia.
- Sugar Beet Seed, History and Development.** T. G. PALMER. 12mo. 120 pp. Price, \$1.35. John Wiley & Sons, New York.
- Trioxymethylene: The Action of Trioxymethylene on Various Hydrocarbons in the Presence of Anhydrous Aluminum Chloride.** V. R. KOKATNUR. 8vo. 45 pp. Price, \$0.50. University of Minn., Minneapolis.
- Water Analysis: A Simple Method of Water Analysis.** J. C. THRESH. 9th Ed. 18mo. 69 pp. Price, 3s. J. and A. Churchill, London.

## RECENT JOURNAL ARTICLES

- Air Washer: High Velocity Air and Gas Washer.** J. L. ALDEN. *The Iron Trade Review*, Vol. 62 (1918), No. 23, pp. 1428-1429.
- Amylo Saccharification Process.** J. S. HORN. *The Chemical Engineer*, Vol. 26 (1918), No. 6, pp. 204-207.
- Analysis of Sulfonated Oils.** G. W. SCHULTZ. *Journal of the American Leather Chemists Association*, Vol. 13 (1918), No. 5, pp. 190-192.
- Atmospheric Nitrate: The Beginning of the Atmospheric Nitrate Industry in America.** O. P. AUSTIN. *The American Fertilizer*, Vol. 48 (1918), No. 10, pp. 33-35.
- Black Dyes.** C. S. WEHRLY. *Color Trade Journal*, Vol. 2 (1918), No. 6, pp. 208-212.
- Calcium Arsenate.** R. H. ROBINSON. *Journal of Agricultural Research*, Vol. 13 (1918), No. 5, pp. 281-294.
- Chemical Industry in War.** HENRY HOWARD. *The Iron Trade Review*, Vol. 62 (1918), No. 19, p. 1188.
- Coal Tar Colors: Application of the Coal Tar Colors to Wood Staining.** F. E. BURNHAM. *Color Trade Journal*, Vol. 2 (1918), No. 6, pp. 227-228.
- Coke: The Determination of Moisture in Coke.** A. C. FIELDNER AND W. A. SELVIG. *The Chemical News*, Vol. 117 (1918), No. 3046, pp. 172-175.
- Condensers: Design and Layout of Condensers.** D. D. PENDLETON. *The Iron Trade Review*, Vol. 62 (1918), No. 22, pp. 1361-1365.
- Copper: New Method of Determining Copper.** JAMES MOIR. *Mining and Scientific Press*, Vol. 116 (1918), No. 21, pp. 725-726.
- Dilution Figures for the Sulfate Process.** JAMES BEVERIDGE. *Paper*, Vol. 22 (1918), No. 13, pp. 11-12.
- Dry Sizing as a Means of Preparing Feed for Concentration.** G. V. BLAND. *Engineering and Mining Journal*, Vol. 105 (1918), No. 20, pp. 908-911.
- Electro-Plating Engineering.** C. B. WILLMORE. *The Metal Industry*, Vol. 16 (1918), No. 5, pp. 209-211.
- Fiber Board.** OTTO KRESS AND G. C. McNAUGHTON. *Paper*, Vol. 22 (1918), No. 11, pp. 11-16.
- Glass: Some American Contributions to the Glass Industry.** E. W. TILLOTSON. *The Kansan Chemallurgist*, Vol. 3 (1918), No. 1, pp. 4-6.
- High Temperatures and Artificial Gas.** E. R. ROSENBAUM. *The American Drop Forger*, Vol. 4 (1918), No. 5, pp. 186-189.
- Iron: Economic Factors in the Iron Industry.** MALCOLM KEIR. *The Iron Age*, Vol. 101 (1918), No. 21, pp. 1342-1344.
- Japanese Development in Steel Making.** T. T. READ. *The Iron Age*, Vol. 101 (1918), pp. 1259-1262.
- Lubrication and Manufacturing Efficiency.** E. E. GRIGNARD. *Textile World Journal*, Vol. 53 (1918), No. 48, p. 69.
- Manganese Conservation Necessary.** C. R. ELLICOTT. *The Iron Trade Review*, Vol. 62 (1918), No. 23, pp. 1439-1441.
- Materials: Requirements in Testing of Materials.** J. F. BEALL. *The American Drop Forger*, Vol. 4 (1918), No. 5, pp. 183-185.
- Milling in Cyanide Solution.** A. W. ALLEN. *Metallurgical and Chemical Engineering*, Vol. 18 (1918), No. 10, pp. 515-519.
- Mine Valuation: Formulas for Mine Valuation.** W. W. WHITTON. *Mining and Scientific Press*, Vol. 116 (1918), No. 20, pp. 691-694.
- Nitrogen and Its Compounds.** HORACE FREEMAN. *Canadian Chemical Journal*, Vol. 2 (1918), No. 5, pp. 123-125.
- Oil Shales: The Economic Position of Oil Shales.** J. C. MORRELL AND G. EGLOFF. *Metallurgical and Chemical Engineering*, Vol. 18 (1918), No. 11, pp. 601-607.
- Oils: A Process for Deodorizing Fatty Oils.** W. P. SCHUCK. *Metallurgical and Chemical Engineering*, Vol. 18 (1918), No. 11, pp. 608-609.
- Paper: Government Paper Bibliography.** H. E. STOCKBRIDGE. *Paper*, Vol. 22 (1918), No. 11, pp. 38-40.
- Petroleum Chemistry in the Schools.** W. F. FARAGHER. *The Kansan Chemallurgist*, Vol. 3 (1918), No. 1, pp. 8-10.
- Potash from Wood Ashes.** C. T. EDGAR. *Metallurgical and Chemical Engineering*, Vol. 18 (1918), No. 10, pp. 537-539.
- Power Requirements of Hydroextractors.** T. A. BRYSON. *Textile World Journal*, Vol. 53 (1918), No. 48, pp. 61-67.
- Pulpwoods: The Principal Properties, Structure and Identification of Canadian Pulpwoods.** H. N. LEE AND R. W. HOVEY. *Pulp and Paper Magazine*, Vol. 16 (1918), No. 20, pp. 446-448.
- Pulverized Coal: Using Pulverized Coal Efficiently.** H. R. COLLINS. *The Iron Trade Review*, Vol. 62 (1918), No. 20, pp. 1243-1245.
- Reforestation: Economic Aspects of Reforestation.** J. W. TOUMEY. *Paper*, Vol. 22 (1918), No. 13, pp. 14-17.
- Refractory Silica Brick Composition.** J. S. McDOWELL. *Mining and Scientific Press*, Vol. 105 (1918), No. 21, pp. 954-956.
- Research Work on the Sizing of Paper.** F. C. CLARK AND A. G. DURGIN. *Paper*, Vol. 22 (1918), No. 10, pp. 11-17.
- Rubber: Defects in Industrial Rubber Goods.** FREDERIC DANNERTH. *Metallurgical and Chemical Engineering*, Vol. 18 (1918), No. 10, pp. 531-534.
- Sodium Cyanide.** W. J. SHARWOOD. *Mining and Scientific Press*, Vol. 116 (1918), No. 19, pp. 655-656.
- Steam Cylinder Lubrication.** W. F. OSBORNE. *Lubrication*, Vol. 5 (1918), No. 7, pp. 2-12.
- Steam Engine Design.** F. S. BAUER. *University of Colorado Journal of Engineering*, Vol. 14 (1918), No. 3, pp. 33-37.
- Steam Turbine Oil Filtration.** E. M. MAY. *Lubrication*, Vol. 5 (1918), No. 7, pp. 12-16.
- Stellite. Alloys of Cobalt, Chromium, Tungsten and Molybdenum.** ELWOOD HAYNES. *Metallurgical and Chemical Engineering*, Vol. 18 (1918), No. 10, pp. 541-542.
- Steel: Its Selection and Treatment.** J. F. KELLER. *The American Drop Forger*, Vol. 4 (1918), No. 2, pp. 52-55.
- Storage of Bituminous Coal.** E. MATHESON. *Paper*, Vol. 22 (1918), No. 11, pp. 35-38.
- Steel: Manufacture, Inspection, Etc., of Crucible Steel.** F. B. LOUNSBERRY. *Journal of the Cleveland Engineering Society*, Vol. 10 (1918), No. 5, pp. 309-314.
- Sulfate of Ammonia: Increasing the Sulfate of Ammonia Supply.** W. B. PHILLIPS. *The American Drop Forger*, Vol. 48 (1918), No. 4, pp. 32-33.
- Sulfur and Copper Oxide Determination.** C. G. MAIER. *Engineering and Mining Journal*, Vol. 105 (1918), No. 8, pp. 372-373.
- Sulfuric Acid: Calumet and Arizona Sulfuric Acid Plant.** C. DEKALB. *Mining and Scientific Press*, Vol. 116 (1918), No. 13, pp. 437-444.

# MARKET REPORT—JUNE, 1918

WHOLESALE PRICES PREVAILING IN THE NEW YORK MARKET ON JUNE 17, 1918

## INORGANIC CHEMICALS

Acetate of Lime.....	100 Lbs.	nominal	
Alum, ammonia, lump.....	100 Lbs.	4.00 @	4.50
Aluminum Sulfate, (iron free).....	Lb.	3 1/4 @	3 1/2
Ammonium Carbonate, domestic.....	Lb.	nominal	
Ammonium Chloride, white.....	Lb.	19 1/2 @	20
Aqua Ammonia, 26°, drums.....	Lb.	nominal	
Arsenic, white.....	Lb.	9 1/2 @	16 1/2
Barium Chloride.....	Ton	65.00 @	85.00
Barium Nitrate.....	Lb.	11 @	12
Barytes, prime white, foreign.....	Ton	30.00 @	35.00
Bleaching Powder, 35 per cent.....	100 Lbs.	1.80 @	2.00
Blue Vitriol.....	Lb.	8.80 @	9.00
Borax, crystals, in bags.....	Lb.	7 1/4 @	8 1/4
Boric Acid, powdered crystals.....	Lb.	13 1/4 @	15
Brimstone, crude, domestic.....	Long Ton	nominal	
Bromine, technical, bulk.....	Lb.	75 @	85
Calcium Chloride, lump, 70 to 75% fused.....	Ton	22.00 @	25.00
Caustic Soda, 76 per cent.....	100 Lbs.	4.35 @	4.40
Chalk, light precipitated.....	Lb.	4 3/4 @	5
China Clay, imported.....	Ton	20.00 @	30.00
Feldspar.....	Ton	8.00 @	15.00
Fuller's Earth, foreign, powdered.....	Ton	nominal	
Fuller's Earth, domestic.....	Ton	20.00 @	30.00
Glauber's Salt, in bbls.....	100 Lbs.	1.25 @	2.50
Green Vitriol, bulk.....	100 Lbs.	1.15 @	1.25
Hydrochloric Acid, commercial, 20°.....	Lb.	2 @	2 1/2
Iodine, resublimed.....	Lb.	4.25 @	4.30
Lead Acetate, white crystals.....	Lb.	17 @	18
Lead Nitrate.....	Lb.	nominal	
Litharge, American.....	Lb.	7 3/4 @	8
Lithium Carbonate.....	Lb.	1.50 @	
Magnesium Carbonate, U. S. P.....	Lb.	20 @	21
Magnesite, "Calcined".....	Ton	60.00 @	65.00
Nitric Acid, 40°.....	Lb.	8 1/2 @	9
Nitric Acid, 42°.....	Lb.	9 @	10
Phosphoric Acid, 48/50%.....	Lb.	8 1/2 @	12
Phosphorus, yellow.....	Lb.	1.05 @	1.30
Plaster of Paris.....	Bbl.	2.00 @	2.50
Potassium Bichromate, casks.....	Lb.	44 @	45
Potassium Bromide, granular.....	Lb.	1.35 @	1.36
Potassium Carbonate, calcined, 80 @ 85%.....	Lb.	40 @	50
Potassium Chlorate, crystals, spot.....	Lb.	39 @	41
Potassium Cyanide, bulk, 98-99 per cent.....	Lb.	nominal	
Potassium Hydroxide, 88 @ 92%.....	Lb.	80 @	82 1/2
Potassium Iodide, bulk.....	Lb.	3.75 @	4.00
Potassium Nitrate.....	Lb.	27 @	30
Potassium Permanganate, bulk.....	Lb.	2.50 @	3.00
Quicksilver, flask.....	75 Lbs.	119.00 @	122.00
Red Lead, American, dry.....	100 Lbs.	10.79 @	12.75
Salt Cake, glass makers'.....	Ton	22.00 @	25.00
Silver Nitrate.....	Oz.	62 1/4 @	65
Soapstone, in bags.....	Ton	10.00 @	12.50
Soda Ash, 58%, in bags.....	100 Lbs.	2.25 @	2.45
Sodium Acetate.....	Lb.	nominal	
Sodium Bicarbonate, domestic.....	100 Lbs.	3.00 @	3.25
Sodium Bichromate.....	Lb.	27 @	28
Sodium Chlorate.....	Lb.	24 @	25
Sodium Cyanide.....	Lb.	40 @	43
Sodium Fluoride, commercial.....	Lb.	17 @	18
Sodium Hyposulfite.....	100 Lbs.	2.50 @	2.60
Sodium Nitrate, 95 per cent, spot.....	100 Lbs.	4.75 @	5.00
Sodium Silicate, liquid, 40° Bé.....	100 Lbs.	2.50 @	3.00
Sodium Sulfide, 60%, fused in bbls.....	Lb.	nominal	
Sodium Bisulfite, powdered.....	Lb.	11 @	12
Strontium Nitrate.....	Lb.	22 @	28
Sulfur, flowers, sublimed.....	100 Lbs.	4.05 @	4.50
Sulfur, roll.....	100 Lbs.	3.70 @	4.10
Sulfuric Acid, chamber 66° Bé.....	Ton	37.00 @	40.00
Sulfuric Acid, oleum (fuming).....	Ton	60.00 @	65.00
Talc, American white.....	Ton	18.00 @	20.00
Terra Alba, American, No. 1.....	100 Lbs.	1.17 1/2 @	
Tin Bichloride, 50°.....	Lb.	28 @	30
Tin Oxide.....	Lb.	1.00 @	1.10
White Lead, American, dry.....	Lb.	9 1/4 @	10 1/4
Zinc Carbonate.....	Lb.	28 @	30
Zinc Chloride, commercial.....	Lb.	15 @	16
Zinc Oxide, American process XX.....	Lb.	14 @	15

## ORGANIC CHEMICALS

Acetanilid, C. P., in bbls.....	Lb.	68 @	70
Acetic Acid, 56 per cent, in bbls.....	Lb.	nominal	
Acetic Acid, glacial, 99 1/2%, in carboys.....	Lb.	65 @	70
Acetone, drums.....	Lb.	nominal	
Alcohol, denatured, 180 proof.....	Gal.	67 @	68

Alcohol, sugar cane, 188 proof.....	Gal.	4.87 @	4.92
Alcohol, wood, 95 per cent, refined.....	Gal.	90 1/2 @	91
Amyl Acetate.....	Gal.	5.25 @	5.30
Aniline Oil, drums extra.....	Lb.	26 @	28
Benzoic Acid, ex-toluol.....	Lb.	3.30 @	3.50
Benzol, pure.....	Gal.	23 @	28
Camphor, refined in bulk, bbls.....	Lb.	1.12 @	
Carbolic Acid, U. S. P., crystals, drums.....	Lb.	48 @	50
Carbon Bisulfide.....	Lb.	8 1/2 @	9
Carbon Tetrachloride, drums, 100 gals.....	Lb.	15 1/2 @	16
Chloroform.....	Lb.	63 @	65
Citric Acid, domestic, crystals.....	Lb.	82 @	83
Creosote, beechwood.....	Lb.	2.00 @	2.10
Cresol, U. S. P.....	Lb.	18 @	20
Dextrine, corn (carloads, bags).....	Lb.	8 @	9
Dextrine, imported potato.....	Lb.	19 @	20
Ether, U. S. P. 1900.....	Lb.	27 @	30
Formaldehyde, 40 per cent.....	Lb.	16 1/2 @	16 1/4
Glycerine, dynamite, drums included.....	Lb.	61 @	62
Oxalic Acid, in casks.....	Lb.	43 @	45
Pyrogallal Acid, resublimed, bulk.....	Lb.	3.10 @	
Salicylic Acid, U. S. P.....	Lb.	90 @	1.00
Starch, cassava.....	Lb.	—	
Starch, corn (carloads, bags) pearl.....	100 Lbs.	6.50 @	7.00
Starch, potato, Japanese.....	Lb.	15 @	16
Starch, rice.....	Lb.	8 @	10
Starch, sago flour.....	Lb.	9 1/4 @	9 1/2
Starch, wheat.....	Lb.	5 @	6
Tannic Acid, commercial.....	Lb.	60 @	75
Tartaric Acid, crystals.....	Lb.	75 @	78

## OILS, WAXES, ETC.

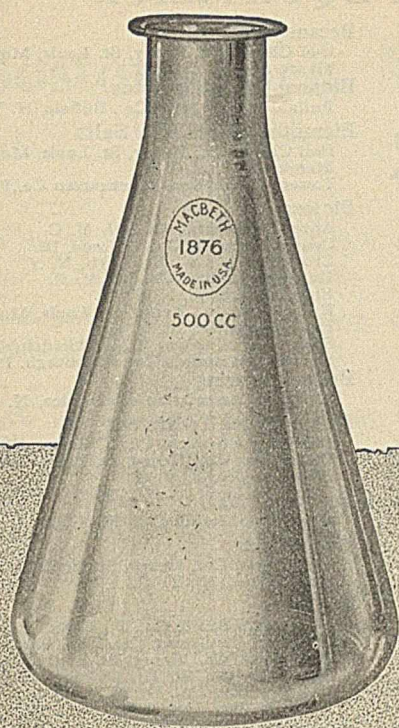
Beeswax, pure, white.....	Lb.	63 @	65
Black Mineral Oil, 29 gravity.....	Gal.	22 @	23
Castor Oil, No. 3.....	Lb.	29 @	33
Ceresin, yellow.....	Lb.	17 @	18
Corn Oil, crude.....	100 Lbs.	16.75 @	17.00
Cottonseed Oil, crude, f. o. b. mill.....	Lb.	17 1/2 @	—
Cottonseed Oil, p. s. y.....	100 Lbs.	21.00 @	22.00
Menhaden Oil, crude (southern).....	Gal.	95 @	1.00
Neat's-foot Oil, 20°.....	Gal.	3.45 @	3.55
Paraffin, crude, 118 to 120 m. p.....	Lb.	12 3/4 @	13
Paraffin Oil, high viscosity.....	Gal.	40 @	41
Rosin, "F" Grade, 280 lbs.....	Bbl.	10.00 @	
Rosin Oil, first run.....	Gal.	56 @	—
Shellac, T. N.....	Lb.	63 @	64
Spermaceti, cake.....	Lb.	32 @	34
Sperm Oil, bleached winter, 38°.....	Gal.	2.23 @	2.25
Spindle Oil, No. 200.....	Gal.	38 @	40
Stearic Acid, double-pressed.....	Lb.	23 @	25
Tallow, acidless.....	Gal.	1.58 @	1.60
Tar Oil, distilled.....	Gal.	35 @	36
Turpentine, spirits of.....	Gal.	63 @	64

## METALS

Aluminum, No. 1, ingots.....	Lb.	33 @	34
Antimony, ordinary.....	Lb.	13 @	13 1/4
Bismuth, N. Y.....	Lb.	3.50 @	3.65
Copper, electrolytic.....	Lb.	23.06 @	
Copper, lake.....	Lb.	23 1/2 @	—
Lead, N. Y.....	Lb.	7.25 @	7.50
Nickel, electrolytic.....	Lb.	55 @	56
Platinum, refined, soft.....	Oz.	nominal	
Silver.....	Oz.	99 1/2 @	
Tin, Straits.....	Lb.	nominal	
Tungsten (W0 <sub>2</sub> ).....	Per Unit	20.00 @	23.50
Zinc, N. Y.....	Lb.	7 1/4 @	8

## FERTILIZER MATERIALS

Ammonium Sulfate.....	100 Lbs.	7.50 @	7.75
Blood, dried, f. o. b. Chicago.....	Unit	6.70 @	6.75
Bone, 3 and 50, ground, raw.....	Ton	37.00 @	40.00
Calcium Cyanamide.....	Unit of Ammonia	nominal	
Calcium Nitrate, Norwegian.....	100 Lbs.	—	
Castor Meal.....	Unit	—	
Fish Scrap, domestic, dried, f. o. b. works.....	Unit	7.20 @	10.00
Phosphate, acid, 16 per cent.....	Ton	16.00 @	17.00
Phosphate rock, f. o. b. mine:	Ton	nominal	
Florida land pebble, 68 per cent.....	Ton	3.50 @	3.75
Tennessee, 78-80 per cent.....	Ton	5.50 @	6.00
Potassium "muriate," basis 80 per cent.....	Ton	nominal	
Pyrites, furnace size, imported.....	Unit	nominal	
Tankage, high-grade, f. o. b. Chicago.....	Unit	6.60 @	



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 Duriron Castings Co., N. Y. C.  
 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., N. Y. C.  
 Elyria Enamelled 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.  
 Devine Co., J. P., Buffalo, N. Y.  
 Jacoby, Henry E., N. Y. C.  
 Schutte & Koerting Co., Philadelphia, Pa.  
 U. S. Stoneware Co., The, Akron, O.

**Acid Pitchers**

Bausch & Lomb Optical Co., Rochester, N. Y.  
 Guernsey Earthenware 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.

**Acid Plants (Complete or in Part)**

Knight, M. A., East Akron, O.

**Acid Proof Brick**

Belden Brick Co., Canton, O.  
 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.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Palo Company, N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Acid Valves**

Cleveland Brass Mfg. Co., The, Cleveland, O.  
 Duriron Castings Co., N. Y. C.  
 General Ceramics Co., N. Y. C.  
 Schutte & Koerting Co., Philadelphia, Pa.  
 United Lined Tube & Valve Co., Boston, Mass.  
 York Manufacturing Co., York, Pa.

**Acid Ware**

General Ceramics Co., N. Y. C.

**Acid Ware (Vitrified Clay)**

Guernsey Earthenware 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, Col. & N. Y. C.  
 Duriron Castings Co., N. Y. C.  
 Elyria Enamelled 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 & Fñelderer 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.  
 Schutte & Koerting Co., Philadelphia, Pa.

**Air-Diffusing Plates**

General Filtration Co., Inc., Rochester, N. Y.

**Air Washers**

Carrier Engineering Corp., N. Y. C.

**Alundum Ware**

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 Norton Co., Worcester, Mass.  
 Scientific Materials Co., Pittsburgh, Pa.

**Ammeters**

Brown Instrument Co., Philadelphia, Pa.  
 General Electric Co., Schenectady, N. Y.

**Ammonia**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.  
 Unger, John S., Chicago, Ill.

**Ammonia Fittings and Supplies**

York Manufacturing Co., York, Pa.

**Ammonia—Salts of**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Ammonia Stills**

Badger & Sons Co., E. B., Boston, Mass.

**Analysis—Gas**

Sarco Co., Inc., N. Y. C.

**Analysis—Gas Apparatus**

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 Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 Eimer & Amend, N. Y. C.  
 Fry Glass Co., H. C., Rochester, Pa.  
 Guernsey Earthenware Co., The, Cambridge, O.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Herold China & Pottery Co., Golden, Col.  
 Kimble Glass Co., Vineland, N. J.  
 Macbeth-Evans Glass Co., Pittsburgh, Pa.  
 Palo Company, N. Y. C.  
 Sargent & Co., E. H., Chicago, Ill.  
 Whitall Tatum Co., Philadelphia, Pa.

Sarco Co., Inc., N. Y. C.

Sargent & Co., E. H., Chicago, Ill.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Tirrill Gas Mach. Ltg. Co., N. Y. C.

**Analytical Apparatus**

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.  
 Eberbach & Son Co., Ann Arbor, Mich.  
 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.  
 Tirrill Gas Mach. Ltg. Co., N. Y. C.

**Analytical Laboratories**

Columbus Laboratories, Chicago, Ill.  
 Dearborn Chemical Co., Chicago, Ill.  
 Sadtler & Son, Samuel P., Philadelphia, Pa.

**Aniline Oil—Salts of**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.

**Antimony—Salts of**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.

**Arsenic—Metallic & Salts**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Arsenic Plants (Acid Proof Stoneware)**

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**Asphalt & Tar Testing Apparatus**

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 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Standard Scientific Co., N. Y. C.  
 Thomas Co., Arthur H., Philadelphia, Pa.

**Autoclaves**

Central Scientific Co., Chicago, Ill.  
 Devine Co., J. P., Buffalo, N. Y.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Tirrill Gas Mach. Ltg. Co., N. Y. C.

**Autoclaves (Glass Enamelled 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**

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**Balances and Weights**

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 Braun Corporation, Los Angeles, Calif.  
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 Daigger & Co., A., Chicago, Ill.  
 Eberbach & Son Co., Ann Arbor, Mich.  
 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.  
 Schutte & Koerting Co., Philadelphia, Pa.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Tirrill Gas Mach. Ltg. Co., N. Y. C.

**Burners—Sulphur**

Schutte & Koerting Co., Philadelphia, Pa.

**Burnishing Pots and Baskets (Acid Proof)**

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**By-Product Machinery**

Devine Co., J. P., Buffalo, N. Y.  
 Swenson Evaporator Co., Chicago, Ill.

**Cable & Wire**

General Electric Co., Schenectady, N. Y.

**Cadmium—Salts of**

Dow Chemical Co., The, Midland, Mich.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Calcium—Salts of**

Dow Chemical Co., The, Midland, Mich.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Calorimeters—Fuel and Gas**

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.  
 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.

**Capsules**

Bausch & Lomb Optical Co., Rochester, N. Y.  
 Braun-Knecht-Heimann Co., San Francisco.

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Buffalo Fdy. & Mch. Co., Buffalo, N. Y.

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**Blowers**

Abbé Engineering Co., N. Y. C.  
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 Crowell Mfg. Co., Brooklyn, N. Y.  
 Duriron Castings Co., N. Y. C.  
 Eimer & Amend, N. Y. C.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Palo Company, N. Y. C.  
 Schutte & Koerting Co., Philadelphia, Pa.  
 Scientific Materials Co., Pittsburgh, Pa.

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Magnesia Association of America, N. Y. C.

**Boiler Water Treatment**

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 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.

**Bolting Cloth**

Abbé Engineering Co., N. Y. C.

**Bone Black**

Heil Chem. Co., Henry, St. Louis, Mo.  
 Toch Bros., N. Y. C.

**Books**

Blakiston's Son & Co., P., Phila., Pa.  
 Chemical Publishing Co., The, Easton, Pa.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Sargent & Co., E. H., Chicago, Ill.  
 Van Nostrand Co., D., N. Y. C.  
 Wiley & Sons, Inc., John, N. Y. C.

**Bottle Blowers**

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**Bottles (Glass)**

Central Scientific Co., Chicago, Ill.  
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 Wheaton Co., T. C., Millville, N. J.  
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 Palo Company, N. Y. C.  
 Scientific Materials Co., Pittsburgh, Pa.

**Burner Guards**

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 Knight, M. A., East Akron, O.

**Burners—Gas and Oil**

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 Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
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 Detroit Heating & Lighting Co., Detroit, Mich.  
 Eberbach & Son Co., Ann Arbor, Mich.  
 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.  
 Schutte & Koerting Co., Philadelphia, Pa.  
 Scientific Materials Co., Pittsburgh, Pa.  
 Thomas Co., Arthur H., Philadelphia, Pa.  
 Tirrill Gas Mach. Ltg. Co., N. Y. C.

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 Swenson Evaporator Co., Chicago, Ill.

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General Electric Co., Schenectady, N. Y.

**Cadmium—Salts of**

Dow Chemical Co., The, Midland, Mich.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Calcium—Salts of**

Dow Chemical Co., The, Midland, Mich.  
 Heil Chem. Co., Henry, St. Louis, Mo.  
 Merck & Co., N. Y. C.  
 Powers-Weightman-Rosengarten Co., Phila., Pa.

**Calorimeters—Fuel and Gas**

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 Braun Corporation, Los Angeles, Calif.  
 Braun-Knecht-Heimann Co., San Francisco.  
 Central Scientific Co., Chicago, Ill.  
 Daigger & Co., A., Chicago, Ill.  
 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.  
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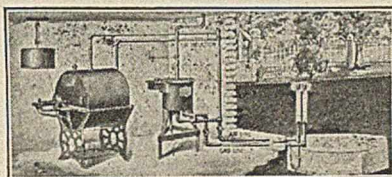
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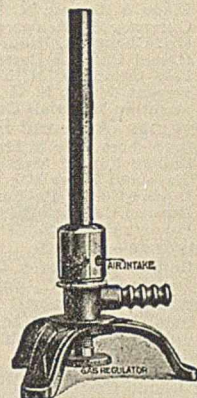
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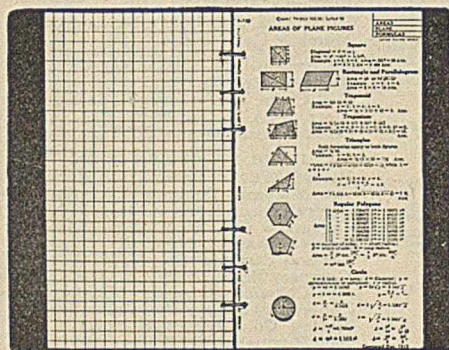
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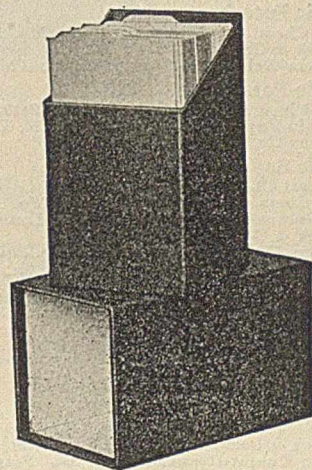
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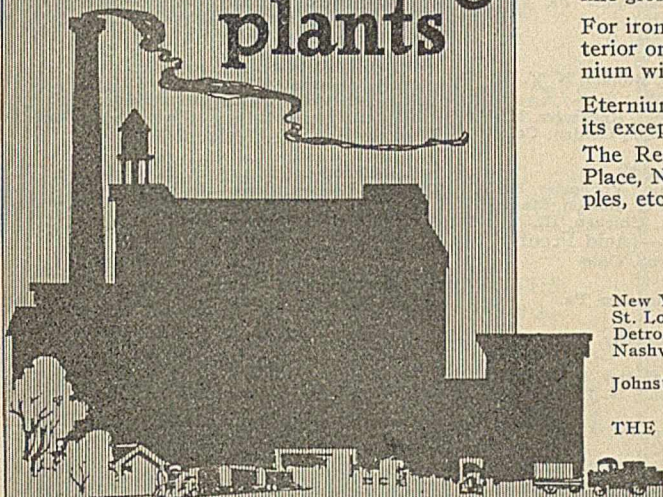
J.I. & E.C.  
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## CLASSIFIED LIST OF CHEMICAL EQUIPMENT—(Continued)

- Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.  
Thermal Syndicate, Ltd., The, N. Y. C.
- Carbon Bisulfide**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.
- Carbon Tetrachloride**  
Dow Chemical Co., The, Midland, Mich.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Carboys**  
Whitall Tatum Co., Philadelphia, Pa.
- Carboy Incliner**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.
- Carboy Stoppers**  
Whitall Tatum Co., Philadelphia, Pa.
- Carboy Stoppers (Acid Proof)**  
Knight, M. A., East Akron, O.
- Casseroles**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Duriron Castings Co., N. Y. C.  
Eimer & Amend, N. Y. C.  
Guernsey Earthenware Co., The, Cambridge, O.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Herold China & Pottery Co., Golden, Colo.  
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Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
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Bethlehem Fdy. & 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., N. Y. C.  
Jacoby, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.
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- Castings—Evaporator & Vacuum Pan**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Caustic Potash**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Caustic Pots**  
Bethlehem Fdy. & Mach. Co., So. Beth., Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Caustic Soda**  
General Chem. Co., N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Mallinckrodt Chemical Wks., St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
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- Cement Filler**  
Toch Bros., N. Y. C.
- Cement Refractory**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.
- Cement Testing Apparatus**  
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Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
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.
- 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.  
Eimer & Amend, N. Y. C.  
Heil Chemical Co., Henry, St. Louis, Mo.  
International Equipment Co., Boston, Mass.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Schaum & Uhlinger, Inc., Philadelphia, Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Sharples Specialty Co., The, West Chester, Pa.  
Tolhurst Machine Works, Troy, N. Y.
- Centrifuges**  
Central Scientific Co., Chicago, Ill.  
Heil Chem. Co., Henry, St. Louis, Mo.
- Centrifugal Dryers**  
Schaum & Uhlinger, Inc., Philadelphia, Pa.
- Centrifugal Engineers**  
Schaum & Uhlinger, Inc., Philadelphia, Pa.  
Sharples Specialty Co., The, West Chester, Pa.  
Tolhurst Machine Works, Troy, N. Y.
- Cerium Oxalate**  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Charcoal**  
Booth Apparatus Co., Syracuse, N. Y.  
Heil Chem. Co., Henry, St. Louis, Mo.
- Chemical and Process Patents**  
Foster, A. B., Washington, D. C.
- Chemical Apparatus**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Central Scientific Co., Chicago, Ill.  
Devine Co., J. P., Buffalo, N. Y.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Schaum & Uhlinger, Inc., Philadelphia, Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Standard Scientific Co., N. Y. C.  
Werner & Pfeleiderer Co., Inc., Saginaw, Mich.
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Kimble Glass Co., Vineland, N. J.
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Braun-Knecht-Heimann Co., San Francisco.  
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Eberbach & Son Co., Ann Arbor, Mich.  
Eimer & Amend, N. Y. C.  
Fry Glass Co., H. C., Rochester, Pa.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Laboratory Supply Co., Columbus, O.  
Macbeth-Evans Glass Co., Pittsburgh, Pa.  
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Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Standard Scientific Co., N. Y. C.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Wheaton Co., T. C., Millville, N. J.  
Whitall Tatum Co., Philadelphia, Pa.
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Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
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Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Knight, M. A., East Akron, O.  
Lummus Co., The Walter E., Boston, Mass.  
Swenson Evaporator Co., Chicago, Ill.
- Chemical Plants (Erected and Supervised)**  
Badger & Sons Co., E. B., Boston, Mass.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
McNamara, M., Everett, Mass.  
Swenson Evaporator Co., Chicago, Ill.
- Chemical Reagents**  
Baker Chem. Co., J. T., Phillipsburg, N. J.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
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.  
Palo Company, N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.  
Scientific Materials Co., Pittsburgh, Pa.
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Powers-Weightman-Rosengarten Co., Phila., Pa.
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Baker Chemical Co., J. T., Phillipsburg, N. J.  
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Braun-Knecht-Heimann Co., San Francisco.  
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Newport Chemical Works, Inc., N. Y. C.  
Palo Company, N. Y. C.  
Pfizer & Co., Charles, N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.  
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Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.  
Scientific Materials Co., Pittsburgh, Pa.
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Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.  
Scientific Materials Co., Pittsburgh, Pa.
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Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
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Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.
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Powers-Weightman-Rosengarten Co., Phila., Pa.
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Knight, M. A., East Akron, O.
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Merck & Co., N. Y. C.  
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- Classifiers**  
Dorr Co., The, Denver, Colo., & N. Y. C.
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Newport Chemical Works, Inc., N. Y. C.
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- Coal Testing Apparatus**  
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Heil Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.
- Cobalt—Metal & Salts**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
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Duriron Castings Co., N. Y. C.  
Knight, M. A., East Akron, O.  
United Lined Tube & Valve Co., Boston, Mass.  
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Braun-Knecht-Heimann Co., San Francisco.  
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Daigger & Co., A., Chicago, Ill.  
Eberbach & Son Co., Ann Arbor, Mich.  
Eimer & Amend, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
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Thomas Co., Arthur H., Philadelphia, Pa.
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- Combustion Tubes and Boats**  
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General Ceramics Co., N. Y. C.  
Guernsey Earthenware Co., The, Cambridge, O.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Laboratory Supply Co., Columbus, O.  
Norton Co., Worcester, Mass.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
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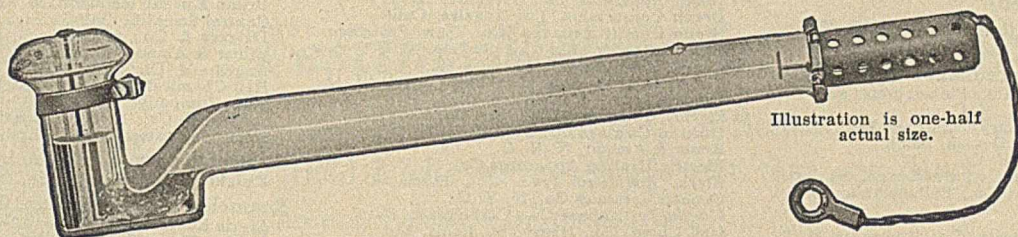


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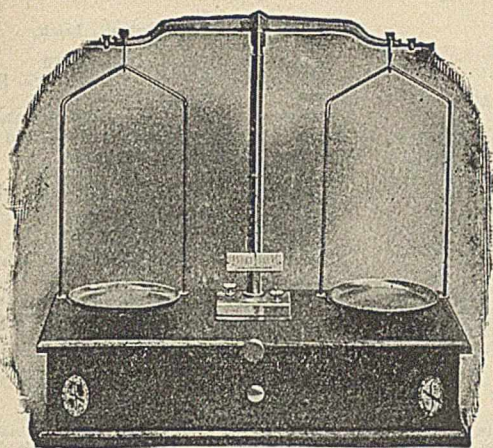
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Fort Worth Laboratories, The, Ft. Worth, Tex.  
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Unger, John S., Chicago, Ill.  
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Raymond Bros. Impact Pulv. Co., Chicago, Ill.  
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Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
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Norton Company, Worcester, Mass.
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Central Scientific Co., Chicago, Ill.  
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Griebel Instrument Co., Carbondale, Pa.  
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Palo Company, N. Y. C.  
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Whitall Tatum Co., Philadelphia, Pa.
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General Filtration Co., Inc., Rochester, N. Y.  
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Unger, John S., Chicago, Ill.  
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Heil Chem. Co., Henry, St. Louis, Mo.  
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Scientific Materials Co., Pittsburgh, Pa.
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Devine Co., J. P., Buffalo, N. Y.
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Carrier Engineering Corp., N. Y. C.
- Dryers—Atmospheric**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Dryers—Centrifugals**  
Schaum & Uhlinger, Inc., Philadelphia, Pa.
- Dryers—Vacuum**  
Badger & Sons Co., E. B., Boston, Mass.  
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**  
Badger & Sons Co., E. B., Boston, Mass.  
Birmingham Mch. & Fdy Co., Birmingham, Ala.
- Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Central Scientific Co., Chicago, Ill.  
Devine Co., J. P., Buffalo, N. Y.  
Eimer & Amend, N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Jacoby, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.  
Palo Company, N. Y. C.  
Werner & Pfeiderer Co., Saginaw, Mich.
- Duriron—Acid-Proof**  
Duriron Castings Co., N. Y. C.
- Dyestuffs**  
Kutroff, Pickhardt & Co., Inc., N. Y. C.  
Merck & Co., N. Y. C.  
Metz & Co., H. A., N. Y. C.  
National Aniline & Chemical Co., N. Y. C.
- Dynamos and Motors**  
General Electric Co., Schenectady, N. Y.
- Efficiency Gas Burners**  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Efficiency Instruments**  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Brown Instrument Co., Philadelphia, Pa.  
Eimer & Amend, 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.
- Electric Heating Devices**  
Central Scientific Co., Chicago, Ill.  
Heil Chem. Co., Henry, St. Louis, Mo.
- 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.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.  
Thwing Instrument Co., Philadelphia, Pa.
- Electrical Measuring Instruments**  
Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
General Electric Co., Schenectady, N. Y.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Leeds & Northrup Co., Phila., Pa.  
Palo Company, N. Y. C.
- Electrical Supplies**  
General Electric Co., Schenectady, N. Y.
- Electrodes—Platinum**  
American Platinum Works, Newark, N. J.  
Baker & Co., Inc., Newark, N. J.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Bishop & Co., J. Malvern 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.  
Eimer & Amend, N. Y. C.  
Engelhard, Chas., N. Y. C.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.
- 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.
- Enamel Paint (Impervious to Acid Fumes)**  
General Bakelite Co., N. Y. C.  
Toch Brothers, N. Y. C.
- Essential Oils**  
Merck & Co., N. Y. C.
- Evaporating Machinery**  
Badger & Sons Co., E. B., Boston, Mass.  
Birmingham Mch. & Fdy Co., Birmingham, Ala.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Company, J. P., Buffalo, N. Y.  
Electric Heating Apparatus 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.  
Zaremba Company, Buffalo, N. Y.
- Evaporating Pans and Dishes**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C. (Acid Proof.)  
Eimer & Amend, N. Y. C.  
Elyria Enameled Prod. Co., Elyria, O., & N. Y. C.  
General Ceramics Co., N. Y. C.  
Guernsey Earthenware Co., The, Cambridge, O.  
Heil 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.  
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U. S. Stoneware Co., The, Akron, O.





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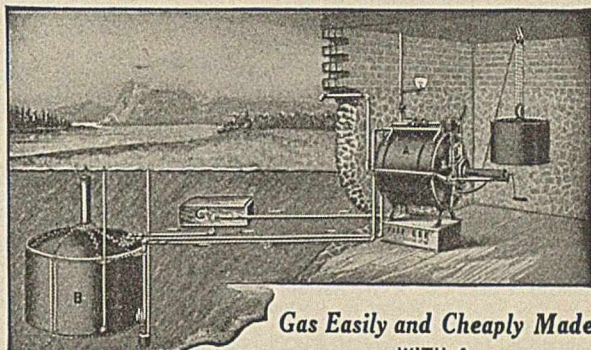
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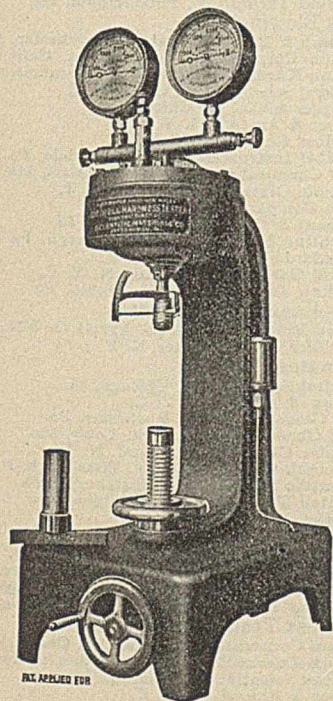
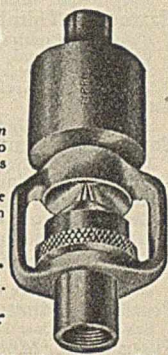
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Devine Co., J. P., Buffalo, N. Y.
- Evaporators—Single and Multiple Effects**  
Birmingham Mch. & Fdy. Co., Birmingham, Ala.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Exhaust and Ventilating Fans**  
General Electric Co., Schenectady, N. Y.
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Duriron Castings Co., N. Y. C.
- Extracting Apparatus**  
Badger & Sons Co., E. B., Boston, Mass.  
Central Scientific Co., Chicago, Ill.  
Devine Co., J. P., Buffalo, N. Y.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Lummus Co., The Walter E., Boston, Mass.  
Schaum & Uhlinger, Inc., Philadelphia, Pa.  
Scientific Materials Co., Pittsburgh, Pa.
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Badger & Sons Co., E. B., Boston, Mass.  
Lummus Co., The Walter E., Boston, Mass.
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Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.
- Extraction Thimbles (Greens)**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Eimer & Amend, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Extractors**  
Schaum & Uhlinger, Inc., Philadelphia, Pa.
- Extractors—Centrifugal**  
International Equipment Co., Boston, Mass.  
Schaum & Uhlinger, Inc., Philadelphia, Pa.
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General Electric Co., Schenectady, N. Y.
- Faucets (Stoneware—Acid Proof)**  
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Knight, Maurice A., East Akron, Ohio.  
U. S. Stoneware Co., The, Akron, O.
- Fertilizer Industries Equipment**  
Schutte & Koerting Co., Philadelphia, Pa.
- Filter Cloth (Metallic)**  
Sweetland Filter Press Co., Brooklyn, N. Y.
- Filtering Media**  
General Filtration Co., Inc., Rochester, N. Y.  
Herold China & Pottery Co., Golden, Colo.
- Filter Paper**  
Angel Co., Inc., H. Reeve, N. Y. C.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
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.  
Whitall Tatum Co., Philadelphia, Pa.
- Filter Paper Clippings**  
Angel Co., Inc., H. Reeve, N. Y. C.
- Filter Paper (Green's)**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Filter Paper (Whatman)**  
Angel Co., H. Reeve, N. Y. C.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.
- 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.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Independent Filter Press Co., Inc., 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.  
Shriver & Co., T. Harrison, N. J.  
Sperry & Co., D. R., Batavia, Ill.  
Sweetland Filter Press Co., Brooklyn, N. Y.
- Filter Pulp**  
Angel Co., Inc., H. Reeve, N. Y. C.
- Filters**  
General Filtration Co., Inc., Rochester, N. Y.  
Norton Co., Worcester, Mass.
- Filters—(Acid Proof)**  
General Filtration Co., Inc., Rochester, N. Y.  
Knight, Maurice A., East Akron, Ohio.
- 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.  
Heil Chem. Co., Henry, St. Louis, Mo.
- Filters, Folded (Greens)**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Eimer & Amend, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Filters—Vacuum**  
Devine Co., J. P., Buffalo, N. Y.  
General Filtration Co., Inc., Rochester, N. Y.
- Filters—Water**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
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.  
Eimer & Amend, N. Y. C.  
General Ceramics Co., 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.  
Schutte & Koerting Co., Philadelphia, Pa.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Filtros**  
General Filtration Co., Inc., Rochester, N. Y.
- Filtros—Vacuum**  
Devine Company, J. P., Buffalo, N. Y.
- Fire Brick and Clay**  
Belden Brick Co., Canton, O.
- Flasks**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Fry Glass Co., H. C., Rochester, Pa.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.
- Fuel Gas Plants**  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Funnels**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Knight, M. A., East Akron, Ohio.  
Whitall Tatum Co., Philadelphia, Pa.
- Furnaces—Annealing—(Hardening and Enameling)**  
Abbé Engineering Co., N. Y. C.  
Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.  
Electric Heating Apparatus Co., N. Y. C.  
General Electric Co., Schenectady, N. Y.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Hoskins Mfg. Co., Detroit, Mich.  
Palo Company, N. Y. C.
- Furnaces (Case Hardening, Rivet and Bolt)**  
Abbé Engineering Co., N. Y. C.
- Furnaces—Electric Laboratory**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
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.  
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.  
Heil 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.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Furnaces—(Forge)**  
Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Electric Heating Apparatus Co., N. Y. C.  
Hoskins Mfg. Co., Detroit, Mich.
- Furnaces (Melting, Oil or Gas)**  
Abbé Engineering Co., N. Y. C.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Electric Heating Apparatus Co., N. Y. C.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Furnaces—Muffle**  
Abbé Engineering Co., N. Y. C.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Electric Heating Apparatus Co., N. Y. C.  
Heil 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.
- Furnaces—Sulphur**  
Schutte & Koerting Co., Philadelphia, Pa.
- Galvanizing Tanks and Jars—Acid Proof**  
Knight, M. A., East Akron, O.
- Gas Analyzers**  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.
- Gas Apparatus**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Palo Company, N. Y. C.  
Schutte & Koerting Co., Philadelphia, Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Gas Burners—Incandescent**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Gas Machines**  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Detroit Heating & Lighting Co., Detroit, Mich.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Gas Pipe & Fittings (Chamotte)**  
Knight, M. A., East Akron, O.
- Gauges—Recording, Suction & Vacuum**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Brown Instrument Co., Philadelphia, Pa.  
Heil Chem. Co., Henry, St. Louis, Mo.
- Gauze—Base Metal**  
Hoskins Mfg. Co., Detroit, Mich.
- Generators (Electro-Oxy-Hydrogen)**  
Shriver & Co., T. Harrison, N. J.
- Glass**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eberbach & Son Co., Ann Arbor, Mich.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.
- Glass Blowing**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Greiner Co., Emil, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Scientific Materials Co., Pittsburgh, Pa.  
Standard Scientific Co., N. Y. C.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.  
Whitall Tatum Co., Philadelphia, Pa.
- Glass Enameled Apparatus**  
Elyria Enameled Prod. Co., Elyria, O., & N. Y. C.  
Pfauder Co., Rochester, N. Y.
- Graduates—Glass**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Fry Glass Co., H. C., Rochester, Pa.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Macbeth-Evans Glass Co., Pittsburgh, Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Whitall Tatum Co., Philadelphia, Pa.
- Grinders**  
Abbé Engineering Co., N. Y. C.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Raymond Bros. Impact Pulv. Co., Chicago, Ill.  
Williams Pat. Crusher & Pulv. Co., Chicago, Ill.
- Hardness Testing Apparatus**  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.
- Heat Exchangers (Multi-Flow Design)**  
Birmingham Mch. & Fdy. Co., Birmingham, Ala.
- Heat Insulation**  
Magnesia Association of America, N. Y. C.
- Heating Apparatus (Laboratory)**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Brown Instrument Co., Philadelphia, Pa.  
Central Scientific Co., Chicago, Ill.

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Distilling 100% within a range of 2° C.

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## Xylol

Distilling 100% between 137° C. and 142° C.

## Mono Nitronaphthalin

Melting-point 50° C., or above.

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Distilling 90% at 160° C.

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Distilling 100% between 150° C. and 200° C.  
Guaranteed flash-point above 100° F.

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17 Battery Place



New York, N. Y.

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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.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Heating Specialties**  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Hot Plates (Laboratory Purposes)**  
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.  
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.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Hydrochloric Acid Apparatus**  
Knight, M. A., East Akron, O.
- Hydro Extractors**  
Schaum & Uhlinger, Inc., Philadelphia, Pa.  
Tolhurst Machine Works, Troy, N. Y.
- Hydrometers**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
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.  
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.  
Taylor Instrument Cos., Rochester, N. Y.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Hygrometers**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Taylor Instrument Cos., Rochester, N. Y.
- Ice-Making Machinery**  
General Electric Co., Schenectady, N. Y.  
York Manufacturing Co., York, Pa.
- Ichthyol**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.
- Impregnating and Drying Apparatus—Vacuum**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Incinerators**  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Incutors—Surgical**  
Central Scientific Co., Chicago, Ill.  
Hell Chem. Co., Henry, St. Louis, Mo.
- Indicating Instruments**  
Brown Instrument Co., The, Phila., Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Taylor Instrument Cos., Rochester, N. Y.
- Indicators—(Speed & Test)**  
Brown Instrument Co., Philadelphia, Pa.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.
- Industrial Burners & Specialties**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Industrial Motor Control Devices**  
General Electric Co., Schenectady, N. Y.
- Injectors for Acids and Gases**  
Knight, M. A., East Akron, O.
- Instrument—Precision Manufacturers**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.
- Iodine—Salts of**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Iron—Metal and Salts**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Iron and Steel Analysis Apparatus—Manufacturers**  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.
- Jars (Hydrometer)**  
Fry Glass Co., H. C., Rochester, Pa.  
Kimble Glass Co., Vineland, N. J.
- Jars, Museum & Specimen**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Whitall Tatum Co., Philadelphia, Pa.
- Jars (for Primary & Storage Batteries)**  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
General Ceramics Co., N. Y. C.  
Guernsey Earthenware Co., The, Cambridge, O.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Whitall Tatum Co., Philadelphia, Pa.
- Jars and Jugs (Acid Storage)**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
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.
- Kettles—Caustic Soda**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.
- Kettles (Chemical Castings)**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C.  
Jacob, Henry E., N. Y. C.  
Sowers Mfg. Co., Buffalo, N. Y.
- Kettles (Lined)**  
Badger & Sons Co., E. B., Boston, Mass.  
Devine Co., J. P., Buffalo, N. Y.  
Elyria Enameled Prod. Co., Elyria, O., N. Y. C.  
Pfauder Co., Rochester, N. Y.  
Sowers Mfg. Co., Buffalo, N. Y.
- Kettles (Steam Jacketed)**  
Badger & Sons Co., E. B., Boston, Mass.  
Bethlehem Fdy. & Mch. Co., So. Bethlehem, Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C.  
Elyria Enameled Prod. Co., Elyria, O.  
Jacob, Henry E., N. Y. C.  
Pfauder 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.
- Kilns (Bone Black)**  
Collwell, Lewis, Chicago, Ill.
- Laboratory Apparatus—Designing & Manufacturing**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Sargent & Co. E. H., Chicago, Ill.  
Schaum & Uhlinger, Inc., Philadelphia, Pa.  
Scientific Materials Co., Pittsburgh, Pa.  
Standard Scientific Co., N. Y. C.
- Laboratory Burners**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Laboratory Chemicals**  
Central Scientific Co., Chicago, Ill.  
Dow Chemical Co., The, Midland, Mich.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Laboratory Gas Machine**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.
- Laboratory Glassware**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.
- Laboratory Supplies**  
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.  
Duriron Castings Co., N. Y. C.  
Eimer & Amend, N. Y. C.  
Greiner Co., Emil, 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.  
Standard Scientific Co., N. Y. C.  
Thomas Co., Arthur H., Philadelphia, Pa.  
Tirrill Gas Mach. Ltg. Co., N. Y. C.  
Whitall Tatum Co., Philadelphia, Pa.
- Laboratory Supplies, Platinum**  
Central Scientific Co., Chicago, Ill.  
Baker & Co., Inc., Newark, N. J.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.
- Lacquer**  
General Bakelite Co., N. Y. C.
- Lactic Acid**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- 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 Lined Pipe and Accessories**  
Badger & Sons Co., E. B., Boston, Mass.  
Schutte & Koerting Co., Philadelphia, Pa.  
United Lined Tube & Valve Co., Boston, Mass.
- Lightning Arresters**  
General Electric Co., Schenectady, N. Y.
- Linings, Cupola**  
Herold China & Pottery Co., Golden, Col.
- Lithopone**  
New Jersey Zinc Co., The, N. Y. C.
- Lubricants**  
Dearborn Chemical Co., Chicago, Ill.
- Machinery—Chemical**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., 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, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
General Filtration Co., Inc., Rochester, N. Y.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Stokes Machine Co., F. J., Philadelphia, Pa.
- Machinery—Mixing and Kneading**  
Abbé Engineering Co., N. Y. C.  
Elyria Enameled Products Co., Elyria, O.  
Jacob, Henry E., N. Y. C.  
Pfauder 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, Cal.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Raymond Bros. Impact Pulv. Co., Chicago, Ill.
- Machinery (Special)**  
Badger & Sons Co., E. B., Boston, Mass.  
Birmingham Mch. & Fdy. Co., Birmingham, Ala.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C.  
Jacob, 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.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Mercury—Salts of**  
Hell Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- 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.  
Palo Company, N. Y. C.  
Scientific Materials Co., Pittsburgh, Pa.
- Metallurgical Laboratory Equipment**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Braun Corporation, Los Angeles, Cal.  
Braun-Knecht-Heimann Co., San Francisco, Cal.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, 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.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Meters—Flow Air, Gas, Water**  
General Electric Co., Schenectady, N. Y.
- Microscopes**  
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.  
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.
- Microtomes**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Hell Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Micro Photo. Apparatus Mfrs.**  
Eimer & Amend, N. Y. C.  
Hell Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.



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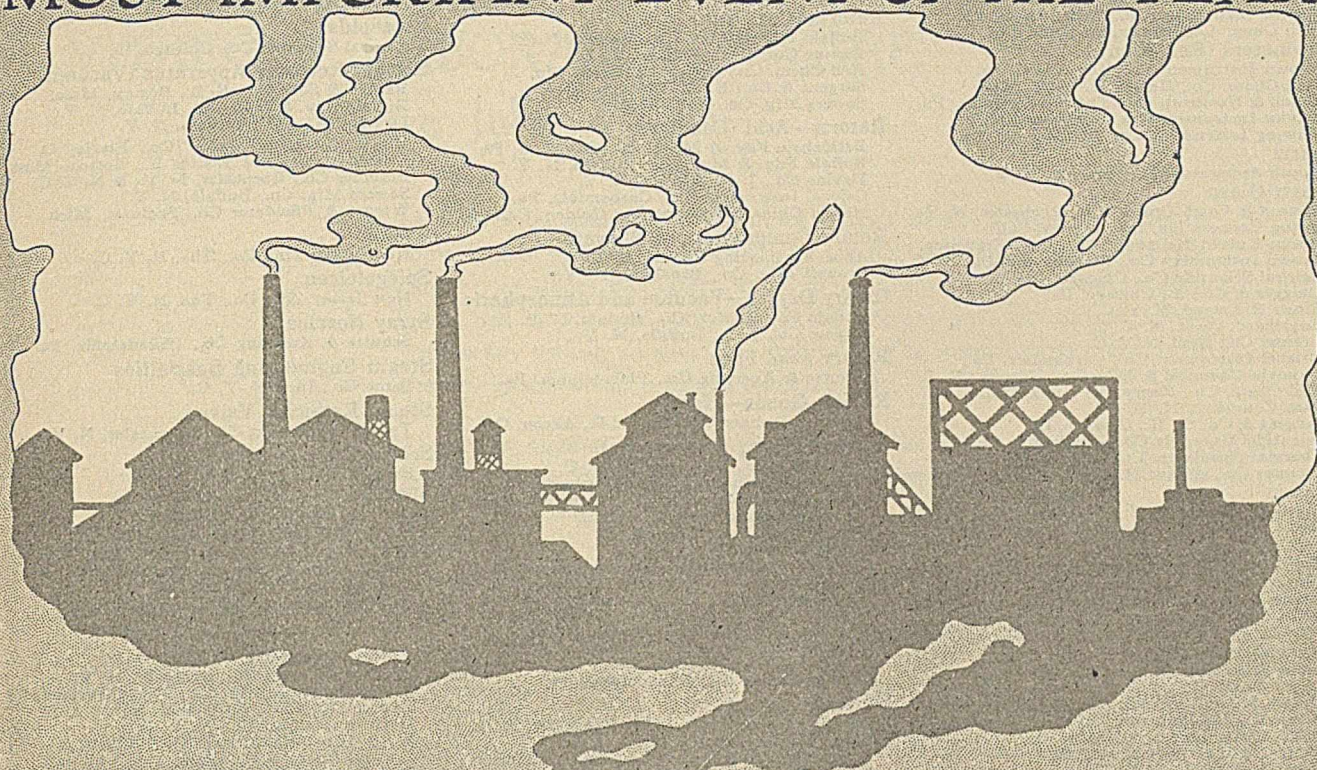
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# GOODRICH

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- Mills—Ball, Pebble and Tube**  
Abbé Engineering Co., N. Y. C.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.
- 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**  
Badger & Sons Co., E. B., Boston, Mass.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C.  
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**  
Central Scientific Co., Chicago, Ill.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Molybdic Acid**  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Scientific Materials Co., Pittsburgh, Pa.
- 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., Philadelphia, Pa.
- Mortars & Pestles**  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Kimble Glass Co., Vineland, N. J.  
Whittall Tatum Co., Philadelphia, Pa.
- Muffles**  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Norton Co., Worcester, Mass.
- Nickel—Salts of**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Nitrating Apparatus**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Schaum & Uhlinger, Inc., Philadelphia, Pa.
- Nitrating Pots**  
Bethlehem Fdy. & Mch. Co., So. Bethlehem, Pa.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C.  
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., South 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., N. Y. C. (Special Alloy.)  
Elyria Enamelled Prod. Co., Elyria, O.  
General Ceramics Co., N. Y. C.  
Thermal Syndicate, Ltd., N. Y. C. (Silica Ware.)
- Nitric Acid Apparatus**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Duriron Castings Co., N. Y. C.
- 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., N. Y. C.  
Schutte & Koerting Co., Philadelphia, 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.  
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.  
Taylor Instrument Cos., Rochester, N. Y.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Optical Instruments**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Thwing Instrument Co., Philadelphia, Pa.
- Optical Pyrometer**  
Heil Chem. Co., Henry, St. Louis, Mo.
- Palo Company, N. Y. C.**  
Scientific Materials Co., Pittsburgh, Pa.
- Oxygen (Pure)**  
Levin, Isaac H., Newark, N. J.  
Packings (Condensing Tower)  
General Ceramics Co., N. Y. C.  
Knight, Maurice A., East Akron, O.  
U. S. Stoneware Co., The, Akron, O.
- Paints**  
Toch Brothers, N. Y. C.
- Panels, Rheostats, Motor Generator Sets and Tungar Rectifiers**  
General Electric Co., Schenectady, N. Y.
- Paper, All Kinds**  
Angel Co., Inc., H. Reeve, N. Y. C.
- Paranitraniline**  
Merck & Co., N. Y. C.
- Partition Rings (Chemical Acid Proof)**  
Knight, Maurice A., East Akron, O.
- Patent Attorneys**  
Brickenstein, John H., Washington, D. C.  
Foster, A. B., Washington, D. C.  
Parker, C. L., Washington, D. C.  
Townsend, Clinton P., Washington, D. C.
- Perchloric Acid, C. P.**  
Genesee Chemical Co., Batavia, N. Y.
- Percolators**  
Badger & Sons Co., E. B., Boston, Mass.  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Devine Co., J. P., Buffalo, N. Y.  
Elyria Enamelled Prod. Co., Elyria, O.  
General Ceramics Co., N. Y. C.  
Pfaudler Co., Rochester, N. Y.  
Whittall Tatum Co., Philadelphia, Pa.
- Perhydrol**  
Heil Chem. Co., Henry, St. Louis, Mo.  
Merck & Co., N. Y. C.
- Pharmaceutical Dryers**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.  
Werner & Pfeiderer Co., Inc., Saginaw, Mich.
- Pharmaceutical Preparations**  
Merck & Co., N. Y. C.  
Powers-Weightman-Rosengarten Co., Phila., Pa.
- Phenol Apparatus**  
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
- Photomicrography**  
Gravelle, Philip O., S. Orange, N. J.
- Photo-Micrographic Apparatus**  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Central Scientific Co., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Thomas Co., Arthur H., Philadelphia, Pa.
- Physical Apparatus**  
Standard Scientific Co., N. Y. C.
- Picric Acid Apparatus**  
Knight, Maurice A., East Akron, O.
- Pipe and Accessories (Acid Proof)**  
Bethlehem Fdy. & Mch. Co., So. Bethlehem, Pa.  
Cleveland Brass Mfg. Co., The, Cleveland, O.  
Duriron Castings Co., N. Y. C.  
Elyria Enamelled Prod. Co., Elyria, O.  
General Ceramics Co., N. Y. C.  
Herold China & Pottery Co., Golden, Col.  
Knight, Maurice A., East Akron, O.  
Schutte & Koerting Co., Philadelphia, Pa.  
Thermal Syndicate, Ltd., N. Y. C. (Fused Silica.)  
United Lined Tube & Valve Co., Boston, Mass.  
U. S. Stoneware Co., The, Akron, O.
- Pipe and Fittings—Lead or Tin Lined**  
Schutte & Koerting Co., Philadelphia, Pa.  
United Lined Tube & Valve Co., Boston, Mass.
- Pipe Coverings**  
The Magnesia Association of America, N. Y. C.
- Pipes—Steam Jacketed**  
Devine Co., J. P., Buffalo, N. Y.
- Pitch for Protective Coatings**  
The Barrett Co., N. Y. C.
- Platinum (Sheet, Wire and Foil)**  
American Platinum Works, Newark, N. J.  
Baker & Co., Inc., Newark, N. J.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Bishop & Co., J., Platinum Works, Malvern, Pa.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Heil Chemical Co., Henry, St. Louis, Mo.  
Palo Company, N. Y. C.  
Sargent & Co., E. H., Chicago, Ill.
- Platinum Ware (Laboratory)**  
American Platinum Works, Newark, N. J.  
Baker & Co., Inc., Newark, N. J.  
Bausch & Lomb Optical Co., Rochester, N. Y.  
Bishop & Co., J., Platinum Works, Malvern, Pa.  
Braun Corporation, Los Angeles, Calif.  
Braun-Knecht-Heimann Co., San Francisco.  
Central Scientific Co., Chicago, Ill.  
Daigger & Co., A., Chicago, Ill.  
Eimer & Amend, N. Y. C.  
Engelhard, Chas., 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.
- Porcelain Ware—Laboratory**  
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.  
Eimer & Amend, N. Y. C.  
Griebel Instrument Co., Carbondale, Pa.  
Guernsey Earthenware Co., The, Cambridge, O.  
Heil Chem. Co., Henry, St. Louis, Mo.  
Herold China & Pottery Co., Golden, Col.  
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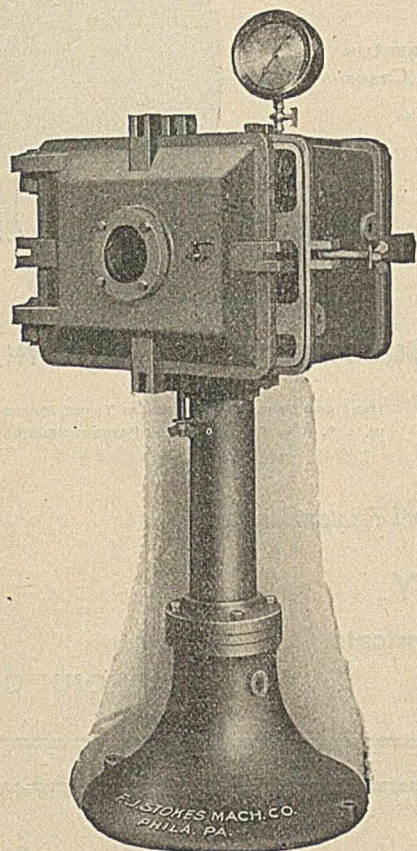
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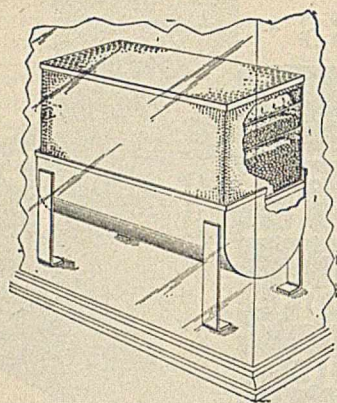
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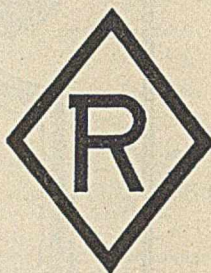
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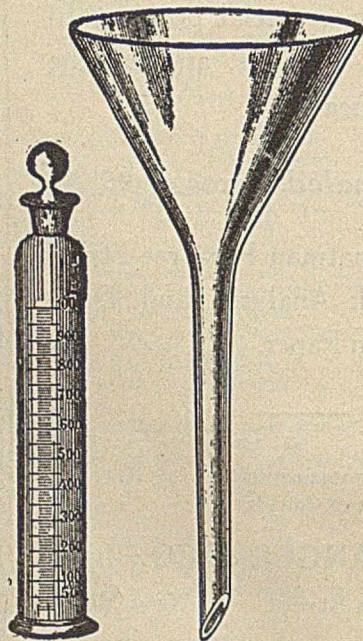
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