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EDITORIALS

THE FORTY-EIGHTH ANNUAL MEETING OF THE AMERICAN CHEMICAL SOCIETY

To the members who have learned from experience the value of our general meetings no appeal need be made to continue in their attendance. To those who go intermittently or who have not as yet attended, we would emphasize the importance of these meetings and of the Rochester meeting (September 9-12) in particular. Our general meetings afford benefits of several kinds. Most important is the opportunity to meet the foremost men in the chemical profession and to talk over matters of mutual interest. Then there is the discussion of papers which is frequently of more value to us than the papers themselves, and the discussions are not published. Again, the Society visits manufacturing institutions of interest to them, and each city offers something unique. Further, the social features, the get-together sessions, the renewal of friendships, all add their quota of attraction.

The chemist going to the general meeting for the first time is liable to feel himself among strangers. He makes a few friends, meets some men from the old school and gets enjoyment from the meetings, but is apt to think that the trip was hardly worth the cost and is doubtful about going to the next meeting. A mistake is made if this is followed out, for the men deriving the greatest benefits from the American Chemical Society meetings are those who go regularly.

We are about to hold our first annual meeting as a separate society on the plan now in force which puts us on our own feet as an independent organization, standing out as the largest chemical society in the world. Every effort is being made by the Rochester Section to offer attractions which will guarantee an attendance in proportion to the importance of the meeting. Rochester is within easy distance of a large majority of our members and has excellent transportation facilities. Both academic and industrial men will be well cared for, the general meeting being scheduled at the great works of the Eastman Kodak Co. and the sectional meetings at the University of Rochester. Factory excursions will be a special feature covering a field from the production of the instruments used by the chemical profession to works making the most modern application of chemistry to industrial problems. Factories, the duplicates of which do not exist in this country or abroad, will be visited. Social features will be prominent and a good program has been arranged for the ladies.

We urge members to be in attendance the first day, for such a unique program has never been planned for this Society. The general meeting at Kodak Park will be followed by a complimentary luncheon at the Kodak Works and an inspection of the plant, this being the only opportunity of making the inspection. In the evening comes the smoker, full of novelty, amusement and good fellowship. A live committee has been working on this event for months for your benefit.

Let the members of the American Chemical Society descend upon Rochester in unprecedented numbers for the first day, Sept. 9th, and make the forty-eighth meeting the best ever! All will be amply repaid.

WOOD ALCOHOL

Since man began handling fire he has been utilizing dangerous substances to his own good purposes. Also the chemist has discovered many substances and shown how they might be used for the benefit of mankind; he has demonstrated the value of cyanides for extracting gold; how strychnine may serve as a heart stimulant; he has proved the value of phenol as a disinfectant; and how sulfuric acid may be used in multitudinous ways. All these substances are dangerous to handle, in fact many of the commoner reagents used in the hundreds of laboratories and factories are poisons. Were partial facts only presented, it is conceivable that they might assemble themselves in astounding array, which, if not properly interpreted, would serve for a cause of impeachment of the entire chemical profession, whose daily routine is one of handling poisons of all kinds, except for the fact that the chemist is supposed to know his business as well as the substances he handles, and to take some precautions for safeguarding himself and the people working with him. It is a matter of business, even if it presented no humanitarian features.

Our attention has recently been directed to attacks made upon methyl alcohol, the production and sale of which constitutes a legitimate chemical business involving the annual manufacture and use of about 10,000,000 gallons of that substance with a capital investment in this country of about \$12,000,000, an industry which employs over 3,000 working people. We find in current literature, read by the many, assertions made by undoubtedly honest, but overzealous and presumably misinformed persons, or persons not sufficiently informed of the facts to be competent to judge according to proportionality, such as the following: "It is unsafe to burn wood alcohol or use it in any way in a room where the air is close. *The bottle should never be uncorked unless the windows are wide open.*" (Italics ours.) Where would all the nearly 7,000 members of the American Chemical Society be if the latter were really true?

There is no doubt about wood alcohol being a poison if taken internally (although we know of cases, perhaps accounted for by idiosyncrasy, where no evil effects have resulted from drinking it) and if breathed as a vapor in a concentrated form for unusually long periods of time. In fact, nearly a thousand cases of poisoning attributed to wood alcohol (mostly due to drinking it) have been reported in the literature since 1899, the time which marked the advent of such preparations as "Columbian Spirits," "Colonial Spirits," etc.—that is to say, methyl alcohol of a high grade of purity. In 1906, due to a general agitation for a tax-free, denatured ethyl alcohol, hearings were held before the Ways and Means

and the Finance Committees of the Federal Congress. The injurious action of wood alcohol on the general health and eyesight of working people handling it in the industries was strongly emphasized at these hearings by manufacturers employing it, workmen and experts.

The United States and practically every state in the union have specific laws against the sale of wood alcohol as a beverage, or as an addition to beverages. In thickly populated communities the fear of detection is lessened, hence cases of adulteration are more numerous, especially where there is foreign cheap labor. That fact (especially in New York and New Jersey) coupled with a few cases of serious poisoning by wood alcohol in varnishing brewers' vats, which are not properly ventilated, caused the New York State Factory Investigating Commission to look into the wood alcohol situation. The Commission invited Professor Baskerville of the College of the City of New York, then Chairman of the Committee on Occupational Diseases in the Chemical Trades of the New York Section and chemical adviser of the Committee on Occupational Diseases of the New York State Labor Association and now Chairman of the Committee on Occupational Diseases of the American Chemical Society, to make a report on "Wood Alcohol." This report, based on a thorough investiga-

tion of the literature and extensive inspection of works of all kinds where wood alcohol is made and used in various ways, we have been privileged to abstract previous to final publication by the State, which abstract appears elsewhere in this issue. The full report may be secured by writing to the Commissioner of Labor, Albany, N. Y.

Wood alcohol presents a unique case for legislation, not only on account of its general resemblance to ethyl alcohol, but especially on account of the word "alcohol," which has a definite meaning to the chemist, but is more associated in the lay mind with "drink."

Methyl alcohol is used extensively as a valuable solvent and in the manufacture of many important materials. Its legitimate use should not be throttled. The present laws in regard to its use as an adulterant of beverages or in any preparation intended for internal use or external application on the human body are in most states now adequate, but they should be more rigidly enforced. Where inadequate, we trust that reasonable legislation, such as is recommended in the report referred to above, may be enacted. At present, however, we insist upon the rigid execution of the laws as they exist before further encumbering the codes.

ORIGINAL PAPERS

THE MICROSTRUCTURE OF STEEL CASTINGS

BY WIRT TASSIN

Received July 23, 1913

INTRODUCTION

This paper deals with the results of some metallographic investigations of steel castings. All of the micrographic work was done in the machine shop on the castings themselves, not on small sections cut from them.

The work was undertaken during the course of what is planned to be a fairly comprehensive study of the relation between structure and physical properties of steel in the mass, and intended for use as a guide by the Inspector and Engineer of Tests.

The present paper gives the results of the work on Tropenas steel castings, and is intentionally made as non-technical as possible.

INFLUENCE OF ANNEALING ON STRUCTURE

A steel casting in the "green," especially when of a complicated shape, is always liable to be under internal strain. In order to relieve these strains annealing is resorted to. This process has a marked effect upon the structure of the metal.

The structure of a steel casting in the "green," *i. e.*, as it leaves the mold, is coarsely crystalline and exhibits to a greater or less degree a regularly arranged network, the meshes of which usually intersect at some angle of an octahedron (Fig. 1). This structure is known as "ingotism."

Heat-treating such a casting breaks down this original crystallization and, if properly carried out, effaces it. Thus in a properly annealed casting the coarse crystal-

line structure gives place to another which is fine-grained and of a uniform distribution (Fig. 2).

The degree to which this change takes place is limited by the temperature reached, its uniformity, the time that it is maintained, the rate of cooling, and the size and shape of the casting.

Given the proper temperature, but let the time be too short to permit of a complete re-arrangement, the structure will be analogous to that seen in Fig. 3, in which the original structure is readily traced by the parallel grouping of the new crystallization. With a still shorter time period there is only a partial re-crystallizing, and more or less of the original network is visible (Fig. 4).

Let the time be long enough, but the temperature be too low, there will be again a partial re-grouping with more or less of the original crystallization present (Fig. 5).

Let the temperature be right but the time period be too long, all traces of the original structure will be destroyed, but in place of the fine granular structure seen in Fig. 2 there will be a much coarser one (Fig. 6) characterized by the large-sized areas of a certain kind.

Let the temperature be too high and the increase in size of certain areas become marked (Fig. 7).

RELATION OF STRUCTURE TO PHYSICAL PROPERTIES

The character of the structure bears a relation to the physical properties of the metal in the casting, the finer the grain, the more uniform its distribution and the freer it is from occluded foreign matter as slag, sulfide, oxide, etc., the better will be the physical properties.

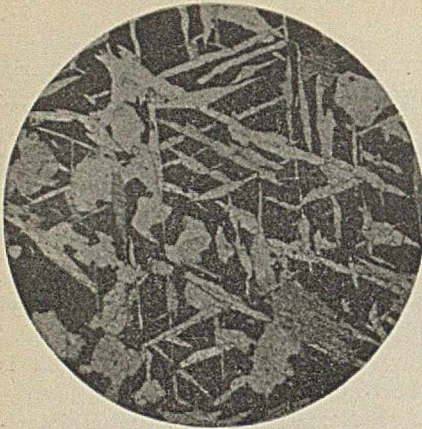


FIG. 1. $\times 65$

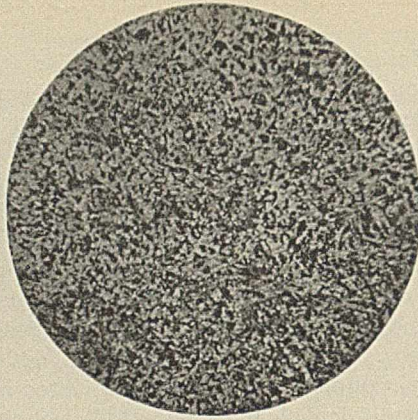


FIG. 2. $\times 65$

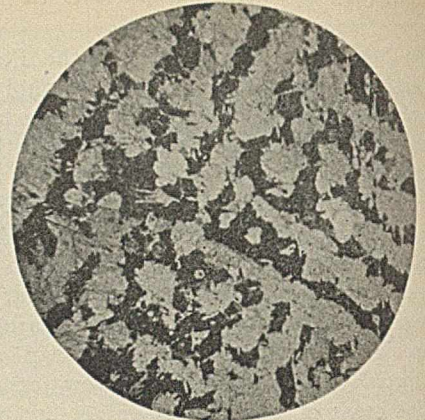


FIG. 3. $\times 65$

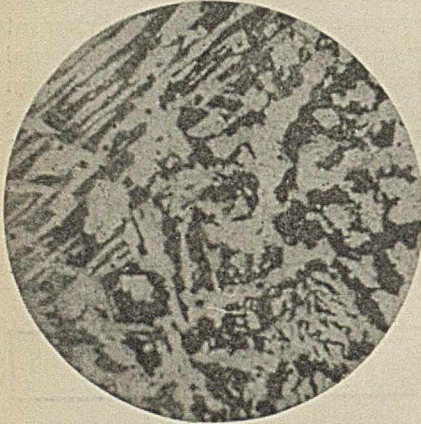


FIG. 4. $\times 65$

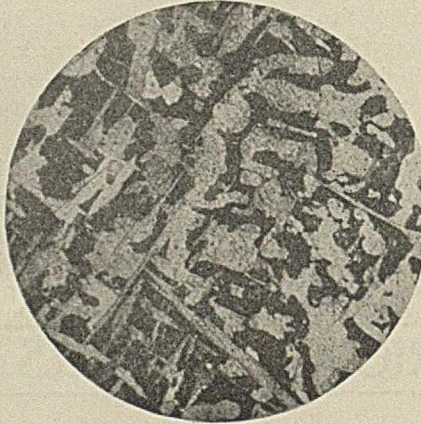


FIG. 5. $\times 65$

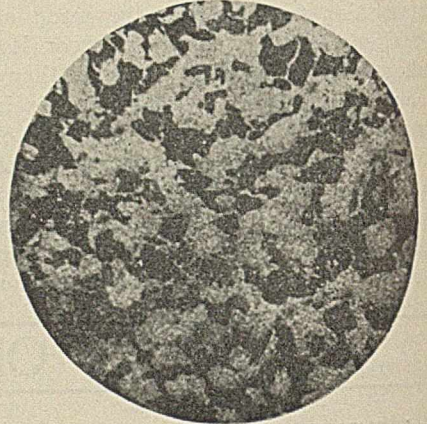


FIG. 6. $\times 65$

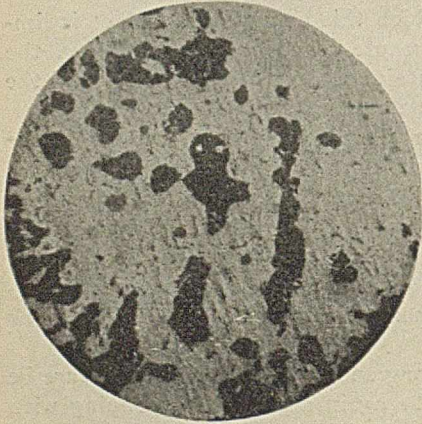


FIG. 7. $\times 65$

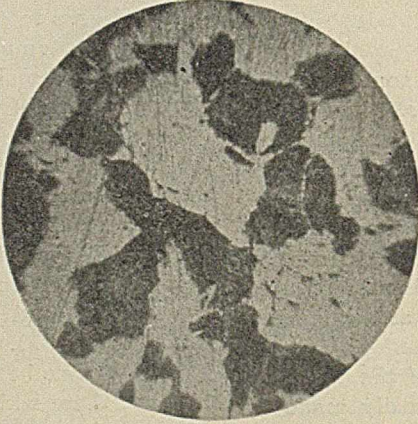


FIG. 9. $\times 150$

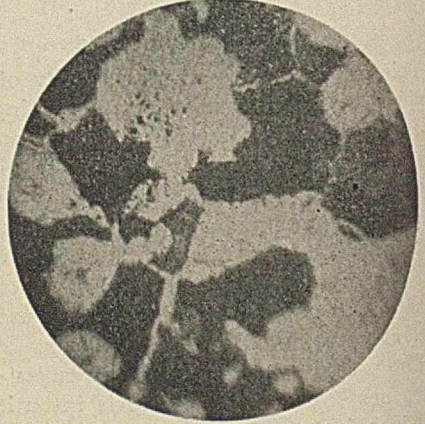


FIG. 10. $\times 150$

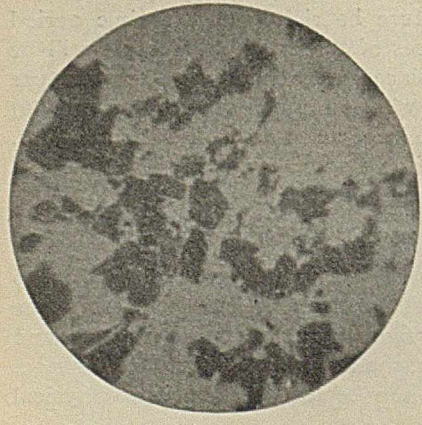


FIG. 11. $\times 150$

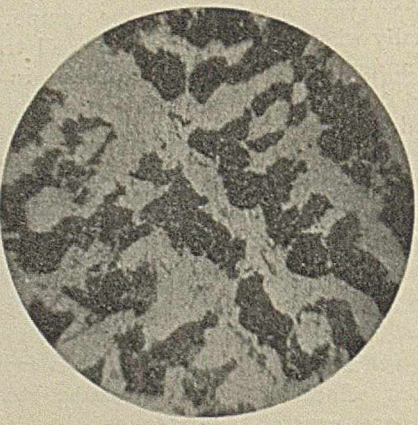


FIG. 12. $\times 150$

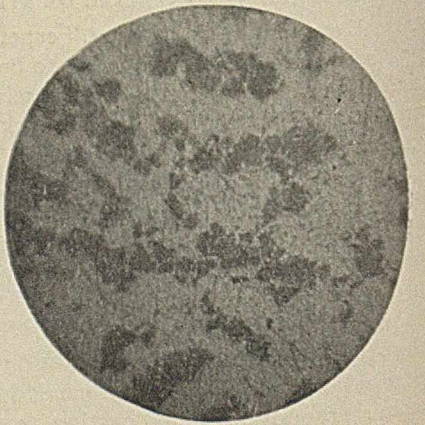


FIG. 13. $\times 150$

The following examples (Figs. 9-15) give the type of the structures as obtained from four or more different places on each casting. The physical values given were obtained from a coupon 5.5" X 3.5" X 1.5" cast on and part of the casting and placed, it was believed, so as to represent neither the best nor the worst of the casting but to give the average of the piece.

The castings were spot polished, etched and photographed in the shop with the portable metallographic outfit (Fig. 8) described by the writer in *Metallurgical and Chemical Engineering*, II, 56-8.

All the photographs are of the same amplification

with a low resistance to shock and to suddenly applied loads. The microstructure of such castings commonly shows the presence of slag, sulfides, oxide and other impurities. Typical illustrations of this are to be seen in Figs. 16 and 17.

The following examples (Figs. 18-20) give the type of the structures as obtained from four or more places on the casting. The physical values were obtained from coupons cast on and part of the casting.

The photographs are of the same amplification with a magnification of 150.

These impurities play an important part as a cause

TABLE I

Mark	Tensile	Yield point	Elong.	Red	Analysis				
					C	Mn	Si	S	P
Fig. 9.....	71,356	38,832	23.75	34.72	0.31	0.63	0.27	0.071	0.044
Fig. 10.....	70,991	38,489	19.25	23.26	0.31	0.61	0.29	0.073	0.043
Fig. 11.....	70,791	39,502	16.75	18.00	0.31	0.59	0.29	0.072	0.040
Fig. 12.....	70,620	39,216	14.00	15.75	0.37	0.60	0.29	0.069	0.040
Fig. 13.....	69,980	39,205	12.00	14.50	0.31	0.60	0.28	0.069	0.043
Fig. 14.....	72,000	33,000	10.75	11.50	0.31	0.67	0.28	0.065	0.040
Fig. 15.....	73,000	40,000	8.50	10.00	0.31	0.62	0.28	0.054	0.044

with a magnification of 150 and are directly comparable with one another.

The castings were made by the same process but in widely separated heats.

of failure. Thus in Figs. 21 and 22, from castings which have failed in service, and which are areas some distance away from the point of actual rupture, it will be noted that there are minute cracks in the grain

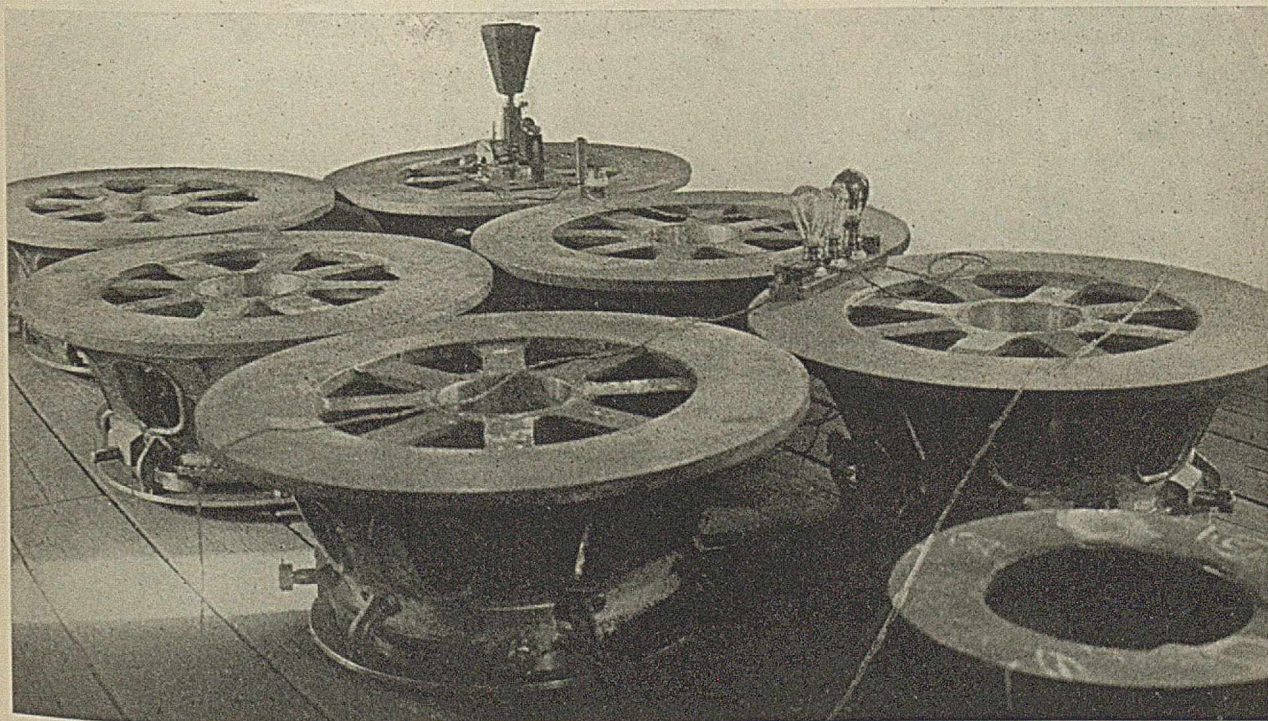


FIG. 8

It will be noted that these structures are all referable to one or the other of the types previously shown as resulting from the conditions of annealing.

Annealing, however, is not the only factor that influences the physical properties, for, given good annealing practice, it is not infrequent to find a casting

and that these cracks start in and follow the line of the impurities. In Fig. 23 the cause of the rupture is clearly indicated.

CONCLUSION

One, more, or all of the several structures here illustrated may be found in any one steel casting.

TABLE II

Mark	Tensile	Yield point	Elong.	Red	Analysis				
					C	Mn	Si	S	P
Fig. 18.....	70,000	36,500	12.50	16.25	0.35	0.65	0.29	0.070	0.040
Fig. 19.....	71,400	37,480	10.50	13.25	0.38	0.68	0.29	0.065	0.042
Fig. 20.....	70,900	35,150	13.75	17.40	0.32	0.64	0.28	0.071	0.045

If, as is generally the case, the coupon be gated to the casting, or be cast on the heavy part, or on the drag, it will represent the best values of the metal, when,

the microstructure of the casting and its physical value.

It can be shown that a casting may be spot polished

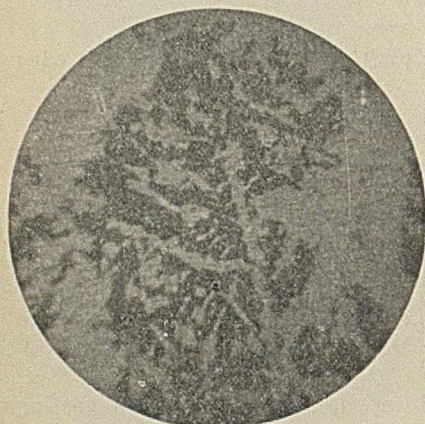


FIG. 14. X 150

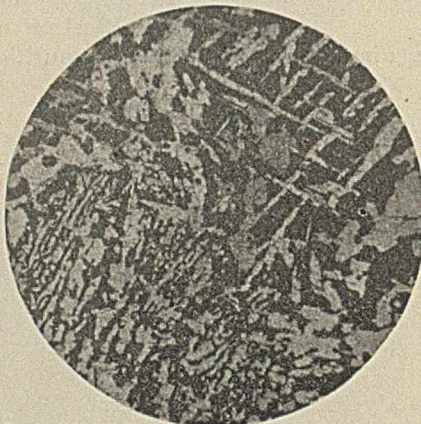


FIG. 15. X 150

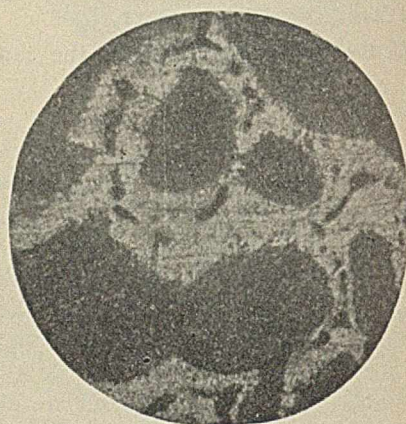


FIG. 16

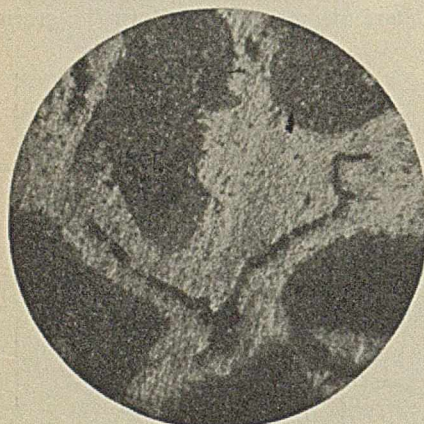


FIG. 17

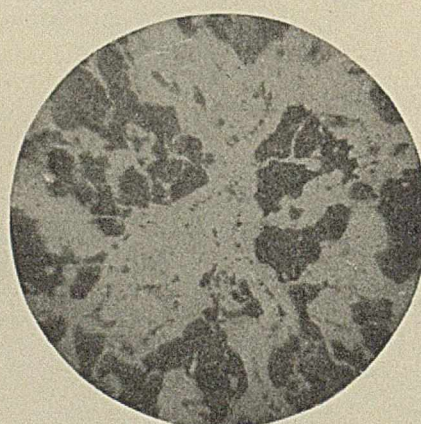


FIG. 18. X 150



FIG. 19. X 150

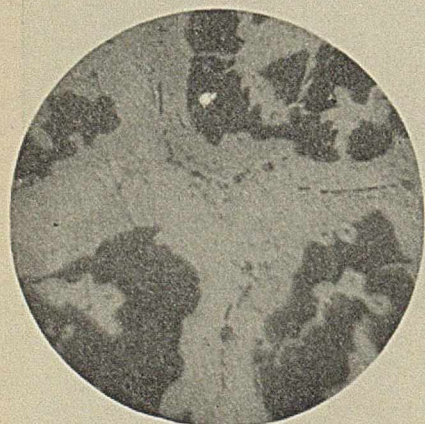


FIG. 20. X 150

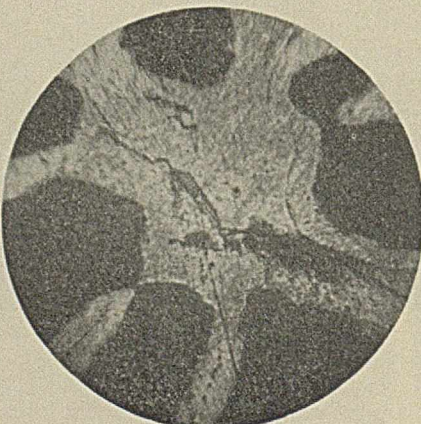


FIG. 21. X 150

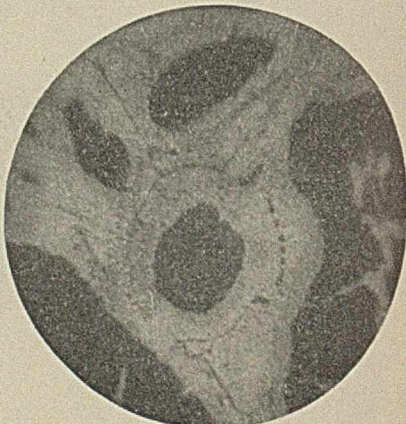


FIG. 22. X 150

in fact, the average value of the casting may be such that it will have but little resistance to sudden strains.

It has been indicated that there is a relation between

in eight or more different places with but little, if any, more expense and time than it takes to prepare a standard test bar.

It follows that, given sufficient experience and a set of standards, the metallographic method will give information that cannot be obtained conveniently by



FIG. 23.

any other manner of test. It becomes, therefore, an additional safeguard in the inspection of important castings.

1423 R St., N. W.
WASHINGTON, D. C.

LUBRICATION WITH OILS, AND WITH COLLOIDAL GRAPHITE

By CHARLES F. MABERY
Received July 14, 1913

In a paper¹ published three years ago, an account was given of some results on the comparative efficiency in lubrication of oil lubricants, and oils carrying colloidal² graphite. It appeared in all the tests therein described that a lower coefficient of friction was given by the use of graphite than by the use of oils alone, that oils supported a much greater pressure with the aid of graphite, and especially that the graphite film was capable of sustaining the friction of a heavy pressure for a long period after the supply of lubricant was shut off. Much attention has since been given to various features of lubrication with colloidal graphite, especially in attempts to ascertain the actual economy of its use in replacing oil lubricants. The observations to be described in this paper present high economic efficiency and a remarkable durability of a graphoid surface. (For a definition of this term see page 722.)

It has long been felt that lubrication with oils under heavy pressures is an artificial system, for the friction is supported by a thin film of oil which must separate completely and continuously the bearing surfaces. If this film be in the least broken, even in minute places, there will be a catch between the metal surfaces with greatly increased friction, as shown by higher temperatures as well as by higher coefficients. This uncertainty in oil lubrication depends on an inherent weakness of the hydrocarbons which constitute the main body of petroleum lubricants, assuming that they have been separated from the crude oil without decomposition in the process of refining; they are few in number, and members of a limited series represented mainly by the general formulas C_nH_{2n} , C_nH_{2n-2} , and C_nH_{2n-4} . The limited number of hydrocarbons in these series is shown by the fact that they may be collected within comparatively narrow limits of temperature during distillation, provided, of course,

¹ THIS JOURNAL, 2, 115; *Journal of the American Society of Mechanical Engineers*, January, 1910.

² At the time of the former publication the term "deflocculated" was used to designate this form of carbon which had been found to be altogether different in its properties from the other well known forms. Further study demonstrated its colloidal quality.

that decomposition is avoided. The stability of these hydrocarbons diminishes in a somewhat regular manner with the increase in complexity of composition, until a point is reached where the oils cannot be distilled without cracking even *in vacuo*. This variation in stability appears in their use as lubricants, especially under irregular conditions of friction and temperature. But so long as the temperature is kept down and the bearings have a properly even surface, the hydrocarbons of suitable viscosity serve as durable lubricants. Under uneven conditions of friction they are liable to immediate decomposition even to carbonization. This complete decomposition is frequently observed especially in the extremely variable conditions of automobile lubrication. In fact the demands of modern locomotion with unprecedented high speeds, such as in automobile racing, uneven loads, and the variable changes of highway traffic, have reached a burden of lubrication that no oils, mineral, vegetable, or animal, are capable of supporting. Carbonization in automobile lubrication is an occurrence of common observation, and oils are rated on the basis of a so-called carbon test, which shows certain differences in stability depending on a difference in the composition of the oils, and also on the method of refining. No oils can withstand the irregular operations of certain automobile practice without carbonizing to a greater or less extent.

Lubrication with oils is based on the quality of oiliness, or greasiness that is inherent in the hydrocarbons poorer in hydrogen mentioned above. It is not strictly defined by viscosity as ordinarily determined. While the molecules have a certain freedom of motion within the body of the oil attended with a consequent inherent friction, they have also an attraction for external surfaces on which they may form an attachment, but preserving their continuity and freedom of motion even under high pressures and high speeds, thus forming, under constant conditions, a continuous and a durable film.

Engler¹ in referring to oil lubricants stated that "Das Schmiermittel par Excellence" is not known, but that for every special use a lubricating oil must be selected on the basis of its viscosity; that since for variable combinations of pressure and speed, there is no definite standard, the viscosity must be determined for any set of conditions in practical operation. It may be said further that viscosity as ordinarily determined is not always reliable for determining the quality of a lubricating oil for any stated condition, for the reason that it is possible to prepare an oil lubricant by compounding a heavy distillate with a lighter one, leaving out the middle fraction, in such a manner as to give any viscosity desired as determined by the viscosimeter. But in the use of such a lubricant there is a tendency of the lighter constituents to creep and evaporate, leaving the heavy constituents between the bearing surfaces. It is evidently possible to determine the presence of any considerable amount of the lighter constituents by determining the flashing point of the oil, but this test is scarcely sufficient to

¹ *Das Erdoel*, Leipzig, 1912, p. 83.

indicate with precision the presence of such proportions of the lighter constituents as may be used to give the desired viscosity. A more accurate means of control is distillation which, if conducted in *vacuo*, should show the smallest proportion of lighter oils. A further aid from a practical point of view, and one that is highly desirable if properly conducted with reference to factory use and conditions, is the trial of an oil on a suitable bearing provided with the means for ascertaining the coefficient of viscosity and for reading temperatures.

On account of the inherent weakness in oil lubricants referred to above, the need is evident of a solid lubricant capable of equalizing the inequalities of the metal surfaces and of such adequate lubricating quality as to avoid intermolecular friction. Of all known materials the substance graphite alone possesses the qualities of a normal lubricant. In its ordinary natural condition it is not possible to mechanically subdivide it so completely that it can penetrate the fine interstices of metallic surfaces and at the same time form a persistent coherent lubricated surface; but in a form of complete purity, free from the mineral constituents of natural graphite and in a condition of minute subdivision, such as is formed by the conversion of Acheson's electric furnace graphite into its colloidal condition, there is available a solid lubricant that fulfills the requirements of economic lubrication. It is so finely divided that it readily permeates metals and by reason of its unctuous quality its own friction is reduced to a practically negligible quantity, thus escaping the internal friction of oil lubricants that is an important factor in the losses of power in factory operation.

The action of colloidal graphite is two-fold: its permanent suspension in oil as oildag or in water as aquadag renders it capable of convenient application and it invariably reduces the viscosity of the oil as a medium of application as shown by many tests with a great variety of oils; its greatest value, however, depends on its readiness to form a graphoid condition on bearing surfaces. It is only necessary that it be suspended in a suitable medium free from any kind of electrolyte to give the finely divided particles freedom of motion. When evenly spread in an oil medium over a bearing surface such, for example, as a Babbitt bearing of the proper quality, the colloidal particles immediately enter the fine metallic interstices and accumulating form a combination somewhat analogous to an amalgamated surface, which needs only to be properly renewed by regular additions of the lubricant to present a bearing surface capable of supporting any reasonable pressure and with the lowest friction that it is possible to obtain.

In the description of the Carpenter machine on which the tests presented in this paper were made, in the former paper referred to above, the necessity of using a hard Babbitt bearing was mentioned. The condition of the journal and of the bearing have been more carefully considered in the recent work, especially with reference to the hardness of the Babbitt, the smoothness of surface, and the even distribution of the

lubricant by the grooved bearing. It is evident that observations of this nature are altogether relative, especially as it is practically impossible that independent bearing surfaces are precisely in the same condition. By the use of a standard lubricant it is of course possible to compare tests made under different conditions.

In the work herein described an especially hard Babbitt was selected, and the bearing surfaces were milled down to true contact in the beginning, and by long-continued use were worn to as extreme a condition of fine smoothness as it is possible to obtain by ordinary mechanical operation. Comparing the extremely low coefficients and temperatures recently obtained with those formerly presented, the difference in the condition of the bearings is evident. While these conditions are necessary in showing small differences of friction, evidently no such strict adherence is necessary in factory operation, although the more closely they are applied in practice the less will be the loss in power. Under any reasonable conditions of operation the use of colloidal graphite as lubricant is certain to reduce the friction very materially and to serve as an important economic element in factory maintenance.

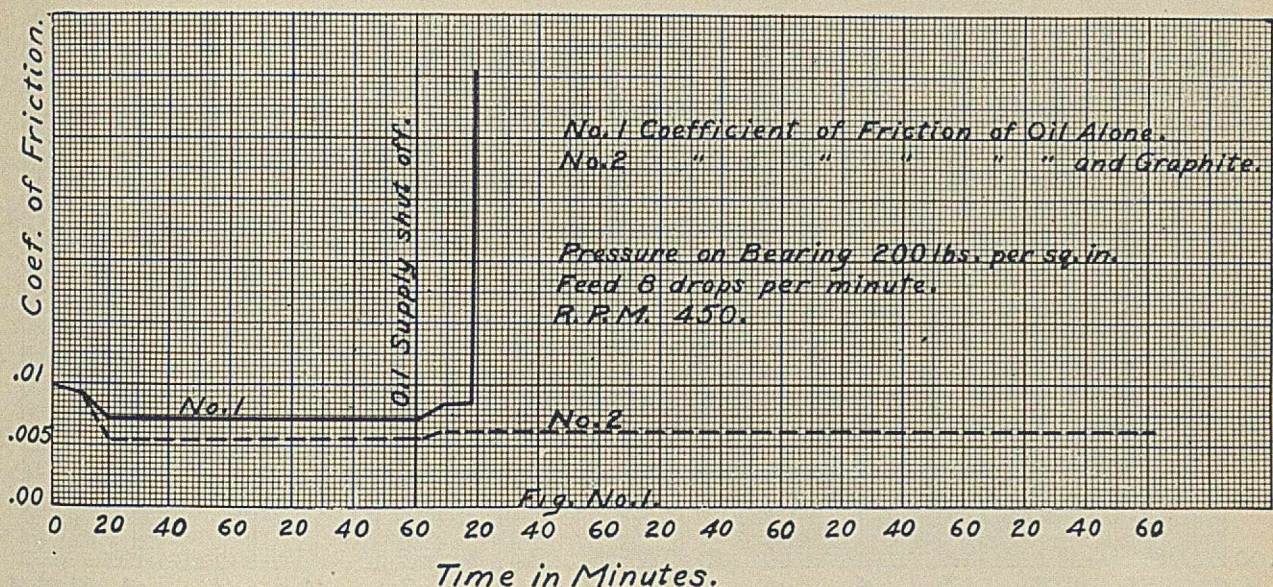
For the purpose of ascertaining with greater precision than formerly the influence of colloidal graphite in reducing friction, observations were made under a variety of conditions, more especially for the purpose of comparing its superior economy over that of oil lubricant. First in the series of tests one of the best automobile lubricants was selected for a test of its frictional capacity alone, and then with different percentages of graphite. The oil was allowed to run from the cup at the rate of eight drops per minute for two hours, with a thermometer inserted in a hole in the bearing for the purpose of reading the temperatures. The pressure selected was 200 lbs. per sq. in. or a total of 1500 lbs. The speed was 450 revolutions per minute (r. p. m.).

Fig. 1 shows the coefficients of friction extending through the period of the test, two hours, and also that the oil film broke seventeen minutes after the supply was shut off.

It should also be mentioned in connection with this observation that a supply of eight drops per minute of the lubricant is the minimum amount of this oil that will support the friction of this pressure under these conditions. This was determined in another experiment, wherein the flow of oil was reduced to six drops per minute; the oil film broke soon after the test was started showing that this quantity of oil was insufficient, a result precisely similar to what was observed in the work three years ago with the same oil and with other oils. The low coefficient of friction in this test is worthy of note, and also its evenness after normal conditions were established, and until the oil film broke.

Fig. 1 also presents the curve for the same oil carrying 0.35 per cent graphite under the same elements of pressure, speed, and supply of oil to the bearing. The low coefficient of friction is apparent which, with

Chart for Motor Oil with and without 0.35% Colloidal Graphite

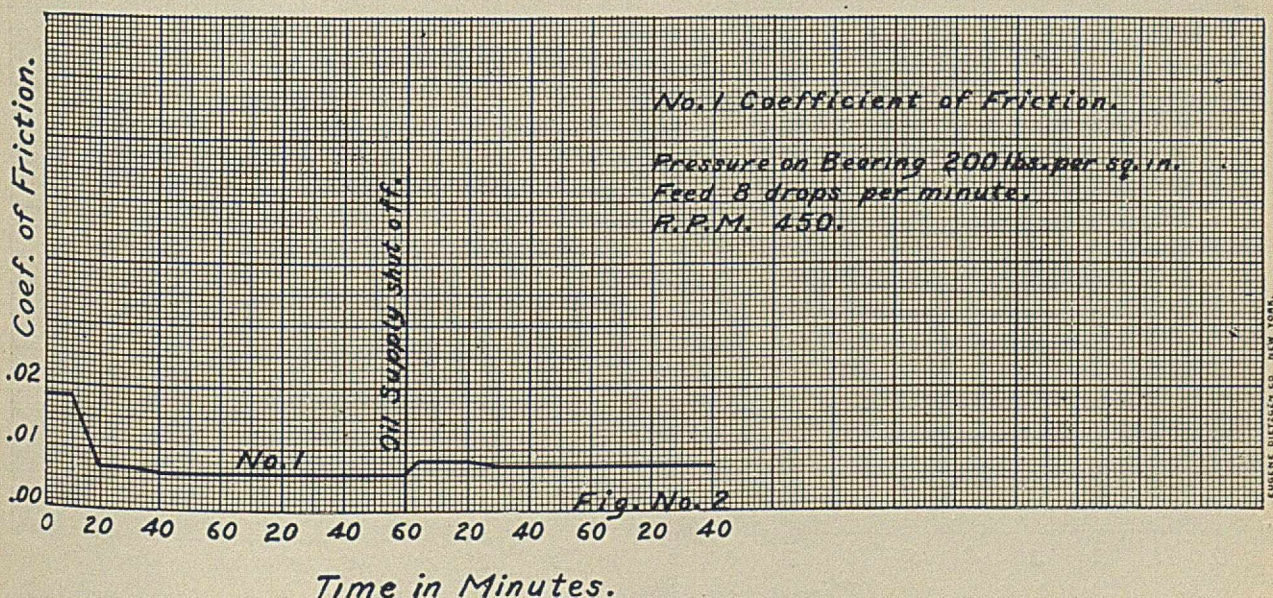


a corresponding lower temperature, indicates clearly the influence of the graphite in reducing the viscosity of the oil, but the observation of especial interest in this test is the permanence of the graphoid surface after the supply of lubricant was discontinued. It appears that this surface supported a pressure of 200 lbs. per sq. in. with an extremely low and even coefficient of friction that continued unchanged during five hours and would probably have continued much longer.

For the purpose of ascertaining whether larger or

pressure of 150 lbs. per sq. in. The results of this test are not essentially different from those of Fig. 2 with 0.35 per cent graphite, although the coefficients are somewhat lower with the larger percentage. There is therefore little to choose between these percentages in establishing the initial graphoid surface; but as will be shown later a suitable surface can be permanently maintained when it is once established by a much smaller addition of lubricant, whether it be used as a smaller percentage of graphite, or by a diminished supply of oil carrying the normal proportion.

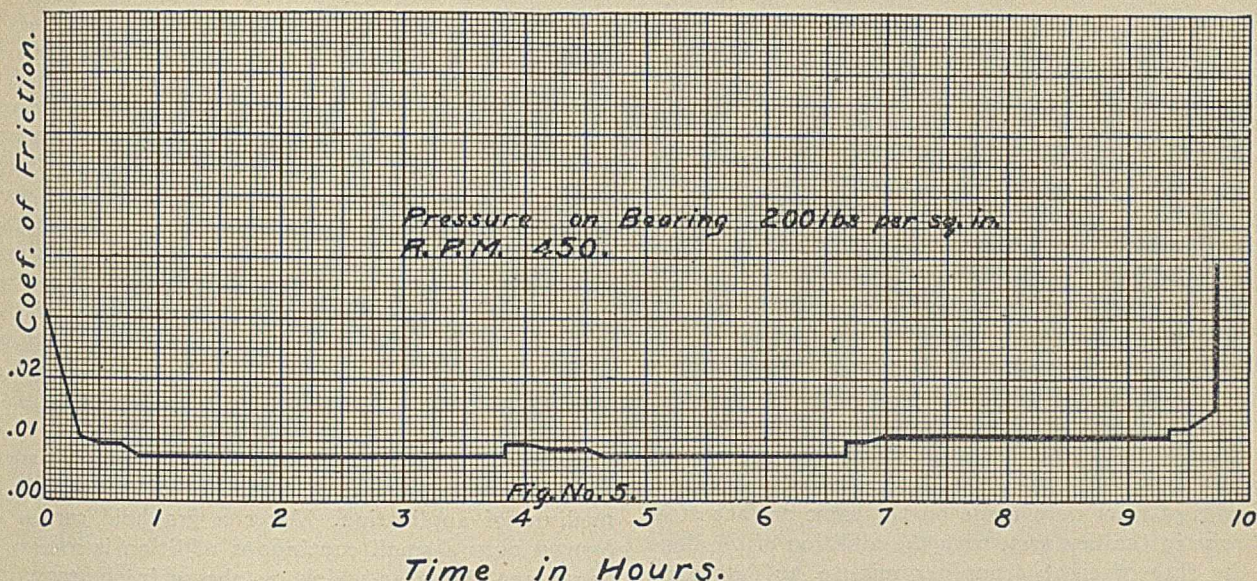
Chart for Motor Oil with 0.25% Colloidal Graphite



smaller percentages of graphite are advantageous, several runs were made with lubricants carrying 0.5 per cent and smaller percentages to 0.1 per cent, but neither of these extremes were satisfactory. Fig. 2 presents the results as to coefficient with the lubricant carrying 0.25 per cent of graphite under a

For the purpose of ascertaining the minimum amount of graphite that will maintain a graphoid condition when once formed on the bearings, a series of tests were made gradually reducing the supply of lubricant all under the same conditions of pressure and speed. Fig. 3 gives the curves after the flow of

Chart for Endurance Test on Colloidal Graphite Alone

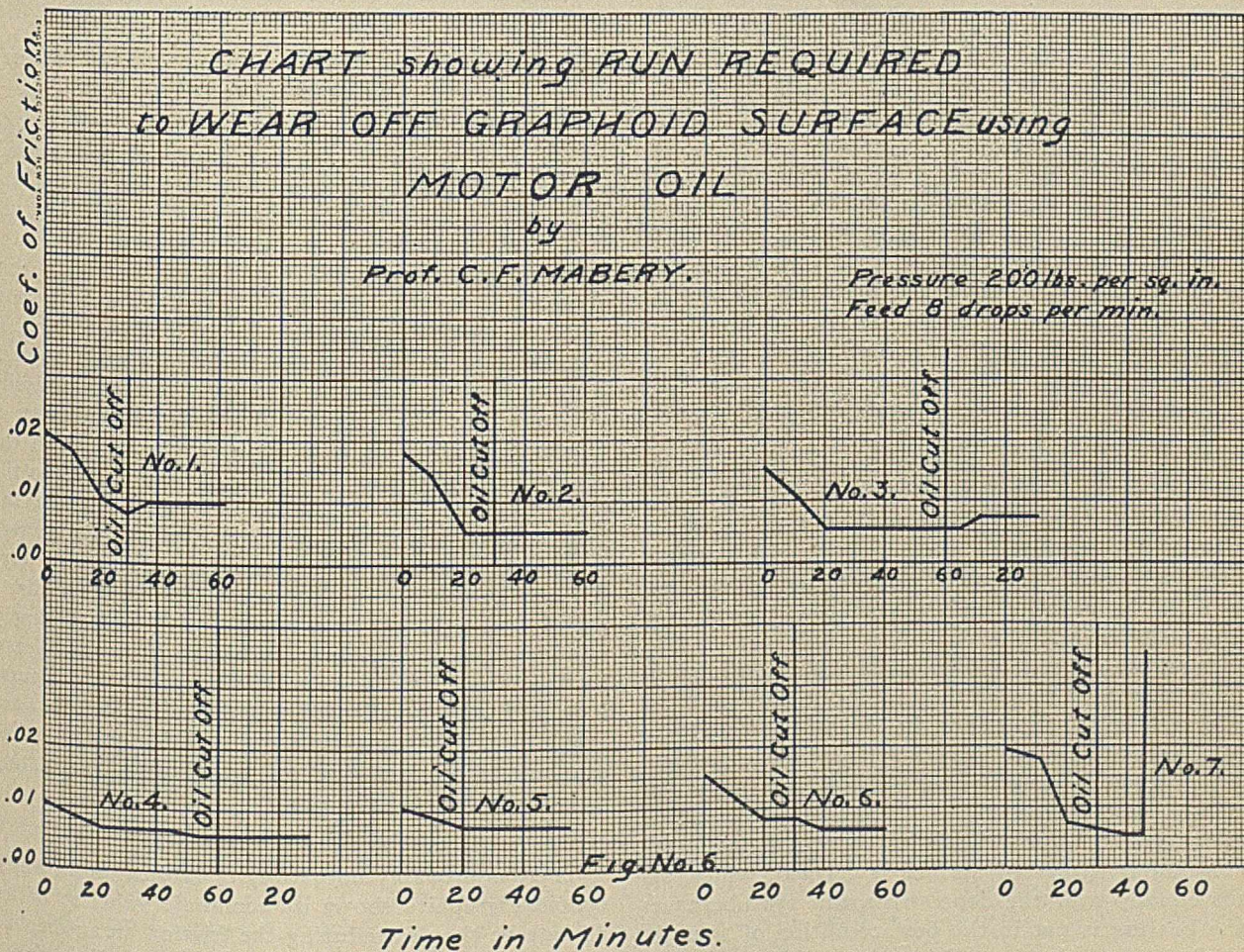


have maintained the graphoid surface, but this flow was practically at the lowest point where it could be accurately measured from the oil cup.

it was shown that a flow of eight drops per minute is the minimum supply of oil alone that will support the friction under these conditions.

It appears, therefore, that the same result in lubrica-

For the purpose of testing still further the quality of



tion is obtained by the use of one-sixteenth of the quantity of oil that is necessary to maintain the same lubrication without the use of colloidal graphite; for

the graphoid surface, at the end of the last test the flow of lubricant was suspended and the machine allowed to run until the bearing caught. The

results of this test are shown in Fig. 5 where it appears that the pressure was supported for nearly ten hours and with a coefficient only slightly higher than in the preceding tests. It should be borne in mind that a break in the continuity of the lubricated surface is indicated suddenly by a great rise on the friction arm and it is caused by the first point or section however minute wherever the graphite becomes worn through, yet there may still be a large section of lubricated surface. This appeared in the next experiment.

The manner in which colloidal graphite is able to support such heavy pressures with low friction has already been explained. After the metallic surface becomes completely saturated with graphite evidently without renewal continued friction would be necessary to remove it completely. It therefore seemed of interest to ascertain how readily it could be removed. A series of runs were made on the same surface after it broke in the last test, with the addition of oil alone at the rate of eight drops per minute, to determine just the point where the graphoid surface could no longer assist in lubrication. In each of the runs the oil was allowed to flow for thirty minutes, and the bearings were then carefully wiped. Fig. 6 shows the effect of the graphite in assisting the oil lubrication without change during six runs, and also that it became exhausted and broke in the seventh run showing that altogether approximately three and one-half hours were required to wear off the graphite until it was no longer an aid in lubrication.

A GRAPHOID SURFACE AND THE CONDITIONS OF ITS FORMATION

In the former paper on this subject¹ the effect of colloidal graphite (then referred to as a "deflocculated form") appears the following statement: "One of the most characteristic effects is that of a surface-veneer, by forming a veneer, equalizing the metallic depressions and projections on the surfaces of journal and bearing." After four years experience with graphite lubrication it appears that the former explanation falls short of defining the intimate relation of colloidal graphite to metallic bearings. No doubt in building up the graphoid surface the entire depressions and projections are saturated with graphite which doubtless enters into a closer state of combination with the metallic surface than that merely of a mechanical veneer.

In the use of colloidal graphite as a lubricant it appears that by reason of the tenuity or fineness of its particles, it is capable under the conditions of lubrication of penetrating the porous surface of metals, and coming into such close contact in their intermolecular structure, of approaching a condition of graphitic combination. It then continues to accumulate until a continuous saturated surface is formed, which extends equally over the depressions and protuberances if the bearing is not in the best condition of smoothness; but in its best condition the graphoid surface formed seems to be nearly frictionless.

Since this relation of metal and carbon is not de-

¹ *Loc. cit.*

finer by any term now in use, the word *graphoid* used above may serve to distinguish it from the term *film* that expresses the state of an oil lubricant on a bearing surface.

As to the greater efficiency of the graphoid surface over an oil film under any conditions of lubrication there seems to be no question. It all depends on establishing the conditions whereby this surface may be readily formed. An oil medium in which the colloidal graphite is permanently suspended can evidently carry it to any form of bearing surface that needs lubrication. Whatever the condition of the metallic bearing the graphite soon combines with it, and the smoother the surface the more readily will a continuous graphoid surface be formed. In all tests it has appeared that the internal friction of an oil is diminished by colloidal graphite, although its larger effect is altogether independent of oils except as a medium of application. After a graphoid surface is formed only a small continuous addition is necessary to replace the wear which, as shown in the tests, reduces the consumption of oil to a small fraction of what is necessary in the use of oil alone. While eliminating the internal friction of oils and rendering viscosity of secondary importance, the graphoid surface is capable of taking care of light and heavy pressures equally well and with a minimum loss of power.

RELATION OF COEFFICIENT OF FRICTION AND VISCOSITY TO TEMPERATURE

Since the curves for temperatures were found to follow closely those of friction practically unchanged, it was not thought necessary to plot them. In all the observations described in this paper, it was observed that the temperature gradually increases with the duration of the test until it reaches a practically constant value not exceeding 65° F., and that for the most part the temperatures were considerably lower. It appears that the friction generates a certain amount of heat until it reaches a normal which is practically constant, and that beyond this point the increase if any is so slight that it is dissipated. In general the lower the coefficient of friction, the lower will be the temperature. This is shown in Fig. 1, where the temperature and the coefficient for oil alone were considerably higher than those for oil carrying 0.35 per cent graphite. It also appears in the other charts especially in the endurance tests, with a small supply of oil. This is doubtless explained, in part at least, by the internal viscosity of the oil which is of course less with the smaller supply of oil aided by the graphite, and it demonstrates a superior quality of the graphoid condition over an oil film, in eliminating practically internal viscosity.

AUTOMOBILE LUBRICATION

There is probably no variety of lubrication in which colloidal graphite shows its economic value to better advantage than in reducing the friction on automobile bearings. On the Babbitt bearings of the cylinder shaft it readily forms a graphoid surface that wears indefinitely, and the self-lubricating quality of this surface reduces friction to the smallest possible value.

It eliminates also any possibility of heating due to an irregular flow of oil. In the wide and sudden variations of highway automobile traffic the bearings are often subjected to greater strain than an oil film can stand, but not a graphitized surface when once well formed. Such protection from undue wear and sudden strains that cannot be avoided in highway locomotion add greatly to the safety and length of service of the finely adjusted mechanism. Assuming properly selected materials in construction, no doubt the most uncertain element in the proper operation and in the economic durability of an automobile is the friction of its moving parts. Its sure control protects the mechanism of the moving parts and materially reduces the expense of operation.

CASE SCHOOL OF APPLIED SCIENCE
CLEVELAND, OHIO

THE ACTION OF VARIOUS SUBSTANCES ON CONCRETE¹

By RICHARD K. MEADE

The following experiments on the action of various substances on concrete were begun some five or six years ago, about the time that the agitation over the destruction of concrete by the alkaline waters of the West was first started and was undertaken not only to see if such action was really likely to take place but also to determine which of the salts ordinarily found in ground waters were the cause of such destruction.

ANALYSIS AND TESTS OF THE CEMENT EMPLOYED

Silica.....	20.20
Oxide of iron.....	2.50
Alumina.....	6.96
Lime.....	62.40
Magnesia.....	3.01
Sulfur dioxide.....	1.60
Loss on ignition.....	2.38

PHYSICAL TESTS

Soundness—Steam O.K.	Cold water O.K.
Boiling O.K.	Air O.K.
Fineness—Passing No. 100.....	94.3%
Passing No. 200.....	97.8%
Setting Time—Initial set—2 hrs. and 15 min.	
Final set—6 hrs. and 30 min.	
Tensile Strength—1 day neat.....	315 lbs.
7 days neat.....	765 "
7 days sand.....	245 "
28 days neat.....	876 "
28 days sand.....	340 "
3 mos. neat.....	885 "
3 mos. sand.....	415 "
6 days neat.....	855 "
6 days sand.....	435 "
1 yr. neat.....	890 "
1 yr. sand.....	510 "

The salts usually found in the so-called "alkali waters" of the West are also those which occur in sea water and are those present in largest amounts in many spring and river waters. They are sodium chloride, magnesium sulfate, calcium sulfate, sodium sulfate and sodium carbonate. In order to test the effect of solutions of these substances on cement mortars, a sample of normal Lehigh Valley cement was selected and from it a large number of sand briquettes were made.

¹Read at the Sixteenth Annual Meeting of the American Society for Testing Materials, Atlantic City, June 26, 1913.

All briquettes were made from a mixture of one part cement and three parts standard Ottawa sand. They were allowed to harden 28 days in air and then immersed in a solution of the salt. The briquettes were piled in such manner that the solution had access to almost their entire surface.

The solutions in all cases except that of the calcium sulfate, which was a saturated solution, were made up of one part of the salt to 100 parts of water, to form practically a one per cent solution. At first the solutions were changed every few days, but after the first month the solutions were changed weekly and after the first year less often. The results obtained are given in the table below:

ACTION OF VARIOUS SALTS ON CEMENT MORTARS

Age in air.....	28 days						
Age in the solution....	0 days	7 days	28 days	3 mos.	6 mos.	1 yr.	2 yr.
TENSILE STRENGTH							
In MgSO ₄	219	268	272	287	196	Disinte- grated	...
In MgCl.....	219	245	300	315	202	115	Disinte- grated
In CaSO ₄	219	227	300	334	314	209	"
In Na ₂ SO ₄	219	257	334	354	378	271	141
In NaCl.....	219	236	268	299	287	310	325
In Na ₂ CO ₃	219	225	277	324	320	337	360

First, it should be remembered that the 28-day strength of briquettes kept in air is much less than that of those kept in water. As will be seen from the results given in the table, the sulfates have a marked action on concrete which seems to be most apparent in the case of the magnesium salt. The action of magnesium sulfate on cement mortars has been discussed quite voluminously of late, and I will not go into it to any length in this paper beyond the fact that we carefully analyzed the affected portion and the unaffected portion of a sand briquette which has been stored in a solution of magnesium sulfate. These analyses follow:

Percentages	After immersion		
	Before immersion	Unaffected portion	Affected portion
Silica.....	75.12	73.96	60.40
Oxide of iron.....	0.52	0.60	0.30
Alumina.....	1.15	1.30	0.64
Lime.....	14.80	14.50	14.21
Magnesia.....	0.70	1.66	3.64
Sulfur trioxide.....	0.33	0.83	5.78
Loss on ignition.....	7.02	7.14	14.97

The large increase in the magnesia and sulfur trioxide and the decrease on the oxides of iron and alumina indicate the elements which react with each other. The loss in silica may be due to chemical action also, but as the surface of the briquettes was very much attacked and the sand grains could be scraped away with the finger, I am inclined to think that the lower silica in the disintegrated portion is probably due to mechanical causes rather than chemical action.

It will be noted that in almost all cases the first effect of the solution was to increase the strength of the briquettes and that signs of disintegration in no cases became evident until after a period of three months in the solution.

Some of the briquettes were even boiled in a 5 per cent solution of magnesium sulfate for several days and in all cases the briquettes were much stronger

after boiling than they were before and fully as strong as briquettes boiled in pure water, showing how slow the action of the sulfates is.

The briquettes which failed were much swollen and presented much the appearance of a baked potato which had burst its jacket.

Various authorities have proposed at different times the use of divers ingredients in concrete exposed to sea water with a view to reacting with the salts of the latter to form insoluble compounds which would protect the concrete. Most persistently suggested of these are the salts of barium which form with sulfates insoluble barium sulfate. I tried both barium chloride and barium carbonate. These were ground very finely and mixed with the cement. I employed 2 per cent of barium chloride with the cement and also 2 per cent and 5 per cent of barium carbonate, respectively. Sand briquettes were made from these mixtures and the test pieces stored in a magnesium sulfate solution containing 10 grams of the salt to the liter. My results follow and, as will be seen, none of these compounds arrest in any way the destruction:

ACTION OF MAGNESIUM SULFATE ON CEMENT MORTARS CONTAINING BARIUM COMPOUNDS

Age in air.....	14 days					
Age in 1 per cent MgSO ₄ solution.....	0 days	7 days	28 days	3 mos.	6 mos.	1 yr.
TENSILE STRENGTH OF BRIQUETTES CONTAINING						
2 per cent BaCl ₂	181	...	213	306	265	146
2 per cent BaCO ₃	115	221	246	311	204	Disintegrated
5 per cent BaCO ₃	166	257	346	346	274	"

Some years ago an English chemist suggested the use of finely ground burnt red brick as an admixture for concrete which was to be used in sea water. After reading this paper it occurred to me that the resistance to sea water claimed for high iron cements might be due to the presence of oxide of iron in the cement. I, therefore, had sand briquettes made up containing oxide of iron in various forms and conditions, *viz.*, red or ferric oxide, magnetic oxide of iron, venetian red (an impure oxide of iron made from low-grade iron ores, so-called "paint ores" of the Lehigh District) and finely ground red brick, using 5 per cent of the weight of the cement of these in each case and placing the briquettes in a 1 per cent solution of magnesium sulfate. The results follow:

ACTION OF MAGNESIUM SULFATE ON CEMENT MORTARS CONTAINING IRON OXIDES, ETC.

Age in air.....	14 days					
Age in solution.....	0 days	7 days	28 days	3 mos.	6 mos.	1 yr.
TENSILE STRENGTH OF BRIQUETTES CONTAINING						
5 per cent ferric oxide...	218	275	310	355	310	125
5 per cent magnetic oxide.	225	280	340	340	280	105
5 per cent venetian red...	165	225	300	345	215	Disintegrated
5 per cent brick dust.....	970	220	275	310	205	"

As will be seen, the additions of iron compounds are in no way beneficial to cements to be employed in sea water.

I next tried waterproofing the mortar on the theory that if the circulation of water through the pores of the mortar could be stopped no chemical action could take place. I employed for this purpose both a high

calcium and a magnesium hydrated lime, road oil as recommended by Page, a mixture of solution of silicate of soda and fish oil (a well-known waterproof compound) and lime soap (the basis of many waterproofing compounds). I also tried dipping the briquettes first in a hot solution of soap and then in one of alum (Sylvester's Process). The result of sand briquettes made from these mixtures and stored in magnesium sulfate solution (10 grams to the liter) will be found below:

ACTION OF MAGNESIUM SULFATE SOLUTION ON WATERPROOFED(?) MORTARS

Age in air.....	14 days					
Age in 1 per cent magnesium sulfate solution.....	0 days	7 days	28 days	3 mos.	6 mos.	1 yr.
TENSILE STRENGTH OF BRIQUETTES CONTAINING						
15 per cent hydrated lime (Ca).....	215	215	315	245	200	120
15 per cent hydrated lime (Mg).....	215	225	320	260	245	105
10 per cent road oil.....	165	200	210	260	210	140
2 per cent lime soap.....	185	210	250	275	230	180
2 per cent oil-silicate of soda	160	200	245	260	225	165
Treated with alum and soap.	220	235	275	265	215	185

It will be noted that while the disintegration is evidently taking place in these test-pieces, all of these compounds seem to arrest it to some extent at any rate and in the case of the lime soap this is quite marked.

I also investigated the action of magnesium sulfate solution on cements high in silica. For this purpose a sample of commercial cement high in silica and low in alumina and one low in silica and high in alumina were selected and sand briquettes were made of this and immersed in a solution of magnesium sulfate containing 20 grams to the liter, or practically a 2 per cent solution. The cements selected had the following analyses:

Percentages	Low-alumina cement	High-alumina cement
Silica.....	23.24	19.86
Iron oxide.....	2.25	2.56
Alumina.....	5.03	7.60
Lime.....	63.55	63.12
Magnesia.....	3.05	3.10
Sulfur trioxide.....	1.51	1.66

As will be seen by the following results, the low-alumina cement resists the action of magnesium sulfate much better than the high-alumina one:

ACTION OF MAGNESIUM SULFATE SOLUTION ON HIGH- AND LOW-ALUMINA CEMENTS

Age in air	14 days					
Age in solution.....	0 days	7 days	28 days	3 mos.	6 mos.	1 yr.
High-alumina cement.	242	318	404	402	230	Disintegrated
Low-alumina cement..	225	307	430	476	472	500

In the above experiments both cements were commercial cements, but the high-alumina cement when received was not quite so finely ground as the other one, so it was ground to practically the same degree of fineness in a small jaw mill (or to 86.2 per cent passing the No. 200 sieve) so that the fineness of the two samples might, in no way, influence the results. Both these cements were made from cement rock and limestone.

In connection with the use of concrete for mine props, where it is often exposed to the action of dilute solutions of sulfuric acid, the following experiment was tried: Sand briquettes were allowed to harden 28

days and then were placed in a solution containing 250 grams of sulfuric acid (H₂SO₄) to the gallon. The solution was changed frequently and the briquettes broken at regular intervals. The disintegration of concrete by such acid water is shown by the following:

Age in the solution..	0 days	7 days	28 days	3 mos.	6 mos.	1 year
Tensile strength.....	226	299	300	280	176	Disintegrated

Several years ago the question of the action of oil on concrete was brought up at one of the meetings of this society in connection with a paper by Prof. Carpenter. In his experiments oil was mixed in with the concrete. In the discussion which followed the reading of the paper, a number of gentlemen suggested that what was needed most was information relative to the action of oil on concrete which had already hardened, in view of the employment of concrete for machinery foundations, engine room and factory floors, etc., where it is subjected to the oil which leaks from the bearings of the machinery. I went home from this meeting and had a lot of sand briquettes made and allowed them to harden 2 weeks in air. These were stored in air, in engine oil, in cylinder oil, and in black oil. They were then broken at stated periods with the following results:

ACTION OF OIL ON CONCRETE

Age in air.....	14 days					1 year
Age in oil.....	7 days	28 days	3 mos.	6 mos.		

TENSILE STRENGTH OF BRIQUETTES KEPT IN

Engine oil.....	253	240	251	232	231
Cylinder oil.....	235	273	221	209	203
Black oil.....	234	222	181	131	Broke in clips
Air.....	248	233	287	303	293

It will be noted that the engine oil and the cylinder oil have practically no effect upon concrete. One would think that as the latter has a considerable proportion of animal oil in its composition it would be apt to effect appreciably concrete exposed to it. On the other hand, the action of the black oil seems strange in view of the fact that it is a straight mineral product. All of these briquettes had absorbed considerable oil, the actual gain in weight of each set at the end of the year being as follows:

	Per cent
Set in engine oil.....	10.6
Set in cylinder oil.....	10.0
Set in black oil.....	12.0

The briquettes in the black oil had not swollen perceptibly and seemed merely to be weak.

The experiments given above were all made upon very small test-pieces and hence the action of the solutions upon them were much more rapid than they would be upon a large mass of concrete, and while in most of the above cases a year was sufficient to completely disintegrate the test-pieces, in a large body of concrete, such as a pier or wall, many years at least would be required to bring about this result. The experiments merely serve to show that even very dilute solutions of the salts of magnesium and the sulfates in general do have a destructive action on concrete and that the generally proposed remedies do not appreciably retard this. They indicate the desirability of

employing low-alumina cements for sea-water construction. The experiments with the oils show that no destructive action is likely to take place where cement is used for floors in machine shops and engine rooms.

202 NORTH CALVERT ST.
BALTIMORE

THE DECOMPOSITION OF FELDSPAR AND ITS USE IN THE FIXATION OF ATMOSPHERIC NITROGEN

By WILLIAM H. ROSS¹

Received July 21, 1913

INTRODUCTION

The extensive search for sources of potash salts which has been undertaken in this country during the past two years has naturally led to renewed efforts in devising methods for its extraction from feldspar and other silicate rocks. The investigations in the use of feldspar as a source of potash have, however, not been confined to the past few years, but date back to the time when the part played by potash as a fertilizer in the production of crops was first recognized. Even as early as 1847 a British Patent, No. 11,555, was issued to Richard Albert Tilghman for a process of obtaining potassium sulfate from feldspar by heating to bright redness for 8 hours, 2 parts of feldspar with 1 part each of limestone and gypsum. Within a few years a number of other patents appeared in England and other countries outlining methods for obtaining potash not only from feldspar, but also from leucite and other eruptive minerals.

The discovery of the importance of potash salts in agriculture took place also simultaneously with the discovery in 1851 of the great salt beds at Stassfurt, Germany, and in 1861 the first factory for refining crude potash minerals was established. The discovery at this time of such an enormous source of soluble potash salts naturally detracted from the interest which was being taken in the extraction of potash from the refractory silicates, and as a result no investigations for obtaining potash from these minerals were patented for 29 years.

The increasing demand in this country for potash fertilizers has again turned attention to the silicate rocks as a possible source. Owing to the commercial application of any successful method which might be devised, most of the investigations have been carried on in secret, and before the publication of any results, patents have been applied for in almost every case covering processes which have been proposed for the extraction of potash from this source. The number of patents of this kind which have been issued during the past few years is surprisingly large. No less than seven have been issued during 1912 alone, and it is known that a number of other patents have been applied for, but have not yet been issued, so that the processes to which they relate are still kept secret.

PROCESSES FOR THE DECOMPOSITION OF SILICATE ROCKS

The total number of patents which are concerned with the extraction of potash from silicate rocks num-

¹ Scientist in Soil Laboratory Investigations, Bureau of Soils, U. S. Dept. Agr.

ber at least 40. That so many patents should relate to processes all distinct from each other is not to be expected, and it is therefore not surprising to find that many have been issued which describe essentially the same process for the extraction of potash from insoluble silicates.

For convenience, these various processes may be considered in 3 groups as follows: (1) Processes which yield potash as the only product of value. (2) Processes which yield potash and some other salable material as a by-product. (3) Processes in which two or more operations are combined in one, yielding a fertilizer containing two or more of the essential plant foods—potash, phosphates and nitrogen in available form.

Of the 40 patents which have been referred to, about 50 per cent make no reference to any products of value which can be recovered other than the potash. From the results obtained in a previous publication¹ it is safe to conclude that processes of this kind cannot prove economical for the reason that the value of the product obtained, if based on the percentage of potash present, as is now universally done in the fertilizer industry, could not cover the cost of the raw materials, and the expense of operation.

The desirability of devising some method of treating feldspar which would produce at the same time some product of value in addition to the potash was early recognized, and about one-half of the patented processes referred to claim to have met this requirement. Among the various by-products which are considered possible of recovery in these processes may be mentioned alumina, silica, pigment, raw materials for the manufacture of glass, and hydraulic cement.

It has been shown² that when 1 part of feldspar and 3 parts of calcium carbonate are ignited to 1300–1400° the alkalis in the feldspar are completely volatilized and the residue has the composition required of Portland cement. Since the shale or clay used in the manufacture of cement contains small quantities of alkalis, it should follow that the percentage of alkalis in the dust which escapes from the kiln during the process of burning should be considerably greater than in the raw materials. This was found to be the case in most plants and in the case of several the concentration of the potash in the flue dust was very marked. In one plant examined the content of potash in the fine flue dust collected by the Cottrell precipitators, recently installed, amounts to as much as 40 per cent, mostly in the form of the oxide. Experiments are now being undertaken with a small cement mill to ascertain the practicability of recovering potash as a by-product in the manufacture of cement by the substitution of feldspar for clay or shale.

It has also been found³ that when 1 part of feldspar and 1.68 parts of lime are digested with water under pressure of about 17 atmospheres, upwards of 90 per cent of the potash in the feldspar passes into solution

¹ Bureau of Soils, U. S. Dept. Agr., *Circ.* 71.

² *Loc. cit.*

³ An article entitled "The Extraction of Potash from Silicate Rocks," giving a more detailed account of the results here referred to, is expected to be published shortly.

in the form of the hydroxide. In this form the potash is worth per unit about 1.8 times the value it commands in the form of the chloride. The residue remaining insoluble has the composition required of Portland cement and would simply require ignition for its manufacture. The cost of the raw materials required for the process is estimated to be about \$6.70 per ton of feldspar, while the products obtained would have a value of about \$13.90, assuming for the feldspar an average potash content of 10 per cent.

The percentage of silica in feldspar is usually about double the combined percentages of potash and alumina present. When consideration is taken of the other reagents added in any treatment of the feldspar, it therefore follows that the insoluble residue remaining at the end of the process will amount to several times the quantity of potash, or potash and alumina present; consequently, in order that the residual material may find a market, when any considerable amount of potash is produced, it must be of such a nature that it can be used on a very large scale. Such may be said to be the case of the raw materials used in the manufacture of glass, of which upwards of one and a half million tons are required annually, but the process which produces cement as a product in the extraction of potash from feldspar has the advantage in that the quantity of cement used in this country is more than 10 times as great. Several of the patented processes of the second group which offer most promise are now being tested out on a comparatively large scale under the direction of the patentees. The results of these trials will determine conclusively the practicability of recovering potash from feldspar by these methods.

Some experiments have also been made in this laboratory on several of the remaining processes of this group, but the preliminary results obtained did not offer sufficient promise to justify continuing the work any further.

The first U. S. Patent (No. 16,111) dealing with the decomposition of feldspar was issued to Charles Bickell for a process which consisted in heating in a reverberatory or other suitable furnace to a light red heat for about 2 hours, 1 part feldspar, 0.5 part phosphate of lime, and 3 or 4 parts of air-slaked lime, all in a finely powdered condition. According to the patent, both the potash and the phosphate become available by this treatment. This process thus belongs to the third group into which processes for decomposing feldspar have been divided, and at first sight seemed to be particularly attractive, for if both the potash and phosphoric acid were rendered soluble as claimed by the patent, the method might be an economical one since, when used as a fertilizer, no separation of the constituents would be necessary and the mixture would be of value for its phosphoric acid as well as for its potash content.

It was found, however, that the calcium phosphate has a passive effect in this treatment of the feldspar and that no greater decomposition was produced in this way than when it was treated with calcium carbonate alone.

The process was further investigated by W. H.

Waggaman of this laboratory who has shown in work soon to be published that when the ignition was made in an open vessel about 56 per cent of the potash was lost by volatilization, and of that which remained very little was water-soluble. None of the phosphoric acid was lost by volatilization under this treatment, but only 40 per cent was rendered citrate-soluble. A better result was obtained when there was added to the mixture about 0.2 part each of hematite and manganese dioxide for each part of feldspar, and the whole ignited to about 1400° for three-quarters of an hour. A limpid fusion resulted which, on cooling, was found to contain all of the potash and phosphoric acid in a citrate-soluble state, but neither was water-soluble.

FIXATION OF NITROGEN

The object of the present investigation was to test the efficiency of ignited feldspar in bringing about the fixation of nitrogen through the agency of the combined alumina or silica present, while there was the possibility that the potash would be set free at the same time. This would thus constitute a process which, like the foregoing, belongs to the third group.

Experiments in the fixation of nitrogen may be said to date back to the year 1785, when Cavendish first noted that an acid was formed when electric sparks were passed through a confined volume of air. The nature of the reaction, however, was at that time unknown. Fifteen years later Sir Humphry Davy observed the formation of nitric oxide when air was passed over platinum wire heated by an electric current. The function of the platinum in this reaction in acting as a catalytic agent was demonstrated by Gay-Lussac in 1864. Since then an immense amount of work has been done along this line. The popular literature on the subject is very extensive and a surprisingly large number of patents have been issued in Germany, Norway, United States, and other countries, on processes and equipment for fixing nitrogen by different methods.

The various processes recommended may be conveniently divided into four groups according as there is produced: (1) nitrates or nitrites; (2) ammonia; (3) nitrids; and (4) cyanids, or related compounds, as cyanamid.

Processes representative of all these groups have been or are being carried out on a large scale. The first and fourth have already developed into large industries. Extensive experiments are now being carried out in this and other countries on processes which relate to the third group, and there is a possibility that the fixation of nitrogen in the form of nitrids may ultimately prove more profitable than in any other way.

That certain metals, when heated, will combine with nitrogen to form nitrids has long been known. Lithium is the most marked in its action in this respect, and takes up nitrogen to form Li_3N even when exposed to moist air. When heat is applied in an atmosphere of nitrogen a violent reaction takes place. Magnesium ranks next to lithium in the tendency to combine with nitrogen. This property of magnesium

is made use of in the well-known method of removing nitrogen in the preparation of argon from the air. Among other metals which possess a marked affinity for nitrogen may be mentioned calcium, titanium, silicon and aluminum.

Instead of using the metal directly in the fixation of nitrogen, there may be used in preference a mixture of the oxide and carbon. In this way reduction of the oxide and fixation of nitrogen may be brought about in the same operation. The most noteworthy experiments in this direction have been made by Ottokar Serpek, who has worked out a technical process for fixing nitrogen on a large scale by igniting bauxite with carbon at $1800-2000^{\circ}$ in an atmosphere of nitrogen.¹ For this purpose a furnace was devised somewhat after the fashion of a rotary kiln having an electric furnace inserted in one section. The rotation of the kiln, while conveying the mixture to the electrical part of the furnace where the reaction takes place, acts also to bring the bauxite and carbon intimately together. The current of nitrogen under slightly increased pressure is passed through the kiln in an opposite direction to the mixed alumina and carbon. As the latter reaches the heated part of the furnace the reaction takes place, whereby there is formed aluminum nitrid (AlN) and carbon monoxide. It is supposed that the aluminum carbide which first has a tendency to form reacts with the alumina to form carbon monoxide and free aluminum which in the nascent state, at the temperature used, readily combines with nitrogen.

The compound thus formed is of a bluish gray color and on issuing from the furnace may contain upwards of 26 per cent of nitrogen. When boiled with alkaline hydroxide or sodium aluminate solution, or with water under pressure, the nitrogen present may be liberated as ammonia, while the aluminum is changed to hydroxide. This may be used over again, but at present the use of fresh bauxite seems to be more economical. In either case the material is rendered anhydrous before passing into the electrically heated kiln by calcining in a separate kiln with the carbon monoxide evolved from the reaction in the furnace kiln and which may be enriched with other combustible gases if necessary.

In some of his patents Serpek suggests that there should be added to the mixture of alumina and carbon about 5 per cent of some metal such as copper or iron which is capable of forming an alloy with aluminum. In other patents an increased yield of nitrid is reported to be brought about by mixing with the nitrogen about 0.1 per cent of some acid gas such as hydrochloric acid or sulfur dioxide. It is further stated that instead of using pure or impure alumina, such as natural bauxite, aluminum borates or silicates may be used for carrying out the process, but no results are given on the use of these minerals as in the case of bauxite.

Experiments along the same line as those outlined in Serpek's patents on the use of alumina for fixing

¹ U. S. Patents 867,615; 884,423; 888,044; 987,408; 996,032; 1,007,495; 1,016,526; 1,030,929; 1,040,439; 1,044,927; 1,044,928; 1,057,286; 1,060,509; 1,060,640.

nitrogen have been made by Tucker and Read.¹ The reactions were carried out in a stationary graphite electric resistance furnace. At temperatures below 1600° the yield of combined nitrogen was small, amounting to less than 3 per cent. The addition of hydrochloric acid gas or of sulfur dioxide to the nitrogen in the furnace gave lower results than when pure nitrogen was used, from which it was concluded that these gases are harmful rather than beneficial. The largest yield of nitrid, amounting to 22.7 per cent calculated as nitrogen, was obtained at a temperature between 1900° and 2000°.

EXPERIMENTAL RESULTS

In undertaking an investigation on the use of feldspar in fixing nitrogen a cylindrical gas furnace was used in the preliminary experiments. This was provided with holes in opposite sides through which was passed horizontally a glazed Royal Berlin porcelain tube 60 cm. long with an internal diameter of 2 cm. With this arrangement the portions of the tube inside the furnace could be repeatedly raised to 1200°, inside temperature, but as the temperature was increased to about 1400°, the tube soon became brittle and cracked. The tube was connected at one end with a hard glass combustion tube containing copper gauze over which, when heated to redness, the nitrogen was led under slightly increased pressure in order to remove the last traces of oxygen. The other end of the tube was closed with a stopper through which passed a glass tube that dipped into a flask containing water. This served as a trap to prevent any back flow of air and also indicated the rate of flow of nitrogen through the apparatus.

The nitrogen used in the experiments was prepared by dropping a strong sodium nitrite solution into a hot saturated ammonium chloride solution. The gas thus formed was stored in a gasometer and before using was dried by calcium chloride and sulfuric acid.

The feldspar used in the experiments was ground to pass a 100 mesh sieve and analyzed as follows:

SiO ₂	64.32%
Al ₂ O ₃	19.64
Fe ₂ O ₃	trace
CaO.....	0.16
MgO.....	0.08
K ₂ O.....	13.72
Na ₂ O.....	2.18
	100.10

This was mixed with sugar cane carbon in different proportions, wrapped in filter paper and inserted in the portion of the tube within the furnace, and then ignited in a current of nitrogen at temperatures up to about 1400°. The fixation obtained in any case was slight, amounting at most to about 1 per cent of the feldspar in the mixture.

Since, as already stated, the potash in feldspar may be set free by igniting with calcium carbonate, a number of experiments were made in which this compound was added to the mixture of feldspar and carbon. When this was done, not only was potash set free by volatilization from the feldspar, but the amount of nitro-

gen fixed at the same time was greatly increased. The porcelain tube in which the ignition was made extended for about 13 cm. outside the furnace, and this portion was therefore comparatively cool. The volatilized potash, however, was found to extend as a deposit not only along this length of the tube, but a portion even passed beyond the tube and was collected in the water trap through which the current was led; as the water contained a little phenolphthalein it acquired a deep pink color as the potash collected.

The percentage of the total potash in feldspar which is volatilized when the latter is ignited in an open vessel for an hour and a half at about 1400° with varying amounts of lime is given in the following table:

Proportion of feldspar to lime	Percentage of potash volatilized
5 to 3.....	6
5 to 4.....	16
5 to 5.....	27
5 to 6.....	61
5 to 7.....	83
5 to 8.....	95

When carbon, however, was added to the mixture of feldspar and calcium carbonate, or its equivalent of lime, and the ignition made in a current of nitrogen, instead of in an open vessel, the proportion of potash volatilized was found to be very greatly increased. Thus, when a mixture of 5 parts of feldspar, 5.4 parts of calcium carbonate, or 3 parts of lime, and 2.5 parts of carbon were ignited in nitrogen for one hour at 1200°, upwards of 50 per cent of the potash in the feldspar was volatilized, while at 1400° the loss of potash by volatilization was found to be complete. The amount of nitrogen fixed at the same time in this and other mixtures was kindly determined by Mr. T. C. Trescott of the Bureau of Chemistry, using the Kjeldahl method. The results given in the following table represent the mean of several determinations:

No. of expt.	Feldspar taken Grams	Carbon taken Grams	Calcium carbonate taken Grams	Temperature of ignition	Potash volatilized in percentage of total present	Nitrogen fixed in percentage of	
						Feldspar taken	Aluminum in feldspar
1.....	4	2	0.0	about 1200°	5.0	0.20	1.9
2.....	6	3	6.4	about 1200	...	0.83	8.0
3.....	4	2	4.3	about 1200	55.7	1.27	12.2
4.....	2	1	2.15	about 1200	...	1.44	13.8
5.....	4	4	4.3	about 1200	...	1.40	13.4
6.....	2	4	2.15	about 1200	38.0	1.45	13.9
7.....	4	2	2.15	about 1200	...	0.50	4.8
8.....	4	2	12.0	about 1200	78.0	0.44	4.2
9.....	4	2	0.0	about 1400	41.3	1.00	9.6
10.....	4	2	2.15	about 1400	98.0	1.68	16.1
11.....	4	2	4.3	about 1400	100.0	6.10	58.5
12.....	2	4	6.0	about 1400	100.0	5.21	50.6

It would seem from these results that varying the proportion of carbon between the limits given does not have much effect, under the conditions of the experiments, on the amount of nitrogen fixed. The minimum quantity of carbon taken was sufficient to combine with all the oxygen in the feldspar. Duplicate results, however, did not agree closely. This was no doubt due to the fact that it was not possible, with the arrangement in hand, to maintain a uniform tempera-

¹ *The American Fertilizer*, 37, No. 9, p. 39.

ture at all times and a comparatively small rise in temperature produced a considerable change in the amount of nitrogen fixed.

The calcium equivalent to the silica, as found by analysis, in 4 parts of the feldspar is equal to 4.3 parts of calcium carbonate. This proportion seems best suited in the fixing of nitrogen since smaller yields were obtained when the proportion of lime to feldspar was increased or decreased beyond this limit. The same results were obtained starting with the oxide of calcium as with the carbonate. Only a very slight amount of nitrogen was fixed when a mixture of lime and carbon were ignited at 1400° in the absence of feldspar.

The increase of fixation with time of ignition is shown in the following table, when 4 parts of feldspar, 2 parts of carbon and 4.3 parts of calcium carbonate were ignited for different lengths of time.

Time of ignition Hours	Temperature	Potash volatilized in percentage of total present	Nitrogen fixed in percentage of	
			feldspar taken	aluminum in feldspar
1.....	about 1200°	55.7	1.27	12.2
2.....	about 1200°	68.9	1.34	12.9
4.....	about 1200°	83.0	1.68	16.1
1.....	about 1400°	100.0	6.10	58.5
2.....	about 1400°	100.0	7.44	71.5

Since the nitrogen in aluminum nitrid amounts to 51.6 per cent of the aluminum, it follows that the nitrogen fixed in the last two experiments is greater than what would correspond to the aluminum in the feldspar. No direct experiments have yet been made to determine in what form the nitrogen is combined, whether as nitrid or carbo-nitrid of silicon or aluminum. When boiled with water the material gave off ammonia very slowly, and even slowly when boiled with sodium hydroxide solution although in this case the ammonia was evolved more rapidly than when the digestion was made with water alone.

Equipment is now being installed to carry on the experiments on a larger scale and at higher temperatures. In the meantime it was thought advisable to publish the preliminary results obtained.

BUREAU OF SOILS
U. S. DEPARTMENT OF AGRICULTURE
WASHINGTON

THE LEACHING OF POTASH FROM FRESHLY CUT KELP

By A. R. MERZ¹ AND J. R. LINDEMUTH²

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Drift kelp usually has a very low content of potash

or rather potassium chloride. It has been currently reported that the loss of potassium chloride from the freshly cut plant proceeds quite rapidly. Theoretically, there should be a distribution of the base between the plant and the water in which it lies, and this redistribution might reasonably be expected to commence at once, and even to be much augmented or accelerated because of the relatively high content of sodium chloride in sea water. This problem is one of considerable economic importance, since it has been proposed in the harvesting of kelp as a raw material for the production of potassium salts, to tow the cut kelp from the groves to the landing and thus avoid lifting the material upon a barge or boat.

In order to obtain some information and data bearing directly on this economic question, Professor W. C. Crandall, of the Scripps's Biological Station, at La Jolla, California, collected two large samples of *Macrocystis* on a recent cruise of the yacht, "Agazzi", of that station, towing these samples and taking subsamples from time to time which were forwarded to the laboratory of the Bureau of Soils, in Washington, for analysis. One of the large samples for the leaching experiment was collected near Pt. Loma and the other near Coronado Island. The subsamples, on their receipt in Washington, had commenced to ferment. This fact, however, could not have any particular influence on the data here given. The tables which follow are self-explanatory. The analytical methods employed have been described in THIS JOURNAL, 4, 431 by Turrentine, and acknowledgment is here made to Mr. T. C. Trescott, of the Bureau of Chemistry, who kindly made the nitrogen determinations for us.

From this examination it is evidently a matter of considerable difficulty to obtain a fair average sample of wet kelp. It also appears that freshly cut kelp, when immersed in sea water, does not, at least at first, lose its potash content very rapidly.

Attention is called to the analysis of "sea lettuce" in Table II. This is not a kelp but a rock weed rather common on the Pacific coast. Algae other than the giant kelps have not usually shown a high potash content, but this analysis indicates that the matter is worthy of further investigation. The difficulties to be apprehended in harvesting rock weeds, however, do not leave much promise for their economic importance at present, even though they should be found to have a high content of potash or other valuable

TABLE I—SAMPLE TAKEN NEAR CORONADO ISLANDS

No.	Time exposed	Per cent K ₂ O	Per cent				Per cent KCl	Per cent N	Per cent I	Remarks
			sol. salts	Per cent org. matter	Per cent ash	Per cent				
1m.....	None } fresh cut	12.48	31.46	65.38	3.16	19.72	0.98	0.16	Dried and sent by mail	
1x.....	None } fresh cut	13.47	31.26	65.98	2.76	21.28	0.79	0.14	Wet, sent by express in jar	
1A.....	3 hrs.	15.56	34.00	62.38	3.62	24.59	0.95	0.13	Wet, sent by express in jar	
1B.....	6 hrs.	17.30	38.26	58.66	3.08	27.33	1.00	0.13	Wet, sent by express in jar	
1C.....	14 hrs.	17.64	38.30	58.30	3.40	27.87	1.07	0.06	Wet, sent by express in jar	
1D.....	17 hrs.	13.35	31.96	54.96	3.08	21.10	0.83	0.10	Wet, sent by express in jar	
1E.....	20 hrs.	10.74	26.36	70.66	2.98	16.97	0.90	0.13	Wet, sent by express in jar	
1F.....	None } fresh cut	18.28	36.78	60.86	2.36	28.89	0.51	0.13	Stems only, dried and sent by mail	
1G.....	None } fresh cut	9.90	25.94	70.18	3.88	15.64	0.84	0.07	Leaves only, dried and sent by mail	

¹ Scientist in Soil Laboratory Investigations, Bureau of Soils, U. S. Dept. Agr.

² Scientist in Fertilizer Investigations, Bur. of Soils, U. S. Dept. Agr.

constituent, and the importance of the matter probably lies in showing that their occasional or accidental



TABLE II—SAMPLE FROM NEAR PT. LOMA

No.	Time exposed	Per cent K ₂ O	Per cent sol. salt	Per cent org. matter	Per cent ash	Per cent KCl	Per cent N	Per cent I	Remarks
2A.....	None	15.82	40.88	47.32	11.80	25.00	2.77	0.18	Wet, sent in jar
2B.....	3 1/2 hrs.	13.43	34.82	56.54	8.64	21.22	2.66	0.19	Wet, sent in jar
2C.....	15 1/2 hrs.	14.96	37.66	52.76	9.58	23.64	2.40	0.15	Wet, sent in jar
2D.....	18 1/2 hrs.	13.05	34.34	60.12	5.54	20.62	1.77	0.15	Wet, sent in jar
2E.....	21 1/2 hrs.	11.00	30.22	62.52	7.26	17.38	1.85	0.16	Wet, sent in jar
2F.....	24 1/2 hrs.	11.59	30.52	59.92	9.56	18.31	2.10	0.14	Wet, sent in jar
2G.....	27 1/2 hrs.	13.68	38.94	50.56	10.50	21.62	2.22	0.24	Wet, sent in jar
2H.....	39 1/2 hrs.	10.31	27.84	62.38	9.78	16.29	1.97	0.14	Wet, sent in jar
2I.....	42 1/2 hrs.	16.39	38.78	53.30	7.92	25.90	2.22	0.21	Wet, sent in jar
2J.....	45 1/2 hrs.	11.77	32.44	60.54	7.02	18.60	1.99	0.22	Wet, sent in jar
2K.....	48 1/2 hrs.	16.69	39.72	54.22	5.96	26.37	1.79	0.19	Wet, sent in jar
2L.....	51 1/2 hrs.	15.77	40.80	45.88	13.32	24.92	2.29	0.22	Wet, sent in jar
2M.....	63 1/2 hrs.	16.74	42.58	45.36	12.06	26.45	2.49	0.21	Wet, sent in jar
2N.....	66 1/2 hrs.	16.75	38.64	55.28	6.08	26.46	1.91	0.19	Wet, sent in jar
2O.....	72 1/2 hrs.	14.67	35.58	58.12	6.30	23.18	1.88	0.19	Wet, sent in jar
2P.....	75 1/2 hrs.	12.61	34.74	55.46	9.80	19.93	2.66	0.22	Wet, sent in jar
2Q.....	87 1/2 hrs.	15.54	40.84	48.92	10.24	24.56	2.47	0.19	Wet, sent in jar
2R.....	90 1/2 hrs.	17.62	41.42	49.48	9.10	27.84	2.16	0.16	Wet, sent in jar
2S.....	93 1/2 hrs.	17.03	42.44	44.50	13.06	26.90	2.28	0.20	Wet, sent in jar
2T.....	96 1/2 hrs.	15.61	37.80	54.42	7.78	24.67	2.16	0.10	Wet, sent in jar
2U.....	99 1/2 hrs.	20.28	46.28	43.96	9.76	32.05	2.12	0.20	Wet, sent in jar
2V.....	111 1/2 hrs.	15.72	38.80	51.44	9.76	24.84	2.26	0.23	Wet, sent in jar
2W.....	135 1/2 hrs.	15.60	39.24	48.74	12.02	24.65	2.57	0.27	Wet, sent in jar
2.....	111 1/2 hrs.	15.27	34.52	57.42	8.06	24.13	1.80	0.18	Dried and sent by mail
M. P.....		30.00	57.10	32.76	10.14	47.41	1.66	0.06	Sea lettuce from Marrowstone Point

presence in harvested kelp is not detrimental to the value of the kelp.

BUREAU OF SOILS
U. S. DEPARTMENT OF AGRICULTURE
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A POSSIBLE COMMERCIAL UTILIZATION OF NELSONITE

By WILLIAM H. WAGGAMAN¹

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INTRODUCTION

In Nelson County, Va., there are large bodies of rock locally known as Nelsonite which consist essentially of the two minerals ilmenite and apatite. The material is not only of scientific interest but in the light of some recent experiments performed in this laboratory may be of considerable commercial importance.

The rock occurs in the foothills of the Blue Ridge near Roseland, Va., about 7 miles northwest of Arrington, a station on the Southern R. R., and 24 miles northeast of Lynchburg. According to Watson² it is probably of pre-Cambrian age and occurs in dikes which are in places over 65 feet thick and 2,100 feet in length as exposed on the surface. The formation can be traced in a general northeast southwest direction for about 7 miles. Watson described it as "a hard rock composed of granular white apatite and black ilmenite." After weathering, however, the rock can be readily disintegrated and a more or less clean separation made of the two minerals by either of the methods described further on in this paper.

ILMENITE

Ilmenite or titanite iron is a black crystalline mineral having feebly magnetic properties. It has a specific gravity when pure of 4.5 to 5.0 and the following formula and composition:

FeTiO ₃	{ Titanium oxide (TiO ₂).....	52.7 per cent
	{ Iron protoxide (FeO).....	47.3 per cent

According to Dana,³ however, the ratio of iron to

¹ Scientist in Fertilizer Investigations, Bureau of Soils, U. S. Dept. Agr.

² "Mineral Resources of Virginia," 1907, p. 300; "Economic Geology,"

U. S. Geol. Survey, 1909, pp. 206-7.

³ "A System of Mineralogy," 1892, pp. 217-219.

titanium varies widely corresponding to the general formula $MFeTiO_3 \cdot NFe_2O_3$.

Ilmenite is rather widely distributed in nature, occurring in beds in gneiss and other crystalline rocks; also in small particles in many crystalline rocks and frequently associated with magnetite. It is used in the manufacture of ferro-titanium for the steel industry. In 1911 there were 51.17 tons of ferro-titanium imported into this country valued at \$22,700, which gives an average price of \$443.61 per ton.

APATITE

Apatite or phosphate rock is a brittle crystalline mineral which varies in color from almost white to dark green-red and brown. There are two main varieties of this mineral; namely, chlor-apatite and fluor-apatite, the latter being much more plentiful than the former. The specific gravity of apatite varies from 3.17 to 3.23 and the formula and composition of the two varieties according to Dana, are as follows:

Chlor-apatite, CaCl.Ca ₄ (PO ₄) ₃ =	{ P ₂ O ₅ = 41.0 per cent
	{ CaO = 53.8 per cent
	{ Cl = 6.8 per cent
Fluor-apatite, CaF.Ca ₄ (PO ₄) ₃ =	{ P ₂ O ₅ = 42.3 per cent
	{ CaO = 55.5 per cent
	{ F = 3.8 per cent

Apatite, however, is seldom found absolutely pure and though widely distributed in nature does not often occur in minable quantities. On treatment with sulfuric acid it yields a very high-grade superphosphate, and if it were not for the fact that the better deposits are rather inaccessible and that careful picking or "cobbing" is necessary before a product sufficiently high-grade for the market is obtained, this mineral would be more extensively used in the fertilizer industry.

SEPARATION OF APATITE AND ILMENITE

At first sight the association of apatite and ilmenite in a rock would seem to seriously affect the commercial value of both of these minerals. In the manufacture

of steel, phosphorus is the most dreaded impurity which can occur in the iron or iron ore used, so that ilmenite containing over 1 or 2 per cent of this element would be rejected as dangerous.

On the other hand, the presence of comparatively small percentages of iron in phosphate rock nearly always causes trouble in the acid phosphate produced therefrom, and when this element is present in large amounts it often renders the acid phosphate unfit for use.

According to Watson¹ the ratio of apatite to ilmenite in nelsonite varies widely from rock consisting largely of the former mineral to that in which ilmenite predominates. Before such a deposit can be economically worked, therefore, it seems essential that a method be devised for making a clean separation of the two minerals.

There are two methods which suggest themselves for effecting such a separation. The first is based on the difference in the specific gravities of the minerals, and the second on the magnetic properties of the ilmenite. The ratio of the specific gravity of apatite and ilmenite is as 3.2 to 4.5. This difference is sufficiently great to warrant a fairly clean separation provided the rock is crushed to uniform size. Unless it is ground very fine, however, a preliminary screening is necessary before suspending the material in water. The various modifications of the methods of separating substances by the difference in their specific gravities are well known and need not be enumerated here.

A separation of the two minerals by means of a magnetic separator has also proved feasible but owing to the rather feeble magnetic properties of the ilmenite it is not entirely removed from the apatite, by this method, and though a preliminary heating enhances somewhat this magnetism, such a procedure would probably prove too expensive in commercial practice.

From a number of experiments with nelsonite it has been pretty well demonstrated that while ilmenite can be fairly easily removed from apatite by either of the methods outlined above it is practically impossible to effect a clean separation by mechanical means, since the nature of the rock is such that there will always be particles of the two minerals which adhere strongly to each other.

We must, therefore, resort to chemical means to bring about this desired end. In the experiments described below it is shown that ilmenite may be almost completely freed from apatite by means of sulfuric acid, with a minimum amount of waste and without involving any great expense. Moreover, all of the products obtained during the process can be utilized commercially.

EXAMINATION AND SEPARATION OF THE MINERALS IN NELSONITE

A typical sample of the rock was submitted to a microscopic examination by Mr. W. H. Fry of this Bureau and found to consist essentially of crystallized ilmenite and partially decomposed apatite. The material was then crushed to pass a $1/2$ mm.

¹ "Mineral Resources of Virginia," 1907.

sieve and a separation made by means of a strong magnet. Owing to the rather feeble magnetic qualities of part of the ilmenite this separation was made at the expense of the apatite which was afterwards found to contain a considerable amount of the former mineral in a finely divided state.

The results of this separation are given below:

		Per cent	
Nelsonite as separated by magnet in the laboratory contained.....	}	Apatite.....	23.8
		Ilmenite.....	76.2

Both the ilmenite and apatite thus separated were analyzed by Mr. C. F. Miller of this Bureau, with the following results:

		Per cent	
Ilmenite as separated by magnet in the laboratory contained.....	}	FeO.....	44.2
		TiO ₂	44.5
		P ₂ O ₅	0.8
Apatite as separated by magnet in this laboratory contained.....	}	Fe ₂ O ₃	6.5
		P ₂ O ₅	33.3
		Ca ₃ (PO ₄) ₂	72.8

Although the amount of phosphoric acid remaining in the separated ilmenite was less than 1 per cent, such a clean separation could hardly be attained in actual works practice. Even this amount of phosphoric acid is objectionable and would probably seriously affect the sale of the material.

Preliminary experiments showed that ilmenite is almost entirely unattacked by dilute sulfuric acid, while it is well known that apatite is acted upon quite energetically by the same acid: so 3 samples of the separated ilmenite of 10 grams each were treated with sulfuric acid of different strengths. The mixtures were warmed slightly and stirred intermittently for 20 minutes. At the end of that time the acid was diluted with water, filtered and washed. The filtrate was made up to 200 cc. and the percentages of iron and phosphoric acid thus dissolved determined in the two solutions. The residual ilmenite was again weighed and also analyzed for phosphoric acid.

The results of these analyses are given in Tables I and II.

TABLE I—ANALYSIS OF SULFURIC ACID EXTRACTS OF ILMENITE

3 samples of 10 grams each treated with 50 cc. of sulfuric acid of various strengths.

Strength of H ₂ SO ₄ used	Amount of P ₂ O ₅ extracted by acid Per cent	Amount of Fe ₂ O ₃ extracted Per cent
50° B.	0.22	0.19
32° B.	0.46	0.29
25° B.	0.41	0.32

TABLE II—ANALYSIS OF ILMENITE AFTER EXTRACTION WITH SULFURIC ACID

Strength of acid used in extraction	Loss in weight of ilmenite Per cent	Amount of P ₂ O ₅ remaining in ilmenite Per cent
50° B.	1.04	0.47
32° B.	1.75	0.31
25° B.	1.58	0.36

From the data given in Tables I and II it will be seen that the apatite remaining in ilmenite after the mechanical separation can nearly all be extracted by means of dilute sulfuric acid (32° B.) without appreciably affecting the ilmenite. The amount of iron

thus dissolved is too small to lower the value of the acid extract which can be used in treating the separated apatite to produce superphosphate.

While this acid extract is considerably weaker than that employed in the manufacture of acid phosphate it could be readily brought to the desired concentration, either by evaporation, or by mixing it with the sulfuric acid which is shipped in tank cars; this latter acid has a strength of 60° B.

The apatite as separated in the laboratory contained a considerable amount of iron, but this impurity should cause no trouble in the making of superphosphate since only the compounds of this element which are soluble in sulfuric acid cause the objectionable reversion to take place or render the acid phosphate too sticky for use.

ECONOMIC CONSIDERATIONS

If both the ilmenite and apatite can be disposed of at a fair price the mining of nelsonite should prove quite profitable. Unfortunately the market for ilmenite is rather limited at present, and it is doubtful whether the steel industry would handle the large tonnage which extensive development could produce. If nelsonite could be depended upon to yield an average of 50 per cent of apatite the exploitation of the rock for this mineral alone would be well worth while, but it is doubtful if the general mine run contains such a high percentage of apatite.

The production of apatite from nelsonite would undoubtedly be more expensive than the mining and preparation of Florida phosphate, since the rock is quite hard and would require rather fine grinding before a separation could be made. On the other hand, the average content of phosphate rock in nelsonite is considerably higher than it is in the Florida deposits. The amount of phosphate rock actually marketed from this latter state is probably not more than 15 per cent of the total material taken from the mines; in other words, every ton of phosphate produced involves the handling of over 6 tons of waste material.

The following figures, while approximate, are conservative and give some idea of the cost of mining and separating the two minerals in nelsonite:

TABLE III—COST OF MINING NELSONITE AND MAKING A MECHANICAL SEPARATION OF THE TWO MINERALS, APATITE AND ILMENITE PER TON

Cost of mining.....	\$1.00
Cost of grinding.....	0.50
Cost of mechanical separation.....	0.25
Total cost.....	\$1.75

Assuming that 1 ton of nelsonite will yield 1/2 ton each of ilmenite and apatite then the cost of preparing this amount of ilmenite for the market will be as follows:

TABLE IV—COST OF PREPARING ONE-HALF TON OF ILMENITE FOR THE MARKET

1/2 expense of mining and separating minerals.....	\$0.88
1/2 ton of sulfuric acid (60° B.) at \$8.00 per ton.....	4.00
Cost of washing ilmenite (1/2 ton).....	0.30
Cost of drying ilmenite (1/2 ton).....	0.15
Total cost of 1/2 ton ilmenite.....	\$5.33

The sulfuric acid extract after concentration can

be used in making acid phosphate by mixing it with an equal weight of the separated apatite.

The cost of manufacturing this product would be approximately as follows:

TABLE V—COST OF MAKING ONE TON OF ACID PHOSPHATE FROM ONE-HALF TON OF APATITE SEPARATED FROM NELSONITE

1/2 expense of mining and separating minerals.....	\$0.88
2/3 ton of sulfuric acid (60° B.) (cost included in expenses for cleaning ilmenite).....
Cost of drying apatite (1/2 ton).....	0.15
Cost of mixing and handling.....	0.50
Total cost of 1 ton acid phosphate.....	\$1.53

The total cost of mining 1 ton of nelsonite (having the above composition) and producing therefrom 1/2 ton of ilmenite (practically free from phosphorus), and 1 ton of acid phosphate (16 per cent P₂O₅) would be (exclusive of interest on investment, insurance and taxes) about \$6.86 per ton—an amount which would be more than covered by the value of the acid phosphate produced.

BUREAU OF SOILS
U. S. DEPARTMENT OF AGRICULTURE
WASHINGTON

QUANTITATIVE DETERMINATION OF ROSIN IN PAPER

By C. FRANK SAMMET

Received July 7, 1913

As the necessity for securing record papers so free from chemicals and deleterious substances that they may be preserved indefinitely becomes more apparent, the importance of the determination of the sizing materials in the papers, particularly rosin, increases. Obviously, the resistance of the cellulose fibers to the action of light, heat, moisture and fumes is greatest in paper free from harmful materials. In carefully prepared specifications for durable papers it is customary to limit the percentage of rosin which may be present, as this substance, especially when used in large quantities, materially hastens discoloration and deterioration. Limits for rosin are now included in government paper specifications.

ALCOHOL METHODS

Of the numerous methods used for estimating the amount of rosin in paper, many are based on its extraction with alcohol, modifications being introduced in the subsequent treatment of the solution containing the extracted rosin.

Turbidity Method.—The turbidity produced by the precipitation of rosin from solution, on diluting the alcoholic extract with water, is compared with the turbidity produced in a like manner in an alcoholic solution, the rosin concentration of which is known. Although in many cases 0.0002 gram differences in the same solution may thus be detected, it was found that in some papers the determinations varied 25 or even 50 per cent from the actual amount of rosin present. Since more than 0.006 gram of rosin precipitated in 100 cc. of water interferes with the delicacy of the comparison, the quantities used must be so small that the percentage error is greatly affected by the uniformity of precipitation, which depends upon such factors as the sequence of operation, time allowed for precipitation and subsequent comparison,

the amount of alcohol present, the foreign materials extracted by alcohol, and the undetermined factors influencing the size of the precipitated rosin particles. It is probable, however, that if the alcoholic extract were freed from foreign materials by evaporation, extraction with ether, and washing with water, as described below, the turbidity method would give more accurate results.

Acidified Alcohol Method.—The rosin is precipitated from the alcoholic extract by dilution with acidulated water, filtered off, washed, dried, and weighed. The sources of error here are incomplete precipitation in the presence of colloidal materials, the escape of minute suspended particles through the filter, and the contamination of the rosin by the co-precipitation of foreign materials.

Direct Evaporation Method.—The acidulated alcoholic solution with which the rosin is extracted is evaporated directly and the residue weighed. This method gives results which, in some instances, may be too high by 60 per cent, because of the contamination by such foreign substances as glue, invert starch, cellulose, soda, aluminum salts, fats and greases, which are also removed from the paper by the acidified alcohol.

*Schumann's Method.*¹—The rosin, having been extracted by a dilute solution of alkali, is precipitated from the extract, filtered off, washed, dried, and weighed. The difficulty of removing the alkali resinates, especially from soft papers which are easily pulped by the action of the alkali, and the contamination of the precipitated rosin by foreign substances extracted by the alkali renders this method unsatisfactory. Were this precipitate of rosin taken up in ether and the ether extract washed with water, then evaporated and weighed, more accurate results could be obtained, although the loss from incomplete extraction and washing would still occur.

ACIDIFIED ETHER METHOD

Acidified ether is the solvent in this method, the ether extract of the paper being directly evaporated, dried, cooled and weighed. It is open to the objection that the resinates are not readily extracted by ether.

ALCOHOL-ETHER METHOD

Because of the objections to the current methods, a procedure reasonably accurate and at the same time simple in execution was sought. The modifications of the method as first devised,² which have been developed by experience, are incorporated in the following procedure:

Cut 5 grams of paper into strips approximately one-half inch wide and fold them in numerous small crosswise folds. Place the folded strips in a Soxhlet extractor and fill with acidulated alcohol diluted to approximately 83 per cent, made by adding to 100 cc. of 95 per cent alcohol 15 cc. of acidulated water containing 5 cc. of glacial acetic acid to 100 cc. of distilled water. Place the Soxhlet flask directly in the boiling water of a steam bath and extract by siphoning from six to twelve times, according to the nature of

the paper. Wash the alcoholic extract of rosin, which may contain foreign materials, into a beaker and evaporate to a few cubic centimeters on a steam bath. Cool, take up in about 25 cc. of ether, transfer to a 300 cc. separatory funnel containing about 150 cc. of distilled water to which has been added a small quantity of sodium chloride to prevent emulsion, shake thoroughly, and allow to separate. Draw off the water into a second separatory funnel and repeat the treatment with a fresh 25 cc. portion of ether. Combine the ether extracts which contain the rosin and any other ether-soluble materials and wash twice, or until the ether layer is perfectly clear and the line between the ether and water is sharp and distinct, with 100 cc. portions of distilled water to remove salts and foreign matter. (Should glue, which is extracted from the paper by alcohol, interfere by emulsifying with the ether, it may be readily removed by adding a strong solution of sodium chloride to the combined ether extracts, shaking thoroughly and drawing it off, repeating if necessary, before washing with distilled water.) Transfer the washed ether extract to a weighed platinum dish, evaporate to dryness and dry in a water oven at from 98° to 100° C. for exactly one hour, cool, and weigh. This length of time is sufficient to insure complete drying; prolonged heating causes a continual loss of rosin.

It may be noted that any alcohol- and ether-soluble fats, waxes or other foreign materials present in the paper will be extracted with the rosin. These substances, however, if present at all, occur in such small amounts in high-grade papers that their influence on the result is negligible. It has been found that the quantity of rosin obtained from 5 grams of paper is sufficient for accurate work, and that a greater amount of paper materially interferes with complete extraction within a reasonable time. The folding of the strips is important, as it prevents the sticking together of the pieces when wet and consequent incomplete extraction which results when the paper is merely torn or cut into small pieces. It is well, although not essential, to saturate the folded strips with acidulated alcohol when they are placed in the Soxhlet flask, as this hastens the breaking up of the resinates.

If the alcohol is too dilute, the extraction of rosin from the paper is incomplete, and large quantities of glue interfere with the subsequent separation with ether. If acidulated alcohol of 95 per cent concentration is used, the acid does not break up the resinates which, therefore, will not be extracted. By placing the Soxhlet flask directly in the boiling water of a steam bath, distillation is hastened and the solvent in the extraction chamber is kept hot by the surrounding steam, which completes the extraction in a minimum length of time.

It was found that neutral 95 per cent alcohol does not completely remove the rosin within a reasonable time, probably because resinates are not readily soluble. A representative instance occurred when 10 grams of paper were extracted in a Soxhlet flask with 125 cc. portions of 95 per cent neutral alcohol by the alcohol-ether method, the following results being obtained:

¹ Sindall, "Elementary Manual Paper Technology," 1910.

² U. S. Dept. Agr., Office of the Secretary, *Report* 89, 1909.

TABLE I—ROSIN OBTAINED FROM NEUTRAL ALCOHOL SOLUTION

No. of extraction	Time of extraction	Rosin obtained
	Hours	Per cent
1	4	1.786
2	3	0.138
3	3	0.094
4	3	0.066
5	4	0.040

It was also found that the percentage removed in successive extractions with neutral 95 per cent alcohol varies with the amount of rosin originally present and with the nature of the paper extracted.

The alcohol-ether method has been subjected to tests, which may best be illustrated by the following typical examples taken from numerous determinations:

Ten grams of rosin were dissolved in a solution of sodium carbonate and made up to 1000 cc. Twenty-five cubic centimeters, representing 0.25 gram of rosin, were withdrawn from this solution, the rosin precipitated by the addition of aluminum sulfate and acetic acid, and the whole then extracted with two 25 cc. portions of ether. The ether extract was washed with distilled water, as directed in the method, evaporated, dried at 100° C. for one hour, cooled, and weighed. The dried material weighed 0.2509 gram, agreeing closely with the amount of rosin taken.

The method was further tested by extracting many different kinds of paper and determining the amount of rosin present. A second extraction was made on the same sample of paper and the rosin then taken up in ether and determined, with the results shown in Table II. Third and fourth extractions with 83 per cent alcohol have not given, in any case, more than 0.04 per cent of rosin.

TABLE II—DETERMINATION OF ROSIN

Kind of paper	Amount of rosin extracted	
	First extraction	Second extraction
	Per cent	Per cent
Ledger.....	1.72	0.02
Bond.....	1.90	0.01
Writing.....	1.20	0.06
Sized and supercalendered printing.....	0.81	0.04
Manila.....	1.48	0.03
Double coated.....	1.57	0.05

The effect of the concentration of acidified alcohol on the extraction of rosin is shown in the following table. Ninety-five per cent alcohol diluted to the approximate percentage given was used in the determination:

TABLE III

Extraction No.	Amount of rosin extracted			
	86 per cent alcohol	83 per cent alcohol	79 per cent alcohol	73 per cent alcohol
Ledger paper:				
1.....	1.67	1.70	1.63	1.53
2.....	0.02	0.02	0.03	0.09
3.....	0.01	0.01	0.02	0.01
Total.....	1.70	1.73	1.68	1.63
Writing paper:				
1.....	2.17	2.03	1.96	2.00
2.....	0.07	..	0.06	0.12
3.....	0.01	0.03	0.03	0.09
Total.....	2.25	2.06	2.05	2.21

These results show that a concentration of 86 or

83 per cent is preferable to one of 79 or 73 per cent, which gives slightly lower results. Even at these latter concentrations, however, the results are within the experimental error of 0.20 per cent, except in the case of the 73 per cent concentration in the first extraction of the writing paper where the percentage error is 0.21.

The variations in results due to sampling are shown by the following figures, obtained by extracting different sheets of the same sample of ledger paper as that reported in Table III.

TABLE IV
Amount of rosin extracted

Extraction No.	Amount of rosin extracted			
	86 per cent alcohol	83 per cent alcohol	79 per cent alcohol	73 per cent alcohol
Sample I:				
1.....	1.66	1.67	1.56	1.65
2.....	0.09	0.02	0.07	0.04
3.....	0.01	0.01	0.04	0.08
Total.....	1.76	1.70	1.67	1.77
Sample II:				
1.....	1.79	1.79	1.70	1.75
2.....	0.04	0.04	0.02	0.02
3.....	0.02	0.04	0.03	0.03
Total.....	1.85	1.87	1.75	1.80

These results are entirely within the experimental error of 0.20 per cent, as far as the 86 and 83 per cent concentrations are concerned.

A comparison of the results obtained by the alcohol-ether method with the results obtained by the alcohol evaporation and acidified ether methods, in which the total extract is dried, weighed and calculated as rosin in each case, gave the following results:

TABLE V
Amount of rosin extracted

Method:	Amount of rosin extracted							
	Machine finished printing Per cent.	Writing Per cent.	Ledger (rag) Per cent	Coated. Per cent	Ledger (wood) Per cent	Bond (wood) Per cent	Wrapping (wood) Per cent	
Acidified alcohol direct evaporation.....	2.87	1.39	1.85	30.6	2.61	2.82	1.79	
Acidified ether direct evaporation.....	2.65	1.22	0.94	2.86	1.66	1.90	1.20	
Acidified alcohol-ether....	2.47	1.18	1.70	0.92	1.25	2.00	1.22	

This table shows that the acidified alcohol extract followed by direct evaporation gives results which are too high, due to the presence in the extract of both soluble and insoluble foreign material. In the alcohol-ether method these materials are eliminated through precipitation and washing.

Acidified ether does not extract as much rosin in the same number of siphonings as acidified alcohol. The method has a tendency to yield low results. In some instances, however, insoluble material is separated from the paper, thereby giving results which are too high.

The amount of material which may be extracted from coated paper by acidified alcohol is indicated when the extract is evaporated directly. To report such material as rosin is obviously erroneous. The acidified alcoholic extract was very opalescent and gave positive tests for glue and casein. Ordinarily,

of course, one would not report such results as rosin. In those cases, however, where the error is less obvious the analyst may frequently fail to detect it.

CONCLUSIONS

The alcohol-ether method has consistently given the most accurate and concordant results in this laboratory in all comparative work. Contrary to our first experience with it, when an insufficient amount of acid was used, extraction with one portion of acidified alcohol, making from six to twelve siphonings, has been found to be sufficient for all practical purposes. The results, when 83 per cent alcohol is used, are within the experimental error of 0.20 per cent, as are the variations due to sampling. No other procedure tried has proved equally satisfactory.

LEATHER AND PAPER LABORATORY
BUREAU OF CHEMISTRY
WASHINGTON

COLORIMETRIC METHOD FOR TITANIUM IN IRON AND STEEL

By C. R. McCABE

Received March 6, 1913

In the colorimetric method for titanium, the color is usually produced in a sulfuric acid solution of the iron or steel. The color of the ferric sulfate is in most cases much more intense than that of the titanium. The color employed for comparison is therefore a blend of two colors in which the color on which we base our method is the subordinate one. If the quantities of iron present in standard and test are the same, it follows that so far as iron is concerned the two solutions have the same color at equal volumes. If comparison be attempted at unequal volumes, even though the divergence be slight, the more concentrated solution contains a more intense iron color than the other. Hence, we must have a standard which contains the same percentage of titanium as the test sample, within very narrow limits.

This consideration led the writer to seek some plan whereby a standard would be always available precisely like the test sample. The plan adopted is to produce the color in a sulfuric acid solution of the iron or steel and then imitate it in a solution of a non-titanium steel by adding a standard solution of titanium from a burette.

PREPARATION OF TITANIUM STANDARD SOLUTION

Place about 100 grams of ferro-titanium in a dish and pour over it about 50 cc. of strong HCl. Heat, and when the solvent action has progressed for a few moments add 5 cc. of nitric acid (sp. gr. 1.2). Continue heating until the acid is about half evaporated, dilute with 15 or 20 cc. water and filter through a large filter paper. Treat the ferro-titanium again with hydrochloric and nitric acids, dilute, and filter in this manner a dozen times or more until the desired quantity of ferro-titanium has been dissolved. Simultaneously with the solution of the ferro-titanium the combined filtrates are evaporated in a large beaker on a hot plate. Evaporate until separation of titanous acid is observed. Pour into several separatory funnels, and add to each 50 cc. ether. Make repeated

ether extractions of the iron until the titanous acid is free of iron as shown by the potassium sulfocyanide test. Combine the several acid solutions and place in a 400 cc. beaker. To the titanous acid emulsion add 150 cc. sulfuric acid (1 : 3). Heat and filter from the insoluble portion, which is usually slight. Dilute the filtrate to about 700 cc., in a liter beaker, add ammonia in excess, boil and allow the precipitate to settle. Decant or siphon away the supernatant solution and wash the hydrated titanous acid free of chlorides by decantation.

Dissolve the titanous acid in 50 cc. of sulfuric acid (1 : 1) and dilute to a liter. Determine the strength of this stock solution by precipitation of 100 cc. with ammonia. Calculate the dilution required to yield a standard solution containing 0.0002 gram of titanium per cc. Remove the requisite quantity of the stock solution and dilute according to the calculation. Finally check the strength of the standard solution by precipitation of 100 cc. with ammonia.

TITANIUM IN IRON AND STEEL

When titanium exists in iron or steel above 0.02 per cent it may be readily determined in less than 1/2 hour by the color method to be described. When present in smaller amount it may be determined with great precision by the writer's more refined color method requiring an ether separation of iron.

PROCEDURE FOR TITANIUM ABOVE 0.02 PER CENT

Put two grams each of the titanium steel and a non-titanium steel into 300 cc. Erlenmeyer flasks. Pour into each flask 80 cc. of sulfuric acid (1 : 3) and heat on a hot plate to complete solution. To each add 4 cc. of nitric acid (sp. gr. 1.2) and continue boiling until the solutions are free of fumes; cool. Transfer the solution of non-titanium steel to a comparing tube, using as little wash water as possible. Pour the test sample into the companion tube and dilute to the same volume as the other. Now observe if the two solutions have colors of the same character and depth, as they should. If the test sample be cast iron, filter the solution into the tube, preferably using a small wad of absorbent cotton because of gelatinous silica. Match the color with that of the non-titanium steel solution. Owing to the fact that cast iron contains a much larger percentage of metal-oids than steel, it is usually found that the volume of the iron solution is a trifle less than that of the non-titanium steel when the colors match. In this case discard sufficient of the non-titanium steel solution to make the volumes equal.

Having obtained solutions showing the same color in equal volumes, introduce into each 2 cc. of 3 per cent hydrogen peroxide. Mix, and observe closely if the test solution shows any deepening of color. If titanium be present in as small an amount as 0.02 per cent, this deepening will be in evidence. It may even be detected with 0.01 per cent present, but in that case it is better to rely on the more refined method to be described.

Titanium if shown to be present in notable quantity is determined by adding from a burette a sufficient

amount of standard titanium solution to the solution of non-titanium steel to imitate the color of the test solution. If but two or three cc. are required, the amount used may be accepted as representing the amount in the test solution without further concern. But if more is required, the equality of volume of the two solutions, so vital to accurate results, must be restored before final comparison. When colors match at equal volumes, the amount of standard titanium solution used indicates the amount of titanium in the test sample.

When the material analyzed is *cast iron*,¹ the insoluble residue retains a small amount of titanium. For ordinary technical purposes this may be neglected. But if an accurate determination is desired, burn the insoluble in a platinum crucible, volatilize silica with hydrofluoric acid and 8 drops sulfuric acid (1 : 1), fuse residue with 1 gram sodium carbonate, dissolve out in dilute sulfuric acid and add to the main solution.

PROCEDURE FOR TITANIUM BELOW 0.02 PER CENT

The fact that titanium does not alloy readily with iron often results in the production of a material in which titanium, though employed in the process of manufacture, exists in minute quantity. This circumstance led the writer to cast about for some better means of determining these small amounts than has heretofore been available. The method below requires about an hour and a quarter, but is very useful where a report more satisfactory than the conventional "trace" is desired.

Weigh 2 grams of a non-titanium steel and the same quantity of the test sample. Place in dishes, add 50 cc. strong hydrochloric acid and heat to complete solution. Add to each 4 cc. nitric acid (sp. gr. 1.2) to oxidize iron. Evaporate each solution to about 10 cc., pour into separatory funnels, and wash the dishes with hydrochloric acid (2 parts strong acid, 1 part water). The total solution in the funnels should be in each case about 25 cc. Add cautiously to each, 50 cc. ether (free of alcohol), shake under a hydrant, and allow the two solutions to separate. Draw off the lower solutions into 400 cc. beakers, making the separation with care so that not more than a drop of the ethereal solution passes into the beaker in either case. It is essential to conduct these ether separations with the twofold purpose of having the minimum amount of iron in the acid solutions and of having the iron content of the acid solutions nearly equal.

Dilute each solution with 225 cc. hot water, add ammonia in slight excess, and boil. Allow the precipitates to settle and filter through 11 cm. filter papers. Wash thoroughly with hot water to remove chlorides.

Open the two filter papers into dishes, tearing off that portion of the filter paper which holds no precipitate. Pour over each 10 cc. of sulfuric acid (1 : 3). Rock the dishes until the precipitate has all dissolved. Filter each into 30 cc. comparison tubes, using small funnels and 7 cm. filter papers. This filtration may be dispensed with, but it is best to re-

move paper fiber. Wash the dishes and pass the washings through the filters. The total solution and washings in each instance should be equal in volume, between 15 and 18 cc.

The solutions now have a light maroon color, depending on the amount of iron present. Into each tube introduce 3 cc. 3 per cent hydrogen peroxide and mix. The maroon color is discharged, leaving the solution practically colorless in the case of the non-titanium steel. But if the test solution contains the most minute quantity of titanium, its presence is revealed by a residual lemon-yellow color. Even though the amount present is so extremely small that the usual manner of comparison leaves the operator uncertain of its presence, a glance through the solution from the top, holding the tube about $\frac{1}{2}$ inch above a white surface, instantly dispels all doubt. If there is as little as 0.001 per cent titanium present, the contrast between the pale green of the non-titanium solution and the yellowish tinge of the test clearly indicates that fact.

By adding the standard titanium solution to the solution of non-titanium steel the percentage of titanium is readily determined.

If the sample is *cast iron*, dissolve it in hydrochloric acid, filter and burn the residue in a platinum crucible. Volatilize silica with hydrofluoric acid and a few drops sulfuric acid, fuse residue with 1 gram sodium carbonate, dissolve out in hydrochloric acid, add to the main solution, and proceed with the ether separation. The insoluble residue can not be neglected when material is being examined for small amounts of titanium.

LIMA LOCOMOTIVE CORPORATION
LIMA, OHIO

VANADIUM IN STEEL BY THE HYDROGEN PEROXIDE COLOR METHOD

By C. R. McCABE

Received March 6, 1913

In 1911, the writer published the details of his original colorimetric method for vanadium in iron and steel¹—a method carefully devised to meet the difficulties occasioned by presence of titanium or molybdenum. Since these conditions are rarely encountered, a more direct and expeditious plan has been substituted for general use. It requires only twenty minutes when chromium is absent, and about double that time when it is present. Results are entirely satisfactory, as many experiments have shown.

The known rules governing all color methods are of unusual importance in the colorimetric vanadium determination. The large sample necessary renders the iron color deep enough to influence the vanadium color very materially. The determination is further limited by the sensitive character of the vanadium color with hydrogen peroxide, it being affected by the acidity of the solution. With the strength of acids commonly used in analytical work, the acidity may be satisfactorily controlled by means of the ordinary measuring apparatus. An excess of hydrogen peroxide partially bleaches the color after having pro-

¹ Cast iron samples should be passed through a 60-mesh sieve.

¹ *Chem. Eng.*, 13, 243.

duced it. Also it is only when vanadium is in the quinquivalent state that hydrogen peroxide imparts to its solution an immediate lasting color.

It is impossible to employ standard steels for the colorimetric vanadium determination with satisfactory results. For this reason the writer has devised a plan which consists in treating a non-vanadium steel precisely like the test, then imitating the color of the test by addition of a standard vanadium solution to the solution of non-vanadium steel, final comparison being made at equal volumes.

STANDARD VANADIUM SOLUTION

The vanadium pentoxide employed in making up the standard vanadium solution may be prepared in the laboratory, though it can now be obtained in the market.

Select a good grade of vanadate of iron containing about 60 per cent V_2O_5 , 38 per cent Fe_2O_3 , and 2 per cent SiO_2 ; put 5 grams of it into a beaker, add 100 cc. strong hydrochloric acid, and heat until dissolved. Add 20 cc. water and 1 cc. hydrofluoric acid. Filter from the slight insoluble residue, using a large filter paper and ribbed funnel. Pour into a separatory funnel, add 50 cc. ether, and shake under a hydrant. Allow the two layers to separate completely. Draw off the acid solution into a beaker, and discard the ethereal solution. Continue the extraction until the solution is free of iron as shown by the potassium sulfocyanide and ether test. Dilute to 400 cc., filter into an 800 cc. beaker and pass air through to remove the ether. Boil on a hot plate and add about 10 grams of potassium chlorate to oxidize the vanadium reduced by the hydrochloric acid. Then add in small quantities sufficient potassium carbonate to neutralize the greater part of the acid. When the proper degree of acidity is reached, a heavy precipitate of hydrated vanadic acid is thrown down. Filter through an 11 cm. filter paper, wash with hot water, and apply suction. Remove the cake, place on a watch glass and dry at $100^\circ C$. Several hours may be necessary to affect complete dehydration. Pulverize the vanadium pentoxide and preserve in a vial.

Place about 0.2 gram of the vanadium pentoxide in a small beaker. Add 30 cc. nitric acid (sp. gr. 1.2) and warm until dissolved. Dilute so that 1 cc. contains 0.0002 gram vanadium, or an amount corresponding to a 0.01 per cent when a two-gram sample is taken for analysis.

VANADIUM IN THE ABSENCE OF CHROMIUM

Place 2 grams of the vanadium steel and the same quantity of a non-vanadium steel in 300 cc. Erlenmeyer flasks. Add exactly 40 cc. nitric acid (sp. gr. 1.2) to each and heat until the steel is dissolved. To each add about 0.1 gram potassium permanganate and digest for two minutes. Then add dilute ammonium bisulfite in sufficient quantity to clarify the solution, and continue heating until SO_2 is all expelled: by this operation carbon is oxidized.

Cool the solutions in running water and transfer to comparing tubes. Bring to equal volumes if they are not already so, and mix. Observe the colors,

which should be of the same character and depth.

Into each tube introduce exactly 1 cc. 3 per cent hydrogen peroxide. Mix the test solution, and observe the vanadium color to gain an approximate idea of the vanadium content of the test.

To the solution of non-vanadium steel add, from a burette, as much of the standard vanadium solution as it is thought may be safely used without passing the amount existing in the test solution. To the test solution add an equal amount of water. Mix the solutions and compare colors. If the test solution appears darker, as will be the case if the proper precaution has been observed, add a further quantity of standard vanadium solution to the non-vanadium steel solution. Add an equal quantity of water to the test solution to maintain the equality of volume. Mix, and again compare. Continue in this manner until the colors match at equal volumes. The amount of standard vanadium solution used indicates the result. Thus, if 20.5 cc. standard vanadium solution are used, the result is 0.205 per cent vanadium.

VANADIUM IN THE PRESENCE OF CHROMIUM

When a vanadium steel contains chromium its color must be compensated to secure a satisfactory comparison. The uncertain color of chromium salts, which varies with the acidity of the solution and other causes not readily determined, renders worthless some apparently simple methods of compensation. The only plan which proved satisfactory, after many experiments, is as follows:

Weigh 2 grams of the chrome-vanadium steel and an equal amount of a plain steel, placing in 400 cc. beakers. Weigh with great care an amount of potassium bichromate corresponding to the chromium content of the test steel and place it in the beaker containing the plain steel. To each add 80 cc. sulfuric acid (1 : 3), and heat.

When nearly in solution, add to each 25 cc. of strong nitric acid and heat for ten minutes. Vanadium is not completely oxidized by a small amount of nitric acid such as would oxidize the iron.

Cool the two solutions in running water, introduce into comparing tubes, and bring to equal volumes. Mix, and observe the colors. Owing to possible error in the chromium determination it is doubly important to know that the solutions show colors of the same character and depth at equal volumes before proceeding with the analysis.

The determination is now finished as when chromium is absent.

For vanadium steels as they commonly occur, this method as described is entirely satisfactory. But when the determination is embarrassed by presence of titanium or molybdenum, or when the equally troublesome case of a steel containing a small amount of vanadium with much chromium presents itself, the more elaborate method mentioned in the first section of this paper may be employed. That method, though it requires more time, has been found to be thoroughly reliable under adverse conditions.

THE DETERMINATION OF ZINC IN TREATED TIES

By FRANCIS C. FRARY AND M. GORDON MASTIN

Received June 16, 1913

It was recently stated in THIS JOURNAL that over nine million ties were treated with zinc chloride in 1911. The railroads are said to be having excellent results with these in practice, and are buying large quantities of them, mostly on a specification of one-half pound of zinc chloride per cubic foot, which means about 0.5 per cent of metallic zinc.

The determination of this small amount of zinc in such material necessitates the use of a rather large sample and the complete destruction of the organic matter. The method commonly used is said to have originated in the laboratories of the Union Pacific Ry., and consists in charring the sample by heating with a small amount of concentrated sulfuric acid, and then adding nitric acid, a few drops at a time, continuing the heating until all organic matter is destroyed and nitrous fumes driven off. The solution is then diluted, iron removed, the zinc precipitated as sulfide and determined in the usual manner as oxide.

This method is said to give satisfactory results with ties except those of oak. Red oak ties in particular are impossible to analyze by this method, for the treatment with acids, no matter how prolonged, will not destroy all the organic matter, but leaves the solution dark colored, and the precipitation of the zinc is so incomplete that less than half of that present is found. In some determinations which we made by this method on the red oak shavings used in the work herein described, we found that the zinc sulfide was precipitated in such a finely divided form that it was not retained by two thicknesses of C. S. & S. white band No. 589 paper, one thickness of which will retain barium sulfate completely.

This difficulty was brought to our attention by Mr. H. M. Newton, of the Kettle River Co., to whom we are indebted for the details of the method of analysis given above and the method of Mr. Stenger, described later. We are also indebted to him and to the Kettle River Co. for furnishing us half of an untreated red oak tie and another half-tie which had been treated "to refusal" with a 4 per cent solution of zinc chloride. The standard method of sampling such material is by boring with a fine one-inch bit, at a point two feet from the end of the tie. To obtain a large quantity of material of about the fineness of such a sample, we sawed the half-tie in two, and reduced half of one of these pieces to shavings by means of a shaper in the university shop. Similar shavings were taken from the untreated half-tie for use with known amounts of zinc.

It is a well-known fact that oxalic acid is made by heating wood shavings or sawdust with caustic alkali, and that care must be taken in the process not to allow the temperature to reach too high a point, or the reaction will be too violent and the acid will be destroyed. It was believed that it would be possible to completely destroy the organic matter in red oak shavings in this way, and it was found that such destruction was both easy and rapid if the process was

properly conducted and a little potassium nitrate added toward the last. The fusion can not be made satisfactorily in an iron dish, as some ferrocyanide is usually formed from the iron of the dish during the fusion, and so much iron goes into solution that its separation becomes tedious. By using a nickel dish, very little of the metal is dissolved, and that can be easily and quickly removed.

The process which has given us the best results is as follows: Two grams of the dried shavings are weighed into a 200-300 cc. nickel dish, moistened with a little water, and heated gently to insure thorough saturation with the water. Two sticks (about 30 grams) of pure potassium hydroxide are now added, and the mixture heated cautiously until the hydroxide is melted. The mass must now be stirred thoroughly with a pure nickel wire, and the heat increased so that it boils vigorously. As the water is boiled off, the fusion becomes almost a salmon-pink (with red oak) and lumps of a gummy substance appear on the surface. As the boiling is continued, these masses disappear, seeming to dissolve, and the color changes to a yellowish brown. The fusion now begins to foam a good deal, and must be continually stirred. When spurts of inflammable gases appear, and the mass is rather viscous, the addition of the nitrate is begun. A piece of potassium nitrate the size of a grain of wheat is added, and the heating and stirring continued about a minute, repeating the addition, heating and stirring as often as may be necessary. The fusion becomes more fluid and lighter in color as the nitrate is added. Usually about half a gram of nitrate will be required.

When all organic matter is oxidized, the fusion becomes perfectly transparent, colorless, and quiet; the dish is then inclined so as to wash down the sides with the fused mass and get any material that might be lodged there and escape oxidation. After cooling, the mass is dissolved in water, 15 to 20 cc. of bromine water added, and the whole boiled about a minute. The nickelic oxide thus precipitated is filtered off on a Gooch crucible, and the filtrate and washings transferred to a 750 cc. casserole and made barely acid with concentrated hydrochloric acid. Sodium sulfite is added, a little at a time, until the first violent effervescence ceases, and then enough hydrochloric acid added to make the amount of free concentrated acid from 2 to 6 per cent of the final volume. Dilute to about 400 cc., heat to boiling, and titrate with potassium ferrocyanide, using a saturated solution of uranium nitrate on a spot plate as an indicator. The conditions for the titration are the same as for any such determination of zinc; the necessary blank must be determined and subtracted from the burette reading.

For use with a sample of the size mentioned, we prefer a solution of about 5 grams potassium ferrocyanide per liter, which is equivalent to about 1 mg. of zinc per cc. The blank with such a solution was found to be about 4 cc., the end point being very satisfactory.

Enough sulfite must be used to reduce all oxidizing compounds present, to prevent them from decom-

posing the ferrocyanide, and care should be taken to keep the volume of the solution and its acidity about as above stated. With practice the fusion can be made in about ten minutes and the whole determination in half an hour. We prefer to standardize our solutions with zinc oxide, made by evaporating a little of a solution of c. p. zinc sulfate in a weighed platinum dish, blasting a few minutes to remove all sulfur compounds, cooling and weighing. The same volume should be used in the standardization as in the regular determination, and about 10 grams of ammonium chloride or 40 grams of potassium chloride added to make the conditions the same, for it is well known that variations in the conditions change the value of the ferrocyanide solution.

Using this method, on the dried shavings from the treated tie, we obtained the following results in eight consecutive determinations: 0.881, 0.901, 0.891, 0.905, 0.896, 0.886, 0.901, 0.891 per cent metallic zinc. Using the untreated shavings, and adding to each determination a solution containing 0.0255 gram of zinc as chloride, the following amounts of zinc were found: 0.0253, 0.0251, 0.0250, 0.0252, 0.0253 gram. There is apparently a slight loss of zinc, probably due to spattering during fusion, being only about 0.02 per cent by weight of the original sample of shavings.

It is not essential to the success of this method that the zinc be determined by titration; after removing the nickel and the bromine it may, of course, be determined by any other method if desired.

So far as we were able to determine at the time this work was taken up, the only other satisfactory method for this determination was an electrolytic one devised by Mr. L. A. Stenger, of the Twin City Rapid Transit Co., of this city, and used in their laboratory. Mr. Stenger describes his method as follows:

"Five to 10 grams of borings or sawdust are taken, about 150 cc. pure water added, then 6 to 8 grams of sodium hydroxide. The mixture is heated nearly to boiling for a few minutes, then electrolyzed with a rotating platinum anode and silver-plated copper cathode for about forty minutes. The cathode area is about 250 sq. cm., and a current of about 5 amperes at six volts is employed. A tall 400 cc. beaker is used to allow some space for frothing, which can be prevented by addition of ether if desired. The cathode with the zinc deposit is removed quickly, washed with hot water and alcohol and dried. After weighing, it is replaced in the solution and the electrolysis continued for five to ten minutes. If little or no increase in weight is found, deposition is considered complete."

Mr. Stenger very kindly determined zinc in a sample of our dried shavings by the above method, and found 0.88 per cent, which is in good agreement with our results. He also tried our method as above outlined on the same sample, and on tamarack and red oak ties, one of which had been treated with both creosote and zinc chloride, and states that the method worked very well.

While we were working on this method, it was found in the laboratory of the Kettle River Co. that complete

oxidation of the wood could be obtained, even with red oak, by using potassium chlorate with the nitric and sulfuric acids in the ordinary method. Mr. Newton very kindly had their laboratory make two determinations on our sample by this method, the results being obtained 0.91 and 0.92 per cent zinc. It is evident that the agreement is as close as could be expected.

We believe that this method of fusion with alkali is adapted for the determination of minute amounts of metals in the presence of large amounts of organic matter of any sort, and have tried it on the determination of tin in canned goods, with success. A larger sample can, of course, be used if a larger nickel dish is at hand.

UNIVERSITY OF MINNESOTA
MINNEAPOLIS

HICKORY NUTS AND HICKORY NUT OIL

By GEORGE O. PETERSON AND E. H. S. BAILEY

Received June 26, 1913

Since the hickory nut crop of the United States is a large one, although no accurate statistics exist in regard to it, it has seemed to us worth while to make a preliminary study of the properties of the oil. The genus *carya* of the walnut or *juglandaceae* family comprises eight species, only two of which, the *carya ovata* or shellbark hickory and the *carya amara* or swamp hickory are here considered. The shellbark nuts are of such commercial value that their oil could hardly be extracted with profit if intended for ordinary table use. The swamp hickory nuts and those called "pig nuts" are abundant and at present not utilized, except as food for hogs and squirrels.

Work on the composition of nuts has been carried on especially in the Iowa,¹ California² and Maine³ Agricultural Experiment Stations, and from two of these the analyses of hickory nuts (presumably *C. ovata*) are presented for comparison with our own analysis.

TABLE I—CHEMICAL COMPOSITION OF HICKORY NUTS

	Maine Sta.	Iowa Sta. meats	P. and B. meats
Refuse.....	62.2(a)	68.0	62.8
Edible portion.....	37.8	30.0	37.2
Water.....	1.4	3.97	3.45
Protein.....	5.8	20.5	13.2
Fat.....	25.5	64.6	70.2
Carbohydrates.....	4.3
Ash.....	0.8	2.2	1.73
Fuel value per pound in calories.....	1265	3226 (calc.)	3310 (calc.)
Crude fiber.....	..	2.3	2.0
N-free extract.....	..	6.40	9.42

(a) Per cents in this column refer to whole nuts.

The methods of analysis used were in general those of the "Official and Provisional Methods of Analysis," *Bull. 107*, Bur. of Chem., U. S. Dept. of Agr., with such modifications as seemed expedient. All desiccations were carried out in air instead of hydrogen.

HICKORY NUT OIL

In extracting the oil from the meats it was decided to use the pressure method rather than the extraction

¹ "The Chemical Composition of Nuts Used as Food," *Proc. Ia. Acad. Sci.*, 10, 108-11.

² "Nuts and Their Use as Food," *Farmers' Bull. No. 332*.

³ *Maine Agr. Expt. Sta.*, 1899, 87.

method, as the former is the commercial method, and the only one used for extracting edible oils. For extracting the oil a cylindrical press was constructed by using an iron cylinder with an inside diameter of $3\frac{1}{4}$ inches and a height of 7 inches into which was fitted a close-fitting piston. The cylinder was set on an iron plate perforated with several $\frac{1}{8}$ inch holes, and holes were drilled in the cylinder near the lower end to facilitate the running out of the oil. When hydraulic pressure was applied to the piston the oil exuded from the holes and collected in a metal pan in which the press had been placed. A pressure of 150 to 300 atmospheres was applied to the piston. A second pressure was applied to the material after breaking up the press cake and heating the press and the press cake to $50-60^{\circ}\text{C}$. The following table shows the yield of oil from the various samples, the total per cent of oil present, and the actual per cent of oil extracted:

TABLE II

Kind of sample	Yield in per cent	Total per cent of oil present	Per cent of oil present extracted
<i>C. Ovata</i> —Whole, cracked, cold-pressed.....	15.8	26.5	59.5
Hot-pressed.....	2.0	..	7.5
<i>C. Ovata</i> —Meats alone cold-pressed.....	30.6(a)	70.2	42.7
Hot-pressed.....	17.5	..	23.2
<i>C. Amara</i> —Whole, cracked, cold-pressed.....	22.1
Hot-pressed.....	3.8

(a) Only 100 atmospheres pressure.

The oils, as at first obtained, were cloudy, and the hot-pressed were darker than the cold-pressed. After filtration through filter paper the oils became bright and clear, and were of the appearance and consistency of Italian olive oil. They possessed an agreeable hickory nut odor and flavor.

The following table gives the constants as obtained from both these oils and for comparison those obtained at the Maine Experiment Station.¹

TABLE III—CONSTANTS OF HICKORY NUT OILS

	<i>C. gmara</i>	<i>C. ovata</i>	Maine Expt. Sta.
Specific gravity at 24°C	0.9119	0.9119	0.9164
Refractive index at 20°C	1.4699	1.4699	1.4696
Hehner number.....	95.6	95.7
Reichert-Meißl number.....	0.48	0.47
Iodine value.....	105.2	106.8	102.8
Saponification value.....	190.0	189.6

The similarity of the two oils is very evident. Perhaps the most important factor for comparison of the different classes of oils is the iodine value, for this is a measure of the unsaturated glycerides present, and thus determines whether the oil is drying, semi-drying or non-drying. The iodine value here obtained for hickory nut oil seems to classify it with the semi-drying oils. Here belong cottonseed oil, corn oil, rape seed oil, Brazil nut oil, and beech nut oil. In the following table is shown a comparison of constants of hickory nut oil with those of cottonseed oil and those of the non-drying oil, viz., olive oil.

These figures show that hickory nut oil resembles very closely cottonseed oil, and would be difficult to distinguish from it.

The hickory nut oil is very agreeable as a salad oil,

¹ *Loc. cit*

TABLE IV

	Hickory nut oil	Cottonseed oil	Olive oil
Specific gravity.....	0.9119 at 24°C .	0.9203 at 19°C .	0.9100 at 24°C .
Refractive index.....	1.4699 at 20°C .	1.4748 at 15°C .	1.4670 at 20°C .
Iodine value.....	106.0	106.0	82.0
Saponification value.....	190.0	192.0	190.0
Reichert-Meißl number.....	0.48	0.6
Hehner number.....	95.7	95.6	95.4

so that if it can be obtained at a sufficiently low cost, it can be used for this purpose. The *C. amara* or the *C. porcine* (pig nut), which are now considered as absolutely of no value, would yield at least a gallon and a half of oil per bushel and the nuts need only be crushed before they are pressed.

SUMMARY

- I. The food value of hickory nuts is high.
- II. The oils from the two species of hickory nuts, *C. ovata* and *C. amara*, are practically identical and are similar to cottonseed oil.
- III. The oil retains the flavor of the hickory nut, and is practically equal to olive oil.
- IV. The possibility of extracting the oil on a commercial basis should be further investigated.

CHEMICAL LABORATORIES
UNIVERSITY OF KANSAS
LAWRENCE

THE ANALYSIS OF MAPLE PRODUCTS I

AN ELECTRICAL CONDUCTIVITY TEST FOR PURITY OF MAPLE SYRUP¹

By J. F. SNELL

The most commonly used adulterant of maple sugar is granulated sugar, which is, of course, chemically identical with the predominating constituent of the pure material. For the detection of such adulteration we are dependent upon measurements of the small quantities of the non-sugar constituents, the percentages of which in the syrup are necessarily decreased by the addition of the practically pure sucrose, together with the proportional quantity of water necessary to convert it into a syrup. The ash, the soluble and insoluble ash, the alkalinities of these, the malic acid value, and the amounts of precipitate produced by lead subacetate and by normal lead acetate are alike lowered by such adulteration, though not necessarily in proportion to the amount of adulteration.²

Sucrose being a non-conductor of electricity, and the salt constituents conductors, it is reasonable to

¹ This test was described at the Washington meeting of the American Chemical Society in December, 1911, but publication was deferred until further experiments could be made, particularly (1) upon the effect of adulteration on the conductivity value and (2) upon the relation between the conductivity of the syrup and that of its ash solution. This later experimental work has been carried out under my direction by Mr. J. M. Scott, to whom my thanks are due for very competent assistance. This assistance was rendered possible by the Dominion Government Grant for the Advancement of Agriculture. Through the courtesy of Prof. Frank T. Shutt, M.A., F.R.S.C., the present paper was read before the Royal Society of Canada at Ottawa, May 28, 1913.

² The effect of such adulteration upon the various analytical values will be discussed in a later paper.

anticipate that maple syrups adulterated with granulated sugar in more than very small quantities will show materially lower conductivities than pure maple syrups. Since with suitable apparatus a measurement of electrical conductivity can be made in a few seconds, a method based upon such a measurement would have a decided advantage in point of rapidity over any of the methods now in vogue.¹

The most rapid methods hitherto proposed have depended upon precipitation with lead subacetate and measurement of the volume of the centrifugally-settled precipitate.² No great delicacy has been claimed for these methods, and even they require more time than a conductivity measurement.

THE CONDUCTIVITIES OF UNDILUTED PURE SYRUP

Measurements of the conductivities of the undiluted syrup have been made upon 43 samples, all of Canadian origin. Eleven pairs of early and late products from the same or adjoining woods were sent me by the makers from various parts of the Province of Quebec in the season of 1911. Thirteen samples from Ontario and six from Quebec, all of the season of 1911, were kindly forwarded by Dr. Anthony McGill, Chief Analyst of the Inland Revenue Department, Ottawa, as fairly representative of the 450 odd samples collected by the Department for the purpose of establishing a standard of purity for maple syrup. Each of these was accompanied by a declaration of genuineness signed by the maker.³

The remaining two samples of the 43 were made from identical sap, one by rapid boiling in a modern evaporator having a corrugated pan, the other in an iron kettle, not entirely free from rust, with very slow boiling.

TABLE I—SUMMARY OF DENSITIES AND CONDUCTIVITIES OF 42 GENUINE CANADIAN MAPLE SYRUPS

	Conductivity	
	Sp. gr. 15° C.	$\kappa \times 10^5$ 25° C.
Mean.....	1.333	18.7
Minimum.....	1.305	9.6
Maximum.....	1.355	33.6
Percentage deviation of minimum from mean...	-2.1	-49
Percentage deviation of maximum from mean...	+1.6	+80

A summary of the variations of the specific gravity and of the electrical conductivity of 42 of these 43 syrups is given in Table I. The syrup omitted from this summary is one of the Inland Revenue Department samples, which was of extremely low density. Its specific gravity was 1.279, its moisture content (according to the analysis made in the Inland Revenue

¹ As long ago as 1889, E. Reichert, *Zeit. anal. Chem.*, **28**, 14 (1899), and more recently, Hugh Main, *International Sugar Journal*, **11**, 334 (1909); *Zeit. Ver. Zuckerind.*, **59**, 783 (1909); *Chem. Abstr.*, **3**, 2249 (1909) and A. E. Lange, *Z. Ver. Zuckerind.*, **60**, 359; *Chem. Abstr.*, **4**, 1554 (1910) have proposed methods of estimating the ash of sugars and syrups from their electrical conductivities. These methods appear to have given good results in the hands of their authors but have been adversely criticized by others—Reichert's, by Fock and Plath, *Zeit. Ver. Zuckerind.*, **39**, 710 (1889); Main's and Lange's, by A. Trenkler, *Oesterr. und Zeit. Zuckerind.*, **39**, 437; *Chem. Abstr.*, **4**, 308 (1910).

² Hortvet, *J. Am. Chem. Soc.*, **26**, 1543 (1904); Bureau of Chem., U. S. Dept. Agr., *Bulletin* **107**, 72; Jones, Vermont Agr. Expt. Sta., *18th Annual Report*, **1906**, 322.

³ See *Bulletin* **228** of the Laboratory of the Inland Revenue Department (1911). The syrups examined were Nos. 184-191, 193-195, 211-214, 263, 278, 281 and 284 of this bulletin.

Laboratory) 38.59 per cent, and its conductivity ($\kappa \times 10^5$) 45.0.

As a general rule, the syrups of high density showed low conductivities and those of low density showed high conductivities. This indicates that in undiluted syrups the concentration of the sugar has more influence upon the conductivity than has the concentration of the electrolyte components.

The syrup made in the iron kettle had a specific gravity of 1.326, and a conductivity of 25.9, while that made from the same sap by rapid, shallow boiling in an evaporator showed a specific gravity of 1.334, and a conductivity of 22.4. This is in harmony with the general rule of lower conductivity accompanying greater density. The minimum conductivity (9.6) was shown by the syrup of maximum density (1.355), while the maximum conductivity (33.6) was found in a syrup of density 1.311.

THE EFFECT OF DILUTION

The results of the above measurements indicate that as the water-content of maple syrup increases, the conductivity increases. In other words, the more dilute the solution the greater the conductivity. This is not in line with the usual behavior of aqueous solutions of electrolytes. In general, the specific conductivity of such solutions decreases with dilution. The thought, therefore, suggested itself that if one continued to dilute maple syrup with water, a point of dilution must eventually be reached at which the conductivity would cease to increase and begin to decrease—a point of maximum conductivity. This proved to be the case, the maximum occurring at a dilution of one volume of syrup to two of water, *i. e.*, in a mixture containing 33¹/₃ per cent of syrup by volume, or 39.6 per cent of syrup of normal density (1.320) by weight.

TABLE II—EFFECT OF DILUTION UPON CONDUCTIVITY OF SYRUP

Syrup I		
Per cent maple syrup by volume	Per cent maple syrup by weight	Conductivity value
5	6.6	62
10	13.0	100
20	25.1	147
25	30.9	163
30	36.5	171
33 ¹ / ₃	40.1	175
40	47.2	169
50	57.3	153
60	66.8	123
70	75.8	99
80	84.3	63
90	92.3	44
100	100.0	32

Syrup II	
Per cent maple syrup by weight	Conductivity value
20	127
30	141
39.9	148
40	147
60	127
83 ¹ / ₃	69
100	26

Table II and Diagrams I and II give results typical of the effect of dilution upon the specific conductivities of maple syrups.

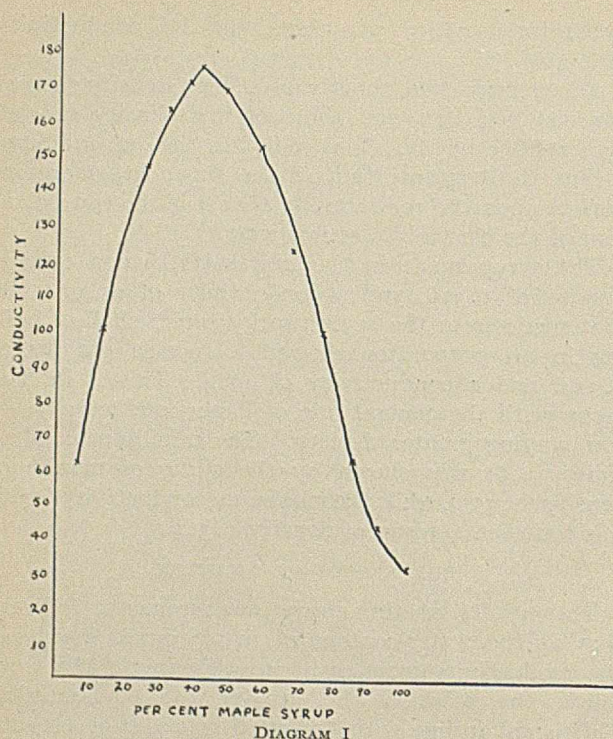


DIAGRAM I

On each side of the point of maximum conductivity there is a considerable range of concentrations, within which the conductivity differs very little from the maximum. This renders it possible to measure the maximum conductivity of diluted maple syrup without observing much precision in making up the mixture of maple syrup and water. This is a point of great practical advantage in relation to the rapidity of the method described below. It is not necessary to weigh the syrup nor to reduce it to a definite density.

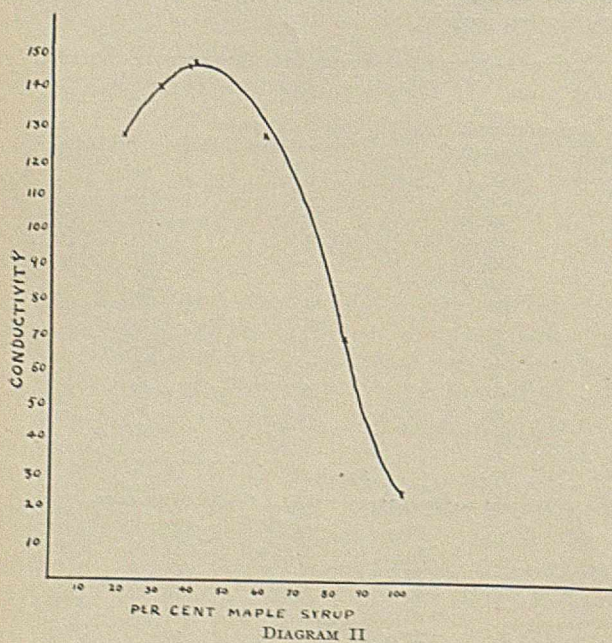


DIAGRAM II

An ordinary 25 cc. graduate can be used to measure the syrup, the two portions of water subsequently measured from the same graduate serving to rinse out the syrup which adheres to the sides.

The range of variation of the conductivity of pure maple syrups thus diluted with two volumes of water is much narrower than that of the conductivity of the undiluted syrups. The mean conductivity at 25° C. ($\kappa \times 10^5$) of the diluted solutions of the 42 syrups of Table I was 153, or over 8 times the mean conductivity of the same syrups in the undiluted state. The minimum was 120, the maximum 203, the latter being the conductivity value of the syrup made in the iron kettle. The minimum deviates from the mean by 21 per cent, as against 49 per cent in the undiluted syrup; the maximum by 33 per cent, as against 80 per cent for the undiluted syrup. We have thus a total range of 54 per cent for the diluted as against 129 per cent for the undiluted syrup. The forty-third syrup, omitted from the summary in Table I, on account of its extremely low density and high conductivity, gave a perfectly normal conductivity value (167) when diluted with two volumes of water.

The maximum conductivity, or more strictly speaking, the conductivity of the mixture of one volume of syrup with two volumes of water is, accordingly, made the basis of the method here proposed.

METHOD

Measure out into a small beaker (or directly into the conductivity cell) a suitable quantity (15 cc.) of the syrup, allowing thorough draining. Using the same graduate, add two successive portions of water, each equal in volume to the syrup taken. Mix thoroughly, pour into conductivity cell, bring to 25° C., and make the measurement. Divide the constant of the cell by the observed number of ohms and multiply the result by 100,000.

Genuine syrups have given values of 110 to 200, but further experience may extend these limits a little.¹

Syrups giving conductivity values distinctly outside these limits may be condemned. Those giving normal values are not necessarily pure and should be further examined by well-established tests.

APPARATUS

The essential features of the apparatus are:

1. A low voltage electrical current operating an induction coil.
2. A conductivity cell of a form suitable for liquids of low conductivity, and with electrodes not easily displaced.
3. A Wheatstone bridge with telephone.
4. A device for exact regulation of temperature.

In the present work a Kohlrausch Universal Bridge bearing the name plate of Messrs. Philip Harris & Co., Birmingham, England,² has been used. The induction coil with which this bridge is fitted was operated by two lead accumulators connected in series. A small Deveau telephone was found to give better results than a Bell. With this very convenient bridge and telephone there was no difficulty in obtaining a sharp minimum of sound. The conductivity cell used at first was of the form represented in Fig. 295 (p. 402)

¹ I have collected nearly 130 syrups of the season of 1913, direct from the sugar bushes of Ontario and Quebec and intend determining the limits of conductivity value in these both at 25° and at 20° C.

² No. 7072 in this firm's Physics catalogue, Vol. 1.

of Ostwald and Luther's "Physiko-chemiker Messungen," 2nd edition (1902). Its capacity was about 50 cc. It had electrodes of 2.5 cm. diameter set 1 cm. apart, which maintained their position satisfactorily,

Eimer & Amend, of New York. The platinum electrodes of this special cell are of B. & S. gauge No. 27, and are 3.2 cm. in diameter. They are adjustable as to distance, but are firmly held in position by set-

TABLE III—SYRUPS MADE IN LABORATORY SEASON OF 1912

No.	Date made	Saps	Density 15° C.	Dry	A	B	C	D	E	F	G	H
				matter from density	Conductivity value	Conduc-tivity value, ash	Total ash Dry basis	Sol- ible ash Dry basis	Insol- ible ash Dry basis	Alkalinity Total ash	Alkalinity Sol- ible ash	Alkalinity Insol- ible ash
1	Apr. 6	Hard and soft	1.329	66.5	150	..	0.95	0.44	0.51	1.18	0.45	0.73
2	Apr. 7	Hard and soft	1.329	66.5	164	61	0.99	0.66	0.33	1.42	0.50	0.92
3	Apr. 10	Hard and soft	1.326	66.0	144	57	0.88	0.38	0.50	1.46	0.46	1.00
4	Apr. 11	Hard and soft	1.327	66.2	153	56	0.95	0.57	0.38	1.42	0.46	0.86
5	Apr. 12	Hard and soft, evaporated on steam bath	1.333	67.1	145	61	1.00	0.58	0.42	1.30	0.47	0.83
6	Apr. 12	Hard and soft, evaporated over flame	1.320	65.0	135	58	0.88	0.59	0.29	1.19	0.46	0.73
7	Apr. 13	Hard and soft	1.325	65.8	138	50	0.85	0.35	0.50	1.30	0.48	0.82
8	Apr. 15	Hard and soft	1.325	65.8	152	68	0.91	0.52	0.39	1.11	0.45	0.66
9	Apr. 18	Hard and soft	1.327	66.2	135	50	0.88	1.41	0.43	0.98
10	Apr. 23	Hard and soft	1.320	65.0	119	48	0.95	0.43	0.52	1.18	0.36	0.82
11	Apr. 25	Hard and soft	1.329	66.5	126	54	0.98	0.50	0.48	1.31	0.47	0.84
12	Apr. 29	Hard and soft	1.327	66.2	155	58	1.09	0.46	0.63	1.61	0.49	1.12
Average results for mixed saps.....				143	56	0.94	0.50	0.45	1.32	0.46	0.86
Percentage deviation maximum from mean.....				14.7	21.4	15.9	32.0	40.0	10.6	8.7	30.2
Percentage deviation minimum from mean.....				16.8	14.3	6.4	30.0	35.6	15.9	21.7	23.3
13	Apr. 15	Hard	1.322	65.4	145	48	0.87	0.41	0.46	1.17	0.41	0.76
14	Apr. 16	Hard	1.327	66.2	152	57	1.00	0.49	0.51	1.29	0.46	0.83
15	Apr. 20	Hard, evaporated over flame	1.322	65.4	146	54	1.02	0.47	0.55	1.39	0.50	0.89
16	Apr. 20	Hard, caramelized in boiling	1.319	64.9	148	50	0.97	0.60	0.37	1.28	0.45	0.83
17	Apr. 22	Hard	1.330	66.6	148	..	1.05	0.48	0.57	1.55	0.52	1.03
18	Apr. 24	Hard	1.325	65.8	141	57	0.96	0.45	0.51	1.45	0.47	0.98
19	Apr. 24	Hard	1.325	65.8	137	52	0.94	0.38	0.56	1.23	0.42	0.81
Average results for hard sap syrups.....				145	53	0.97	0.47	0.50	1.34	0.46	0.88
Percentage deviation maximum from mean.....				4.8	7.0	8.2	27.7	14.0	15.7	13.0	17.0
Percentage deviation minimum from mean.....				5.5	9.4	10.3	19.1	26.0	12.7	10.9	13.6
20	Apr. 16	Soft	1.321	65.2	134	54	0.90	0.47	0.43	1.18	0.40	0.78
21	Apr. 20	Soft	1.322	65.4	135	58	0.81	0.42	0.39	1.10	0.44	0.66
22	Apr. 22	Soft	1.330	66.6	148	..	0.92	0.54	0.38	1.27	0.55	0.72
Average results for soft sap syrups.....				139	56	0.88	0.48	0.40	1.18	0.46	0.72
Percentage deviation maximum from mean.....				6.5	3.6	4.4	12.5	7.5	7.5	19.6	8.3
Percentage deviation minimum from mean.....				3.6	3.6	8.0	12.5	5.0	6.8	13.0	8.3
General average.....				143	55	0.94	0.49	0.46	1.31	0.46	0.85
Maximum.....				164	68	1.09	0.66	0.63	1.61	0.55	1.12
Minimum.....				119	48	0.81	0.35	0.29	1.10	0.36	0.66
Percentage deviation maximum from mean.....				14.6	23.6	16.0	30.6	37.0	22.9	19.6	31.8
Percentage deviation minimum from mean.....				16.8	12.7	13.8	28.6	37.0	16.0	21.7	22.4

TABLE IV—FULL ANALYTICAL DATA FOR TWELVE SYRUPS OF TABLE I

Number	Run	A	C	D	E	F	G	H	Canadian Lead No.	Modified Winton Lead No. (a)
		Conductivity value	Total ash Dry basis	Soluble ash Dry basis	Insoluble ash Dry basis	Alkalinity Total ash	Alkalinity Soluble ash	Alkalinity Insoluble ash		
1	Early	133	0.80	0.46	0.34	1.04	0.46	0.58	3.36	1.74
2	Early	149	0.82	0.59	0.23	1.01	0.58	0.43	3.10	2.11
3	Early	152	0.89	0.61	0.28	1.05	0.53	0.52	2.95	2.04
4	Early	139	0.86	0.61	0.25	0.93	0.48	0.45	2.82	1.85
5	Early	147	0.78	0.50	0.28	0.89	0.45	0.44	2.93	1.96
6	Early	145	0.89	0.58	0.31	1.08	0.56	0.52	3.30	2.18
7	Early	139	1.09	0.61	0.48	1.03	0.51	0.52	2.64	1.56
8	Early	162	1.14	0.79	0.35	1.20	0.64	0.56	3.39	2.33
9	Late	187	1.38	0.93	0.45	1.63	0.91	0.72	3.29	2.04
10	Late	149	0.86	0.57	0.29	1.12	0.56	0.56	3.44	2.40
11	Late	138	0.89	0.60	0.29	1.01	0.50	0.51	2.70	2.10
12	Late	158	1.14	0.62	0.52	1.14	0.54	0.60	3.62	1.88
Average.....		150	0.96	0.62	0.34	1.10	0.56	0.53	3.13	2.02
Per cent deviation of maximum from mean.....		24.7	43.8	50.0	52.9	48.2	62.5	35.8	15.6	18.8
Per cent deviation of minimum from mean.....		11.3	18.8	25.8	32.4	19.1	17.9	18.9	15.6	22.8

(a) Determined upon 25 g. dry matter instead of upon 25 g. syrup. Bull. 228, Laboratory of the Canadian Inland Revenue Department.

as was demonstrated by repeated determinations of the cell constant. Later, a special cell of the same type with extra heavy electrodes and with a thermometer set in the cover was made for me by Messrs.

screws. The thermometer range is 20° to 30°, graduated in tenths. This type of cell has proved perfectly satisfactory for the purpose.

In routine work it might be advisable to have a

considerable number of cells fitting the same pair of electrodes. These could be filled with the different samples and set in a thermostat to come to the required temperature, when measurements of all could be made in rapid succession. I have not used a thermostat in this work, but have regulated the temperature by placing the conductivity cell in a beaker of water of a suitable temperature, a fraction of a degree above or below that desired, and stirring the contents of the cell with the electrodes until the thermometer within the cell showed the correct reading.

The cell constant was determined by use of 0.1, 0.02 and 0.01 M potassium chloride solution, made up at 18° C. from the ignited C. P. salt, assuming for these¹ at 25° C. the conductivities 1289, 276.8 and 141.2 × 10⁻⁵.

RANGE OF VARIATION OF THE CONDUCTIVITY VALUE IN GENUINE SYRUPS

In addition to the 43 syrups referred to above, the conductivity value has been determined on 22 syrups made in this laboratory in the season of 1912 (see Table III). These syrups were all made from the sap of ten trees in the woods of the Macdonald College farm—six hard and four soft maples. They represent the runs of different days from April 6th to April 29th; some were made from the sap of the soft maples alone, others from the hard maples only, though the majority represent the mixed sap of the ten trees. They were boiled down in basins on gas stoves with the exception of one (No. 5), which was evaporated on the steam bath. These 22 syrups show a range of 119 to 164, average 143. This is a total range of 31.4 per cent. This is about the same range of variation as is shown by the total ash, and a much lower range than those shown by the soluble ash, the insoluble ash and the alkalinities.

Table IV includes analytical data for twelve of the Quebec syrups included in the summary given in Table I. These were examined to determine whether this apparent advantage of the conductivity method held for syrups of various origin, as well as for those from a single bush. It will be seen that among these 12 syrups the range of the conductivity value is much narrower than that of the total ash and narrower also than that of any of the other data except the Canadian lead number.

If this narrowness of range shall be found to be characteristic of the conductivity values of pure maple syrups in general, it will be a point of advantage for this method.

I have also examined three syrups made at the Vermont Agricultural Experiment Station under the supervision of Prof. C. H. Jones, who advised me that they were prepared with great care, "every opportunity being given for the malate of lime to settle out." These syrups were exceptionally light in color. As received, they were of exceptionally high density, as indicated by Prof. Jones' refractometer readings, *viz.*, No. 1 at 24.75° C., 1.4632; No. 2 at 21.5° C., 1.4651; No. 3 at 24.75° C., 1.4623. For my own analyses they were diluted with water, boiled to a boiling point of

219–220° F., and filtered through double S. & S. No. 597 filters.¹ Their densities at 15° C. were then 1.325, 1.321 and 1.332, respectively. Two of these Vermont syrups gave lower conductivity values than any of the Canadian syrups, *viz.*, 110 and 115. The third gave a value of 122. The ash values of these three syrups were also exceptionally low. Complete data for these syrups, as obtained by Professor Jones and myself, are given in Table V.

TABLE V—ANALYSES OF THREE VERMONT SYRUPS BY C. H. JONES AND BY J. F. SNELL

No.	Analyst	Per cent		Alkalinity	Alkalinity	Conductivity	Malic acid
		Total ash	Insoluble ash				
1	Jones	0.70	0.37	0.35	0.45	...	0.44
	Snell	0.71	0.59	0.37	0.44	115	..
2	Jones	0.68	0.33	0.39	0.44	...	0.45
	Snell	0.83	0.32	0.42	0.50	122	..
3	Jones	0.64	0.25	0.36	0.35	...	0.39
	Snell	0.70	0.23	0.37	0.38	110	..

The minimum value yet found in a genuine syrup is 110. The maximum of 203 obtained with the syrup prepared in the iron kettle has not been reached in any other pure syrup. The highest value yet found in a syrup actually prepared for the market is 197. The limits of variation of genuine syrups may, therefore, be tentatively set at 110 to 200.

The mean conductivity of the 68 syrups may most fairly be estimated by giving the mean of the 22 syrups made from the trees on the Macdonald College farm a weight of 2—the same as one pair of early and late run syrups. We thus obtain for the 48 syrups—46 actual and 2 theoretical—a mean conductivity value of 150. This is approximately equal to the electrical conductivity of an 0.01 M aqueous solution of potassium chloride (141.2).

THE RELATION OF THE CONDUCTIVITY VALUE TO THE ASH DATA

Since the salt components of maple syrup are undoubtedly responsible for the electrical conductivity, relations between the conductivity value and the ash data are worthy of investigation. From the investigations of Hortvet,² Jones³ and Bryan⁴ it is clear that maple syrup ash consists largely (*viz.*, to the extent of about 85 per cent) of the carbonates of calcium and potassium, the two metals being present in about chemically equivalent quantities. The three investigators are in close agreement as regards the lime content which is in the neighborhood of 22 per cent. Bryan finds an average of 38 per cent of potash in 100 syrups, Jones one of nearly 35 per cent in 6 syrups, two of which were composites of a number of samples, while Hortvet finds about 31 per cent in each of two syrup ashes. Hortvet alone has determined carbonic acid. His results show an average of about 30 per cent in the two samples. The proportions of these three constituents of the ash may accordingly be placed at the following figures as a rough estimate:

¹ Jones, Vt. Agr. Expt. Sta., 18th Annual Report, 1905, 328; also Bulletin 167, 470 (1912).

² J. Am. Chem. Soc., 26, 1541 (1904).

³ Vt. Agr. Expt. Sta., 18th Annual Report, 1905, 331.

⁴ Bur. of Chem., U. S. Dept. Agr., Bulletin 134 (1910).

¹ Ostwald-Luther, *Physico-chemische Messungen*, 2nd edition, p. 407.

	Per cent Molecules	
K ₂ O.....	35	0.37
CaO.....	22	0.39
CO ₂	30	0.68
Sum.....	87	—

} sum, 0.76

These results indicate that organic salts of potassium and calcium are the chief electrolytes of the syrup.¹ Potassium, having a higher ionic conductivity than calcium, may be expected to contribute a little more than an equal share to the conducting power of the

Considered by themselves, the ratios of Table VI would indicate that the closer relationships are those between, on the one hand, conductivity and *weight* of the *total* ash and on the other hand conductivity and *alkalinity* of *soluble* ash. It is to be remembered, however, that these syrups were all from the one bush, and were manufactured on a small scale in the laboratory. As will be pointed out later, they are also peculiar in their ratios of the alkalinities of soluble and insoluble ash. Table VII, which refers to more representative

TABLE VI—RATIOS OF THE ANALYTICAL DATA FOR 22 SYRUPS MADE IN LABORATORY (SEE TABLE III)

Number	A/B	A/C	A/D	A/F	A/G	A/H	B/D	B/G	E/G	G/H	
	Conduc- tivity syrup + Conduc- tivity ash	Conduc- tivity syrup + Total ash	Conduc- tivity syrup + Soluble ash	Conduc- tivity syrup + Alkalinity total ash	Conduc- tivity syrup + Alkalinity sol. ash	Conduc- tivity syrup + Alkalinity insol. ash	Conduc- tivity ash + Soluble ash	Conduc- tivity ash + Alkalinity sol. ash	total ash + Alkal. sol. ash	Alkal. sol. ash + Alkal. insol. ash	
Mixed sap of hard and soft maples	1	158	341	127	333	205	2.62	62	
	2	2.52	166	248	115	328	178	92	122	2.84	54
	3	2.53	164	379	99	313	144	150	124	3.17	46
	4	2.73	161	268	108	333	178	98	122	3.09	53
	5	2.38	145	250	112	309	175	105	129	2.77	57
	6	2.33	153	229	113	293	185	98	126	2.59	63
	7	2.76	162	394	106	288	168	143	104	2.71	57
	8	2.24	167	292	137	338	230	131	151	2.47	68
	9	2.70	153	...	96	314	138	...	116	3.28	44
	10	2.48	125	277	101	331	145	112	133	3.28	44
	11	2.33	129	252	96	268	150	108	115	2.79	56
	12	2.67	142	337	96	316	138	126	118	3.29	44
Average mixed sap.....	2.52	152	297	109	314	170	116	124	2.91	0.54	
Per cent deviation of maximum from mean.....	9.5	9.9	32.7	25.7	7.6	35.3	29.3	21.8	13.0	9.7	
Per cent deviation of minimum from mean.....	11.1	17.8	22.9	11.9	14.6	18.8	20.7	16.1	15.1	29.2	
Hard maple sap	13	3.02	167	354	124	354	191	117	117	2.85	54
	14	2.66	152	310	118	330	183	116	124	2.80	55
	15	2.70	143	311	105	292	164	115	108	2.78	56
	16	2.96	152	247	116	329	178	83	111	2.84	54
	17	...	141	308	95	285	144	2.98	50
	18	2.48	147	313	97	300	144	127	121	3.09	48
	19	2.63	146	361	111	326	169	137	124	2.69	52
Average hard maple.....	2.74	150	315	109	317	168	116	118	2.86	53	
Per cent deviation of maximum from mean.....	10.2	11.3	14.6	13.8	11.7	13.7	18.1	5.1	8.0	5.7	
Per cent deviation of minimum from mean.....	9.5	6.0	21.6	12.8	10.1	14.3	28.4	5.9	5.9	9.4	
Soft maple soap	20	2.48	149	285	114	335	172	115	135	2.95	51
	21	2.33	167	321	123	307	205	138	132	2.50	67
	22	...	161	274	117	269	206	2.31	76
Average soft maple.....	2.41	159	293	118	304	194	127	134	2.59	65	
Per cent deviation of maximum from mean.....	2.9	5.03	9.6	4.2	10.2	6.2	8.7	0.7	13.9	16.9	
Per cent deviation of minimum from mean.....	3.3	6.3	6.5	3.4	11.5	11.3	9.4	1.4	10.4	21.5	
General average.....	2.58	154	302	110	313	172	117	123	2.85	0.55	
Maximum.....	3.02	167	394	137	354	230	150	151	3.29	76	
Minimum.....	2.24	125	229	95	268	138	92	104	2.31	44	
Per cent deviation of maximum from mean.....	17.1	8.4	30.5	24.5	13.1	33.7	28.2	22.8	15.4	26.7	
Per cent deviation of minimum from mean.....	13.2	18.8	24.2	13.6	14.4	19.8	21.4	15.4	18.9	26.7	

syrup. But its preponderance is not such as to justify a confident prediction that the conductivity value will be more closely related to the soluble ash (consisting mainly of potassium carbonate) or its alkalinity than to the total ash or its alkalinity.

I have, therefore, made calculations of the ratios of several of the data of Tables III and IV and present these ratios in Tables VI and VII.

¹ Magnesium and sodium salts are present in minor proportions.

samples, shows no material difference in the variability of the ratios of the conductivity value to the different ash data. If anything, the conductivity is more closely related to the total alkalinity than to the separate alkalinities of either the soluble or the insoluble ash. The range of variation of any of these ratios is somewhat wider than that of the ratios of the total alkalinity to the soluble alkalinity.

TABLE VII—RATIOS OF THE ANALYTICAL DATA FOR THE 12 QUEBEC SYRUPS OF TABLE IV

Number	A/C Conductivity + Total ash syrup	A/D Conductivity + Soluble ash syrup	A/E Conductivity + Alkalinity syrup total ash	A/G Conductivity + Alkalinity syrup soluble ash	A/H Conductivity + Alkalinity syrup insol. ash	F/G Alkal. total + Alkal. sol. ash	G/H Alkal. sol. ash + Alkal. insol. ash
1	166	289	128	289	229	2.26	79
2	182	253	148	257	347	1.74	135
3	171	249	145	287	292	1.98	102
4	162	228	149	290	309	1.94	107
5	188	294	165	327	334	1.98	102
6	163	250	134	259	279	1.93	108
7	128	228	135	273	267	2.02	98
8	142	205	135	253	289	1.88	114
9	136	201	115	205	260	1.79	126
10	173	262	133	266	266	2.00	100
11	155	230	137	276	271	2.02	98
12	139	255	138	293	263	2.11	90
Average.....	159	245	139	273	284	197	105
Per cent deviation of max. from mean	18.2	18.0	18.7	19.8	22.2	14.7	28.6
Per cent deviation of min. from mean	19.5	18.0	17.2	24.9	19.4	11.7	24.8

CONDUCTIVITY VALUE OF THE ASH

Among the data of Tables III and VI is included for 19 syrups a figure designated "Conductivity Value of the Ash." This was determined according to the following method: 5 grams of syrup are ashed in a platinum dish. The ash is boiled with 30 cc. water for two minutes, filtered through a 7 cm. filter and washed with hot water to a volume of nearly 50 cc. (the residue is ignited and weighed as the insoluble ash). The cooled filtrate is made up to exactly 50 cc. and the conductivity measured at 25° C. The solution is washed out of the conductivity cell and titrated for alkalinity of soluble ash.

This method departs from the conventional one for determination of soluble and insoluble ash in maple products in using only 50 cc. of hot water on the ash of 5 grams of syrup, instead of 100 cc. Possibly it might be preferable to adhere to the conventional method of separating soluble and insoluble ash and to make the determination of conductivity value of ash in a volume of 100 cc. instead of 50 cc.

To determine whether there is any material difference between the results obtained with the two different quantities of wash water, Mr. Scott has repeated the determinations upon syrups Nos. 2, 5 and 11, using 100 cc. of wash water. The results obtained by the two methods are compared below:

Syrup No.	Date made	Alkalinities as determined with use of					
		50 cc. wash water			100 cc. wash water		
		Sol. ash	Insol. ash	Ratio	Sol. ash	Insol. ash	Ratio
2	Apr. 7-9	50	92	0.54	46	82	0.56
5	Apr. 12	47	83	0.57	52	86	0.60
11	Apr. 25	47	84	0.56	52	84	0.62
Average,				0.56			0.59

I infer from these results that the difference between the two methods of washing is of little consequence.

¹ In the A. O. A. C. provisional method for saccharine products in general, only 60 cc. of water are used. See Bur. of Chem., *Bulletin* 107, 68 (1908). Hortvet followed this method.

As intimated above, the syrups upon which the determination of the conductivity of the ash was made are peculiar in their low ratio of alkalinity of soluble to that of insoluble ash. They average 0.54. On the 94 syrups examined by Jones, the average value of this ratio is 0.94; on the 13 upon which Hortvet made these determinations it is 0.85; Bryan's general average for 481 syrups is 0.77, although for the 100 syrups selected by him for ash analysis, I find the average to be 0.87; and the average for the 12 syrups of Tables IV and VII of the present paper is 1.05. It is clear, therefore, that the syrups made in the laboratory (Tables III and VI) are of exceptional character. Under these conditions I do not feel justified in drawing any conclusions from the results obtained in the determination of the electrical conductivity of the ash solution. This property is, however, worthy of further study.

TABLE VIII—CONDUCTIVITY VALUES OF NON-MAPLE SYRUPS

Number	Description	Specific Conductivity	
		gravity	value
1	Corn syrup	1.40	2.51
2	Golden syrup	1.44	414
3	Molasses	...	656
SYRUPS DILUTED TO DENSITY OF MAPLE SYRUP			
No. 1 diluted	Corn syrup	1.339	253
4	Corn syrup	1.320	359
5	Corn syrup	1.320	209
No. 2 diluted	Golden syrup	1.320	427
6	Golden syrup	1.320	392
7	Golden syrup	1.320	403
8	Molasses	1.320	1121
9	Molasses	1.320	1280
10	Molasses	1.320	604
11	Molasses	1.320	1250
12	Cane sugar syrup from granulated sugar	1.314	0.5
13	Cane sugar syrup from pale brown sugar	1.329	178
14	Cane sugar syrup from pale brown sugar	1.333	185

THE CONDUCTIVITY VALUES OF NON-MAPLE SYRUPS

Table VIII gives the conductivity values of a number of non-maple syrups. It will be seen that syrup made from granulated sugar is practically a non-conductor, that syrups from partially refined cane sugar may give values within the limits found for pure maple syrups, and that all of the others give conductivity values distinctly above the limits for maple. It is clear, therefore, that values either above or below the limits for genuine maple syrup may be produced by adulteration. Syrups yielding values either abnormally high or abnormally low may be condemned without further examination. But it is clear that the possession of a normal conductivity value is not in itself adequate evidence of the purity of a syrup.

USEFULNESS OF THE METHOD

In Table IX are given the results of analysis of 34 syrups as found upon the market in the Provinces of Saskatchewan, Alberta and British Columbia in 1911 and 1912. The modified Winton lead number was determined upon the quantity of syrup containing 25 grams of dry matter.

It will be seen that the conductivity method would condemn 15 of these samples, and that every one of

TABLE IX—SYRUPS PURCHASED IN THE CANADIAN WEST

Number	Sold as	Specific gravity	Per cent Moisture	Per cent Total ash Dry basis	Per cent Insoluble ash Dry basis	Per cent Alkalinity soluble ash	Modified Winton			
							Alkalinity insoluble ash	Lead Number (Dry basis)	Conductivity value	
1	Pure	1.309	36.8	0.13	0.03	0.11	0.11	0.46	23	
2	Pure	1.337	32.2	0.09	0.05	0.06	0.13	0.10	24	
3	Pure	1.304	37.6	0.67	0.29	0.34	0.46	1.15	113	
4	Pure	1.315	35.8	0.62	0.31	0.34	0.52	1.37	114	
5	Pure	1.332	33.0	0.76	0.34	0.47	0.60	1.99	134	
6	Pure	1.315	35.8	0.95	0.50	0.42	0.58	2.38	150	
7	Pure	1.323	34.4	0.84	0.44	0.46	0.70	1.96	143	
8	Pure	1.318	35.3	0.91	0.43	0.42	0.74	2.09	159	
9	Pure	1.326	34.0	0.41	0.20	0.23	0.38	0.80	79	
10	Pure	1.332	33.0	0.82	0.42	0.50	0.66	1.92	133	
11	Pure	1.316	35.6	0.07	0.04	0.08	0.10	0.23	21	
12	Pure	1.326	34.0	0.85	0.30	0.38	0.42	1.40	123	
13	Pure	1.344	31.1	0.64	0.18	0.28	0.28	0.99	90	
14	Pure	1.352	29.8	0.72	0.30	0.35	0.45	1.61	117	
15	Pure	1.340	31.7	0.93	0.33	0.48	0.50	1.58	142	
16	Pure	1.323	34.5	0.93	0.38	0.49	0.61	2.06	152	
17	Pure	1.330	33.4	0.33	0.14	0.20	0.26	0.84	60	
18	Pure	1.335	32.5	0.43	0.19	0.23	0.32	0.83	66	
19	Pure	1.315	35.8	0.50	0.22	0.29	0.38	1.09	85	
20	Pure	1.320	35.0	1.09	0.40	0.50	0.72	2.25	160	
21	Pure	1.333	32.9	0.70	0.33	0.38	0.58	1.68	128	
22	Pure	1.327	33.8	0.74	0.21	0.31	0.46	1.33	93	
23	Pure	1.331	33.2	0.54	0.28	0.32	0.42	1.41	97	
24	Pure	1.340	31.7	0.59	0.23	0.30	0.46	1.29	94	
25	Pure	1.324	34.3	1.22	0.43	0.49	0.72	2.33	145	
26	Pure	1.325	34.2	0.93	0.52	0.47	0.83	2.56	149	
27	Pure	1.326	34.0	0.85	0.33	0.50	0.57	2.11	146	
28	Pure	1.329	33.5	0.62	0.21	0.32	0.48	1.26	94	
29	Pure	1.335	32.5	0.65	0.25	0.40	0.57	1.36	113	
30	Pure	1.329	33.5	0.35	0.15	0.16	0.33	0.86	64	
31	Compound	1.337	32.2	0.10	0.04	0.09	0.09	0.20	24	
32	Compound	1.351	30.0	0.56	0.14	0.31	0.23	0.72	136	
33	Maple flavor	1.351	30.0	0.21	0.13	0.15	0.17	0.47	48	
34	Maple flavor	1.339	31.9	0.30	0.25	0.36	0.42	1.68	129	
Standards of purity:										
Canada.....				35.0	0.60	0.12	1.20	..
Vermont.....				..	0.77	0.23
Ordinary limits for genuine syrup:										
Minimum.....				..	0.75	0.23	0.30	0.36	..	110
Maximum.....				..	1.35	0.80	0.66	0.94	..	200

these 15 is also condemned by a complete analysis. Syrups 32 and 34, however, which would pass the conductivity test, are condemned by the other determinations, and Nos. 3, 4 and 29, which are near the limit in conductivity value and which, being of the same brand as Nos. 9, 19, 21, 22, 24 and 28, are probably adulterated, are also near the limits of the Canadian standard in respect to the other analytical data and are below the Vermont standard on total ash. When one considers that all the ordinary analytical work on the 15 samples might be omitted, the usefulness of the conductivity method is apparent.

SUMMARY

1. A rapid method of detecting adulteration of maple syrup with commercially pure sucrose is described.
2. The "conductivity value" is defined as 100,000 times the specific conductivity at 25° C. of a mixture of one volume of syrup with two volumes of water.
3. The limits of conductivity value for pure maple syrup are tentatively set at 110 to 200.
4. The relation of conductivity value to ash data is discussed.
5. The usefulness of the test is illustrated.

MACDONALD COLLEGE
QUEBEC

THE COMPOSITION OF DIFFERENT VARIETIES OF RED PEPPERS¹

By L. M. TOLMAN AND L. C. MITCHELL

Received May 26, 1913

CAYENNE OR CHILLI

General Description.—Cayenne or chilli is a small fruited pepper, a variety of *Capsicum frutescens* L., a species of *Capsicum*, which is a genus of the family *Solanaceae*, indigenous to the American tropics, but now grown or cultivated in nearly all tropic and subtropic countries. It is characterized by its extreme pungency and the small size of the pods. The leading commercial varieties (1911) are African and Japanese.

The African cayenne or chillies come chiefly from the ports of Mombasa and Zanzibar, British East Africa, and are usually designated in the trade by the name of the port from which shipped; they are from 10 to 15 mm. in length, dark, dull red in color, and extremely pungent; they are ground for use. A few of the samples contained some unattached stems and calyxes.

The Japanese chillies come from the port of Kobe, Japan; these are from 15 to 40 mm. in length, bright red in color, clean, containing very few stems or calyxes, and are used chiefly in the unground condition for the preparation of the so-called "chilli sauce."

Cayenne or chilli contains a fixed, bland oil, found

¹ Authors' abstract of *Bull.* 163, Bureau of Chem., Dept. of Agr.

in both pods and seeds, but more abundantly in the latter, considerable resinous and mucilaginous material, a red coloring matter confined to the shell, and the active principle, capsaicin, to which the pungency is due. The capsaicin is present in both

Analyses.—In addition to the African and Japanese varieties which make up the major portion of the importations, two other apparently different varieties were examined. N. Y. 25482 is a nearly round cayenne or chilli from India, 15 to 25 mm. in length and 10 to

TABLE I—RESULTS OF ANALYSES OF CHILLIES, OR CAYENNE PEPPERS

Serial No.	Original						Sifted					
	Total ash Per cent	Ash insoluble in 10 per cent hydro- chloric acid Per cent	Sand-free ash Per cent	Volatile ether ex- tract Per cent	Non-volatile ether extract Per cent	Crude fiber Per cent	Total ash Per cent	Ash insoluble in 10 per cent hydro- chloric acid Per cent	Sand-free ash Per cent	Volatile ether ex- tract Per cent	Non-volatile ether extract Per cent	Crude fiber Per cent
African (Mombasa) chillies or cayenne:												
N. Y. 24584....	6.45	1.56	4.89	..	17.91	26.41	5.98	0.82	5.16	..	18.34	26.75
N. Y. 24605....	5.34	0.44	4.90	..	18.59	27.26	5.51	0.46	5.05	..	18.45	27.97
N. Y. 24606....	7.38	1.73	5.65	..	17.70	28.45	5.71	0.72	4.99	..	18.60	28.11
N. Y. 24610....	7.44	1.03	6.41	..	19.00	26.13	6.16	0.72	5.44	..	18.46	27.44
N. Y. 24701....	4.88	0.45	4.43	0.24	19.36	25.26	4.80	0.52	4.28	0.28	19.02	26.21
N. Y. 24802....	7.72	2.59	5.13	0.41	16.43	27.25	5.80	0.72	5.08	0.47	16.41	27.22
N. Y. 24803....	5.51	0.52	4.99	0.74	17.03	28.81
N. Y. 24855....	5.81	1.00	4.81	1.21	18.60	27.39	5.71	0.64	5.07	0.85	18.63	27.63
N. Y. 24885....	5.48	0.55	4.93	0.89	18.60	27.86	5.18	0.44	4.74	1.11	18.14	28.03
N. Y. 25027....	5.51	0.78	4.73	0.51	16.81	26.57	5.13	0.50	4.63	0.49	17.28	26.77
N. Y. 25028....	6.96	1.63	5.33	0.49	15.88	26.70	5.64	0.74	4.90	0.56	16.87	27.08
N. Y. 25029....	5.83	0.76	5.07	0.73	16.21	26.54	5.59	0.65	4.94	0.65	16.59	26.48
N. Y. 25031....	7.45	2.25	5.20	0.52	17.93	24.98	5.86	0.85	5.01	0.26	17.33	26.98
N. Y. 25190....	5.94	0.93	5.01	0.65	16.09	27.27	5.56	0.77	4.79	0.44	16.82	26.72
N. Y. 25195....	5.72	0.65	5.07	0.54	15.90	28.00	5.31	0.50	4.81	0.58	16.07	27.72
N. Y. 25245....	5.64	0.86	4.78	0.74	17.95	25.48	5.33	0.66	4.67	0.75	17.40	27.25
N. Y. 25348....	5.76	0.73	5.03	0.75	16.22	26.59	5.19	0.54	4.65	0.72	16.53	26.44
N. Y. 25399....	5.53	0.73	4.80	1.09	17.14	26.73	5.38	0.59	4.79	0.74	17.83	25.87
N. Y. 25470....	7.07	1.95	5.12	1.59	16.52	26.30	5.75	0.59	5.16	0.63	17.41	26.80
N. Y. 25481....	6.21	1.19	5.02	1.72	15.91	26.16	5.66	0.74	4.92	1.37	16.40	27.40
N. Y. 25566....	8.41	3.03	5.38	0.73	16.00	25.21	5.55	0.62	4.93	0.76	17.01	26.12
N. Y. 25634....	5.35	0.50	4.85	0.28	16.89	26.12	4.86	0.37	4.49	0.25	16.81	25.39
I. S. 3817-C....	6.66	1.42	5.24	1.15	17.60	28.70	5.24	0.55	4.69	0.71	19.68	28.85
I. S. 3819-C....	5.77	0.87	4.90	0.97	18.76	27.93	5.58	0.73	4.85	0.40	18.38	28.16
I. S. 3820-C....	6.18	1.09	5.09	0.91	16.99	27.97	5.38	0.55	4.83	0.66	18.13	28.00
I. S. 3822-C....	17.62(a)	11.38(a)	6.24(a)	0.76(a)	14.73(a)	24.50(a)	5.74	0.72	5.02	0.50	18.34	27.55
I. S. 3823-C....	7.31	2.26	5.05	0.96	16.65	28.24	5.73	0.73	5.00	0.82	16.65	29.46
Average....	6.31	1.24	5.07	0.81	17.26	26.86	5.51	0.63	4.88	0.64	17.52	27.23
Maximum....	8.41	3.03	6.41	1.72	19.00	28.70	6.16	0.85	5.44	1.37	19.68	29.46
Minimum....	5.34	0.44	4.73	0.78	15.88	24.98	4.86	0.37	4.49	0.25	16.07	25.39
Japanese (Kobe) chillies or cayenne:												
N. Y. 24819....	5.08	0.38	4.70	1.59	20.41	24.89
N. Y. 25004....	5.21	0.58	4.63	0.32	19.04	25.17	5.06	0.38	4.68	0.34	19.54	24.91
N. Y. 25010....	5.69	0.53	5.16	0.35	20.51	24.84	5.44	0.49	4.95	0.25	20.00	24.42
N. Y. 25011....	5.41	0.77	4.64	0.29	18.08	23.66	5.08	0.48	4.60	0.32	18.15	24.72
N. Y. 25012....	5.18	0.66	4.52	0.20	18.39	25.32	5.12	0.51	4.61	0.22	17.67	25.87
N. Y. 25013....	5.76	0.73	5.03	0.19	20.17	24.39	5.34	0.46	4.88	0.20	19.09	24.06
N. Y. 25014....	6.20	1.07	5.13	0.28	20.26	24.11	5.70	0.51	5.19	0.25	20.57	25.05
N. Y. 25026....	5.86	0.57	5.29	0.88	23.21	24.18	5.79	0.53	5.26	0.97	22.89	24.65
N. Y. 25398....	5.26	0.41	4.85	0.24	20.77	22.83	5.10	0.30	4.80	0.10	20.14	22.86
N. Y. 25408....	5.55	0.41	5.14	1.01	18.51	24.60	5.34	0.32	5.02	0.88	18.88	24.35
N. Y. 25409....	5.45	0.31	5.14	0.72	21.11	24.00	5.50	0.27	5.23	0.96	20.73	24.95
N. Y. 25410....	5.41	0.33	5.08	0.75	20.68	23.58	5.26	0.33	4.93	0.60	20.39	23.70
N. Y. 25567....	5.32	0.57	4.75	0.09	19.43	23.89	5.15	0.37	4.78	0.05	19.21	23.86
N. Y. 25678....	5.40	0.36	5.04	0.59	17.10	24.28	5.36	0.39	4.97	0.20	17.81	24.07
N. Y. 25679-A.	5.52	0.54	4.98	0.27	20.14	22.82	5.40	0.47	4.93	0.20	19.10	24.44
N. Y. 25679-B.	5.90	0.40	5.50	0.40	19.44	23.80	5.82	0.47	5.35	0.25	20.01	23.18
I. S. 3818-C....	5.71	0.44	5.27	1.39	21.69	25.96	5.40	0.37	5.03	1.14	21.20	26.08
Average....	5.52	0.53	4.99	0.56	19.94	24.25	5.37	0.42	4.95	0.43	19.71	24.45
Maximum....	6.20	1.07	5.50	1.59	23.21	25.96	5.82	0.53	5.35	1.14	22.89	26.08
Minimum....	5.08	0.31	4.52	0.09	17.10	22.82	5.06	0.27	4.60	0.05	17.67	22.86
Cherry:												
N. Y. 25482....	6.62	1.23	5.39	1.18	16.17	26.20	5.82	0.51	5.31	0.77	17.56	25.77
Sweet, red pepper:												
B. 4484.....	6.04	0.40	5.64	0.72	12.80	25.31	5.93	0.42	5.51	0.75	13.64	24.90
Siftings:												
I. S. 3821-C....	62.63	51.53

(a) Not included in average, maximum, and minimum.

the seeds and the shell, but the larger part is found in the placentae. The red coloring matter is soluble in ether, petroleum ether, carbon bisulfid, and chloroform, but very slightly soluble in alcohol.

15 mm. in width, belonging to *C. frutescens baccatum* L., and known to the trade under the name "cherry." The color of the pods varies from dark red to a brownish yellow; the sample was clean and had no

stems or calyxes. B. 4484 is a capsicum from Japan and is from 45 to 65 mm. in length, bright red in color, clean, and had the stems and calyxes attached to the pods. Analysis indicates this to be a sweet, red pepper.

In order to determine the characteristic taste and flavor and to find out definitely what part or parts of the pod contained the capsaicin, pods from different samples were broken up into the following parts: shells, seeds, and placentae. The shells from the Japanese chillies have a slightly bitter, slightly acid, bland taste (similar to that of pimenton) and are mildly pungent; the seeds, a sweetish, bland, mildly pungent taste; and the placentae extremely pungent, containing nearly all the active principle, capsaicin; the ground product has a very pungent taste. The shells from the African chillies have a slightly acid, pungent taste; the seeds, a sweetish, bland, pungent taste; and the placentae, extremely pungent; the ground product has a very pungent taste.

The samples were taken from the various imports as they were received by the regular United States Treasury samplers by means of coffee triers which were inserted into the packages or bales and the samples allowed to flow out into paper bags.

In each instance an analysis was made of the original and of the part which has been carefully sifted with an ordinary coarse 3 mm. hand sieve to remove as much sand as possible. The two analyses are placed on the same line, so that a ready comparison may be made.

The African or Mombasa chillies have the appearance of being dirty or sandy, and the analyses plainly show this condition. They are said to grow wild in the interior of tropical East Africa and are gathered by semicivilized people. They are dried on the ground and roofs of the huts or houses, thus subjected to the various conditions of the weather. They contain dirt, sand, stems, etc., and necessarily need cleaning before being ground for use. Attention is called to one sample, I. S. 3822-C, showing a most remarkable amount of sand and dirt present, which apparently could readily be sifted out, as the analysis of the sifted product shows a normal condition. The results on the sifted portions are remarkably uniform, showing only slight variations between the maximum and the minimum.

The Japanese or Kobe chillies differ in appearance, being very clean-looking and of a much brighter color. The results plainly show this difference, as the amounts of sand and ash are materially lower. Many samples contained less than one-half per cent. of sand, while in the Mombasa chillies practically none of the 50 samples ran as low as that.

I. S. 3822-C is so abnormal in its ash and ash insoluble in acid that none of the results are included in the average, maximum and minimum results. Note how the sifting reduces the mineral matter and increases the nonvolatile ether extract and crude fiber, making them normal.

PAPRIKA

General Description.—Paprika is a large-fruited

pepper, grown in Hungary, a variety of *Capsicum annum* L., a species of *Capsicum*, which is a genus of the family *Solanaceae*. When powdered, it has a deep red color and a sweetish, mildly pungent flavor. Its origin is somewhat obscure, but it apparently originated in America, whence it can be traced from Spain, through Greece and Turkey, to Hungary. It has been variously designated as Turkish pepper or paprika, Hungarian pepper or paprika, or garden pepper.

According to Csonka and Varadi,¹ there are five grades of Hungarian paprika known to commerce, as follows:

Rosenpaprika or Rózsapaprika.

Königspaprika or Királypaprika.

Merkantilpaprika.

Kranzpaprika.

Geschnittener Paprika.

The two last grades are practically unknown in the United States, as all of the paprika coming into the United States is ground.

Rosenpaprika, or Rózsapaprika, is the best grade of paprika made in Hungary, and has been known under this name since 1860, obtaining its name from its beautiful red color. It is produced with the utmost care from special, selected pods, the spotted and damaged parts being cut out; the placentae, the stalks, and the stems removed, and the seeds and shells washed many times before being ground. The ground product has a bright red color and is distinguished by its pleasant taste. It will be noted that even in the highest grade of paprika the seeds are ground with the shells.

The second grade, or Königspaprika, is prepared by grinding the whole pod without selecting any of the pods, and includes not only the seeds, but also the stems, in the amount naturally occurring with the pod.

The Mercantile grade is produced from the spotted pods remaining from the selection for the finer grades and from the pods containing flaws, the lower grades of the Mercantile being ground with the stalks, stems, and other waste parts. This grade has, as a rule, a much more pungent taste and has a pale yellow color.

Szigeti² classes paprika in four grades, making a special grade above the Rózsapaprika, which he calls Ectes paprika; otherwise, he classifies them in practically the same manner as the previous authors. He calls attention to the fact that the Mercantile grade consists of scraps and waste from the other grades, and that the product is more pungent and has a yellow color, as distinguished from the higher grades.

Analyses.—The samples which were examined by the authors, the results of which are given in Table III, were received under the seal of the Royal Hungarian Ministry of Agriculture through the Department of State. They consisted of large wreaths, were of a bright red color, sound, from 6 to 10 cm. in length and from 2.5 to 4.0 cm. in diameter, and of a conical shape.

¹ *Der Szegeder Paprika und der Szegeder Paprikahandel*, 1907.

² *Zts. Landw. Versuchsw. Oesterr.*, 5, 1208-22 (1902).

In order to get at the composition of the different parts of the paprika, each sample was subdivided into five different portions, as follows: *First*, ground shells, stems, seeds and placentae; *second*, ground shells, seeds, and placentae; *third*, ground shells alone; *fourth*, ground seeds and placentae alone; *fifth*, ground stems alone. In the preparation of these products, they were ground separately and passed through a 0.5 mm. sieve, with the exception of the seeds, which were passed through a 1 mm. sieve.

The results of this separation in percentage of shells, seeds and placentae, and stems, are as follows:

TABLE II—SEPARATION OF HUNGARIAN PAPRIKAS FOR ANALYSIS

(21 samples)	Seeds and placentae		
	Shells Per cent	Placentae Per cent	Stems Per cent
Average.....	56.4	36.1	7.5
Maximum.....	63.7	43.2	9.4
Minimum.....	50.5	28.1	6.0

The percentage was determined so that the effect of the addition of any of the parts in excess of the normal might be calculated, and thus a check had on the actual determinations made on the various grindings.

The percentage of these various parts is also of value in the microscopic examination of the ground product, in order to determine whether or not there is an abnormal percentage of seeds or stems present. It will be seen from this table that the percentage of stems never exceeds 10 per cent.

Table III contains the results obtained by the authors on the various samples of Hungarian paprika, separated into their various parts and ground.

TABLE III—ANALYTICAL RESULTS ON GROUND HUNGARIAN PAPRIKA

Serial No.	Volatile at 70° C. in vacuo Per cent	Total ash Per cent	Ash insoluble in 10 per cent hydrochloric acid Per cent	Sand-free ash Per cent	Total ether extract Per cent	Non-volatile ether extract Per cent	Volatile ether extract Per cent	Crude fiber Per cent	Seeker's method		Index of refraction at 40° C.	
									Non-volatile ether extract Per cent	Iodine number		
Shells, seeds and placentae, and stems:												
Average.....	3.47	5.63	0.28	5.36	14.49	14.04	0.42	21.93	12.61	132.6	1.4806	
Maximum.....	3.76	6.03	0.33	5.73	16.56	16.43	0.89	22.76	15.00	134.0	1.4854	
Minimum.....	3.29	5.08	0.24	4.82	12.87	12.21	0.08	20.69	10.86	129.8	1.4758	
Shells, seeds and placentae:												
Average.....	3.51	5.22	0.26	4.96	15.62	15.28	0.34	21.56	13.91	131.9	1.4799	
Maximum.....	4.16	5.56	0.31	5.25	17.43	17.35	0.90	23.18	15.08	133.2	1.4834	
Minimum.....	3.11	4.66	0.20	4.41	14.50	13.94	0.07	20.47	12.64	129.0	1.4756	
Shells:												
Average.....	3.63	6.60	0.28	6.33	6.34	6.07	0.28	23.19	4.68	140.1	(a)	
Maximum.....	4.00	7.11	0.37	6.84	7.19	6.90	0.44	24.07	5.26	149.5	(a)	
Minimum.....	3.44	6.29	0.22	6.04	5.38	5.14	0.16	22.20	4.14	133.2	..	
Seeds and placentae:												
Average.....	3.97	3.63	0.26	3.38	27.02	26.96	0.06	20.76	23.53	133.5	1.4696	
Maximum.....	4.23	3.80	0.30	3.59	27.71	27.56	0.15	21.60	24.96	134.0	1.4699	
Minimum.....	3.73	3.38	0.21	3.13	25.99	25.97	0.00	20.89	22.53	133.1	1.4691	
Stems:												
Average.....	5.22	10.59	0.85	9.74	2.77	2.54	0.23	30.67	
Maximum.....	5.78	11.35	1.20	10.67	3.40	3.05	0.35	32.86	
Minimum.....	4.66	9.84	0.66	9.18	2.25	2.11	0.15	29.34	

(a) Not read.

PIMENTON, OR PIMIENTO¹

General Description.—The pimenton, or pimiento, is a large-fruited pepper, a variety of *Capsicum annum* L., grown in Spain, and, under the standard

¹ "Pimenton" should not be confused with "pimento" or "pimenta" which are applied to Jamaica pepper or allspice. "Pimenton" is the definite term used to designate the Spanish product when ground. The usual term in the trade is Spanish paprika.

given in Circular 19, Office of the Secretary, would be classified, when ground, as paprika. The succulent pericarp of this pepper is much used for stuffing of olives, while the dried pod is ground as a spice. A large quantity of this pepper imported to the United States is used largely on account of its high coloring properties. The taste and flavor are quite different from the Hungarian paprika.

The samples which were examined during this study were received under seal from the American consul-general at Barcelona, Spain, and consisted of large wreaths, the pods of which were of a bright red color, sound, and from 2.5 to 4 cm. in length, and from 4 cm. to 6 cm. in diameter, having a nearly round shape.

Analyses.—Five different grindings were made from each set of peppers. In the *first* case, the shells, seeds and placentae, and stems, were ground together; in the *second*, the shells, seeds and placentae; in the *third*, the shells alone; in the *fourth*, the shells and placentae; in the *fifth*, the stems alone. Table IV gives the percentage composition of the various parts of the pods used in the above ground samples:

TABLE IV.—SEPARATION OF SPANISH PIMENTON FOR ANALYSIS

Serial No. (18 samples)	Seeds and placentae		
	Shells Per cent	Placentae Per cent	Stems Per cent
Average.....	55.3	36.0	8.7
Maximum.....	58.1	37.4	10.9
Minimum.....	53.1	34.9	6.0

The percentage was determined so that the effect of an addition of any of the parts in excess of the normal might be calculated and thus a check had on the actual determinations made on the various grindings. The percentage of these parts is also of value in the

microscopical examination of the ground product in order to determine whether or not there is an abnormal percentage of any of the parts.

Table V gives the analytical results on the various ground samples.

CONCLUSIONS

Cayenne.—The first point of special interest to be

TABLE V—ANALYTICAL RESULTS ON GROUND SPANISH PIMENTON, OR PIMENTO

Serial No.	Volatile at 70° C. in vacuo Per cent	Total ash Per cent	Ash insoluble in 10 Per cent hydrochloric acid Per cent	Sand-free ash Per cent	Total ether extract Per cent	Non-volatile ether extract Per cent	Volatile ether extract Per cent	Crude fiber Per cent	Seeker's method		Index of refraction at 40° C.	
									Non-volatile ether extract Per cent	Iodine number		
Shells, seeds and placenta, and stems:												
Average	5.06	7.39	0.35	7.04	12.34	11.87	0.47	20.13	10.34	136.7	1.4805	
Maximum	5.98	7.86	0.48	7.54	13.06	12.58	0.69	20.59	10.81	137.3	1.4818	
Minimum	4.31	6.98	0.29	6.69	11.93	11.30	0.10	19.53	9.81	136.0	1.4776	
Shells, seeds and placenta:												
Average	4.83	6.98	0.32	6.66	12.91	12.47	0.44	19.49	10.67	136.1	1.4801	
Maximum	5.09	7.35	0.40	6.98	13.85	13.34	0.60	20.34	11.30	137.2	1.4810	
Minimum	4.52	6.60	0.24	6.26	11.83	11.58	0.25	18.76	9.80	134.5	1.4792	
Shells:												
Average	4.83	7.99	0.32	7.67	7.02	6.30	0.71	18.04	4.46	141.0	1.4963	
Maximum	5.02	8.46	0.37	8.09	7.52	6.81	1.05	18.70	4.67	143.7	1.4968	
Minimum	4.74	7.55	0.26	7.29	5.85	5.44	0.41	17.26	4.26	136.7	1.4958	
Seeds and placenta:												
Average	3.92	4.79	0.27	4.52	23.32	23.10	0.21	22.37	19.99	129.9	1.4699	
Maximum	4.12	5.02	0.31	4.76	24.89	24.58	0.42	24.08	21.35	130.8	1.4702	
Minimum	3.59	4.43	0.22	4.12	22.15	21.82	0.05	19.90	19.11	128.1	1.4695	
Stems:												
Average	5.35	14.53	0.73	13.81	1.76	1.44	0.31	29.43	
Maximum	5.98	15.77	1.07	14.91	2.45	2.05	0.49	30.98	
Minimum	4.86	13.00	0.58	12.36	1.48	1.22	0.15	27.66	

noted is the fact that the Mombasa, or African, chillies now coming into this country are exceedingly dirty, and must necessarily be very carefully cleaned before they are ground. This condition is undoubtedly brought about by the fact that they grow wild in the interior of Africa, and are gathered by semi-savage tribes and dried under various conditions.

It is also likely that the conditions which control the production of this product may vary in the course of a number of years, so that the cayenne on the market may vary from time to time as the source of the chillies varies.

At the present time, a considerable amount of small, round-shaped chillies, known as "cherries," grown in India, is being brought into this country, and practically none of the true Zanzibar chillies is being received, although some chillies are still shipped from Zanzibar which are undoubtedly the same as those coming from Mombasa, and their source is the interior of Africa and not the island of Zanzibar.

The sand present in these products appears to be entirely of an extraneous nature, and they can be sifted so that an entirely uniform product is obtained. An examination of the tables shows that the sand-free ash of these products, regardless of source, is extremely uniform, and as a criterion of the product it would appear that this figure would be of great value.

The Kobe, or Japanese, chillies are comparatively clean and need but little sifting to produce a satisfactory product. These, however, are used in the preparation of "chilli sauce," and not for grinding purposes.

A careful study of the various tables shows that in judging the various varieties of cayenne—African, Japanese, and Indian—they should be compared with data obtained upon chillies from the same source.

Hungarian Paprika.—From a study of the literature and the information available in the course of this investigation, it would appear that the word "paprika" has generally referred in the past to a product made in Hungary, but now it is applied to similar products produced in Spain and America; it is clear that the

terms "Rosenpaprika" and "Rózsapaprika" are limited to a well-defined product which has been made in Hungary for a long period of time and consists of the highest grade of pods ground without the stems.

Further, it is clear that the grinding of the stems with a product of this kind is simply for the purpose of producing more weight and acting as a filler, and that it does not add any valuable qualities to the product, as it contains no flavoring principles. It is also clear that the grinding of the seeds and placenta with the pods produces an improved product, and all of the facts seem to indicate that it is a well recognized and legitimate process.

The results show that paprika must be judged from data obtained upon samples of paprika from a known source.

Pimenton, or Spanish Paprika.—It appears from this investigation that this product is commonly known as "Spanish paprika," or "pimenton." It is quite different in its flavoring qualities and value from Hungarian paprika, and should be properly distinguished from it. It has very little pungency and its flavor is different from that of the Hungarian product.

Further, the analytical results upon the pimenton show that the different conditions under which it is grown have affected, to a greater or less extent, the amount of ash and other ingredients present, clearly showing that in the judging of these products it is necessary to compare the Hungarian paprikas with data upon Hungarian paprikas, and the Spanish pimentons with data upon Spanish pimentons.

It is shown that it is possible to distinguish by analytical methods and by characteristic properties, such as taste and odor, between the Hungarian paprikas and the Spanish pimentons. They have different uses, the pimenton being largely used for its coloring value, rather than for its flavor.

SOME DISTINCTIONS BETWEEN PEPSIN AND PEPTASE¹

By DR. R. WAHL

The far-reaching influence of peptonizing ferments in the brewing industry on the stability of the product, together with the fact that very little has been published on the nature and properties of the vegetable ferment, has led me to investigate the peptase of barley malt. Although I shall not attempt to go into experimental details and data to-day, the time being short and some of our experiments being still incomplete, I will state in a general way only some of the differences between pepsin and peptase, which have become apparent in the course of my work.

One of the chief differences between these two is that while pepsin (the proteolytic ferment derived from the animal organism) is comparatively easy to obtain in a dry form approximating purity, the same does not hold true of the vegetable peptase. So far, to my knowledge, the peptase has not been isolated, and our own experiments dealing with the obtaining of dry, stable, peptic preparations of vegetable origin have not been very successful. Although I have devised a process which enables me to produce malt extracts of high peptic strength, as outlined in my communication to the Eighth International Congress of Applied Chemistry, September, 1912, which has proven of great practical value to the brewing industry, all attempts to reduce the extract to a dry form without the sacrifice of the greater part of the peptic strength, have failed.

Another interesting difference is that while pepsin acts best in the presence of mineral acid—preferably hydrochloric—the peptonizing power of the peptase of malt is greatly enhanced by the presence of lactic acid, preferably of a bacterial nature. It seems evident that the peptic enzyme contained in malt is locked up (combined with some base, presumably of albuminous nature) and becomes active through liberation by bacterial acidity. The digestive power must be generated, as it were, and I find that bacterial lactic acid is the specific activator, having tried a large number of other acids, both mineral and organic, but with comparatively little effect. The remarkable fact is that the lactic acid necessary to activate the peptase is provided during the growth of the malt, having been introduced into the steeping water with the barley. The secretion in the animal organism of pepsin and its specific activator, hydrochloric acid, which has even led some authors to the belief that active pepsin is not pepsin in the free state, but a compound of pepsin with hydrochloric acid, is thus seen to offer quite a close parallel.

The optimum temperatures of these two proteolytic ferments, that is, the temperature at which the proteolytic action proceeds most favorably, also differ. While this temperature lies in the vicinity of 50° C. in the case of pepsin, I have found the most favorable temperature for the most thorough digestive action of the peptase to be as low as 35–40° C. Again, peptase in solution is rapidly destroyed at 60° C.

¹ Presented at the meeting of the Chicago Section of the American Chemical Society, June 13, 1913.

while pepsin is not. Furthermore, the peptase of the barley grain is capable of inverting albumen at the low temperature of say 2° C. while the proteolytic action of the pepsin at that temperature is practically nil. All of the heat-coagulable albumen contained in a cold water malt extract will be completely peptonized, which means, converted into the non-coagulable form, by permitting this extract to stand for a few days at this temperature, *viz.*, 2° C.

Finally, most of the customary tests employed in physiological chemistry to measure the proteolytic activity of gastric and similar juices, give no satisfactory results when applied to the peptase of barley grain. I have reference to such methods as those devised by Jacoby, Volhard, etc., and the well-known fibrin digestion test. So far the gelatin test depending upon the liquefaction of gelatine by proteolytic ferments, suggested by Schidrowitz, and which I have slightly modified, has proven the one most applicable and most reliable in measuring the peptic action of malt extracts.

Another almost equally reliable criterion of the peptic strength is auto-digestion—the amount of coagulable albumen remaining in a malt extract after the same had been subject to the action of the peptonizing enzyme for a given period and at a given temperature being determined by means of heat coagulation.

At the beginning of this paper I have mentioned the application of these peptic malt extracts to practical brewing, and would like to add that by means of this principle the sensitiveness of the bottled product to low temperatures is minimized. Influences that have such detrimental effects on beer which has not attained stability to a high degree, are high temperatures, like pasteurization and summer temperatures, low temperatures, and light. All these effects are due to the presence, in unstable beer, of colloidal albumen, *i. e.*, albumen in a semi-soluble or unstable condition, and this albumen is influenced and gradually broken down by light or heat vibrations, and reverts to an insoluble state, as shown by chilling. By employing the digestive principle contained in malt, the colloidal albumen remaining in the beer after proper storage and natural treatment throughout, can be transformed before the beer reaches the bottle, into albumenoids that are desirable in every way, that is, into the permanently soluble and stable forms known as albumose and peptones.

At some future time I will make public the experimental data and details of these tests, which I have now been conducting for a number of years.

1135 FULLERTON AVE.
CHICAGO

DETERMINATION OF TOTAL FORMALDEHYDE IN FUMIGATORS AND COMMERCIAL SOLUTIONS¹

By JACK J. HINMAN, JR.

Many methods have been devised for the estimation of formaldehyde, most of which are designed for small amounts or mere traces, although the methods for the

¹ Read before the Indiana Section of the A. C. S., Indianapolis, June 13, 1913.

determination of formaldehyde in considerable amount are by no means few.

I have extended Craig's table of methods intending to give the principal ones which are said to be applicable to the analysis of strong solutions and solid polymers.

FORMALDEHYDE METHODS

Class I—Specific reactions:

- a. Ammonia to hexamethylenetetramine:
 - Legler, *Ber.*, **32**, 2841 (1899).
 - Trillat, *Compt. rend.*, **116**, 891 (1893); M. Klar, *Pharm. Zeit.*, **40**, 438 (1895).
 - Cirelli, *Arch. farm.*, **8**, 581-93.
 - Hermann, *Chem. Ztg.*, **35**, 25.
 - Schiff, *Chem. Ztg.*, **27**, 14 (1903); Gaillot, *Ann. chim. anal.*, **18**, 17-18 (1913); Malle, *Pharm. J.*, June, 1905, p. 844.
- b. Potassium cyanide:
 - G. Romijn, *Z. anal. Chem.*, **36**, 18-27 (1897).
- c. Sodium hydroxide:
 - Legler, *Ber. d. deut. Ges.*, **16**, 133.
- d. Hydrazine sulfate:
 - E. Riegler, *Z. anal. Chem.*, **36**, iii, 115 (1887).
 - Rimini and Jona, *Giorn. Farm. Chem.*, **61**, 49-56.
- e. 4,4'-Dihydrazinodiphenyl hydrochloride:
 - Lebbin, *Pharm. Zeit.*, **42**, 18 (1897).
- f. Catechin:
 - Clauser, *Ber.*, **36**, 106 (1903).
- g. Sodium bisulfite:
 - C. Kleber, *Pharm. Rev.*, **22**, 1894 (1904).
 - M. Ripper, *Bull. soc. chim.*, [3] **31**, 691 (1904).
- h. Sodium sulfite:
 - Seyewetz and Gibello, *Chem. News*, **91**, 135, 2365.
 - Lemme, *Chem. Zeit.*, **27**, 896 (1903).

Class II—Addition products with elimination of water:

- a. Hydroxylamine:
 - Brochet and Cambier, *Compt. rend.*, **120**, 449 (1895).
- b. Aniline:
 - Trillat, *Compt. rend.*, **116**, 891 (1893).
 - M. Klar, *Pharm. Zeit.*, **40**, 548 (1895).
- c. Hydrazine hydrate:
 - Curtius and Pulvermacher, *Chem. Zeit.*, **26**, 701 (1902).
 - A. Pfaff, *Ber.*, **36**, 2360 (1903).
- d. Phloroglucin:
 - Clowes and Tollens, *Ber. d. chem. Ges.*, **32**, 2841.

Class III—Oxidation and reduction:

- a. Acid silver nitrate:
 - Grützner, *Arch. Pharm.*, **234**, 634 (1896).
- b. Alkaline silver nitrate:
 - L. Vanino, *Z. anal. Chem.*, **40**, 720 (1901).
- c. Acid potassium permanganate:
 - L. Vanino and E. Zeiter, *Z. anal. Chem.*, **40**, 587 (1901).
- d. Alkaline potassium permanganate:
 - H. M. Smith, *Analyst*, **21**, 148 (1896).
- e. Sodium hydroxide and hydrogen peroxide:
 - Blank and Finkenbeiner, *Ber.*, **31**, 2979 (1898).
 - Haywood and Smith, *J. Am. Chem. Soc.*, **27**, 1183 (1905).
 - Fresenius and Grünhut, *Z. anal. Chem.*, **44**, 13 (1905).
- f. Iodine:
 - Romijn, *Z. anal. Chem.*, **36**, 18 (1897).
 - Fresenius and Grünhut, *Z. anal. Chem.*, **44**, 13 (1905).
- g. Calcium oxychloride:
 - W. Brautigam, *Pharm. Zentr.*, **51**, 9156.
- h. Potassium bichromate:
 - Nicloux, *Bull. soc. chim.*, [3] **17**, 839.

Class IV—Physical methods:

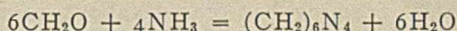
- a. Refractometer:
 - Richter and Jansen, *Chem. Weekblad.*, **9**, 1049.
- b. Specific gravity.

Investigations by A. G. Craig,¹ B. H. Smith,² Wallnitz,³ Romijn,⁴ and others have shown many of these to be untrustworthy. The general conclusions seem to be that of these methods the preferable ones are the potassium cyanide, iodine, ammonia, hydrogen

peroxide, and sulfite types. The potassium cyanide method is best adapted for dilute solutions and R. H. Williams¹ has shown the iodine method to be most readily influenced by normal impurities. For these reasons I chose methods from the three remaining types, since the solutions and polymers I wished to analyze were both strong and impure. Under the ammonia type I used Legler's and Schiff and Malle's procedures and repeated the latter, using ammonium sulfate instead of ammonium chloride. I also used the Blank and Finkenbeiner hydrogen peroxide method of the U. S. P., and Haywood and Smith's modification as given in *Bulletin 107* (revised) of the U. S. Bureau of Chemistry. The sulfite methods were those of Seyewetz and Gibello and C. Kleber. I also compared the refractometer tables of B. Wagner² and the calculation formula of Richter and Jansen based upon the refractometer reading. The references to these methods are given in the table above.

These methods were used in the analyses of four samples of commercial formaldehyde solutions, eight types of formaldehyde fumigators and a sample of Merck's trioxymethylene. In addition to normal impurities the fumigators contained various other substances such as naphthalene, phenol, glycerine and fats and all were more or less contaminated by rust. They were prepared for analysis, after removal from the tin cases, by separating the paraffin covering, and cutting into small pieces after scraping off as much iron rust as possible. The small pieces were mixed and preserved in rubber-stoppered bottles.

The ammonia method is based upon the reaction between ammonia and formaldehyde to form hexamethylene tetramine and water:



The gravimetric method has been found to be worthless as the tetramine loses weight indefinitely.

Legler's Method.—Weigh about 2 cc. of the solution (or 0.5 to 0.7 gram of polymers) into a glass-stoppered Erlenmeyer flask and add 50 cc. approximately normal ammonium hydroxide. Grease the stopper and let the tightly closed flask stand over night. Prepare two blanks using the same amount of the ammonium hydroxide solution and allow to stand the same length of time under the same conditions. Run in a slight excess of normal sulfuric acid and titrate back with normal sodium hydroxide, using rosolic acid as the indicator. The difference between the amount of normal acid used to neutralize the blank and the amount used in the case of the sample gives the amount of true normal ammonium hydroxide. Correct for the acidity of the sample.

1 cc. normal NH_4OH = 0.045 gram formaldehyde.

This method is known to give low results. Rosolic acid is not a satisfactory indicator. Loesekann recommends methyl orange titrated to full red.

Schiff and Malle's Method.—Introduce 20 cc. of neutral 20 per cent ammonium chloride solution into a glass-stoppered Erlenmeyer flask. Make a 1 to 10

¹ R. H. Williams, *J. Am. Chem. Soc.*, **28**, 596 (1905)

² B. Wagner, *Sonderhausen*, 1903: booklet accompanying Zeiss' immersion refractometer.

¹ A. G. Craig, *J. Am. Chem. Soc.*, **23**, 638 (1901).

² B. H. Smith, *Ibid.*, **25**, 1028 (1903).

³ Wallnitz, *Deut. Gerber Ztg.*, **1**, **4**, **6**, **8**, **12**; through *J. Chem. Soc.*

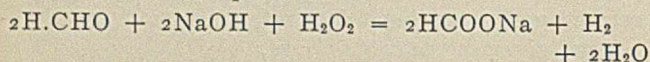
⁴ Romijn, *Zeit. anal. Chem.*, [1] **36**, 18-24.

dilution of formaldehyde and add 20 cc. to the ammonium chloride in the flask. Follow by 25 cc. of normal sodium hydroxide, grease the stopper and close tightly. Allow to stand one hour and titrate excess alkali with normal sulfuric acid, using rosolic acid as indicator. Correct for the acidity of the sample. When analyzing the solid formaldehyde use 0.5 to 0.7 gram and allow to stand over night.

1 cc. normal NaOH consumed = 0.045 gram formaldehyde.

This method gave higher results than did the Legler method. Solution of the polymers in the liquid was not always complete after 12 hours' standing, however.

The hydrogen peroxide methods are based upon the oxidation of formaldehyde to formic acid and the neutralization of sodium hydroxide by it. The reaction which takes place is



According to Haywood and Smith another reaction may accompany this one, *viz.*:



This secondary reaction they claim leads occasionally to incorrect results and their method is intended to avoid this error. As a rule this method gave no lower results.

Blank and Finkenbeiner's Method (U. S. P.).—Three cc. of the solution or about 1 gram of the solidified formaldehyde is weighed into a glass-stoppered Erlenmeyer flask of about 500 cc. capacity: 50 cc. of normal sodium hydroxide solution and 50 cc. of hydrogen peroxide of known acidity are added, the latter run in slowly and through a funnel resting in the mouth of the flask. After foaming has ceased (about 10 minutes) rinse the funnel and walls of the flask with recently boiled distilled water and titrate the excess of alkali with normal sulfuric acid and litmus indicator. Correct for acidity of sample and the hydrogen peroxide used.

1 cc. normal NaOH consumed = 0.03002 gram formaldehyde (on O = 16 basis).

Haywood and Smith's Method (Bull. 107).—Put exactly 50 cc. normal sodium hydroxide into a 500 cc. Erlenmeyer flask and add 50 cc. of pure hydrogen peroxide of known acidity. Add 3 cc. of formaldehyde, whose specific gravity has been determined, through a pipette having an upper and lower graduation, holding the pipette so that it almost reaches the liquid in the flask. Put a funnel in the neck of the flask and heat on water bath for five minutes, shaking the flask occasionally. Cool to room temperature, wash funnel and sides of flask with recently boiled distilled water and titrate with normal sulfuric acid, using litmus indicator. Correct for the acidity of the sample and the hydrogen peroxide. When examining polymers use about 1 gram and heat on bath 5 minutes or until dissolved. This usually will not exceed the five minutes.

1 cc. normal NaOH consumed = 0.03002 gram formaldehyde.

The hydrogen peroxide methods have been recom-

mended as among the best for commercial solutions. The Blank and Finkenbeiner procedure usually gives results somewhat higher than the Haywood and Smith, however. The end point is fairly sharp.

The sulfite methods are based on the formation of the addition product, formaldehyde sodium bisulfite, when formaldehyde and sodium bisulfite are allowed to interact.

Seyewetz and Gibello's Method.—Prepare a 20 per cent solution of (dry basis) sodium sulfite and titrate 20 cc. to find the alkalinity. Dissolve 0.5 to 0.7 gram of the polymeric form in another 20 cc. of the solution in the cold and titrate with normal sulfuric acid to the disappearance of the red color. For the liquid form weigh about 2 cc. into a flask. Correct for the acidity of the sample and the alkalinity of the sodium sulfite solution. Use no more phenolphthalein than is necessary.

1 cc. normal acid = 0.03002 gram formaldehyde.

C. Kleber's Method.—Prepare a strong solution of commercial sodium bisulfite and add sodium hydroxide nearly CO₂ free, until the odor is destroyed. Dilute the solution so that 30 cc. equals 50 cc. normal sodium hydroxide, using phenolphthalein as indicator.

Use 5 cc. of formaldehyde solution, the specific gravity of which is known, and titrate with the bisulfite solution, using phenolphthalein until the red color just vanishes. Warm if necessary.

When examining polymers, warm about 2 grams in a little water and run in the bisulfite solution until the substance is entirely dissolved and the red color has disappeared.

1 cc. bisulfite solution = 0.05 gram formaldehyde.

These two sulfite methods are rapid, cheap and accurate. They approach the Haywood and Smith method quite closely. The Seyewetz and Gibello method resembles the Lemme method and the method of the 5th German Pharmacopoeia which have been highly recommended by Hampshire and Furnival,¹ Doby,² and Yamoto and Nakajima.³

The refractometric methods for formaldehyde solutions are not so accurate as the chemical methods on account of the interference of methyl alcohol and the polymers.

Richter and Jansen's Method.—The immersion refractometer should read 15.5 at 15° C. Make a one-to-one dilution of the formaldehyde solution with distilled water. Take the immersion refractometer reading of this solution at 15° C., subtract 15.5 from it, divide by 3.17 and multiply by two. This gives grams per 100 cc. Divide by the specific gravity to get per cent by weight.

B. Wagner's Table.—The immersion refractometer should read 15.0 at 17.5° C. Dilute as in preceding method. Take immersion reading at 17.5° C. Look up on table and obtain grams per 100 cc. Divide by specific gravity to obtain per cent by weight.

The dilution was necessary to get the reading within the scale of the instrument. The results were in good

¹ Hampshire and Furnival, *Pharm. J.*, **89**, 133 (1912).

² Doby, *Zeit. angew. Chem.*, **20**, 353-6 (March 1).

³ Yamoto and Nakajima, *J. Pharm. Soc. Japan*, **1910**, 871; through U. S. Hygienic Lab., *Bull.* **84**.

agreement among themselves. The result on the first sample was higher than the Blank and Finkenbeiner result, however.

No attempt was made to allow for errors introduced by the added substances, naphthalene, phenol, etc. The fats in sample L. 214 caused such variation in results that I did not complete the work with it.

The strength of solutions, the variation in measurement of volumes, the small amount of substance and the loss in weight during weighing tend to introduce inaccuracies. A difference of 0.1 cc. of normal solution means a difference of from 0.13 to 0.19 per cent in the result.

PERCENTAGE OF FORMALDEHYDE AS DETERMINED BY THE DIFFERENT METHODS

No.	Condition			Schiff and Malle				Ammonium sulfate		Blank and Finken'r		Haywood and Smith		Seyewetz and Gibello		C. Kleber		Richter and Wagner	
				Legler	Malle	sulfate	Finken'r	Smith	Gibello	C. Kleber	Wagner	Jansen							
Old stock	Liquid	Slightly polymerized		36.11	36.57	36.05	37.20	37.09	35.67	36.44	37.42	37.34							
X. 40	Liquid	Clear		36.30	37.98	37.95	38.31	37.70	37.50	37.90	37.83	37.65							
X. 43	Liquid	Clear		35.46	36.79	36.42	36.73	36.27	36.17	36.74	36.25	36.28							
X. 46	Liquid	Clear, greenish	Blk. sed.	36.88	37.16	37.16	37.67	37.02	36.84	37.45	37.60	37.52							
Trioxymethylene	Powder			90.10	94.81	94.52	95.55	95.42	94.14	94.43							
L. 208	Solid	Hard	Naphthalene	87.46	89.30	89.51	91.55	89.86	88.81	90.39							
L. 209	Solid	Soft, waxy	Phenol and glycerine	61.94	64.00	64.06	64.90	64.63	63.96	63.84							
L. 210	Solid	Soft, waxy		61.73	62.95	62.91	64.75	63.69	63.01	63.45							
L. 211	Semi-solid	Mushy	Much rust	32.61	...	32.87	32.73							
L. 212	Solid	Soft, waxy		62.78	62.93	63.16	64.43	63.45	63.08	62.97							
L. 213	Solid	Hard		91.09	93.32	93.81	95.71	94.41	94.29	94.37							
L. 214	Solid	Soft, greasy	Fats	46.32	40.53	45.12	45.71							
L. 215	Solid	Soft, waxy		64.73	66.32	...	66.64	66.46	66.51	66.59							

The end point with rosolic acid is difficult; the ammonia methods require standing for a considerable period of time and in the case of the Legler method the preparation of blanks is necessary.

The litmus end point is better than that of rosolic acid. The time required for the hydrogen peroxide methods is much less and the results are easily checked.

The end point in the sulfite processes is sharper than in either of the other types. The titrations are direct

instead of titrations of excess material. For formaldehyde solutions they require the minimum of time, but when polymers are to be titrated the Kleber method is the most rapid of all. Sulfite methods are, of course, not applicable to the polymers gamma and delta polyoxymethylene and alpha trioxymethylene as described by Auerbach and Barschall.¹

For an occasional analysis, the methods of Haywood and Smith and Seyewetz and Gibello seem to be preferable, while in a plant producing formaldehyde or where a large number of samples are to be analyzed, Kleber's method would be the most desirable.

Legler's method gives too low results and Blank and

Finkenbeiner's gave me the highest ones. Haywood and Smith's method gave slightly lower percentages than Blank and Finkenbeiner's, closely followed by Kleber's and the remaining methods.

My thanks are due to Mr. Rex Rudicel who assisted me in making the analyses.

CHEMICAL LABORATORY
CITY BOARD OF HEALTH
INDIANAPOLIS

LABORATORY AND PLANT

AN AUTOMATIC PIPETTE

By C. H. McCHARLES

Received July 7, 1913

A description of an automatic pipette was given in THIS JOURNAL by T. O. Smith (Vol. IV, p. 47). The apparatus described there has been used for a number of years in the laboratories of this station, but for a purpose quite different from that for which it was originally recommended. The lower end of tube C was placed above the zero mark on the burette and the tube C acted as a safeguard against overflow in case the three-way cock should be accidentally left partly open into A and B.

Smith's statement that "it is obvious that if the capillary tube is drawn out as fine as possible and the burette and container brought near the same level, the accuracy of the pipette is practically that of the burette" should be somewhat amplified. The automatic feature of this apparatus rests in the fact that liquid will rise in C to a point on a level with the surface of the liquid in A. This latter is not at all con-

stant in use, and its variation amounts to as much as six inches in smaller stock bottles and a foot in larger ones. If the tube C be drawn out so small that the volume of liquid contained in six inches to one foot of it shall be negligible in accurate pipette measurements, it would be so small that capillarity and cleanliness of its inner walls would influence the automatic level; and passage of air through it, especially when wet, would be altogether too slow for practical purposes.

The automatic pipette shown here in Fig. 1 differs from the above mentioned one only in that the point to which the liquid rises in C is constant. When liquid rises in D to the level H, the air supply to the syphon between A and B is closed and no more liquid enters D.

The dimensions given were found to be convenient, but the only required ones are:

1. E should be long enough to include the levels F and G as shown.

¹ Auerbach and Barschall, *Arb. Kais. Gesundheitsamt*, **27**, 183-230; *Chemical Abstracts*, **2**, 1125.

2. E should be of sufficient volume below G to prevent air from entering the syphon between A and E while filling the burette.
3. The side opening I prevents air bubbles from entering the syphon.
4. D should be of sufficient volume below H to prevent breaking the syphon between D and B. A four or six ounce salt-mouth bottle serves the purpose.
5. C must extend above H.

deterioration. This could be obtained from any glass-blower.

NUTRITION LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY

IMPROVED GOOCH CRUCIBLE HOLDER

By LORIN H. BAILEY

Received June 30, 1913

There have been developed in the Bureau of Chemistry several forms of Gooch crucible holders. Each form, while differing somewhat in detail from the others, has for its foundation principle the substitution of a solid rubber holder for the rubber-covered glass Gooch holder which has been in almost universal use. The particular form shown in the accompanying illustration has been in use in the Plant Chemistry Laboratory for about a year and has given good satisfaction. It has also met the approval of a number of visiting chemists.

This holder is made to fit an ordinary 2-inch funnel which is inserted in a one-hole rubber stopper in a regular suction flask. The upper edge of the holder projects over the edge of the funnel and thus makes the seal. The lower edge of the holder rests on the side of the funnel and supports the holder when the suction is on the flask. The opening is made to conform to the size of the 25 cc. porcelain Gooch crucible, and the lower surface of the crucible projects beyond the holder so that the filtrate does not come in contact with it. These holders can be cut from No. 12 rubber stoppers, which was at first done, or they can be obtained already molded from some of the large chemical-apparatus supply houses.

BUREAU OF CHEMISTRY
WASHINGTON

NOTE ON PRODUCER GAS ANALYSIS

By JAMES G. VAIL

Received June 26, 1913

In the analysis of producer gas with the Williams modification of the Orsat apparatus or with Morehead apparatus the residual gas after the absorption of CO_2 , illuminants, O and CO in the usual manner is often too lean to be exploded without the addition of hydrogen. As there may be a certain amount of inconvenience and some uncertainty as to the purity of the hydrogen, I have found the following procedure more satisfactory for enriching the gas to a point where it may readily be made to explode. After the usual absorptions CO_2 , illuminants, O, CO, the residual gas is passed into the CO_2 absorption pipette or into the reservoir provided for the purpose in the Morehead apparatus and the measuring burette filled with water (acidified with sulfuric acid) which has previously been cooled to room temperature. A current of 6 or 8 volts passed between the electrodes used for the explosion will quickly produce enough of

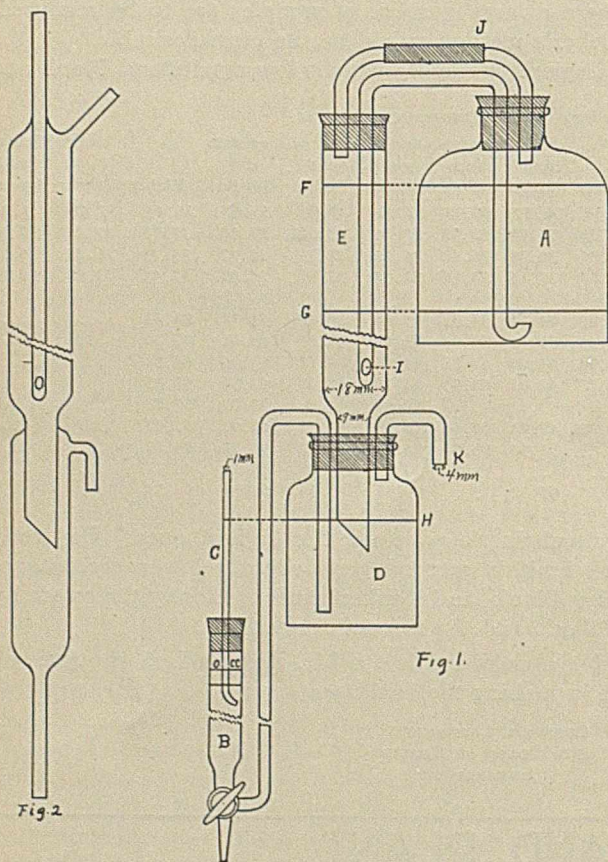


Fig. 1 represents an arrangement for use of liquids which must not be permitted to come in contact with rubber. A silver stopcock permits the use of lye. When this precaution is not necessary the container A may be stoppered with a one-hole stopper bearing a glass tube corresponding to the lower end of E, which in turn is inserted in D inverted. A and D are now inverted and placed on a support and B connected as in Fig. 1. This eliminates E and its connections.

In order to fill the syphon shown in Fig. 1, the air passage is disconnected at J and pinched. Apply suction to a rubber tube connected to K, and bearing a pinch-cock, till D is filled. Close the pinch-cock, connect J, fill B by suction applied to C, and remove the rubber tube from K.

By regulating the height of the lower end of C, the burette may be adjusted to deliver any desired volume.

Fig. 2 represents a form of apparatus which would eliminate two rubber stoppers and all inconvenience arising from the leaks which they develop upon

a mixture of hydrogen and oxygen in molecular proportions to sufficiently enrich the gas. This volume is read off and a sample added and the whole diluted with air and, after thorough mixture, exploded in the usual manner.

This simplifies the calculation of results as the volume

of hydrogen-oxygen mixture added may be subtracted from the total contraction due to explosion and the H and CH₄ and N calculated as though it had not been necessary to enrich the gas.

CHESTER, PA.

ADDRESSES

THE CHEMICAL INDUSTRIES AND THE UNIVERSITIES¹

By MARSTON TAYLOR BOGERT

Before this audience it is wholly unnecessary to dwell upon the tremendous importance of chemistry to the material growth and prosperity of the nation, or its vital bearing upon the health and happiness of the individual. Suffice it to point out that the amount of a country's appreciation of the value of chemistry in its development and the extent to which it fosters the manifold applications of this science in its every-day life is generally a measure not only of the industrial progress and supremacy of that country, but also of its real civilization.

Two of the most important factors in the advancement of chemistry are the universities and the chemical industries, and it is consequently a matter of concern to all of us whether they are working together at present to the best advantage or not. My own observation leads me to believe that they are not, and this address is intended primarily to indicate some of the lines along which it seems to me there might be much closer coöperation.

As the majority of my fellow-members are themselves manufacturers, or are connected in one way or another with our chemical industries, I shall endeavor to present the case principally from the university side, and I trust that what I shall have to say will be judged from this point of view.

Robinson has defined education as "the process of fitting the individual to take his place and do his part in the life of his age and nation," and no educational institution at the present day can discharge its responsibilities faithfully unless it accords, in its equipment and in its curricula, adequate recognition to so comprehensive a science as chemistry, which in its wide sweep, in one way or another, touches almost every phase of human life and endeavor. That our universities are realizing this more clearly every day seems evidenced by the frequent construction of new laboratories and the steadily increasing size of the chemical staff. It is worth noting that, almost without exception, additional laboratory accommodations are immediately taken up by an increased number of students. As indicative of the extent to which chemistry enters into our modern university curricula, I may be permitted to refer to my own Alma Mater, Columbia University, where this subject is taught under seven of the eleven constituent faculties; and, as proof of the demand for higher education in this same field, I would call attention to the doctorates conferred in 1911 (the latest compilation I have seen; *Science*, N. S., 36, 129, Aug. 2/12) by the leading universities of the United States, when more than twice as many were awarded in chemistry as in any other subject (78 out of a total of 492), the nearest competitor being English (with 34).

The universities are active in everything which concerns the life and welfare of the nation, and it can be truly said of them, in the words of Terence, "humani nil a me alienum puto." So far as the chemical industries are concerned, it should not be overlooked that the universities, in addition to training chemists, also educate men for the non-chemical positions, whether clerical, financial, or commercial; and that everything which they accom-

plish to increase the security of the rights of the individual or of property, or which ministers to the general prosperity, comfort and happiness of the nation, is obviously of direct advantage to the chemical manufacturer as well, and in all such matters therefore the universities have the right to expect cordial and active coöperation on the part of our chemical industries.

In the training of men for the chemical profession we are all immediately interested, and in this direction the universities are doing the best they can with staff and equipment available and with the raw material represented by the student body. Equipment and staff are increased as funds are provided and entrance and graduation requirements steadily advance.

The chemical industries can and do coöperate by giving employment to properly trained and qualified graduates; and higher training, as represented for example by a higher degree should, other things being at all approximately equal, secure for a man a higher initial salary. It goes without saying that the salary should in any event be a fair one, commensurate with the service the man is expected to render, and with good chance of promotion for demonstrated efficiency and ability. In many cases, chemists employed by manufacturing concerns are required to execute iron-clad contracts by which they definitely waive all patent rights to anything they may discover while in the employ of the company. These discoveries then become the property of the employer, who is under no legal obligation to remunerate his chemist therefor. Employers, however, who are really desirous of assisting the universities in their work of training chemists, will see to it that their chemists are well rewarded for all discoveries of value.

Many of our leading chemical manufacturers have coöperated with the universities very helpfully with advice and suggestions in arranging the most effective curriculum for the training of the industrial or engineering chemist. But the curriculum is not everything, and even with the best one possible it is not an easy matter to turn out men who, in addition to adequate scientific training, are clear-headed and masterful, honorable and dependable, sober and of high moral tone, of broad and creative scholarship and conspicuous inventive ability, of unwearying energy, and with the necessary health and physique to stand plenty of hard work. Of course, such paragons are scarce and much sought for in all lines of human activity. Nor is it ordinarily feasible for a university to turn out men who are experts in the manufacturing details of the various lines of chemical industry. The best they can do is to give their men the necessary broad, theoretical and practical foundation upon which all lines of chemical manufacturing rest. Munitiae can be learned only in special schools or, preferably, in the plant itself, and manufacturers can help here by giving a good man a chance to learn these details. Many a high-class university graduate has lost his first position because of his employer's impatience in this respect. And yet, if the manufacturer but knew it, he is often throwing away gold and accepting dross in exchange. The properly trained chemical graduate is, in the vast majority of cases, a far more valuable man to a chemical concern than the employee who knows only mechanical details and has not had the benefit of any real scientific education. At first the latter may appear to greater advantage because of his familiarity with

¹ President's address at the thirty-second annual general meeting of the Society of Chemical Industry, Liverpool, England, July 16, 1913. Reproduced from the *Journal of the Society of Chemical Industry*, 14, 720.

the processes involved, but he will be very speedily outstripped by the university man given at all a similar initial endowment. One is likely to be a mere rule-of-thumb man, whose actions are determined wholly by previous experience and who is completely lost when anything new and totally unexpected occurs. The other is the more resourceful, and will seek the underlying cause of the difficulty and independently endeavor to work out a remedy. Each has his place in our industrial life, but the scientifically trained man has surely the brighter and more promising future before him, and will rise to higher levels of usefulness.

On the other hand, university teachers often appear insufficiently posted on the type of men needed in chemical industry, and ignorant of the fact that a man admirably suited for the research laboratory is not necessarily equally well fitted for the career of an engineering chemist or works manager. To my mind, no greater unkindness can be done to a student than to secure for him a position for which he is not properly qualified, only to see him struggle along through the best years of his life and awaken finally, perhaps too late to begin another career, to a keenly painful realization of the fact that he is but a square peg in a round hole. It is a heavy burden of responsibility, and one not to be shirked or avoided, this advising of students about to embark upon the uncertain sea of life's work for which port to steer, but it is also a splendid opportunity for high personal and public service and should be so esteemed. Few talents are more valuable to the teacher than the ability to gauge accurately a student's character and capabilities, and this genius is likewise potent in the business world, for I have heard more than one great captain of industry say that he owed his success to his knowledge of men more than to any other one thing. Employers are entitled to a perfectly frank and accurate statement concerning an applicant's qualifications—a statement which sets forth the candidate's weak points as well as his strong ones, and it does not take them very long to find out from whom they may expect such statements.

But I would not have you overlook, nor do I intend to dismiss quite so briefly, the importance of manual training, for it is a subject very much to the fore with us in the United States just now and of vital concern to all manufacturers. Every factory, in addition to capable management, up-to-date mechanical equipment and suitable research laboratories, must have in its employees technical skill equal to that of its competitors, domestic and foreign, or it cannot hope to win in the struggle. To obtain in our workmen this high degree of manual dexterity and industrial efficiency, there is need for active coöperation between our educational institutions, our factories and our labor unions. It is far better to market the results of high-grade labor than merely to realize on raw material. To illustrate the point, let me quote from a recent report of a special committee of the National Manufacturers' Association of the United States, citing conditions now obtaining there:

"We sell our cotton to Switzerland at 14 cents a pound, with scarce any labor in it. We buy it back in the form of fine handkerchiefs at \$40 a pound, all labor. We export bar iron and import razor blades; export hides and import gloves; export copper and import art bronzes." In the year 1911, 56 per cent of the total exports of manufactured articles from the United States carried only from 3 to 15 per cent of factory labor.

Perhaps the greatest opportunity of all for coöperation between the universities and the chemical industries, and the one where in many countries such coöperation is conspicuously lacking, is in extending the boundaries of human knowledge by original investigation and research. Here, too, the universities and technical schools are doing what they can, with insufficient funds and equipment, and with an overburdened staff, to serve the community and the chemical industries, both by conducting original research themselves, and by throwing open their great libraries and occasionally certain of their laboratories for the

solution of problems of financial value to the manufacturer. The latter, in return, can assist by supplying raw material, additional funds, and knowledge gained in the laboratories of the plant.

When a chemical industry has problems to be solved, these problems can be attacked either inside or outside of the plant. If the policy of the management is that all chemical problems are to be studied only within the establishment, a research laboratory or at least a research chemist must be provided for the plant or for the company. At present, in the United States, probably not more than 75 or 100 manufacturing establishments have research laboratories or employ research chemists. In Germany, and perhaps also here in England, such research laboratories in association with chemical industries are much more common. The great laboratories of the Badische Anilin und Soda Fabrik and of the Elberfeld Company are good examples of the importance attached to such research work in Germany, and it would be difficult to adduce any stronger argument in support of its value than the marvellous achievements of these huge concerns.

A frequent difficulty encountered in the employment of research chemists or the establishment of a research laboratory is that many a manufacturer does not appear to grasp the need or importance of such work, nor know how to treat the men in charge so as to secure the best results. The owner may not even fully understand just what is the cause of his manufacturing losses or to whom to turn for aid. If he finally engages a chemist he is apt to look upon him as a sort of necromancer who should be able to accomplish wonders, and if he cannot see results in the course of a few months is likely to consider the investment a bad one and regard chemists as a class as a poor and useless lot. It is not unusual for the chemist to be told to stick to his laboratory and not go prowling about the works, and he must also face the natural opposition of workmen to any innovations and reckon with the jealousies of foremen and of various superior officials so that, like the policemen in the "Pirates of Penzance," often his "lot is not a happy one."

From the standpoint of the manufacturer, one weighty advantage of the policy of having all problems worked out within the plant is that the results secured are not divulged, but are stored away in the laboratory archives and become part of the assets and working capital of the corporation which has paid for them; and it is usually not until patent applications are filed that this knowledge, generally only partially and imperfectly, is made known to the public. When it is not deemed necessary to take out patents, such knowledge remains buried.

In this matter of the dissemination of knowledge concerning industrial chemical practice, it must be evident to all that there is but little coöperation between the manufacturers and the universities. The crux of the difficulty lies just here. Chemical manufacturers, as just stated, are quite naturally averse to publishing any discoveries made in their plants, since "knowledge is power" in manufacturing as elsewhere, and new knowledge gained in the laboratories of the corporation may often very properly be regarded as among the most valuable assets of the concern. The universities, on the other hand, exist for the spreading of knowledge, and from their standpoint the great disadvantage of the above policy is this locking up of knowledge, for it effects a serious retardation of the general growth and development of the science in its broader aspects, and renders it much more difficult for the universities to train men properly for such industries, since all textbooks and general knowledge available would, in all probability, be far behind the actual manufacturing practice. I believe that all familiar with the situation will admit that this is a real problem and one constituting a serious handicap to those endeavoring to give up-to-date instruction in industrial chemistry. One immediately harmful effect that the above policy has upon the universities

is that it not infrequently results in some of their most capable investigators spending many of the best years of their lives and considerable sums of money in attempts to solve problems, the clues or answers to which had been discovered and filed away long before in the laboratory records of the factories.

Of course, the universities must also contend with the fact that the larger manufacturing establishments can usually offer much better salaries to skilful chemical investigators, and thus withdraw from university work some of the best men.

All who have had any experience in such matters know how difficult it is to secure papers for the programs of our Sections, or for our Journal, from men engaged in industrial chemistry, the usual reply the manufacturer gives to such solicitations being to the effect that he is in business to make money and is not running an educational institution.

It is not to be expected that the manufacturers will promptly publish to the world, upon demand, their most cherished "trade secrets," and it would of course be useless to make any such request. But in almost all great chemical industrial establishments which have been in operation for any length of time, information has been accumulated in the course of the work which could be made known without injury to the firm concerned, and to the considerable advantage of the chemical world-at-large. For example, it not infrequently happens that a corporation permanently abandons one line of work, either because local conditions have made it no longer profitable or because it wishes to devote itself to other more lucrative ones. In such cases there seems no good reason why the knowledge gained in connection with the abandoned process or product should not be made public. Or, it may be, the analytical laboratory finds some convenient rapid method to take the place of a more laborious one, or devises new apparatus for old laboratory processes. Other cases might be cited were there time.

A very welcome form of coöperation, therefore, would be that of keeping the university teachers better posted on present-day manufacturing practice, so that they could point out more clearly the applications to industry of the principles they were endeavoring to inculcate, and call attention to the rapid approximate commercial analytical processes and short cuts used in the factories in addition to the longer and more accurate methods generally taught in analytical laboratories. Were the teacher more familiar with industrial wastes and by-products, he could often select problems for his advanced research students with reference thereto and thus considerably heighten their interest in the work.

It is well said in the Reports of the Twelfth Census of the United States that "Probably no science has done so much as chemistry in revealing the hidden possibilities of the wastes and by-products in manufactures. This science has been the most fruitful agent in the conversion of the refuse of manufacturing operations into products of industrial value. . . . Chemistry is the intelligence department of industry." Yet the teacher is often uninformed concerning the character and amount of the by-products going to waste in his immediate neighborhood, a careful study of which might lead not only to financial reward for the manufacturer as well as for himself, but might also save us much of the present pollution of our streams and of the air we breathe.

It is not only now very desirable, but will soon become imperative for our manufacturers to avail themselves more freely of the assistance of the experts in our universities and technical schools. That nation will unquestionably make the longest strides in commercial development which sells goods carrying the maximum of brains and the minimum of raw material. Germany has already learned this, and is rapidly forging ahead as the natural result. England and America must follow that lead or ultimately find themselves far in the rear. Any country, like my own, thanks to a wonderful endowment of natural re-

sources, and to protective tariff walls, may, for a time, appear to be holding its own in the world's trade without adopting the above policy; but it is only because of these peculiar local conditions that we have been spared the embarrassment of close international comparisons. Our tariff walls are already tottering, and the lavish use of natural resources is but a spendthrift squandering of a priceless and irreplaceable heritage, and the end of that, too, is in sight with a continuance of present methods. The day is already at hand, for America at least, when manufacturers must scrutinize more closely the efficiency of their plants with reference to raw material consumed, for human intelligence is the one resource which increases with use, forever and immeasurably, and the cultivation of which furnishes the only substantial basis of prosperity, progress and happiness.

In the United States we have been wont to boast of our huge agricultural exports, but, as President Wallace said at a recent Conservation Congress, "the nineteenth century farmer was no farmer at all; he was a miner, mining the fertility of the soil, and selling it for the bare cost of the mining;" since every bushel of wheat exported carries with it approximately 27 cents worth of phosphorus, every bushel of corn 13 cents, every pound of cotton 3 cents, figures which equal the supposed profits of the transaction. How we are to replenish our stock of phosphorus, if once exhausted, is not yet clear, although, so far as we now know, the race itself must perish without an adequate supply of this element.

There are but four great manufacturing nations in the world—England, Germany, France, and the United States. Outside these four, and looking to them for their manufactured articles, stand the one and a half billion human beings in the rest of the world. The prizes to be won in this international trade are beyond compare. They are to be measured not only in money, but also in intellectual advancement, in closer bonds of international friendship, mutual respect and esteem, in national spirit, and in heightened civilization.

Any manufacturer who feels unable to coöperate as suggested above, should at least be willing to support generously those agencies upon whom now falls the chief burden of disseminating knowledge—notably our educational institutions and our chemical journals, such as the Journal of this Society.

Or, if he still demurs, there are other lines open. Few manufacturing concerns feel inclined or called upon to establish extensive libraries within their plants, and it would appear, therefore, to be to their own immediate interest to provide whatever funds might be needed to supply the library of the nearest university or technical school with all books likely to be of service to the various experts of the plant. This also would bring them into closer personal touch and sympathy with the officers of instruction there.

Any manufacturer having an important chemical problem to solve in connection with his business, and without the proper staff or equipment to attack it within the plant, must look outside for assistance, and he then has the choice of the private commercial or research laboratory on the one hand, or the universities and technical schools on the other.

The advantages and disadvantages of the private research laboratory, at least in so far as they relate to the availability of the new knowledge thus discovered, are much the same as those already pointed out for the factory research laboratory.

If the manufacturer elects to refer his problem to the university or technical school, such reference is most likely to take the form of an industrial fellowship and there is much to be said in favor of these fellowships. They allow the donor to keep secret for two or three years the results secured, after which they can be published. They also secure to him patent rights. They give highly specialized training to good men and often secure for them permanent employment and a share in the profits of their discoveries. It should be obvious at the outset that a

fellowship of this character can be successful only when there are close confidential relations subsisting between the manufacturer and the officer in charge of the research. No coöperation can be really effective unless based upon a thorough mutual familiarity with conditions and an abiding faith in the integrity and sincerity of purpose of each other. It is likely to prove a poor investment for a manufacturer to seek the aid of an investigator if he is unwilling to take such expert into his confidence. I have occasionally had manufacturers apply to me for the solution of a problem affecting their industry and, when I asked how they were actually carrying out the process in the factory, have had them say politely that they preferred not to answer any questions concerning the operations of their factory and that all I was asked to do was to see what I could accomplish in my own laboratory. Of course the answer to such a proposition should be a decided negative, since its acceptance would almost invariably result to the mutual dissatisfaction of both parties concerned.

It would seem manifest that no one man is competent to direct original research in all lines of industrial chemistry, and that the wisest course for a manufacturer to pursue when contemplating the establishment of a research fellowship would be to secure the aid of the particular university officer whose experience and personal achievements entitle him to be considered an expert in that special field, and who has the necessary equipment and location to attack such a problem with good chances of success, and then to arrange to provide him with such funds as may be required for assistants or for raw material.

In the case of divergent reports from chemical experts, the university laboratory is the natural court of appeal for an impartial verdict.

The most urgent need of the day is, in my judgment, the endowment of chemical research, either by the establishment of research professorships or, still better, by founding great chemical research institutes in affiliation with our chief universities for the study of problems both in pure and in applied chemistry. The splendid Kaiser Wilhelm Institute for Chemistry, recently opened in Berlin, thanks to the active and generous assistance of His Majesty the German Emperor, is the first of its kind in the world, and its advent should be hailed with delight as indicating the dawn of a new and brilliant era for our science. It is worth noting that the policy of this research institute is the man first, the equipment afterwards, and the group of distinguished investigators now gathered together there witnesses to the wisdom of this plan. If we would not be outstripped by our friends across the water, we must have similar establishments here in England and that, too, speedily. The benefits which will accrue to the nation from the labors of such research institutes manned by our keenest investigators are truly inestimable, and it is to be hoped that this remarkable opportunity will appeal to the imagination and the generosity of other far-sighted benefactors of mankind. Much has been done, and worthily done, in all of our great cities for the cause of medicine, for example, whereas chemistry, except in Germany, still appeals in vain for similar recognition. Yet the dependence of medicine, and of life itself, upon chemistry is seen more clearly every day, and he who now still has doubts upon the subject has but to read the recent remarkable presidential address of Dr. Schäfer before the Dundee meeting of the British Association for the Advancement of Science (1912), in which, after calling attention to the few elements and simple inorganic compounds making up living substance, he says: "The combination of these elements into a colloidal compound represent the chemical basis of life; and when the chemist succeeds in building up this compound, it will, without doubt, be found to exhibit the phenomena which we are in the habit of associating with the term 'life.'" He further suggests the possibility "that heredity also is one of the questions, the eventual solution of which we must look to the chemist

to provide," and, as already indicated, the maintenance of any human life whatever upon this planet of ours depends absolutely upon the labors of the chemist.

In the sphere of industrial chemistry such a research institute might well take the form of laboratories "especially equipped with the standard appliances for executing industrial operations, and provided with all the means for the prompt, proper and accurate determination of experimental data, in a way and on a scale which would make this data available for factory application," as has been suggested already by Professor Whitaker, the Chairman of our New York Section (*THIS JOURNAL*, 3, 797), and since, as he points out, few if any laboratories now exist where researches can be undertaken "which involve single and multiple effect distillation, evaporation, filtration, calcination, condensation, absorption, drying, controlled temperature reactions, vacuum and special atmosphere reactions," and the like.

The really important thing is to bring together the problem and the man competent to solve it. Skilful investigators are often wholly unaware of many of the important industrial problems calling for solution, and the great manufacturing concerns are often equally ignorant concerning the men in the country best qualified to solve these problems. If it were possible to establish some sort of a clearing-house committee, composed of representatives of the universities and of the manufacturers to bring investigator and problem together, it could render immense service to the country. If the research institutes proposed above were established, their Boards of Direction might perhaps perform such a function. In fact, the matter is so important that there would seem ample justification for the general Government itself to take cognizance of it, since it concerns the prosperity of the whole nation, and refer it to an appropriate committee or board, in conjunction with its other activities for the benefit of our manufacturing industries.

Some of our wisest and most far-sighted manufacturers feel that there is urgent need for what might be termed an Industrial Efficiency Corporation, with a staff of experts in all lines of chemical manufacturing, to whom any manufacturer might refer for solution all problems affecting his plant. If the details of such an organization can be satisfactorily worked out, this corporation can render much of the service indicated above, but it too will have to overcome the reluctance of many manufacturers to allow any other man, or group of men, to know what they are actually doing in their factories.

There is room also for better coöperation in the matter of patents. The university teacher is usually at a heavy disadvantage in endeavoring to obtain a fair trial of his inventions and a reasonable share in the profits. In this connection the establishment here of the Letters Patent Insurance Co., Ltd., and in the United States of the Research Corporation, are of interest.

The latter is described as "an experiment in the public administration of patent rights" (F. G. Cottrell, *THIS JOURNAL*, 4, 864). It was incorporated February 26, 1912, as a stock company, under the laws of the State of New York, with offices at 63 Wall Street, New York City, its declared purposes being:

(a) To receive by gift and to acquire by purchase or otherwise, inventions, patent rights and letters patent either of the United States or of foreign countries, and to hold, manage, use, develop, manufacture, instal, and operate the same, and to conduct commercial operations under or in connection with the development of such inventions, patent rights and letters patent, and to sell, license, or otherwise dispose of the same, and to collect royalties thereon, and to experiment with and test the validity and value thereof, and to render the same more available and effective in the useful arts and manufactures and for scientific purposes and otherwise.

(b) To provide means for the advancement and extension of

technical and scientific investigation, research and experimentation, by contributing the net earnings of the Corporation, over and above such sum or sums as may be reserved or retained and held as an endowment fund or working capital, and also such other moneys and property belonging to the Corporation as the Board of Directors shall, from time to time, deem proper, to the Smithsonian Institution, and such other scientific and educational institutions and societies as the Board of Directors may, from time to time, select in order to enable such institutions and societies to conduct such investigation, research and experimentation.

(c) To receive, hold and manage, and dispose of such other moneys and property, including the stock of this and of any other corporation, as may, from time to time, be given to or acquired by this Corporation in the furtherance of its corporate purposes, and to apply the same, and the proceeds or income thereof, to the objects specified in the preceding paragraph."

To summarize briefly, the purposes of the Corporation are to control patents for the benefit of the public, and to aid research.

It is expected that the duties of the Corporation will be largely administrative, the actual practical work in connection with the patents being conducted either in coöperation with the manufacturing plants or with the universities and technical schools. These administrative expenses for the first year are estimated at \$10,000, and have been provided for by subscriptions to the stock of the Corporation, which stock it is expressly stipulated shall pay no dividends whatever, but may be repurchased at any time at par by the Board of Directors, and no stock can be disposed of without first giving the Board an opportunity to exercise this option. After the first year, it is expected that the undertaking will be self-supporting.

It appears to be the intention of the Corporation for the first few years to consider only such patents as may be offered as outright gifts, and Dr. Cottrell, in the article cited, says, "present indications are that the latter (the Corporation) will find itself well occupied even by these offers alone." In other cases, patentees may be willing, it is hoped, to assign to the Corporation their patent rights within certain geographical boundaries, retaining the rest for themselves; or perhaps present to the Corporation the rights to use the patents in connection with certain lines of manufacturing and retain for themselves the rights for other lines.

The real actuating cause in the establishment of this Corporation has been the fine self-sacrificing generosity of Dr. Cottrell himself in turning over his own valuable patents on the electrostatic collection of smoke and fumes, thus voluntarily relinquishing what promises to be large profits. Too much cannot be said in praise of such a splendid example of altruism, and it is to be hoped that many others may be found to follow this example of real patriotism. It has been urged that had such an organization been in existence at the time the Diamond Match Company generously threw open to all its sesquisulfide patents, it might have been willing to turn them over to this Research Corporation for the benefit of chemical investigation.

The experiment is an exceedingly interesting one and surely we all wish it the greatest measure of success.

True, in the United States (Patent Act of March 3rd, 1883) and perhaps also here in England, patents may be formally issued as free to the public without royalty of any kind, and for these patents the Government collects no fees. One reason why such regulations have not accomplished as much as their advocates hoped for is that they fail to provide adequate protection to the manufacturer who invests the large sums necessary in plant, equipment and advertising, to market successfully a new article.

There is also need for coöperation in some countries in securing an efficient and equitable system of patent laws, and in obtaining fair and impartial legislation where our chemical industries are

concerned; coöperation which, on the one hand, shall protect our manufacturers from unjust laws or regulations and which, on the other hand, shall effectually safeguard the community against any inroads upon their rights by the manufacturers.

For example, a Government might propose legislation classing as adulterated any foodstuff containing 10 per cent of arsenic. If the university experts knew that such a percentage of arsenic was normally present in certain perfectly wholesome and unadulterated natural foods, it would be their duty to call the attention of the law-making body to the fact, that the proposed legislation might be amended accordingly.

To take the other side: if it appeared that any line of industry, by combinations at home and the erection of a tariff wall against imported articles, was effectually stifling all competition in order that it might continue old-fashioned, wasteful and inefficient methods of manufacture, and charge the public high prices for their goods, that public is entitled to redress and will surely obtain it sooner or later, and it would be the duty of the university officers to make clear to the community the extent to which they were being imposed upon by the offending manufacturers.

In the building up at our larger universities of museums illustrating the development and present status of industrial chemistry, there is afforded a field for coöperation not yet very extensively made use of. The exhibits themselves must, in the nature of the case, be contributed chiefly by the manufacturers, and should include full sets of samples of all products—initial, intermediate and final—as well as working models of the mechanical parts of the process, so that the student may see represented before him all the various steps in the manufacture: the university, on its part, to provide suitable housing and display cases, to look after the proper assorting, arrangement, distribution and labelling of the material, and the necessary care and upkeep of the same. What such collections mean to the teacher of chemistry, no one appreciates better than the Columbia University man, for there we have had for more than a generation the extensive Chandler Museum of Chemistry, contributed largely by our chemical manufacturers, and gathered together patiently and with tireless energy and enthusiasm by that Nestor of American chemists, the first American President of this Society, our own dear Dr. Chandler.

Such museums are not only for the benefit of the university but are for public exhibition as well, and in addition to these university museums, or replacing them, are the great industrial museums of the world like the Deutsches Museum of Munich and the South Kensington of London, and it is a genuine inspiration to see these places thronged with young and old watching closely the operation of the working models exhibited, examining critically the various products, or listening to the explanatory remarks of an accompanying teacher or of an attendant. But we need to add to the equipment of those museums we now have and to assist in the creation of others, for they illustrate most graphically the commercial and industrial resources of the country and provide a telling object lesson of the significance of chemistry in the utilization and development of those resources.

When the layman sees before him a barrel of coal tar, it is to him only a black, evil-smelling viscous mass; but when he sees growing out of that barrel, a tree-like arrangement of the innumerable substances obtainable therefrom—first the primary products, then the secondary, and so on, until at the ends of the branches he surveys all the wonderful dyestuffs, drugs and perfumes manufactured from this same foul mass—it takes on for him a wholly new aspect and interest.

A block of wood is a very commonplace article, but when that block of wood stands side by side with some of the beautiful and useful products which chemistry can conjure therefrom, the observer begins to have some inkling of the magic of science and its potent influence in the progress of civilization.

Lest I weary you, I will but touch upon one other opportunity for coöperation, and that is in the organization of chemists' clubs, where the members of the profession may regularly foregather and the university man rub shoulders with his manufacturing brother. A chemists' club is a most excellent reagent for blending together in one homogeneous, harmonious whole all brands of chemists, and it is quite true that a happy union of theoretical and practical chemists can often be accomplished most pleasantly by clubbing them into it. Our Chemists' Club in New York has brought together, in one big contented family, all the chemists of that metropolitan district, and its influence has extended over the length and breadth of the United States. After all, friendship is the main thing between brother chemists, and I had rather have a colleague's intimate and warm-hearted friendship for me personally than merely his respect and admiration for my scientific achievements.

To recapitulate briefly, the university exists for the community, and its supreme desire is to render ever better and more efficient service. It is the servant of our chemical industries and their agent in the training of the men needed by these industries. It seeks closer coöperation from them in disseminating knowledge and in extending its boundaries, in endowing chemical research and chemical publications, in aiding our chemical libraries, in the matter of patents and legislation, in the creation of great museums of peaceful arts, in the organization of chemists' clubs and the provision of suitable buildings therefor, and in countless other ways, and if this short and very imperfect presentation of the subject serves in any way to arouse increased interest in these matters, it will have been worth while. Efficiency is the only sure path to supremacy in any field of human endeavor, and closer coöperation between our universities and our industries will go far to assure increased efficiency to both.

COLUMBIA UNIVERSITY
NEW YORK CITY

DEPRECIATION AND OBSOLESCENCE¹

By RICHARD K. MEADE

The capital invested in any manufacturing enterprise may be appropriately distributed among the following items:

- (1) Plant.
- (2) Working capital.
- (3) Good will and patent rights.
- (4) Laboratory and office buildings and their equipment.
- (5) Mill site, real estate, mines, quarries, etc.
- (6) The cost of financing.

Of these various items, a few may be expected to increase in value as time goes on without any special provision other than that ordinarily attended with successful operation of the business. Other items become less even under the most careful management. "Good will" of course increases in value under successful management. The working capital may or may not be increased according to the policy of those at the head of the organization. The real estate will increase or decrease in value according to local conditions. The value of patent-rights grows less as the time of the monopoly granted under them diminishes. On the other hand, where processes are employed under a royalty, the expiration of the life of the patent will allow these to be used without such expense. The deposits of raw material, of course, decrease in value as they are drawn on to supply the plant. That portion of the capital which was devoted to raising the rest represents nothing of actual tangible value, although it is something with which practically all manufacturing enterprises have to reckon.

The value of the plant unquestionably gets less as time passes and the only way in which it can be maintained at anything like its original value, is by continually repairing the machinery and apparatus and by replacing worn-out parts by new ones. Even

¹ Paper read at the Boston meeting of the American Institute of Chemical Engineers, June 27, 1913.

when this is done, however, there comes a time when such replacement of parts will not be sufficient to keep the plant operating economically either because the cost of the repairs themselves is too great or else because the plant has reached that point of general dissolution when the breakdowns are so continual that the loss of time and output occasioned thereby is too great a handicap to be overcome.

In every plant the expense of keeping the machinery in satisfactory working condition is taken care of by the current expenses, but very few plants have made any provision against the time when the entire plant must be rebuilt. Unquestionably, such a fund should be provided, or, in other words, if the plant is to be self-perpetuating, a fund should be set aside during the life of the plant for its replacement when it goes out of commission.

Most manufacturing enterprises frequently meet with extraordinary expenses, due to the breaking down of individual units. These breakdowns may come within a year or so of the starting up of a new plant, whereas the actual life of the plant itself may be many times this. If these expenses are allowed to come in the ordinary monthly cost sheets, these latter will show irregularities and will not serve so well as an indication of the operation of the plant during the period in which they occur; consequently it is desirable to have a certain fund available which can be used for such unit renewals.

These two funds, one for replacing units as they go out of commission and the other for rebuilding the entire plant, constitute the depreciation account. The former may be termed "current depreciation" and the latter "plant renewal depreciation." The former is to be used for the replacement of any individual units and the latter for the replacement of the entire plant.

To draw a parallel from the cement industry, let us take, for example, a cement kiln: this is a long, horizontal cylinder, revolving slowly on rollers and heated by a jet of pulverized coal or oil so that a temperature is maintained in it of about 2500° F. The lower part of the lining is subjected to the scoriifying action of the charge, and this part needs to be replaced about once a year. The cylinder itself, however, will last at least fifteen to twenty years. The replacement of the lining of the kiln should be taken care of by the current depreciation. There comes a time, however, when the outside shell of the kiln becomes worthless and when the entire apparatus must be replaced, and for this purpose we should be able to draw on a plant renewal fund. The proper plan, therefore, would be to estimate the average cost per year of repairing the lining and set aside a sum sufficient to cover this, crediting this sum to the current depreciation account. At the same time the life of the shell should be calculated and a sum sufficient to cover the replacement of this at the end of its usefulness should be set aside and credited to the plant renewal fund. In the purely chemical industries, the lead chamber offers an illustration of a piece of plant apparatus which must be replaced some day practically *in toto*.

Sometimes a machine is renewed part by part, as these wear out, so that at the end of a period of years there is probably, in its make-up, not one part originally present in the machine when it was first put on its foundations. Certain types of pulverizing machinery offer a good illustration of this. For example, with mills of the type of the Griffin and the Fuller-Lehigh mills, which are subject to constant repair, it is a fact that any of these mills which have been properly kept in repairs will be practically as good at the end of ten years' continuous operation as they were on the day on which they were installed. On the other hand, with the tube mill, for example, the cost of repairs is much lighter, but there will come a day when the entire mill must be replaced and the cost of such replacement will practically represent the cost of a new tube mill.

Depreciation of the value of a machine may also be caused

by the working out of other and more improved machines, by the discovery of a more economical process for the working out of the finished product from the raw materials in which this machine is not used, or even by the demand of the trade for a product that the original machine is not capable of producing. Such depreciation is now generally considered as a separate and distinct item known as "Obsolescence."

Where enlargement of the plant is made necessary by an increase in the demand of the product, this can be properly financed by increasing the capital if necessary. Such enlargements are in no way concerned with depreciation because for depreciation the original plant must actually depreciate in value. When new apparatus taking the place of the old has a greater capacity than the original apparatus part of the value of the former may properly be added to the capital account, but a part of it should certainly be taken care of by depreciation.

At the present time, competition in practically all industries is much keener than it was ten years ago and profits are much less. The introduction of the cost system has enabled manufacturers to determine accurately the factory cost of producing their goods, and many large manufacturers have been willing to undertake business using this system as a basis and figuring from this the prices of their product, allowing a fair return on the capital invested. As a result of this blind following of the cost system and the failure to take into consideration depreciation, etc., prices have often been figured far too low. It can be safely said that one of the first effects of keen competition has been to make manufacturers rely too closely upon the cost system in fixing prices. Ordinarily installed as a method of furthering the economic operation of the plant, it has of late years become the basis of prices themselves. Of course, the mill cost is an item in fixing the price at which a product may be sold but if a manufacturer considers only the cost of labor, fuel and supplies in making his prices he will sooner or later come to grief.

In many concerns, the failure to carry a depreciation account and the distribution in dividends to stockholders of money which should have been set aside for plant renewal has resulted in their ultimate financial distress.

Replacements are often made necessary by fire, floods, storms, explosions, etc. Such replacements can be properly made from the depreciation fund because new apparatus is installed and the opportunity can be taken to equip with improved forms of the latest design in place of the older type.

Obsolescence cannot be stated and figured so scientifically as depreciation. Certain things, however, can be borne in mind in considering this. Obsolescence, as a general thing, grows less as a process grows older. In every industry, the change and improvements which occur in its early years are much greater than those which occur later. Much of the obsolescence which has occurred to plants in the last few years has been due rather to need for greater outputs than to any actual efficiency of the earlier apparatus itself as compared with the later forms. In many industries, the general concession seems to be that plants have now reached the most economic size for a unit.

I might express my idea of obsolescence in the form of a curve in which one ordinate is the rate of obsolescence and the other time. Such a curve will take the form of a hyperbola. It is easy to conceive, however, that there may be a break in this curve due to some discovery which would revolutionize the art of manufacture in this industry. Such discoveries, however, have seldom been of such a nature as to put out of business old established concerns which could not adopt them immediately. Time is required to perfect the workings of a new process and most plants which are under competent management are informed of the discovery in sufficient time to make preparation for its adoption should it become universal.

As examples of radical changes in processes and the slowness

of the newer and theoretically much more efficient process to displace the older process, may be mentioned the electrolytic method for soda and the contact process for sulfuric acid. In spite of its apparent simplicity and marked advantages over the older methods, the electrolytic process for soda has not yet overtaken the older Solvay process. This latter process is fifty years old, and although it has now displaced the Le Blanc process to a great extent, it has required half a century in which to do it.

In the case of the contact process, the improvements in lead chamber practice seem to keep pace with the advances made in the operation of the former, so that ample time will be given manufacturers to change from the older process to the new should the latter prove to be the more efficient.

Most processes change detail by detail. Much apparatus in the old one could be used in the new; notably buildings, power plant equipment, conveying machinery, etc. The most radical change which could occur would probably be the substitution of a furnace process in place of a wet reaction one, or from a simple chemical process to an electrochemical one.

As an example of such a possible change, processes are being investigated at the present time for rendering available the phosphoric acid in phosphate rock by furnace methods. This, if successful and universally adopted, would decrease the consumption of sulfuric acid fifty per cent and cause the sulfuric acid plant, now a prominent part of many large fertilizer works, to become worthless and necessitate the installation of rotary kilns and pulverizing mills in its place.

The scarcity of labor might at any time cause machinery to become obsolete, owing to the need of replacing men by machines. In certain sections where labor is now cheap, it is easy to foresee conditions where an increase in wages might necessitate the entire reconstruction of a plant.

Obsolescence generally determines the value of the mill site since this is of course subject to no physical deterioration and is affected only by the need of moving the plant to a more advantageous site relative to climate, markets, raw materials, labor or transportation facilities. In certain industries, such, for example, as the manufacture of Portland cement, the extinction of the raw materials would affect the value of the mill site because when the raw materials are exhausted it will be necessary to move the plant to some other location. This form of obsolescence, however, can be calculated with the same degree of accuracy as depreciation.

How to handle depreciation best is a question which is open to considerable discussion. The ideal plan is unquestionably to set aside from the profits a certain sum each year. This amount should be derived by a very careful study of machinery and buildings and other appliances and assets of the company, including the patent rights. In every business, there are good years and bad years and it will be found, therefore, to be impracticable to set aside a fixed sum each year because there are times when even the most careful management will fail to make both ends meet, so that the good years must bear the heavy burden.

The depreciation account, as we have said, should be divided into two parts, one for use immediately and one as a reserve. The former should be used to repair breakdowns and make renewals of machinery of an extraordinary character and the latter for the renewal of the entire plant. The first is designed principally in order that the manufacturer's cost sheets may not vary too widely from month to month, owing to unusual repair items, and may serve as a reliable guide to the operation of the plant during various periods. In some industries, the repairs are small and the replacements are light, and in these, such a drawing account may not be necessary, but in most chemical, metallurgical and mining plants there should be set aside such a fund. There should also be set aside a sum representing the depre-

ciation of the raw material deposits if there are any.

In plants which are financed entirely by the use of bonds, there is usually a clause in these latter which provide for a sinking fund sufficient to retire the bonds after a certain length of time. Plants which are financed in this way do not need a "plant renewal depreciation fund," because if their bonds have been retired, new securities can be issued and in this way sufficient funds may be raised to cover the cost of the plant renewal.

If depreciation is placed at too high a figure, manufacturers will be inclined not to consider it or, if they do, stockholders will be cut off from dividends which are rightfully theirs. The fact that the surplus is an asset of the company and consequently the value of its securities is increasing is not to be considered as answering this argument because many trust funds, etc., are invested with a view to bringing in steady revenues to the investors.

The question of depreciation is not simply one for the Board of Directors and the financial committee only. It is also a problem for the plant superintendent, the engineer, the chemist, and the geologist. It is only by a careful and scientific study of the subject that a proper figure can be arrived at. No two industries present the same conditions and probably no two plants manufacturing the same commodity will have the same life; hence depreciation is his particular problem with every manufacturer.

Obsolescence is always an unknown quantity and in a new industry it is much harder to gauge the rate of this than in an old one. Generally speaking, in most established industries the amount set aside for depreciation will also cover obsolescence.

Whenever the successful working of the plant is dependent upon a limited supply of raw materials, the life of the plant is fixed by the length of time the raw materials will last rather than by the wearing quality of the machinery. The depreciation fund in this case is, of course, the cost of replacing the plant and property.

As the first step in fixing the depreciation fund, the expert should consider the question of raw materials. If a local supply, how long will it last? Even if brought from a distance, the possibility of having to go still farther for it should be considered. Can new property be had at the cost of old? If the quality of the raw materials is decreasing, making it less easy to fashion the finished product as time goes on and possibly causing increased equipment, this should also be considered and provided for.

Next, the present equipment of the plant should be considered. Is it efficient and how soon will it have to be replaced? Is it already obsolete or does it represent present-day efficiency? Each piece of apparatus should be tabulated according to its probable life and value. Apparatus which needs to be replaced at the end of a year or so should be classed under the current renewal fund, and that with a longer life under the plant renewal fund.

From the plant renewal fund list should be excluded all apparatus which breaks down part by part, such, for example, as bucket-elevators, screw-conveyors, Griffin-mills and all small apparatus whose replacement represents a comparatively small sum, such, for example, as acid-eggs, cast iron evaporating pans, niter pots, etc.

Most apparatus has a salvage value even if sold only for its metal to the junk dealer. The amount of this may be calculated and deducted from the cost of new apparatus or it may be neglected altogether and considered merely as furnishing a margin of safety.

Let us take, for example, a cement plant of 3,000 bbls. daily capacity built, say, in the last year or two, and equipped with ball and tube mills for grinding the clinker and Fuller-Lehigh mills for grinding the raw materials, electrical drives, etc.

The capital raised for this will probably be distributed as follows:

Plant and quarry equipment.....	\$1,000,000
Property.....	100,000
Office and laboratory.....	15,000
Working capital, bags, etc.....	235,000
Cost of financing.....	150,000
Total.....	\$1,500,000

Let us consider the amount of raw material to be sufficient to last at least forty years. In considering the amount to be charged off for the extinction of the raw materials, the question is not merely one of present value but rather of the cost of replacing the property by a new one at the end of its usefulness. Since the life of the property is far greater than that of the plant, the latter will probably have been rebuilt several times before the extinction of the raw materials.

In the plant itself unquestionably much of the apparatus would have been replaced piece by piece in the course of current renewals.

The estimate of the cost of such a plant, showing the distribution, is given below:

COST OF BUILDINGS AND EQUIPMENT, 3,000 BBL. PER DAY PORTLAND CEMENT PLANT	
Buildings.....	\$200,000
Crushers, screens, etc.	25,000
Driers, coal and raw materials.....	25,000
Raw mills (Ball mills and Fuller mills).....	70,000
Kiln and equipment.....	75,000
Coolers.....	20,000
Clinker mills (Ball and Tube mills).....	75,000
Coal mills (Rolls and Fuller mills).....	14,000
Elevators and conveyors.....	50,000
Bins and stock boxes.....	40,000
Shafting, pulleys and belting.....	20,000
Foundations, tunnels and concrete work.....	40,000
Power plant equipment.....	175,000
Motors.....	40,000
Wiring and electrical work.....	30,000
Machine shop, carpenter shop and blacksmith shop equipment.....	10,000
Miscellaneous equipment.....	41,000
Quarry equipment.....	30,000
Tracks, coal trestle and yards.....	20,000
	<hr/>
	\$1,000,000

The above estimate includes the cost of installing the machinery and also its proportion of the engineering work, superintendence, clerical work, accident liability, insurance and other items incident to construction.

If the mill and kiln buildings are of steel, it would be safe to estimate their life, if properly painted and taken care of, at from forty to fifty years. Depreciation on steel mill buildings is usually considered at 2 to 2½ per cent. If the stone-house and the stock-house are of concrete with steel roof trusses, their life should be even longer. The roofs and sidings to the building would probably not have a longer life than fifteen to twenty years, so that the average life of the building could be safely placed at thirty years. It is hardly likely that they will become obsolete as modern machinery generally is more compact and, even if otherwise, steel buildings can be very readily enlarged.

No figures are obtainable upon the life of kilns. Some seven or eight years ago the size of the kiln was changed so that the long kilns now in use are none of them older than about ten years. These kilns do not show signs of going generally to pieces, although in some cases a lower section (25') may have to be riveted on. I think that 20 years would be a safe estimate of the life of an 8' × 125' kiln.

The driers and coolers are subject to considerable wear and tear—the latter particularly and the rotary coolers wear faster than the upright ones. The life of a stone drier on hard lime-

stone is generally about fifteen years and of a coal drier not over ten years. The crushers will last at least twenty years. Ball and tube mills should last this long also. Both, of course, need lining frequently. Griffin, Maxecon, Sturtevant, Hammer and Fuller mills are practically built over every few years. I think that with the ball and tube-mill installation 20 years would be liberal, as I know of mills still running efficiently which are nearly this old and which are probably good for many years more. The quarry equipment is subject to great wear and tear and I do not believe the life of steam shovels, cars and engines will be much more than ten years.

On engines, boilers and power plant equipment the figures generally used are from 5 to 6²/₃ per cent., or a life of from 16 to 20 years, and motors in cement plants, where they are subject to dust, cannot be expected to have much over 15 years of efficient life.

The bins and stock-boxes are quite an important item in the cost of a cement plant. These are not subject to appreciable wear and their life can safely be figured at 30 to 40 years. Furthermore, they are not subject to obsolescence and, in most plants with whose remodelling I have been connected, the bins in the old plant have been used in the new one.

Some of the concrete is in the foundations for the machinery and buildings and some of it in dams, retaining walls, tunnels, coal-trestles, etc., about the plant. Of itself, it is practically indestructible, but it is of course subject to obsolescence, particularly the foundations which may not suit the new machinery to be installed.

Machine shop tools are usually figured as having a life of 20 years, and accurate figures are obtainable on these. The tools in the cement plant machine shop are simple (lathe, drill-press, planer, etc.) and are not subject to very great obsolescence.

Wiring and electrical work, if substantially done, as it should be, if the figure given on the estimate is adhered to, may be safely assumed at the figures used by various electrical railroads, etc., for indoor wiring, viz., 20 years.

Shafting, belting and pulleys will, for the most part, be taken care of by repairs, but these are also subject to obsolescence in that they may not suit new machinery.

Elevators and conveyors are subject to rapid wear and their renewal will come under repairs. The casings, metal troughs, etc., will have a long life, consequently they should be taken care of in the plant renewal fund.

The miscellaneous equipment is mostly made up of small items, many of which are subject to renewal as repairs, etc.

Rearranging the items in the above estimate of the cost of the plant according to their probable life, we see that in arriving at the proper figures for depreciation we must provide for \$80,000 at the end of 10 years, \$230,000 at the end of 15 years, \$277,500 at the end of 20 years, \$240,000 at the end of 30 years, and \$187,000 at the end of 40 years, when the raw materials become extinct. The latter sum includes the value of the office and laboratory. The amount to be set aside each year is found not by dividing \$80,000 by 10, etc., but by calculating the sum which, placed at compound interest, will net \$80,000 at the end of ten years. It is inconceivable that such a fund set aside each year would not bear interest. If invested in the business itself in the form of enlargements, etc., the depreciation fund should be credited by its earnings. A depreciation fund could be safely invested at 5 per cent interest and the interest so obtained reinvested with the new installations to the fund as they are set aside. So it seems perfectly proper in setting aside such a fund, to assume that it will bear compound interest for say at least 5 per cent.

A table calculated by Mr. John W. Hill, and first published in *Engineering News*, June 25, 1894, for calculating this sum, will be found in Kent's "Mechanical Engineers' Pocket Book," page 16.

PLANT COST ITEMS ARRANGED ACCORDING TO PROBABLE LIFE

Practically Indestructible		
Concrete work.....	\$40,000	\$40,000
30 Years		
Buildings.....	\$200,000	\$240,000
Bins and stock boxes.....	40,000	
20 Years		
Crushers, screens, etc.....	\$ 25,000	\$277,500
Kilns and equipment.....	75,000	
Clinker mills.....	75,000	
Raw ball mills.....	15,000	
Coal rolls.....	2,000	
Machine shop equipment.....	10,000	
Wiring and electrical work.....	30,000	
Shafting, pulleys and belting (25 per cent).....	5,000	
Elevators and conveyors (40 per cent).....	20,000	
Miscellaneous equipment (50 per cent).....	20,500	
15 Years		
Power plant equipment.....	\$175,000	230,000
Motors.....	40,000	
Stone driers.....	15,000	
10 Years		
Coolers.....	\$ 20,000	80,000
Coal driers.....	10,000	
Tracks.....	20,000	
Quarry equipment.....	30,000	
Taken Care of by Current Renewal Fund		
Fuller mills.....	\$ 67,000	132,500
Shafting, pulleys and belting (75 per cent).....	15,000	
Elevators and conveyors (60 per cent).....	30,000	
Miscellaneous equipment (50 per cent).....	20,500	

In the above case the sums to be set aside annually are as follows:

To provide \$ 80,000 in 10 years.....	\$ 6,360
To provide \$230,000 in 15 years.....	10,666
To provide \$277,000 in 20 years.....	8,392
To provide \$240,000 in 30 years.....	3,612
To provide \$187,000 in 40 years.....	1,548
Annual depreciation fund.....	\$30,578

As the above plant will probably manufacture from 900,000 to 1,000,000 bbls. per year, this sum will be equivalent to from 3.01 to 3.4 cents per bbl. of cement produced.

On the extinction of the raw materials, two courses are open: either to retire entirely from business or to secure some other source of these and build a new plant at the new location. If the first course is followed, the depreciation should be increased by a sum sufficient to retire all of the securities at the end of forty years. The working capital at this time will most of it represent bills receivable and hence no provision need be made for retiring this. That portion represented by bags will probably be cut in half, but this will be offset in most cases by receipts from the sale of land, old machinery, buildings, etc.

The amount of depreciation to be fixed upon, therefore, in case plans are made to go out of business, will be the amount necessary to provide \$250,000 in forty years or an annuity of \$2,070, equivalent to from 0.23 to 0.21 cent per barrel, making the total depreciation in this event from 3.24 to 3.61 cents per bbl.

If new property is to be acquired, it is probable that this will be purchased as the opportunity presents itself to acquire desirable deposits of raw materials rather than at the expiration of the life of present holdings, and it is also probable that the transaction would be financed by the surplus profits of some period of good prices rather than by any fixed annuity.

In fixing a price for the cement, however, the cost of the raw materials, as represented by the purchase price of the property, should be included. Or considering the life of the deposit as

forty years, or 40,000,000 barrels, 0.25 cent should be added to the cost of production to cover the "in the ground value" of the raw materials.

In my estimation, the sum of 10 cents per barrel urged by some cement manufacturers recently is much too high. While it may be the part of wisdom to set aside some such sum as a "contingent fund," there seems to be no need of any such figure for depreciation alone.

202 N. CALVERT ST.
BALTIMORE

THE CORROSION OF LEAD¹

By RICHARD H. GAINES

The problem to determine what particular metal or combination of metals is best suited for a given purpose in engineering construction is sometimes one of great difficulty. A mistake in judgment may be at the expense of economy or durability or may even lead to a disaster. The decision of the engineer, to be correct, must be based on a thorough knowledge of the physical properties of the metals under consideration and this is generally gained by experience.

In many constructions, the choice of some of the materials is determined more by their durability under conditions of working than by their strength or other properties, a more expensive and weaker material being sometimes chosen in preference to a cheaper and stronger one on account of superior resistance to corrosion. Examples of this are afforded by the frequent use of special bronzes in places where, except for their durability, the stronger and cheaper iron and steel would be preferable.

Incidental to a recent study to aid in determining whether it would be safe to substitute lead-lined steel pipe for bronze pipe where a material of great durability was required in portions of a water conduit, some experiments were made, and facts collected from the experience of others, bearing on the corrosion of lead. From the data obtained it was concluded that for the particular purpose in view, the relative merits of bronze pipe and lead-lined steel pipe would barely admit of comparison. Under whatever conditions lead withstands the action of water or acids, the bronzes are similarly unaffected, while under other conditions of service in which the bronzes are practically untouched by corrosive action, lead would be destroyed. Lead-lined steel pipes no doubt have their uses in the chemical manufactures, and may also find extensive application for the conveyance of hard waters, or acid waters of a certain character in mines, but they cannot be recommended for use in connection with city water supplies. Owing to its peculiar physical and chemical properties, lead affords a doubtful protection to iron or steel conduits from corrosion, and under some conditions might prove an absolute disadvantage if not an actual menace to the life of the pipe.

THE ACTION OF ALKALIES

A peculiarity of lead is that it may be corroded either by alkalies or acids. Thus, lime mortar, lime putty and lime water will attack lead; if the mortar is very alkaline the effect will be greater. Proof of this was established by experiment as well as from observation of lead service pipes in wet concrete.

The following experiment was made to test the effect of an alkaline seepage water on lead. A weighed sample of lead was immersed for a period of eight days in seepage water that leaked through the concrete walls of a tunnel. The sample was taken out daily, washed, dried and weighed, and the loss in weight noted. At the end of the test period, it was found that progressive corrosive action had taken place, amounting to 0.132 per cent in eight days, or at the rate of about 6 per cent a year.

Several examples of the corrosion of lead service pipes in wet concrete were observed, one of which it will suffice to men-

tion here. In a bathing establishment in Brooklyn two lead pipes passed up through a concrete floor—one of the pipes was for hot and the other for cold water. Owing to the free use of water on the floor the concrete was wet much of the time. Here the lead pipe for the conveyance of hot water was nearly destroyed by corrosion in five years, while the cold water pipe was also deeply corroded. No evidence was found of leaking electric currents in the building to aggravate this action.

CORROSION IN CONCRETE

A series of experiments was made to determine what loss lead suffers from corrosion as compared with steel, and some other metals when embedded alone, or in contact, in very damp concrete.

Specimens of the following metals were weighed and embedded singly and in contact in damp concrete and allowed to remain therein for two months. At the end of the test period the concrete blocks were broken up and the metals taken out, carefully cleaned, dried and reweighed, the loss in weight representing the amount of corrosive action in each specimen.

METALS:	SINGLE SPECIMENS		Loss	
	Grams Weight		Grams Loss 2 mo.	in grams per sq. ft.
	Jan. 2, 1912	Mar. 4, 1912		
Manganese bronze.....	73.6655	73.6475	0.0180	0.32
Steel.....	39.1556	39.1481	0.0075	0.135
Lead.....	92.3806	91.7146	0.6660	11.99
Copper.....	73.7630	73.7272	0.0358	0.64
Monel metal.....	62.9667	62.9646	0.0021	0.04
METALS IN CONTACT:				
Steel.....	37.0380	37.0270	0.0110	0.20
Manganese bronze.....	70.6864	70.6700	0.0164	0.30
Steel.....	38.1746	38.1700	0.0046	0.08
Lead.....	90.0399	89.5408	0.4991	9.08
Steel.....	38.8055	38.8031	0.0024	0.03
Copper.....	70.1913	70.1348	0.0565	1.04
Steel.....	38.6396	38.6271	0.0125	0.23
Monel metal.....	60.1092	60.1074	0.0018	0.30
Steel.....	34.5625	34.4626	0.0999	2.48
Lead.....	83.8596	83.5163	0.3433	6.77
Copper.....	68.9752	68.9626	0.0126	0.31
Steel.....	34.7072	34.8026	0.0676	1.68
Fiber.....
Copper.....	64.1247	64.1122	0.0125	0.31

The resulting losses show but slight differences when in contact and alone, and, therefore, give no evidence of electrolytic action when the metals are coupled in concrete. With the exception of the lead the corrosion loss of all the metals was small, that of the monel metal almost nil.

The action of the lead confirms results obtained from actual experience and shows that this metal may suffer very appreciably in an alkaline as well as acid medium. The corrosive action in this case was observed to be solvent in character, no protective coating being formed to hinder further change.

THE EFFECT OF ACIDS

Sir William Crookes once said that lead was the first cousin of thallium which Lothar Meyer called the ornithorhynchus of the metals. This strange jurassic animal, as some of you may know, possessed the body of a mammal and the beak of a bird. Whether such a doubtful order of relationship in the family of elements is true or not, it is a fact that the chemical properties of lead are very peculiar and they present some remarkable contrasts. While lead resists the action of sulfuric and hydrochloric acids in a far higher degree than iron, zinc or tin, it is readily attacked by weak organic acids and is slowly dissolved even by pure water.

With respect to corrosion the behavior of lead is decidedly inconsistent. Notwithstanding that lead is very slightly acted on by some of the strong mineral acids in the cold, it is soon extensively corroded when exposed to the action of moist air in the presence of carbonic acid and becomes eventually con-

¹ Paper presented before the New York Section of the American Chemical Society, Chemists' Club, May 9th, 1913.

verted into a mass of white lead. Since carbonic acid is produced abundantly in the decay of animal and vegetable matter, metallic lead is much affected when kept in contact with such substances in the presence of air, the oxygen of which unites with lead to form an oxide which then combines with carbonic acid. The oldest process for the manufacture of white lead depends upon the corrosion of the lead by contact with decaying organic matter in this manner. Bloxam, in his work "Metals and their Properties," relates, "In breach loading cartridges where grease is employed as a lubricator, the bullets have sometimes become partly converted into white lead and thus increased so much in bulk as to burst open the copper case of the cartridges, and render it useless."

THE CORROSION OF LEAD IN THE SOIL

The impression prevails among many engineers that in the absence of stray currents, the corrosion of lead in the soil is negligible. This is not always true. While in some soils lead is very slowly affected by corrosion, in others an active chemical change or deterioration of one kind or another takes place; for example, "The lead of old coffins is sometimes found to have become almost entirely converted into an earthy-looking mass of basic carbonate of lead" (Bloxam, "Properties of Metals").

The Russian engineer, A. N. Schensnovitsch, of Odessa, recently sent by his government to New York to examine our pier and dock system, told the writer of an interesting case with which he was familiar of the destruction of lead water pipes by corrosion, that occurred in St. Petersburg during the seventies, before the days of industrial electricity or other conditions that could give rise to electrolysis. According to this engineer, in 1860-61, or thereabouts, a quarter of the city of St. Petersburg was served with water by a system of lead mains. In from ten to fifteen years these pipes had become so pitted by corrosion as to necessitate extensive changes. The cause of corrosion at the time was attributed to some local peculiarity of the soil, the exact nature of which was never ascertained. Mr. S. stated that the whole system was subsequently replaced by cast iron pipes, which had proved durable and satisfactory.

The superintendent of the water works of a suburban town in this vicinity told the writer that several years ago he had occasion to renew a number of lead service pipes on account of deterioration from corrosion. As he expressed it, "The pipes were pretty well used up, presenting the appearance of just having passed through a severe attack of small-pox. The lead moreover had the appearance of having lost all its vitality. There was no indication of electrolysis playing any part here."

In several cases reported by recent engineering periodicals, accounts are given of the lead pipe in the earth "losing its vitality, becoming brittle and porous, rendering renewal necessary after a comparatively short period of service, or the substitution of some other kind of pipe." Cases are also reported where it was deemed necessary to lay the lead pipe in wooden troughs, to surround it with pitch, or otherwise protect it from the corrosive influences of the soil.

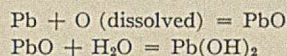
THE CORROSION OF LEAD IN WATER

So far as ordinary atmospheric corrosion is concerned, lead is one of the most durable of the common metals, undergoing no change in dry air or in water perfectly free from air. It is, moreover, but slightly affected by hard waters or dilute solutions of either hydrochloric or sulfuric acids. The latter acids quickly form coatings on the surface of the metal, which protect it from further action. Lead, however, is readily dissolved by water high in nitrates and by dilute nitric acid. It is also more or less rapidly corroded by weak organic acids in contact with moist air. Certain waters which actively corrode lead are those with a slightly acid reaction, from peaty swamps. There are certain microbes associated with peat which are acid-producing, and impart acidity to waters which have been in contact with peat.

Soft waters are particularly unsuited for conveyance in lead pipes, and in such waters containing nitrates derived from decaying vegetation, the amount of lead dissolved is large. Every plumber knows that pure soft water will attack, and sometimes destroy lead pipes. For this reason it has often been found necessary to line lead pipes with tin.

When lead pipes are acted on by water the corrosion is more or less irregular, some portions being hardly attacked, while at others, holes may be eaten through. This does not seem to be due to any irregularity in the metal, but rather to merely accidental circumstances. One authority quotes various experiments to show that lead water pipes should be kept full of water all the time to prevent deterioration.

The corrosion of lead by pure aerated water takes place as follows:



If exposed to the action of clean, soft water containing the normal quantity of dissolved oxygen, the lead is oxidized to hydroxide, which dissolves. After a time this is converted, by the action of the atmospheric carbon dioxide, into the basic carbonate of lead, $2\text{PbCO}_3, \text{Pb(OH)}_2$. Lead hydroxide is then again formed, and thus the corrosive action may be continued.

Potable waters always contain a certain amount of salts in solution, and the corrosive action depends to some extent upon the nature and quantity of the salts present. The waters which act least on lead are those containing carbonate of lime, phosphate of lime, and in a less degree, sulfate of lime. The waters which are most liable to act upon lead are those obtained from upland gathering grounds, waters which are soft and usually of remarkable purity. When highly oxygenated, the latter form no protective coating against solvent action. On account of the quantity that may be thus dissolved, lead has always been considered a more or less dangerous metal in connection with water supply for drinking purposes. Acting as a cumulative poison, its salts produce serious results if taken into the system even in very minute quantities for a length of time. Although indirectly related to the subject of corrosion, the experience of certain of the towns of Lancashire and Yorkshire in England should never be forgotten where the use of lead water pipes is under consideration. In these towns, lead poisoning was prevalent for a considerable period, doing immense damage to the health of the inhabitants before its real nature was recognized and its cause discovered. Incidentally it may be said that the compounds of lead are poisonous practically in proportion to their solubility. Thus the metal itself and its sulfide being incapable of absorption as such into the system, are practically innocuous, while the soluble salts, nitrate, chloride and acetate are active irritant poisons. The oxide, sulfate and carbonate are much less active, but continued exposure to lead fumes or to any atmosphere in which these substances are present in the form of dust brings on first "lead colic," and later, if exposure to the dust or fume is continued, a more chronic form of poisoning called "lead palsy" accompanied by great emaciation.

THE CORROSION OF LEAD BY ELECTROLYSIS

Instances are commonly reported of the electrolysis of lead pipes. In some of these cases, such as have occurred at St. Paul, Omaha and Paterson, N. J., all the phenomena of electrolysis were no doubt present. In others, damage from stray currents has by no means been proved, while the observed phenomena rather suggest ordinary chemical corrosion. In several cases of lead pipe deterioration called electrolysis, it was found that molecular changes of another character had taken place in the metal, similar to what Milton and Larke (Proc. Inst. C. E.) described as decay.

The electrolysis of either lead or lead-lined steel pipe, however, is a very real trouble, and the interest attaching to it here is the ease with which it may occur and the rapidity with which

serious damage may be done. A few years ago the Westinghouse Electric Company showed by experiments at Pittsburgh how readily buried lead pipes could be corroded by electrolysis. This company also made a series of tests in different soils to compare the corrosive action of stray currents on embedded lead and steel pipes. It was demonstrated that in some soils lead pipes were even more rapidly destroyed by electrolysis than steel. This accords with actual experience reported from several cities. At St. Paul, where considerable damage to lead water pipe occurred, presumably from electrolysis due to escaping current from electric railway lines, cast iron pipes in the path of the same currents are said to have been little affected by corrosion. In New York City an engineer connected with the water department told the writer that in the few instances in which there had been any trouble from electrolysis of water pipes in Manhattan, "the lead joints were the first to go."

147 VARICK ST.
NEW YORK CITY

WOOD ALCOHOL¹

The comprehensive report on the chemistry, technology and pharmacology of methyl alcohol by Dr. Charles Baskerville, the issuance of which by the New York State Factory Investigating Commission is most opportune, owing to the prevailing hysteria regarding the use of wood alcohol and the need of reasonable changes in the present laws, is divided into the following sections:

Chapter I: "Wood Alcohol: What It is;" Chapter II: "Manufacture of Wood Alcohol;" Chapter III: "The Uses of Methyl Alcohol;" Chapter IV: "Is Methyl Alcohol a Poison?" Chapter V: "Investigational Evidence and Recommendations;" Chapter VI: "Legislation Pertaining to Methyl Alcohol;" Chapter VII: "Conclusions and Recommendations;" Appendix A: "Affidavits of Employees as to Injury from Wood Alcohol Used as a Solvent for Shellac in Hat Stiffening;" Appendix B: "Free Alcohol;" Appendix C: I. "Summary of Cases of Poisoning by Drinking Wood Alcohol and Preparations Thereof;" II. "Summary of Cases of Poisoning by the Inhalation of Wood Alcohol;" and Appendix D: "Wood Alcohol Manufacturers in the United States."

THE GRADES OF WOOD ALCOHOL

The commercial wood alcohol on the market in 1896 was the same vile-smelling, greenish yellow, nauseous liquid as in 1850, and only quite recently was the deodorized product placed upon the market. Purified methyl alcohol is now sold under various names by American manufacturers, viz.: "Columbian Spirits," "Colonial Spirits," "Manhattan Spirits;" in Canada it appears under the names of "Green Wood Spirits" and "Standard Wood Spirits;" in Germany the newest variety is "Pro Spirit." The crude methyl alcohol of commerce is also termed wood spirit, wood naphtha, or pyroxylic spirit; it is a complex liquid, containing variable proportions of methyl alcohol, acetone, methyl acetate and formate, dimethyl-acetal, allyl alcohol, aldehyde, methylamine, oil, water, etc. The "tailings" contain furfural, methyl-ethyl ketone, and allyl acetate, with small quantities of paroxanthine. The best commercial wood spirit contains about 95 per cent or more of real methyl alcohol, the common varieties from 75 to 95 per cent, while some samples may contain only 35 to 40 per cent. The crude alcohol is a greenish yellow fluid, possessing a characteristic odor and nauseous taste; it is generally sold of a specific gravity of from 0.796 to 0.875 at 60° F. It is a good solvent and its

¹ Abstracted from "Wood Alcohol: A Report on the Chemistry, Technology and Pharmacology of and the Legislation Pertaining to Methyl Alcohol," prepared for the New York State Factory Investigating Commission by Dr. Charles Baskerville, Professor of Chemistry and Director of the Laboratory in The College of the City of New York, and Chairman of the Committee on Occupational Diseases, American Chemical Society.

miscibility with water depends upon the amount of impurities present.

Dr. Baskerville indicates that methyl alcohol is contained in the steam distillates from a number of plants, in the fermented juice of fruit, and in the products of certain fermentations; and that methyl esters occur very frequently in volatile plant oils. He considers that these occurrences might require more than passing significance in chemically establishing the origin of wood alcohol in a matter of law, especially in connection with a question as to the presence of methyl alcohol in flavoring extracts and medicines intended for internal use.

THE MANUFACTURE OF WOOD ALCOHOL

The destructive distillation of wood is almost the only method of practical interest for obtaining methyl alcohol in the United States, although in Europe it is sometimes manufactured by the destructive distillation of peat¹ and also as a by-product from vinasse.² Methyl alcohol may also be prepared by a variety of synthetical reactions³ and recently a process has been developed for obtaining it as a by-product in the manufacture of wood-pulp by a soluble sulfite⁴ process.

The production from peat, vinasse and wood-pulp is inconsiderable, and more or less incidental; and the synthetical methods are at present too expensive to be practiced on a commercial scale. The commercial demand for methyl alcohol is, therefore, essentially met by submitting wood to destructive distillation, the methyl alcohol, together with many other products, being obtained from the liquor condensed from the vapors evolved. The residue from the distillation is charcoal, which finds employment as a domestic fuel and in the smelting of various ores.⁵

The manufacture of methyl alcohol in the United States on a commercial scale is considered under the following heads:

A. The manufacture of the crude product.

- (1) The raw materials and their preparation.
- (2) The process of destructive distillation of the wood.
- (3) The separation and distillation of the crude product.

¹ For description of process, see W. A. G. von Heidenstam, *English Patent* 2,292, Feb. 5, 1900.

² Vinasse is the residue remaining after the distillation of fermented beet-root molasses.

³ The chief methods proposed are:

A. From methane: (1) By chlorination in reflected sunlight (Berthelot, *Compt. rend.*, **45**, 916); (2) Incomplete oxidation (Maumené, *Bull. soc. chim.*, [2] **19**, 243); (3) Oxidation with ozone at ordinary temperatures (*ot.*, *Ann. chim. phys.*, [7] **13**, 77); (4) By passing methane and air over finely-divided copper (Glock, *Ger. Pat.* 109,014 of 1898; Coquillon, *Z. Spiritusind.*, **23**, 182); (5) By oxidation with hydrogen peroxide, ferrous sulfate, monopersulfuric acid, combined or alone (Lance and Elworthy *Eng. Pat.* 7,297, March 26, 1906); (6) By oxidation at 30–50° C., using tan bark as a contact substance (Hausmann & Co., *Ger. Pat.* 214,155, Sept. 26, 1906).

B. By the dry distillation of calcium formate (Lieben and Rossi, *Gaz. chim. ital.*, **1871**, 164; Lieben and Paterno, *Ann.*, **167**, 293; Friedel and Silvia, *Bull. soc. chim.*, [2] **19**, 481).

C. By the electrolysis of an organic salt: (1) From potassium malonate (Peterson, *Z. phys. Chem.*, **33**, 714); (2) From potassium acetate (Hoefler and Moest, *Z. Elektrochem.*, **10**, 833); (3) From sodium acetate, perchlorate, sulfate and potassium carbonate and bicarbonate (Moest, *Chem. Centr.*, **74**, 1, 370).

D. From formaldehyde: (1) By the action of potassium hydroxide (Lieben, *Monatsh.*, **22**, 302); (2) By the action of light on a mixture of it with water (Inghilleri, *Z. physiol. Chem.*, **73**, 144).

E. From glycerine and glycerates: (1) By distillation of glycerine with soda (Fernbach, *Bull. soc. chim.*, [2] **34**, 146); (2) By the action of carbon dioxide on sodium glycerate at 180° C. (Loebisch and Looss, *Wein. Anz.*, **1881**, 252).

F. From prussic acid (Linnemann and Siersch, *Chem. Centr.*, **39**, 390).

G. From acetone by saturation with hydriodic acid or hydrochloric acid (Friedel, *Compt. rend.*, **46**, 1165).

H. By the action of a zinc salt [Zn(C₂H₃)₂] upon mono-chlor-ether (Henry, *Chem. Centr.*, **62**, II, 680).

⁴ See Meyer, U. S. Pat. 407,422, July 23, 1889; Stora Kopparbergs Bergslags Aktiebdag, French Pat. 402,331, April 23, 1909; Bergstrom, *Papier-fabrikant*, **8**, 970.

⁵ Chiefly iron ores, for the production of Swedish pig-iron.

B. The refining of the crude product. Purification and rectification, especially the separation of the acetone.

Hard woods, preferably birch, beech, maple, oak, elm, and alder, are best for the purpose, and recently processes have been developed for the destructive distillation of sawdust and wood-waste.¹

The wood used is thoroughly seasoned for 1 to 2 years and cut in 50-inch lengths.²

The destructive distillation is carried on in large iron retorts at a temperature³ of 400–500° F. These retorts are usually made of steel of varying dimensions, and are provided with large, tightly fitting doors, and are also provided with outlet pipes about 15 inches in diameter, or the gasified products may be taken out through several delivery tubes on the side of each oven. The retorts are set in pairs in brickwork and batteries of from 2 to 20 pairs are common. The cord wood is fed through the door and carefully stacked so as to completely fill the retort, or steel cars are loaded with the wood and run into the retort. In the larger works the retorts are constructed of brick and are of 50 cords capacity. They are provided with heavy iron doors, which may be sealed after opening. The doors are tightly closed and the retorts are heated from below by burning wood, coal, or charcoal, supplemented by the tar, red oil, and gas which are by-products in the industry, or the ovens may be heated by burning natural gas.

The gasified products of the distillation are run through condensers. The non-condensed gaseous products are returned and burned under the retorts.

The products condensed are run into tanks, and the tar is allowed to settle out; the pyroligneous acid containing acetic acid, methyl alcohol, acetone, allyl alcohol, phenols, etc., remains on top. The pyroligneous acid is a dark red-brown liquid, having a strong acid reaction and a peculiar empyreumatic odor; its density varies from 1.02 to 1.05. It is used to a limited extent in the manufacture of an impure acetate of iron, known as "black iron liquor" or "pyrolignite of iron," but is usually treated to separate the methyl alcohol (of which it contains about 4 per cent.), acetone, and acetic acid. This is done by fractional distillation. The acetic acid is recovered by passing the vapors through milk of lime, whereby "gray acetate of lime" is obtained, or the pyroligneous acid is neutralized with lime before distilling off the alcohol, which latter process is technically called "making a tub." Usually three stills of about 2500 gallons each are employed and from them are obtained distillates containing 15, 42 and 82 per cent wood alcohol, respectively. It is a closed process, but not necessarily a continuous one. The commercial product thus obtained is of 82 per cent strength by the Tralles alcoholometer, and contains varying amounts of acetone and other substances.

A graphic presentation of the process is shown in the accompanying diagram.

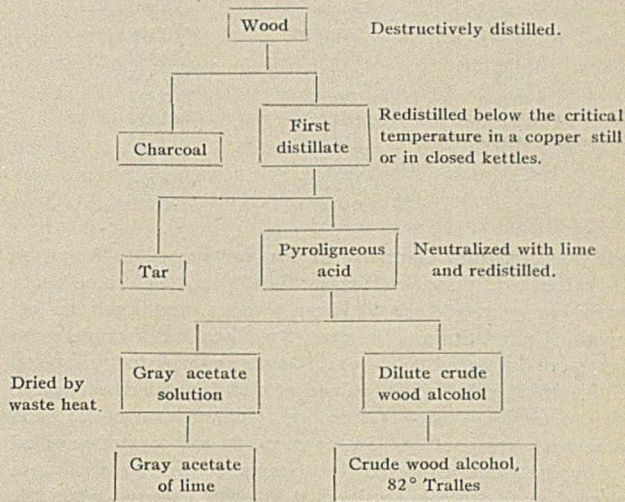
This crude material is usually shipped to a centrally located refinery in tank cars, iron drums, or in barrels, for purification and rectification. This is accomplished by further distillation from lime or caustic alkalis. The acetone cannot be removed by simple distillation and various methods for its separation have been proposed, *viz.*: conversion into chloracetones of high boiling points; conversion into chloroform and volatilizing by distilling the mixture with chloride of lime; crystallizing the methyl alcohol with calcium chloride and separating; forming the methyl ester, separating, decomposing by caustic potash

¹ See Bergman, U. S. Pat. 504,264, Aug. 29, 1893; Fischer, *Z. angew. Chem.*, 1900, 192; Orejavacer, Chem. Fabrik. Muller, French Patent 357,432, Sept. 2, 1905; Walker, *J. Soc. Chem. Ind.*, 30, 934; Norton, U. S. Consular Rep., Nov., 1911; *Chem. Eng.*, 16, 1.

² In most distillation plants, the wood is divested of its bark.

³ No decomposition occurs below 160° C.; between 160–275° C., the pyroligneous acid is formed; about 275° C., the yield of gaseous products becomes marked; between 350–450° C., liquid and solid hydrocarbons are formed; and above 450° C. little change occurs.

and obtaining the alcohol by distillation. Various types of rectifiers are used. The final product thus obtained is commercial wood alcohol, which is usually sold at 95 per cent strength by Tralles' alcoholometer, but may contain from 10 per cent to 20 per cent acetone and varying proportions of other organic impurities. Sometimes commercial wood alcohol is still more highly rectified and refined up to strengths of from 97 to 98 per cent by volume of real methyl alcohol, and is then sold under various trade names.



THE EXTENT OF THE INDUSTRY

The manufacture of acetate of lime and methyl alcohol was started in the United States about 1867, by James A. Emmons and S. A. Saxon, of Crawford County, Pa., although the manufacture of pyroligneous acid was begun by Ward at North Adams, Mass., in 1830. In 1874, George C. Edwards established the Burcey Chemical Works at Binghamton, N. Y., to refine the crude wood spirit produced by various manufacturers. In 1900, there were 99 establishments in the United States producing some of the crude substances enumerated above; of these, 84 were regular wood-distilling establishments, and produced 4,191,379 gallons of crude alcohol, having a value of \$1,660,061; 81,702,000 pounds of acetate of lime, having a value of \$926,358; and 14,428,182 bushels of charcoal, having a value of \$612,009. These works employed \$4,858,824 of capital and 1,268 wage earners. There were nine establishments reporting the production of the crude material and the refining of the alcohol in the same factory; and these establishments produced 637,856 gallons of refined alcohol, having a value of \$370,513; 5,134,000 pounds of acetate of lime, having a value of \$54,928; and 2,726,120 bushels of charcoal, having a value of \$114,663. They employed \$760,150 of capital and 254 wage earners. During the years 1909–10 there were 147 establishments engaged in wood distillation in the United States, employing 3,039 wage earners and \$13,017,000 of capital. Of these, 117 were engaged in wood distillation of hardwoods, chiefly birch, beech, and maple. The quantity of hardwood consumed during the year was 1,257,997 cords, or 86.7 per cent of the total of all woods consumed in distillation, and the total value of the product was \$9,737,000.

At the present time there are 63 manufacturers of wood alcohol in the United States.

The larger part of the export of wood alcohol for the past six years went to Germany, the United Kingdom, and Belgium. During 1910, wood alcohol valued at \$5,696 was imported into the United States.

Among the European nations, Austria-Hungary and Sweden are large producers of crude wood spirit, while Germany is the principal marketer of purified wood spirit. The crude wood

CRUDE MATERIALS PRODUCED, 1880 TO 1910

Year	No. of establishments	Wood alcohol		Acetate of lime		Charcoal	
		Gallons	Value	Pounds	Value	Bushels	Value
1880	17	\$86,274	6,593,009	\$156,892	\$31,770
1890	53	1,116,075	688,764	26,778,415	315,430
1900	93	4,945,963	1,976,986	86,826,000	981,286	17,154,302	726,672
1910	147	8,468,083	2,582,253	170,000,000	3,600,000	42,000,000	2,700,000

EXPORTATION OF WOOD ALCOHOL, 1900 TO 1911.

Year	Gallons	Value
1900	540,799	\$320,306
1904	1,194,466	585,359
1905	1,097,451	603,385
1906	780,222	466,467
1907	2,150,311	862,819
1908	1,958,630	819,753
1909	1,100,495	383,788
1910	1,691,000	760,000
1911	2,040,000	898,000

spirit imported into Germany in 1911 amounted to 8,759,300 kg., valued at 5,694,000 marks: 4,168,700 kg. came from Austria-Hungary, 268,700 kg. from Sweden, and 4,318,800 kg. from the United States. In 1910, 8,726,900 kg. of crude wood spirit were imported. In 1911, Germany exported 1,739,200 kg. of purified wood spirit, valued at 1,267,000 marks. France took 919,300 kg., Italy 138,700 kg., Holland 236,300 kg., and Switzerland 170,500 kg. In 1910, 1,782,400 kg. of purified wood spirit were exported by Germany. In 1910, France imported methyl alcohol valued at 1,819,006 francs, while the exportations for the same year were valued at 106,480 francs. In 1911, 291,600 kg. of methyl alcohol, valued at 379,080 lira, were imported by Italy, while but 500 kg., valued at 650 lira, were exported.

THE USES OF METHYL ALCOHOL

A. For Denaturing Ethyl Alcohol.¹

B. In the Chemical Industries.

The chief uses of wood alcohol in the chemical industries are as follows: (1) As a solvent; (2) as an extraction agent; (3) as a raw material for the production of formaldehyde; (4) in syn-

¹ Denatured alcohol is employed in large quantities in the arts and crafts and in the chemical industries; and in recent years it has displaced wood alcohol to a large extent. It is used as a solvent for shellac in the manufacture of all kinds of wood products, such as passenger cars, carriages, furniture, pianos, organs, billiard tables, burial caskets, toys, whips, trunks, pipes, etc. Shellac is used as a binding material in the manufacture of lead pencils and electric motors and generators. It enters intimately into the manufacture of stiff, silk, straw and felt hats, in which cases the shellac is incorporated in the body of the hat by the aid of alcohol. Similarly, it enters into the manufacture of lacquers, enamels, etc., used to enamel the surface of metals such as hardware, iron, brass beds, gas and electric fixtures, lamps, brass musical instruments, bird cages, clocks, watches, toys, etc.

The manufacture of celluloid, xylonite, artificial silk, collodion, etc., are dependent upon the availability of denatured alcohol. It is used in large quantities in the manufacture of smokeless powder, fulminate of mercury, and other explosives. An important use of industrial alcohol is in the manufacture of ethyl ether and ethyl chloride. Another utility of the alcohol is in the production of dyes, where it enters as a solvent, a medium of interaction, or as a chemically active body. There is also the manufacture of fine chemicals, a very important industry in which alcohol enters.

The use of denatured alcohol as an illuminant, fuel and in gas engines is of growing importance. Hundreds of patents have been granted for all sorts of alcohol lamps, and numerous alcohol heating mechanisms have been put on the market, especially in Europe.

The withdrawals of alcohol in the United States in the fiscal year 1910-11, for the purpose of denaturing, upon which no taxes were paid, amounted to 11,062,060 proof gallons, and there remained at the end of the year, in bonded warehouses, 2,213,390 proof gallons. In 1910-11, Germany produced 91,765,078 gallons of alcohol, of which 37,141,633 gallons were consumed in the industrial arts; and in 1910, 3,962,412 gallons of denatured alcohol were made in England. The Swiss importations of denatured spirit amounted to 6,993,900 kg. in 1911; of this quantity 6,246,500 kg. came from Austria-Hungary, a large producer of methyl alcohol.

thetic chemistry for the introduction of the methyl (CH₃) group; and (5) as a reagent in chemical laboratories.

(1) Wood alcohol is a good solvent for fats, volatile oils, camphor, resins, gums, alkalies, and various salts. Because of its cheapness, it is used as a solvent for varnishes, lacquers, stains, finish, and shellac. It is also used as a solvent in the manufacture of certain inks, photographic materials, celluloid, xylonite, and similar products, and incidentally in the manufacture of transparent soap. In celluloid, xylonite, etc., the alcohol does not appear in the finished product, while in varnish, shellac, etc., the wood alcohol remains as such and may produce its deleterious effects on workmen employing these in the industries.

(2) As an extractive, wood alcohol is used in the manufacture of smokeless powder, fulminate of mercury, nitrocellulose, and other explosives. Thus, dried, pulped military guncotton is freed from lower cellulose nitrates by extraction with wood alcohol.

(3) Formaldehyde, which has an extensive use, is produced from methyl alcohol by oxidizing with air by the aid of a catalytic agent. It is prepared on a large scale by passing a mixture of methyl alcohol vapor and air over a glowing platinum spiral or heated copper gauze. Formaldehyde may also be prepared by the electrolysis of dilute methyl alcohol and when ozone is passed through it. Commercial formaldehyde is a 40 per cent water solution, containing from 12-15 per cent of methyl alcohol.

(4) Methyl alcohol is used in synthetic chemistry for the introduction of the methyl group (CH₃) into various substances. In the manufacture of artificial perfumes and flavors, it was discovered that the presence or addition of the methyl group converted certain odorless and tasteless bodies into those possessing strong perfumes or flavors. Thus, methyl benzoate (C₆H₅-COOCH₃) is prepared by distilling wood spirit with benzoic acid and sulfuric acid; it is used in perfumery, under the names "Essence Niobé" and "Peau d'Espagne." Methyl beta-naphtholate (CH₃O.C₁₀H₇), known in commerce under the names "Nerolin" and "Yara-yara," is prepared by boiling beta-naphthol with methyl alcohol and zinc chloride; it is one of the strongest smelling perfume materials. Methyl cinnamate (C₆H₅.CH : CH.COOCH₃) is prepared by distilling methyl alcohol with sodium cinnamate and sulfuric acid; it is used as a flavoring extract in confectionery and as a perfume. Methyl anthranilate, a very fragrant perfume, is prepared by condensing anthranilic acid with methyl alcohol. Methyl alcohol is also used in the preparation of vanillin, etc. It is used in a similar manner in the manufacture of various aniline dyes, viz., methylene blue, green, and violet.

(5) Methyl alcohol is used as a reagent in chemical laboratories for the detection of salicylic acid, the determination of boric acid, in the preparation of grape-sugar, and as a substitute for ethyl alcohol for various purposes.

C. In Pharmaceutical and Medicinal Preparations.

Methyl alcohol is employed in the preparation of synthetic drugs (methylal, methyl esters, etc.); solid medicinal preparations, surgical dressings, cattle medicines, plant washes, and in capsules and other medicinal appliances improperly in toilet preparations (bay rum, witch hazel, "Florida water," etc.), and in essences, extracts (extract of lemon, orange, etc.), tinctures (tincture of iodine, etc.), liniments, lotions and embrocations, patent medicines, proprietary and domestic medicines.

D. In the Arts and Crafts.

Methyl alcohol finds application as a solvent in the manufacture of hats (stiff, silk and straw), electrical apparatus, gas and electric fixtures, furniture, pianos and organs, cabinet work, picture molding, burial caskets, passenger cars, wagons and other vehicles, boots and shoes, toys, whips, lead pencils, brushes, rattan goods, brass beds, various kinds of metal hardware, incandescent mantles, artificial flowers; in dyeing establishments; in cleaning in laundries, etc., and in the painting industry.

E. Unclassified.

Other uses of methyl alcohol are as a fuel and illuminant. It is also employed for a variety of domestic purposes (cleaning, etc.).

THE ABUSES OF METHYL ALCOHOL

The data collected by Dr. Baskerville indicate that the drinking of liquors containing methyl alcohol is responsible for most of the deaths and blindness attributable to methyl alcohol. The "deodorized" methyl alcohol resembles ethyl alcohol so closely that the ordinary layman can hardly distinguish the difference between the two, and, as it costs less than one-fourth as much as grain alcohol, people are tempted to use it as a substitute for ethyl alcohol in adulterating whiskey, essences, extracts, bitters, washes, liniments, balsams, perfumes, etc. The victims are generally those who indulge in the commoner forms of whiskey, rum and wine, although persons not addicted to the use of intoxicating drinks are undoubtedly often affected innocently, from drinking Jamaica ginger, lemon extract, essences, bitters, medicines, etc., whose chief menstruum is "deodorized" wood alcohol. Happily these abuses are growing less through the operation of the National Pure Food and Drugs Act of June, 1906.

THE POISONOUS NATURE OF METHYL ALCOHOL

After critically reviewing the ideas which have prevailed at various periods regarding the toxicity of methyl alcohol and the investigations which have been conducted on this point, Dr. Baskerville is led to conclude that methyl alcohol is less poisonous to lower plants and infusoria than ethyl alcohol, but for higher animals, and especially for man, it is a severe toxic agent. Its poisonous properties are doubtless due, first, to a specific action, and then to its oxidation in the body, first to formaldehyde and then to formic acid. It is furthermore a cumulative poison. It produces toxic effects whether it is taken internally or inhaled through the lungs.

It is a matter of little practical importance, he continues, as to whether *chemically pure* methyl alcohol is toxic or not, as economic reasons prohibit its extended use in the arts and manufacturing industries, although the purest material is used in certain products. The numerous cases of poisoning prove beyond doubt that the methyl alcohol of commerce is decidedly toxic.

CONCLUSIONS AND RECOMMENDATIONS

After a consideration of the published cases of poisoning by the use of methyl alcohol (a summary of these is given); the evidence which has been presented by experts, physicians, manufacturers and workmen as to the injurious action of wood alcohol when drunk, inhaled, or used externally; the recommendations which have at various times been made by individual experts and organizations to limit the sale and use of methyl alcohol; and the legislation, both in the United States and in foreign countries pertaining to methyl alcohol, Dr. Baskerville arrives at the following conclusions:

1. (a) While there may be differences in opinion as to the actual toxicity of absolutely pure methyl alcohol, the preponderating evidence indicates that its physiological action spells injury. The toxicity of the ordinary methyl alcohol of commerce, commonly called wood alcohol, even though it be of a higher

grade of purity, is a recognized fact, whether its morbid action is due to the concomitant impurities or not.

The use of methyl or wood alcohol in any food, condiment, flavoring extract, or liquid capable of being used in whole or partially as a beverage, or internally as a medicine, should therefore be prohibited by law. This is covered in part at present by Article 8, Agricultural Law 201, 1909, State of New York.¹

(b) In connection with the New York law, attention is directed to a lack in clarity, and, in consequence, likely injustice from attempts at its execution. The use of the word "methylated" is ambiguous. In the loose English sense it means ethyl alcohol which has been denatured with methyl alcohol, but it has an even wider meaning from what might appear as its real intent. One instance is cited to illustrate the point. Methyl salicylate, oil of wintergreen, is a recognized product of manufacture used for flavoring, which is a "methylated" preparation. It is identical with oil of wintergreen prepared from natural sources. If the synthetic preparation were properly labelled, it would not be a case of misbranding, morally, within the meaning of the section,² yet a court could very properly hold that it was, because synthetic oil of wintergreen is a "methylated preparation." The wood alcohol is no longer present as such and the physiological action of the wood alcohol is no longer observed. The above is true of many synthetic perfumes, flavoring extracts, and drugs of recognized medicinal value which are welcomed as legitimate commercial products of the chemist's art.

2. Wood alcohol should not be permitted in preparations such as perfumes, witch hazel, bay rum, eau de cologne, liniments, washes, etc., which are intended primarily for external use on the human body. It is remarked that "tippling" with these preparations is not uncommon.

The question of allowing ethyl alcohol which has been denatured with methyl alcohol to be used for this purpose, namely, in liniments, etc., might appear as one open to argument primarily on the basis of dilution. The denaturing formulas now in effect by Federal consent do not encourage but discourage the use of industrial alcohol for such purposes. The opinion is expressed that it would be better to err on the safe side and to make the restriction only as the occasion arises.

3. In the manufacture of wood alcohol the workmen are liable to come into contact with the vapor only in neutralizing the acetic liquor with lime and in filling the shipping containers. In the case of the former, the common practice now is to carry out the operation in closed vats which are opened only when the lime is added. General requirements for *ample ventilation* should meet these difficulties, which, in fact, do not now exist in the works inspected in New York State.

4. Wood alcohol is a valuable solvent used as such extensively in the arts in two important ways:

(a) It is used as a solvent in the *course of the manufacture* of many substances, but does not appear in the product when the latter is put upon the market. It is not destroyed but evaporates or is saved in part by condensation. It can exert its deleterious action during the process

- (1) by the workmen inhaling its vapor,
- (2) by the workmen constantly dipping their hands and arms into the liquor, or
- (3) by the workmen drinking the wood alcohol.

Ample ventilation will meet the first difficulty. Standards

¹ This article is as follows: "200. No person, firm, association or corporation shall, within this state, manufacture, produce, sell, offer or expose for sale any article of food which is misbranded within the meaning of this article."

² "201. Definition of adulterated or misbranded food: An article of food shall be deemed to be adulterated if it contains methyl or wood alcohol, in any of its forms, or any *methylated preparation* made from it."

² No. 200, *loc. cit.*

should be determined in each case by the Board of Health or such board with authority as may be designated.

In the second case the workmen should be provided with impervious long gloves or the processes should be operated mechanically; if a closed process is used, the first difficulty is also largely met.

Education only can solve the third problem.

(b) Wood alcohol serves as a solvent or menstruum of the material which is placed on the market in a liquid form (varnishes) and exerts its action upon the users, depending upon multiform conditions. In works where these materials are used in large quantities and the solvent evaporates, *ample ventilation* should be required. In many cases, however, the workmen are more or less isolated. They may and should be warned of the danger attendant by suitable labels on the vessels containing the liquid they are to apply.

5. All bottles or vessels used for transporting or selling products containing wood alcohol should be required to bear a prominent display label stating that it contains a "Poison."

RECOMMENDATIONS AS TO LAWS

The existence of the following laws is recommended:

1. A law prohibiting the presence of wood alcohol in any form of material intended for internal use.

2. A law prohibiting the presence of wood alcohol in preparations intended for external use on the human body.

3. A law requiring *ample ventilation* in works where wood alcohol is made or used in manufacturing products wherein the wood alcohol remains as such; the same law should apply where the products containing wood alcohol are used up, as, for example, in varnishing vats in breweries.

4. A law requiring containers in which wood alcohol is marketed to bear suitable display labels of warning.

These laws should be so drawn as not to inhibit use of wood alcohol in manufacturing products in which methyl alcohol supplies a constituent part, but does not remain as wood alcohol therein.

SOME PECULIAR FUNCTIONS OF THE RETAINED EXPERT¹

By W. M. GROSVENOR

One dislikes to use the name "expert" for three excellent reasons. It is as much abused by careless use as the name lady—likewise most applied where it least belongs. It savors of a holier-than-thou snobbery that is obnoxious to the plain common sense that recognizes every man as expert in some direction, from ditch digging to diplomacy. Finally, in the legal sense experts have sometimes taken advantage of their peculiar position as opinion witnesses to a sinister extent. An illustration from the files of the patent office is not without interest. In the early days of telegraphy an application was filed for a new kind of relay that, to make it very sensitive, possessed a long arm pivoted near one end between strong electromagnets and making relay contacts at the other. The examiner found nothing to anticipate it, but wrote the applicant to know, before allowing the application, why it was entitled "Expert Relay." The applicant answered that he called it a relay because it was for repeating forward telegraphic impulses and an expert because it possessed a tongue suspended in the middle and adapted to lie with equal facility in either direction according to the nature of the influence brought to bear.

This, however, is not the peculiar function of the expert that I desire to present. Aside from the so-called "expert" notorious chiefly for holding to one opinion or avoiding another, there is the real expert in getting, marshalling and using facts—sometimes quite tangible facts, like the type of man nature made him, but natural selection draws the narrower specialist into the employ of some one company or plant and further narrows

¹ Paper read at the Boston Meeting of the American Institute of Chemical Engineers, June 25-28, 1913.

him, while selection likewise urges the broader (sometimes shallower) man into general consultation work that further broadens his activity and experience until the particular science in which he was especially trained becomes but a subordinate part of his activity. It is the latter expert that interests us at the moment. To avoid the word "expert" I suggest we call him a retainer—a perfectly good English word implying honorable and somewhat independent service and loyalty.

For such work I believe chemical engineers to be peculiarly fitted, judging by the number who have succeeded in this field. Nor is this surprising when we consider that a general knowledge of Chemistry to-day begins at the very foundation of matter and energy and of necessity involves the fundamentals of mathematics, physics, mineralogy and biology, that as engineers by training or adoption the practical development of all this theory has been made compulsory and that all of this work has been done with the insight and thorough understanding that the chemical type of mind induces.

We read these days of scientific detectives—some fiction—but there is a sufficient basis of scientific fact to satisfy certain dynamiters. The scientific detective has certainly arrived and is making good. What I wish to point out and then to illustrate by a few examples is that the retainer has practically the same functions for industrial life and uses practically the same weapons as the scientific detective. It is his privilege to foresee and prevent, or to detect and penalize the industrial waste, deceit, misrepresentation, infringement, and forced bankruptcy that correspond to loss, fraud, blackmail, theft and murder in commercial and social life. Likewise his chief weapons are common sense, patience and experience and to him no knowledge comes amiss. Both men are detectors of fact guided by combined theory and experience and must check up by experiment or test.

Whatever the employer's problem, the retainer can bring to its solution two invaluable contributions: *first*, the independent, impersonal point of view of one neither involved in the petty detail of the business nor prejudiced by preconceived ideas or parental relation to the matter; *second*, a broad experience with different managements and ways of getting results, different plants and different branches of industry. If it is also true, as of the Italian bandit, "He had studied human nature and he knew a thing or two," he can be almost invaluable to his employer—and quite valuable to himself. To illustrate:

A retainer was faced with a problem of daily hoisting 3,000 tons of sulfuric acid about 80 ft., and because he had not grown up with the problem but met it fresh with other things in mind, he cut the usual cost down 60 per cent by putting in a balanced pair of ten-ton mine skips.

Another retainer without special foundry or crane experience was asked to reduce the excessive number of accidents in one foundry handling a large number of very heavy castings; he studied the problem a few days and concluded it was a matter of temperament; that the crane men were casually selected from the mechanical force predominating in nervous or energetic rather than cautious men. A new gang was carefully selected for cautious combativeness from various points in the plant and before trying them out the duties and dangers were carefully explained. So well were they selected that all the first lot threw up the new job before trying it. Another lot with milder explanation took over the cranes and the accidents ceased almost immediately.

A new type of shellac drier had just been installed and tested. It reduced the drying time from 48 hours to 3 hours and the floor space from 6000 square feet to 200 square feet, but the shellac packed and gradually built up on the sides of the drier, making no end of trouble. The retainer of the drier company, who knew nothing about shellac and had not heard of this particular drier, was sent for, looked the drier over, felt of the shellac

to be dried and in various stages of dryness by the old process, made a short preliminary test, and then ordered the circuit opened for the first hour of each three-hour treatment to reduce the initial humidity. The packing promptly ceased.

A northern manufacturer of muriatic acid called up on the phone to say that his plant was crazy. It took in raw material but did not produce acid, so the absorption water grew weaker and had to be shut off. The retainer knew the plant, instructed pressure tests with a draft gauge to be made at certain points, and reported by phone. When the report came in he asked, "How was the weather last night?" "Bitter cold," came the answer. "Thaw out the crystalline hydrochloric hydrate from your air precooler with warm water," was the advice.

A certain man was retained by one of the textile coating firms and after making his first trip through the plant asked for the largest single items of expense for material not actually present in the finished goods. Volatile solvents used with the coating material were first mentioned to the amount of \$400 a day. In reply to his question why this was not recovered it was stated that this had been tried elsewhere and failed for two reasons, *i. e.*, only 30 per cent recovery had been made but, far more important, the quality of the goods had been injured. He offered reasons why the tests must have been carried out under improper conditions to show such results, and advised testing again. He condenses two years' development into a sentence and says, "The first little test box showed 50 per cent recovery and improved the quality of goods, and the commercial installation on formal test showed 80 per cent recovery, better quality and 25 per cent reduction in labor for the operation."

A large garage company desired to improve their competitive position and sought advice. On examination, no chance for considerable improvement appeared until petrol sales were examined and found to be a source of considerable revenue and a possible drawing card if the petrol were treated and carefully clarified. A special high-grade petrol was made a great talking point thereafter and customers found it actually much more efficient—not surprising when the patent finally explained that a small but effective quantity of organic nitrobody such as picric acid was dissolved therein.

The salesman of one of the glue companies met a series of complaints regarding goods purporting to be glued up with their product, made by some of their customers, each of whom did not, however, buy enough glue to account for his entire output. Whenever any of the output of this group of customers went wrong in the hands of the final consumer, the fault was laid to this company's glue and the trade name suffered. The fault was mentioned to their retainer. Certain peculiarities of their extraction process were taken advantage of and a simple color test devised for distinguishing their glue when in place. A small bottle of reagent and a pocket magnifier in the hands of each salesman enabled him to disprove the calumny on the spot wherever he met it and quickly made it a dangerous statement to continue making.

In one instance the same company was threatened with damages on account of several large lots of bad goods made with their product. The first claim was about \$4,000. The retainer was asked what he thought the trouble was. Examination at the factory of the record samples covering this period indicated the shipments of glue to have been excellent in quality. Examination of a couple of pieces of the goods complained of showed bad results and suggested dampness. Stained microscopic sections of standard and specially made joints were compared with sections of the bad work shipped out by the user of the glue. Measurements and appearance proved conclusively that the joint was too wet when made, either green wood or overwatered glue and almost beyond question the latter. On the evidence informally submitted the claim was dropped.

A manufacturer of feather boas complained bitterly of the dye

used on the feathers he purchased. It made sore the satiny necks of the wearers, and their feelings, and the retailer. His retainer showed him that the trouble lay nearer home in the angle to which the feathers were trimmed, sharp instead of square.

The makers of a special grade of treated rubber goods who had just had their patent allowed, were planning infringement suits, and through their counsel retained a technical patent adviser. The great economy and advantage lay in the ability to minutely form the goods before treatment, making allowance for the alteration in size during treatment and avoiding all the difficulty of mechanically working the goods after treatment. The process of treatment alone had been described in detail and claimed, though the use of it to make formed articles had been described as one of the advantages. The retainer referred to the literature and found more or less similar treatments though not adjustable as regards change of size produced. He asked a conference of principals and counsel, pointed out the possible confusion of the issue by expert testimony regarding the treatment process, compared with the great simplicity of the case if the patent had a different underlying theory, suggested the theory of a process of producing formed articles of rubber, describing and claiming the novelty of the series of steps, *i. e.*, first roughly forming them, precisely shaping them with allowance for change of size, then treating with corresponding conditions to produce the desired change of size as well as texture. The special treatment claims would be retained at subsidiary to the general invention. Both principals and counsel seized upon the new theory, decided to abandon and refile, deferred suit to be brought under the new patent, and found suit unnecessary because in the new form of the patent there was no chance for defense.

A manufacturer of highly polished sheets had occasionally found trouble with the sort of bloom that appeared irregularly without apparent reason on the polished surfaces. Suddenly the trouble began to increase and he submitted it to his retainer with samples of the bloom sheets. The samples arrived almost simultaneously with the telegram saying the trouble was serious and still increasing. The samples were at once put into the laboratory for analysis, tests and micro examination, a series of questions framed and dictated over the long distance phone. Some of these questions must have seemed ridiculous at the factory. Among the series of twenty or thirty were, "Has the trouble been most noticed in the past in summer, fall, winter or spring?" and "Does the present trouble seem to vary with your weather?" The answers came back after a little and among them were "Noticed more in winter," "Worse on sunshiny, windy days," with the final emphatic comment that seconds had reached \$600 per day. Meanwhile, the work in the laboratory had developed a series of puzzles. That night, after going carefully over the questions and answers, the retainer sent a night lettergram instructing the factory to carefully ground electrically to water or steam pipes every part of one machine and report results. Next afternoon, while still working on the puzzle in the laboratory, the retainer received the telegram—"Grounding stopped trouble immediately; proceeding with grounding of other machines." Just why static charges caused the trouble is too much of a story.

The legal and business representative of a small company operating a secret and very profitable process in the West, came east to sell out on a basis of \$50,000 cash plus royalty to a larger company manufacturing their chief raw material. The business was opening up much larger than the westerners had the means to handle and they were afraid of a rumored combination of the makers of the raw material that might squeeze them hard. They shrewdly figured to get their little wedge into that combination in the nascent state. The president of the Eastern Company was immensely interested in the product, the sample

correspondence and orders, but was not buying a pig-in-a-poke, and could get no secrets from the westerner. Thinking it over in the evening, he called the company's retainer on the phone and asked him to take a hand, so it was arranged that the president would turn over the westerner to him for lunch the next day. The retainer took him out to lunch and started to get acquainted, found that he was dealing with a lawyer, and thought he saw a way. So he discussed the difficulty of negotiating sales of secrets from some experience and carefully avoided asking awkward questions. Finally the western lawyer began to realize the difficulty of the merchandise he had come to market and asked the retainer if any arrangement could be suggested to overcome the dead lock. "You might look up my personal rating—that of my company you probably have—then if you want to make me a director of your company on one share of stock you could take my acknowledgment of all information received by me in confidence as director of your company and have me and anyone to whom I revealed it pretty well secured under recent decisions." Naturally, the lawyer jumped at the suggestion, telephoned to the president, and by four o'clock the details were all agreed upon and an appointment made for the following week in the western city. The president and his retainer went west as arranged, only to find that at the last minute the other western man had waxed nervous over the possibility of having to sue the bigger eastern company to recover, and wouldn't accede to the director's suggestions. All day they discussed possibilities, with no result, and at 5 o'clock the president said there was no use staying over. The westerners, too, were ready to give up the deal. But the retainer had a few questions to ask. The westerners said they could guarantee the manufacturing cost, yes; and the selling price for 6 months, yes; and gross sales for the first 6 months equal to the

full amount of cash payment demanded if they had the goods to sell; and they could together get an indemnity bond in that amount as security for this guarantee. The retainer then took the president outside and pointed out that if their sales force were put on his staff their guaranteed cost, selling price and 6 months' sales would yield a profit equal to half the purchase price—or the bond would forfeit—and that there was practically no chance of the second 6 months not showing as well as the first. The president stayed over and the deal was fixed that way.

I believe the general point has been sufficiently illustrated, *i. e.*, that in retainer work the chemical engineer has open to him a sphere of activity in connection with industrial life for which he is peculiarly fitted by training and experience and which parallels in a fascinating way that of the solver of human problems in commercial and social life. One such chemical engineer exhibited to me what I would regard as a treasure, a comment forwarded by the president (whom he knows very well) from a company that has retained him for years.

"The chemist says he doesn't know as you help him so much, but you ask questions and suggest things that set him thinking, and somehow he gets first-rate results. The engineer says you ask more fool questions than anyone that comes around, and he doesn't know how you figure it out, but he does something you suggested and the trouble stops."

May we all be equally fortunate in effects and leave the chief chemists and engineers to their various conclusions. The men at the factory work far better if they feel that a large share of the success depends on them, as, indeed, it does. They are welcome to think it and to their credit, as long as the employer gets the results.

50 EAST 41ST ST., NEW YORK

CURRENT INDUSTRIAL NEWS

By W. A. HAMOR

THE CATALYTIC PRODUCTION OF PETROL

Some particulars are given in the *Times Engineering Supplement* of a catalytic process for the conversion of petroleum almost entirely into petrol. In this method, which is controlled by Messrs. Planes, Ltd., of Birkenhead, England, the petroleum is placed in a high still having a conical bottom passing through the fire bars. A continuous feed of petroleum and of hydrogen under pressure is passed into the still from the bottom, and contained in the still is the nickel used in Normann's process for hydrogenating oleic acid. Strong agitation is maintained by means of a stirrer in the still. The still is heated to a little above the temperature at which petrol or whatever hydrocarbon is wanted becomes a gas. The vapors of petrol or other light hydrocarbon, as the case may be, are carried up by the hydrogen and the heavier hydrocarbons mechanically carried up are condensed in the upper part of the still and fall down, while the light vapors, with the hydrogen, are passed to the condenser. The hydrogen from the condenser is returned to the still. The petroleum, a saturated compound, is thus split up into a saturated hydrocarbon, say, petrol, and a heavier unsaturated hydrocarbon; the latter, it is claimed, at once takes up hydrogen in the presence of the catalyst, and becomes saturated, when it is again broken up into petrol and an unsaturated hydrocarbon, which is again hydrogenated. The process is continued until the nickel becomes inactive and the petroleum in the still becomes overloaded with tarry matters. The tarry compounds and the nickel are then removed, and the latter is regenerated.

THE MANUFACTURE OF COLD-PROCESS SOAPS IN EGYPT

Nadalini (*Chem. Trade J.*, 52, 596) points out that if the cold process for soap-making has now fallen entirely into disrepute

in Europe and America, in Egypt it is very much in vogue for the production of the cheapest quality of household soaps. This is due to the fact that it is possible to produce a soap of good appearance containing many different and cheap fillers, by means of a very simple plant and little knowledge; the Egyptian production is now very great.

The amount of insoluble matter which these soaps contain varies from 20 to 35 per cent and consists principally of talc, calcium carbonate, clay, flour, and various earths. The employment of cottonseed oil is general; the use of coconut oil or other oils of that class containing glycerides of the lower fatty acids has become rare. The caustic soda used is generally the ordinary 60 per cent, at a strength of from 30 to 38° Bé. An ordinary crutcher, frames, a banjo slabber, a cutting machine, and a very simple wooden hand-stamp are used in the manufacture of these soaps, supposed to be an imitation of the Syrian soaps, so well known to the natives. The materials are mixed at the same time into the crutcher, except that gelatine is usually added at the end of the operation, this requiring one hour's stirring. The soap once framed is slabbed after 3 to 4 days, and then is cut into tablets of about 150 grams. Soaps made in Egypt either by the Syrian or the cold process are never neutral; the neutralizing material used is gelatine, rarely sodium bicarbonate, and the use of ammonium compounds or bibasic phosphate is absolutely unknown.

THE PULP-WOOD CONSUMPTION IN CANADA

According to *Paper*, June 18, 1913, p. 34, the bulletin now being issued by the Forestry Branch on the pulp-wood consumption in Canada for 1912 is a compilation of statistics obtained from the sixty pulp mills now operating in Canada. Of the many facts revealed by these figures, there are at least three points

which should be of special interest to the general public, to the forester, and to the trade, respectively.

In 1912, nearly two million cords of pulp-wood were cut in Canada, 47 per cent of which was manufactured into pulp by Canadian mills, an increase of 3 per cent over 1911. This means that over half the pulp-wood cut in Canada is exported in unmanufactured form, principally to the United States. It is estimated in the bulletin that, had this exported pulp-wood been manufactured into pulp in Canada, the increase in the national dividend from this source would be over six and a half million dollars. Yet all the provinces, with the exception of Nova Scotia, have placed restrictions on the exports of raw material, and the fact that, in spite of these restrictions, the quantity of wood exported in the raw state increased from 1911 to 1912, as well as the falling off in Canadian prices for pulp-wood during this period, seems to indicate overproduction on the local market.

Of perhaps special interest to the forester are the statistics showing 40 per cent increase in the use of balsam fir in the manufacture of wood pulp. We quote from the bulletin: "News print can be satisfactorily made from a mixture containing over 40 per cent balsam fir and 60 per cent spruce, and the prejudice against balsam fir is rapidly disappearing." Balsam fir is especially adapted for pulp, the wood being free from resin and having very long, strong fibers. It is, however, good for little else, and is at present viewed by foresters as a "weed-tree." As it is one of the most common species of Canadian trees, the development of a market for this wood will be viewed with satisfaction for both the forester and the private owner.

Of interest to the trade is the successful introduction of the sulfate process in the manufacture of chemical pulp. In this process sulfate of soda is added as a source of alkali and to make good the mechanical losses—10 per cent approximately—in the process of recovery of the soda. Three large Quebec mills used this process in 1912 with excellent results. Spruce, with a small percentage of balsam fir, was the wood used.

SOME PROBLEMS OF THE RUBBER INDUSTRY

Potts (*J. Soc. Chem. Ind.*, 32, No. 11, 564) indicates four leading groups of problems in the rubber industry: (1) Quality of natural raw rubber; (2) Synthetic rubber; (3) Vulcanization; and (4) Regeneration.

The viscosity of Hevea latex diminishes on dilution with water in such a manner as to suggest that it is an emulsion rather than a suspension. Since, however, rubber is an emulsoid colloid, and therefore always a liquid, this does not enable us to draw any conclusion as to the state of polymerization in the latex. The nature of coagulation is not yet fully elucidated, although it is of absolutely vital importance as regards the quality of the rubber produced. On comparing a sample of Plantation rubber with fine hard Para, the latter is preferred. The Para appears harder and "nervier" on the mixing rollers, and in any case it is usually more consistent in quality. Many explanations of this have been advanced, such as the youth of the trees on the Plantations, the acetic acid process of coagulation as compared with the smoking process, etc.; but at present no chemical explanation can be given. Research is also required to determine the nature of the factors affecting quality of raw rubber, and its velocity of vulcanization.

Potts emphasizes the importance of having a raw rubber which shall vulcanize at a constant speed, and, if possible, of guaranteeing this by careful tests before buying. Rubber from which the resins or proteins have been removed will not vulcanize as readily as ordinary technically pure rubber, and the conclusion has been drawn that synthetic rubber will be bad on the ground of being too pure. The rubber manufacturer will not, however, be in any way embarrassed on that account and will be able to add as much resin as is needed, consistent, of course, with specification work.

With regard to the regeneration of waste vulcanized rubber, to Potts' knowledge, so far it has not proved possible to obtain from waste vulcanized rubber a product like raw rubber and containing no sulfur. The combined sulfur is bound with extraordinary strength. What can be accomplished is to remove the free sulfur, some or all of the mineral fillers, and the fibrous materials, and the reclaimed rubber thus obtained by many processes is a valuable adjunct in cheap mixings.

THE EXPORTATIONS OF CAMPHOR FROM JAPAN, 1910 TO 1912

During the last three years, crude and refined camphor valued at the following amounts was exported from Japan (including Formosa), according to *Die Chemische Industrie*, 36, No. 11, 355:

Destination	1910	1911	1912
Hongkong.....	\$ 6,500	\$ 15,438	\$ 10,956
British India.....	226,000	285,500	322,000
Straits Settlements....	2,450	4,034	4,900
England.....	327,700	327,700	362,000
France.....	434,000	398,000	251,500
Germany.....	178,000	260,000	244,500
United States.....	393,000	358,000	228,500
Canada.....	6,000	6,000	10,400
Australia.....	32,000	47,000	37,850
Other Countries.....	26,000	24,000	57,800
Total.....	\$1,631,650	\$1,725,672	\$1,530,406

"SILIT" AND ITS APPLICATIONS IN THE CHEMICAL INDUSTRIES

"Silit II" is regarded by H. Grossmann (*Chem. Ind.*, 36, No. 10, 304) as a most interesting product. It consists essentially of silicon carbide, and is obtained, according to German Patent 257,468, of July 21, 1911, in the following manner: Silicon, carbon and a binding material, such as colophonium or paraffin, are made up into a plastic mass, shaped in molds of silicon carbide, and finally heated in an atmosphere of carbon monoxide or carbon dioxide to from 1,400° to 1,500° C. At this high temperature the silicon combines with carbon monoxide: $\text{Si} + \text{CO} + \text{C} = \text{SiCO} + \text{C}$. That SiCO is formed has been actually demonstrated by Schützenberger and Colson. If the mass is heated to from 1,600 to 1,700° C., or is heated in an electric furnace, the free carbon combines with the oxygen of the SiCO and escapes as carbon monoxide; there remains silicon carbide. It is obvious that an excess of carbon is always necessary, for without this excess the reduction of the oxycarbide to carbide becomes possible only at a temperature of 2,800° C., and at this temperature a considerable amount of the silicon is lost through volatilization and the molds fuse.

Owing to its great stability at high temperatures, even as high as 1,400° C., and other properties which permit of its mechanical working, "Silit" may be employed successfully for heating purposes. Like silundum, "Silit" may be used for electrically heated radiators and hot-plates; a high heat may be obtained from these radiators and hot-plates, without any attending unpleasant odors or accidents. "Silit" tubes have been successfully employed in the illuminating industry as rods in arc lamps; they have also been used as combustion tubes, and as furnaces for tempering steel pens and other instruments.

Under ordinary atmospheric conditions, "Silit" furnaces resist a temperature of 1,200° C. for a long period of time. If the furnace is surrounded with a jacket of carborundum, it will resist a temperature of 1,250 to 1,300° C. and a current of 45 volts and 7 amperes for 75 hours, and a heat of 1,400° to 1,450° C. for 5 hours.

"Silit II" and "Silit III" are resistant to the action of chemical reagents, particularly acids and chlorine. "Silit II" cannot be employed as an electrode in the electrolysis of alkali chlorides because of the action upon the carbide of the nascent

oxygen liberated; it may, however, be used in the electrolysis of hydrochloric acid.

"Silit III" consists of silicon carbide and silicon carbonitride. According to German Patent 176,001, of January 4, 1905, it is obtained as follows: Silicon and carbon compounds, or silicon, silicon carbide and an agglutinant containing carbon, are thoroughly mixed and then heated in an atmosphere of nitrogen. The product possesses extraordinary density, hardness and resistance to fluctuating temperatures. It can replace carborundum in the manufacture of whetstones.

THE SCHOOP METALLIZING PROCESS

Sometime ago the Schoop metallizer was described in this column (see THIS JOURNAL, 4, 853). At that time no practical results were available; these are, however, brought out in a paper by Albert Neuburger, read before the Märkischer Bezirksverein (*Z. angew. Chem.*, 26, No. 44, 384).

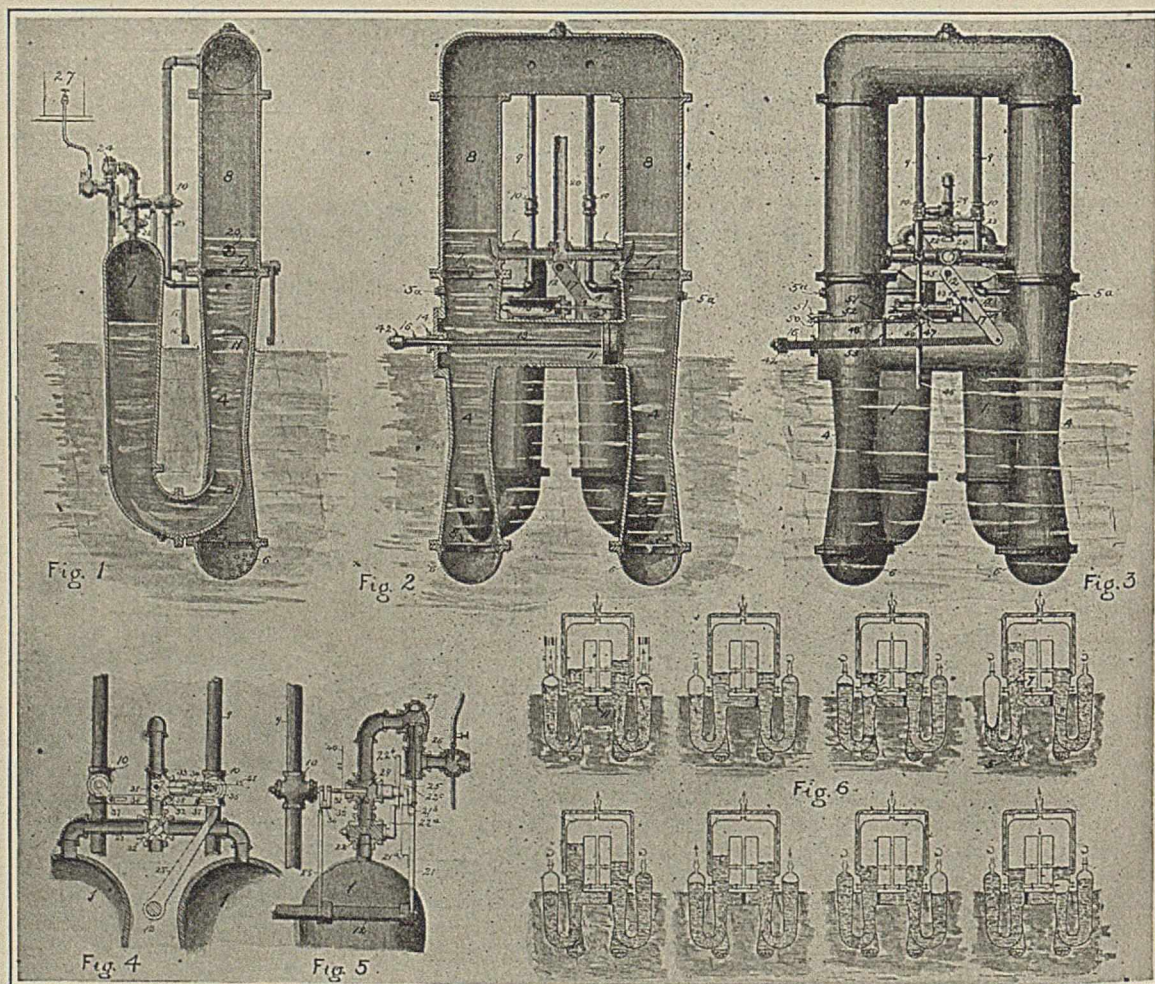
By the old process of galvanizing, not every metal can be made to adhere to another metal without the previous application of a binding metal. In case non-metallic objects are to be coated with metal by this process, they must first be coated with graphite

when a suitable metal is used, rendered uncorrodible. This metallizer draws the metal into wire, fuses it, and, by the aid of an air turbine, converts it into a dust-like state; the metal mist is then projected with great velocity upon the object to be coated. Neuburger reports that the fine metal particles adhere to the body and form a solid layer. He considers that Schoop's advice may be used with all metals and for coating all kinds of objects. He has found that the projected metal is very hard; for example, the density of tin deposited by the Schoop metallizer is 14.2 as compared with the density of 9.5 in the case of ordinary fused tin. It is stated that the device may be employed in the construction of accumulators, in the chemical industry (in metallizing glass, quartz glass or iron vessels, etc.), in the canning industry, in the textile industry (for metallizing fibers), etc.

RECENT ACTIVITY IN THE EXPLOSIVE PUMP ART

A review of the more recent internal combustion pumps appears in *Scientific American*, 108, No. 22, 496, from which the following is taken.

The Smyth pump, as shown in the accompanying engravings,



FIGS. 1 TO 5—DETAIL OF THE SMYTH INTERNAL COMBUSTION PUMP FOR RAISING WATER
FIG. 6—THE CYCLE OF OPERATION OF THE PUMP

and then placed in baths in which the necessary operations of plating are performed. Structures such as iron bridges, railway stations, etc., are rendered resistant to corrosion by galvanizing or sherardizing, but they remain open to corrosion at the rivet holes. According to Neuburger, by means of the Schoop metallizer even the rivets and rivet holes are metallized, and,

is a duplex one, both parts being alike. A combustion chamber, 1, the lower end of which is merged into a nozzle, 3, extends into a casing, 4, which constitutes the suction pipe of the pump. The lower end of this casing is provided with a check valve, 5, and a strainer, 6, and the upper end is separated from an air chamber, 8, by the check valve 7. As seen in Figs. 1 and 2, the discharge

of the pump is through a pipe, 20 which connects with each air chamber, 8. Two pipes, 9, having valves, 10, to control the flow of air or water, connect the air chamber 8 with the casings 4 just below the valves 7. A water piston, operated by the explosive charge, is located in each chamber, 1, and the two casings 4. Air is introduced into the air chamber by means of valves 5a.

To regulate the position of the water piston in the chamber 1, an independent piston, 11, free to move in response to heat pressure, is provided in the pipe connecting the two casings 4. This piston is actuated by the power piston 19, in the motor cylinder 15, through the link 18, lever 17 and rock shaft 12 and link 16 connected by the yoke 42 to the rod 13, which passes through a stuffing box, 14.

As seen in Fig. 3, the motive fluid for operating the motor 15 is introduced and exhausted to and from the cylinder by a four-way valve, 43, through pipes 45, 44 and 46. This valve is operated by the link 16, carrying the tappet 53, which actuates the levers by riding over the cam surfaces 52. These levers 51 operate in turn, by means of the spring-operated tappets 50, the sliding tappet lever 48, which oscillates the lever 47 of the valve 43.

The apparatus for introducing an explosive mixture is shown in detail in Figs. 4 and 5.

A fuel reservoir, 27 (see Fig. 1), is connected to a vaporizer, 26, which communicates with the combustion chambers 1 by a pipe controlled by the poppet-valve 24 and pipes 23 leading to a four-way valve, 22. The sparking device, located in the chamber 29, consists of a rocking wiper, 30, secured on the shaft 31, which is provided with an operating lever, 32, and a flat spring, 33, attached to an insulated rod, 34. The wires 40 and 41 connect the sparking device with a battery not shown. The tappet arm 28 on the shaft 12 operates both the valves 10 and the wiper 30 by means of the slotted link 36 carrying the pins 37 and 38.

The valve 22, which controls the flow of gases to or from the combustion chambers, is also operated from the rock shaft 12 by the tappet 21, the latter having a cam surface, 21a, which operates one arm of a loosely pivoted bell crank tappet lever, 22c. This arm of the tappet lever 22c engages with the slotted link 22b, which is loosely connected to the handle 22a on the stem of the valve 22. The other arm of the lever 22c constitutes a tappet which, by engaging the part 21b of the tappet 21, operates the spring-actuated valve stem 25 of the check valve 24, so that the tappet 21 serves the double function of reversing the valve 22 and keeping the valve 24 open. The operation is now evident.

Motion is imparted to the piston 11 by the motor 15, which causes the water piston to move in the chamber 1 and make room for the charge in this chamber. The valve 22 being properly disposed, the action of the piston 11 and the travel of the water piston cause successive inflow of explosive charges and scavenging of the spent gases.

If the explosive charge is assumed to be in chamber 1, the water piston will extend nearly to the valve 7, separated from the water and pressure in the air chamber 8 by this valve and by whatever air has been permitted access through the valve 5a. At this point the valve 10 is opened by engagement of the tappet arm 28 with the link 36 and the compressed air, or water under pressure from the air chamber, consequently flows beneath the valve 7. Thus the water piston is forced back against the charge, compressing it to the pressure of the air chamber, which is, of course, that of the head. The charge is prevented from escaping by the closing of valve 24.

Ignition now takes place, resulting from the engagement of the tappet pin 37 with the end of the slot in the link 36, thus rocking the wiper 30 past the spring 33 by the connection of the link 36 to the wiper arm 32. The slot in the link 36 permits the valve 10 to close the moment the wiper arm 32 passes out of

engagement with the spring 33. The expansion of the gases in the chamber 1 causes the water piston to be driven with great energy and speed through nozzle 3.

The spaces vacated in the chamber 1 and casing 4 are filled instantly (practically simultaneously with the expansion) by air through valve 5a. That portion of the air admixed with the spent gases in the casing 4 of the expansion chamber will be cut off and separated from that in the charged chamber 1 by the incoming water through valve 5. The portion of air and gas in the casing 4 is thus trapped beneath valve 7, ready to be driven into the air chamber at the next operation of the water piston. That portion of the spent gases and air in the chamber 1 will pass out as exhaust. The cycle of operation of the Smyth pump is shown in Fig. 6, and described as follows:

1. Explosive charge being taken in the left-hand chamber and discharging the spent gases from the other chamber, the piston 11 being midway of its stroke, traveling to the right.
2. A fresh charge in the left-hand chamber and the right-hand chamber completely occupied by the water piston, the spent gases of the previous explosion having been driven out by the water.
3. Charge in the left-hand chamber compressed to less than half of its original bulk by the admission of air pressure between the water piston and the check valve 7.
4. Expansion of charge due to explosion and the travel of the water into the air chamber, preceded by the volume of air from beneath the valve 7. The inrush of the water through the lower check valve 5 induced by the injector, gravity and atmospheric pressure is also shown.
5. Left-hand chamber almost filled with water.

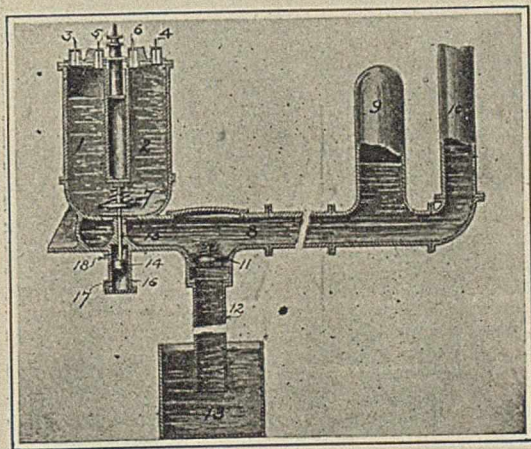


FIG. 7.—THE CHANCE APPARATUS FOR OPERATING INTERNAL COMBUSTION PUMPS AND COMPRESSORS

6. Condition similar to 1, but reversed as to direction of the flow of gases and the movement of the water piston.
7. Charge in the right-hand chamber. The left-hand chamber is completely scavenged of spent gases from previous explosion.
8. Explosive charge in the right-hand chamber ready to be ignited, thus completing the cycle.

It has been the custom in explosive pumps having two combustion chambers to cause the charge to be drawn into one chamber by the continued travel of the liquid piston after the waste gases in the other chamber have dropped to low pressure. In pumps of this character the continued travel of the liquid piston must be sufficient to draw in the new charge; the expulsion of the waste gases is accomplished by the return stroke of the piston and must be completed before the return stroke can compress the charge in the other chamber.

The necessity for prolonging both the out stroke and the return stroke of the piston to permit the drawing in of the charge and the expulsion of the waste gases, introduces elements of

time by which the capacity and speed of operation of the pump are correspondingly reduced.

H. M. Chance and T. M. Chance, of Philadelphia, Pa., have devised a method of operating such pumps whereby the rapidity of action is increased by decreasing the time necessary for each stroke and return stroke.

The apparatus, as seen in Fig. 7, comprises two combustion chambers 1 and 2 provided with inlet valves 3 and 4 for the introduction of the combustible mixture and two exhaust valves 5 and 6 for the discharge of the waste gases.

A spring-pressed valve, 7, which controls the communication between the two chambers 1 and 2, is operated by the rod 15 and piston 14 in the cylinder 16. This cylinder communicates with the liquid in the chambers 1 and 2 by the passage 17, and the connection 18 leads to a source of pressure slightly greater than that at which it is desired to exhaust the waste products of combustion. The usual conduit 8 is connected to the air chamber 9, delivery pipe 10 and suction pipe 12 with inlet valve 11. This suction pipe 12 is connected with the source of supply 13.

Let it be assumed that the chamber 1 contains a compressed combustible charge which has just been ignited, the valve 7 being open, the liquid in the conduit 8 is given a high velocity,

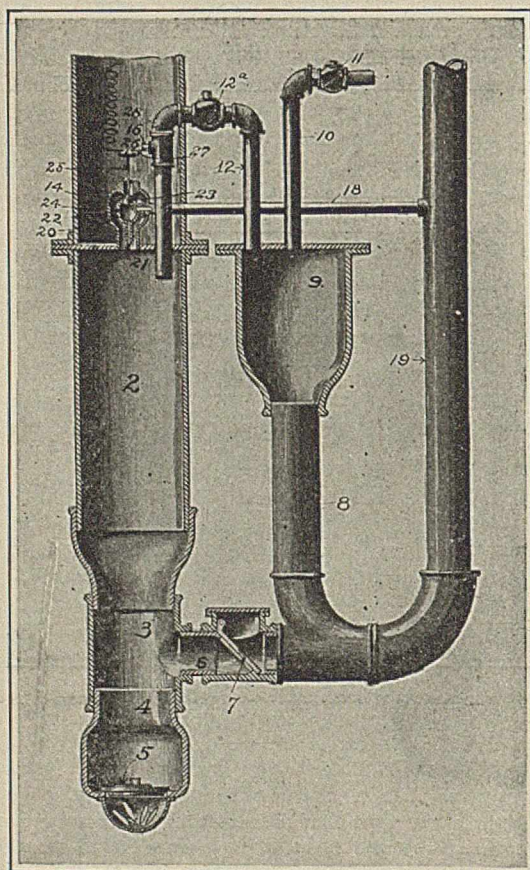


FIG. 8—THE WHITE PUMP FOR UTILIZING FULL STATIC PRESSURE IN COMPRESSING THE CHARGE

When the pressure in the chamber 1 falls below the predetermined pressure at which it is desired to open the exhaust, the valve 5 is opened and the valve 7 closed by the piston 14, operated by a pressure slightly greater than that now existing in the chamber 1, the scavenging taking place at atmospheric pressure. The liquid in the chamber 2 now falls by gravity; the inlet valve 4 opens; and a new combustible mixture enters the chamber 2. At the same time the liquid rises in the chamber 1, expelling the waste gases. The valve 4 then closes.

Upon commencing the return stroke the liquid in the conduit 8 opens the valve 7, forcing the liquid into the chamber 1 and

closing the valve 5 by the impact of the liquid. The momentum of the liquid in the conduit 8 now causes the liquid to rise in the chamber 2, compressing the charge in that chamber. The cycle is then repeated.

It will thus be seen that a new charge of combustible mixture is introduced while the products of combustion are being discharged, obviating the necessity for prolonging both out stroke and return stroke, and in this manner increasing both the speed and the capacity of the pump.

In explosive pumps it has been the custom to compress the combustible charge in the same chamber in which it is exploded. A recent invention of C. E. White, of San Francisco, Cal., is a pump in which the charge is first drawn into a separate chamber and forced thence into the usual combustion chamber by the pressure of the liquid in the delivery pipe. By this means, it is claimed, the full static pressure is utilized in compressing the charge.

The White pump, as shown in Fig. 8, comprises a combustion chamber, 2, connected to the supply pipe 4 having the usual inlet valve 5, by the tee 3, which communicates with the delivery pipe 19 through the pipe 6, in which is placed a check valve, 7. This delivery pipe 19 is connected to the gas chamber 9 by a pipe, 8.

The gas supply pipe 10, having the check valve 11, communicates with the chamber 9, the latter being connected with the chamber 2 by the pipe 12 having a check valve 12a.

The exhaust valve 14 comprises a cup, 20, provided with a bored extension, 21, and a diaphragm, 22. This diaphragm 22 is provided with a valve, 23, adapted to enter a seat, 24, the stem 25 of the valve 23 is provided with a contact, 26, to bridge the terminals 27 and 28 to close the circuit of the spark plug 16. A pipe, 18, connects the delivery pipe 19 with the cup 20.

Let it be assumed that the pump is filled with liquid and an explosive mixture, the contact 26 closes the ignition circuit through the terminals 27 and 28 and the charge is exploded. The liquid is thus forced through the pipe 6, closing the valve 5 and opening the valve 7. The liquid in the chamber 9 and pipe 8 moves downwardly, creating a suction in the chamber 9, whereupon the valve 11 opens and causes a fresh supply of gas to be drawn into the chamber 9. As the liquid moves upwardly in the pipe 19, the pressure on the diaphragm 22 is removed, and the contact 26 is moved downwardly, permitting the escape of the waste gases.

After the explosive force is spent, the liquid in the pipe 19 closes the valve 7 and the liquid piston in the pipe 8 and the chamber 9 forces the explosive mixture into the chamber 2 and compresses the charge against the water, which has by this time risen in the chamber 2.

The cycle is then repeated.

In internal combustion pumps it has been necessary after each explosion to draw in a fresh charge of gaseous mixture from an outside source. This has necessitated gas and air connections which are impracticable when such pumps are used in mines and in many other instances.

To obviate this difficulty, Poore and Harvey, of London, England, have recently constructed a device which generates its own gas by the electrolysis of water. No pipe connections whatever except those used for suction and delivery of the water are thus needed.

In Fig. 9, the current for decomposing the water is supplied by the dynamo *L* to the contact *L*¹, which makes a sliding contact with the chamber *G* at prearranged periods of time. The upper part *B* of the body *A* of the pump forms the explosion chamber. The gases formed by the electrolysis of the water are mingled with air introduced in the chamber *B* through the air valve *M*, and are exploded by the spark plug *K*¹.

The ignition circuit comprises a primary winding, *J*, having a battery, *J*¹, and switch, *J*², and a secondary winding, *K*. The

float *F*, carried by the lever *H*, which is pivoted at *H*¹, operates to close the firing circuit.

The apparatus is operated by closing the switch *J*² and turning on the decomposing current. The float *F* is then in its lowermost position, and the contacts *L*¹ are in contact with the chamber *G*.

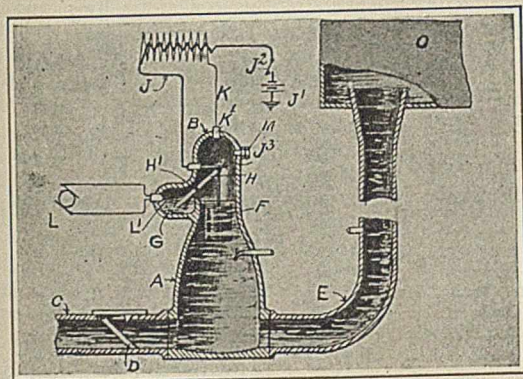


FIG. 9—POORE AND HARVEY DEVICE FOR GENERATING ITS OWN GAS BY THE ELECTROLYSIS OF WATER

As the water rises, the float *F* moves the chamber *G* out of contact with the contact *L*¹ and, rising still farther, causes the lever *H* to contact with the contact *J*², thereby closing the primary circuit. The charge is then fired by the plug *K*¹.

The force of the explosion expels the water up the delivery pipe *E* into the tank *O*, and also draws a fresh supply of water through the suction pipe *C*. The return movement of the water closes the check valve *D* and compresses the new charge of explosive mixture. The cycle is then repeated.

The pipe *N* is used to form a spray for condensing the vapor caused by the explosion.

THE ALKALI INSPECTOR'S REPORT FOR 1912

The forty-ninth annual report of the Chief Inspector of Alkali, etc., Works for England, Ireland and Wales, which was issued during July, is reviewed in *The Chemical Trade Journal*, 53, 49.

The number of works registered under the Act during 1912 was 1,305, of which 73 were "alkali works." The general average amounts of acid escaping from the exits for waste gases in works under inspection showed slight variations from the preceding year. The acidity of gases of all chimneys, however, showed a distinct increase, being equivalent to 1.218 grains of sulfuric anhydride per cubic foot, as compared with 1.192 grains during 1911. This increase was mainly due to those works wherein ores containing metallic sulfides were calcined.

For many years past there has been a slow but continued reduction in the number of works registered as sulfuric acid works. This has been due to the increase in aggregate production accompanied by the closing of works of small capacity, concurrently with increased productive capacity in other works already in operation. The various forms of mechanical burner came into more extended operation during 1912. The use of fans also—so efficient in maintaining constant working conditions—became more widely adopted. There is a general tendency to appreciate the advantages of increased Gay-Lussac tower space, and in many works this has been increased. The "Opl" system continued in constant use, and during 1912 a second installation of this system was put into successful operation. So far as regards the escape of noxious gases, both of these sulfuric acid plants gave every satisfaction; but it is still too soon to express an opinion as to wear and tear. Mention is made of a fatality which occurred in a sulfuric acid works during the removal of packing from a Gay-Lussac tower, owing to the presence of oxides of nitrogen

Those sulfuric acid works in which the catalytic or contact processes were used were conducted during the year without any marked change in methods of working, nor was there any change in the number of works, although extension of plant effected an increase in the capacity for production. The methods previously in operation for the concentration of sulfuric acid continued in use. Undue escapes of acid gases were observed coming from over-heat pans on several occasions; these irregularities were associated with the methods of firing and with the disposition of the fireplaces. The Kessler system continued to yield exit gases low in total acidity and to operate in a regular way. The use of fans instead of the original steam-jet form of draught-inducer was further extended. The cascade system gained in favor and is now in widely extended operation; silica ware and tantiron continued to be used as materials of construction of the evaporating vessels. The Gaillard tower continued in use, generally with satisfactory results as to escaping gases, but in one works there was much trouble.

The chemical manure works were kept actively in operation during 1912. 14,115 tons of guano, 520,270 tons of mineral phosphates, and 123,580 tons of nitrate of soda were imported during the year. The exports of ammonium sulfate amounted to 287,000 tons, as compared with a home consumption of 90,000 tons. The use of mechanical methods for dealing with products was extended during the year, and the direct method of producing ammonium sulfate was put into more extended employment, chiefly the coke-oven works. The loss of ammonia during the handling and storing of ammoniacal liquor is very considerable. Attention continued to be given during 1912 to the formation of ammonium sulfate from gases containing ammonia and hydrogen sulfide, without the use of sulfuric acid, but so far as practical production in England is concerned it cannot be said that permanent success has yet been obtained.

The bulk of the chlorine was produced in alkali works, and, as these works were seriously affected from the temporary scarcity of fuel, the production of chlorine was restricted for a time. The mechanical arrangements for the production of bleaching powder continued to work satisfactorily, and their use was further extended.

A further increase in the number of tinsplate flux works was noted, the total registered being 73. Great activity was apparent in the earlier part of 1912, but towards the end of the year the demand for tinsplates was decidedly lessened. The methods in use in the recent past for minimizing the escape of hydrochloric acid from these works proved satisfactory in all cases in which efficient means were provided for washing the furnace gases or for washing the scruff before this was charged into the furnace for roasting. The general average escape of hydrochloric acid fell from 0.219 in 1911 to 0.170 in 1912.

The number of sulfide works under inspection increased from 69 in 1911 to 78 in 1912. During the year four works in which certain sulfide colors were manufactured came under consideration and were found to require registration in this class. Much improvement inside these works as well as to their immediate neighborhood has been effected as the result of inspection, and what was formerly an objectionable escape of a noxious gas has been entirely removed. This desirable result has been attained partly by the provision of means for preventing access to the air of any hydrogen sulfide evolved, and partly by reducing the amount of noxious gases requiring to be dealt with through improvements in methods of manufacture.

The total number of lead poisoning cases reported from all industries in 1912 was 587, of which number 44 were fatal, as against 669 (37 fatal) in 1911. Only 5 cases of arsenic poisoning occurred, and 3 of these were due to the handling of birdskins imported from the West Indies; a fourth case occurred to a workman employed in a paper mill in dissolving block tin in concentrated hydrochloric acid. Although helmets and air

tubes, or similar apparatus, with compressed oxygen or air, are supplied in many works, folds of flannel still seem to be the more general form of respirator.

WANTED: NEW USES FOR BROMINE

A prize of \$2,500 is offered by the Deutsche Bromkonvention G. m. b. H., Leopoldshall-Stassfurt, Germany, to the discoverer of a process or compound leading to a new or an increased consumption of bromine. The following are some of the conditions attached to this competition: The new discovery must represent a technical innovation and must not adversely affect existing uses of bromine. The process must be applied in practice at the latest one year after the awarding of the prize. The process must, in the opinion of the jury, lead to a considerable increase in the European consumption of bromine at a suitable price. Competitors must send in their processes by January 1, 1914, at the latest.

THE RADIUM MARKET

There has recently been a marked reduction in the prices of radium preparations. Earlier in the year radium bromide sold for \$105.60 per milligram in Germany; and, in April, a New York firm offered for sale any part of 800 milligrams of radium bromide, gauging from 25 per cent to 90 per cent purity, to be delivered in tubes of 10 to 125 milligrams capacity, at \$90 to \$100 per milligram. In July, however, the K. K. Montan-Gesellschaft in Wien, which produces radium salts from pitchblende, made sales at \$43.20 to \$52.80 per milligram, depending upon the activity. The recent decrease in price is thus explained in *Chemie*, 6, No. 1530: During the last three years, mesothorium, as well as radiothorium, began to be employed in place of radium, especially in medicine. Mesothorium is obtainable at a cost of \$32.40 per milligram in Germany and, in addition, may be procured four times more active than radium. While the life of mesothorium is short, by mixing it with radium salts a preparation of long life may be obtained. The total production of radium bromide per year is between 2 and 3 grams. In 1911, the radium preparations produced by the Austrian Radiumpraeparatefabrik amounted to 14.146 grams, containing 2.647 grams of pure radium chloride, valued at \$214,900.

The Radium Hill Company conducted operations in 1912 in Australia which resulted in the production of 2.5 milligrams of pure radium bromide; it is reported that 350 milligrams more are in the laboratory in various stages of purification. Recently two American firms have been organized for the preparation of radium salts, etc.; these are the Radium Company of America, of Philadelphia and Sellersville, Pa., and the Radium Chemical Company, of Pittsburgh, Pa. The latter company is marketing radium salts and applicators, radium drinking water, radium bath water, radium compresses, and radioactive earth.

THE MANUFACTURE OF COPPER WIRE BY ELECTRO-DEPOSITION

According to the *Brass World*, W. E. Gibbs has devised a process for the manufacture of copper wire by electrode position. Previous attempts to accomplish this object have been unsuccessful.

The Gibbs method requires a fine copper wire as a core for making the desired product, and the additional copper is deposited on it while it moves through a tank containing the solution. The fine copper wire is made endless and passed through a regular plating solution containing sulfate of copper and a little sulfuric acid. The wire passes over grooved rollers operated from outside while passing through the tank. After leaving the tank, the wire passes through a small rinsing tank to remove the solution, and then goes to a reel around which it passes a number of times, returning again to the plating tank. The

plating may thus be continued until the required thickness is obtained.

THE MELTING POINTS OF ELEMENTS OF ATOMIC WEIGHT 48 TO 59

Burgess and Waltenberg¹ have determined the melting points of the refractory elements of the iron group by means of the micro-pyrometer,² obtaining the following results:

Metal	Melting point with micro-pyrometer	Purity Per cent.	Probable melting point of pure element
Nickel.....	{ 1452°(a) 1449(b)	99.83 ₅	1452° ± 3
Cobalt.....	{ 1477 ± 2 1478 ± 1(c)	99.95 ₁	1478° ± 5
Iron.....	{ 1533 ± 1(d) 1528(b)	99.98 ± 01	1530° ± 5
Manganese.....	1255	97 to 98	1260° ± 20
Chromium.....	1520	98 to 99	1520° to > Fe
Vanadium.....	1720	97 to 98	1720° ± 20
Titanium.....	1794 ± 12	99.9+	1795° ± 15

(a) Assumed value on platinum strip; also observed value on iridium strip.

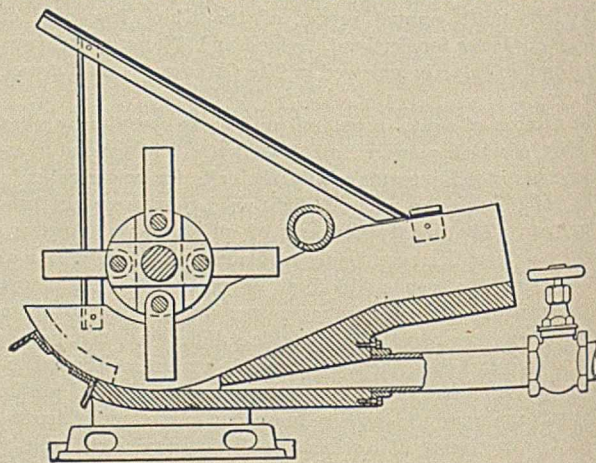
(b) Crucible melts in electric furnace.

(c) Crucible melts in electric furnace; also on iridium strip with micro-pyrometer.

(d) Five samples all agreeing to within 3°.

THE GRANULATION OF SLAG

Recently G. Juntzen described a method of granulating slag by means of a jet of air before the German Metallurgical Association. In this procedure, which has been in use for over a year at the Buderus Works, where about 100 tons are produced daily, the slag run from the furnace is conducted through a conduit to a rotating drum 45 feet in length and 6 feet in diameter. Upon entering this drum, it is met by a jet of compressed air, which throws it against the sides, which are kept cooled by the exterior circulation of water; in this way the disseminated



DEVICE FOR GRANULATING MOLTEN SLAG

slag always encounters cool surfaces. Scrapers within the drum prevent adhesion of the slag, which eventually is removed at the bottom end of the cylinder in small grains, said to be without lumps. It is claimed that the slag thus reduced is more satisfactory for cement-making than that granulated by water; it is dry and also more dense. Such granulated slag is suitable for use as sand.

In the process of J. T. Bergwith for the granulation of blast-furnace slag, the attempt is made to produce a dry granulated slag, notwithstanding the employment of water.³

¹ *J. Wash. Acad. Sci.*, 3, No. 13, 371.

² *Bull. Bureau of Standards*, 9, 475.

³ *Eng. Min. J.*, 96, 55.

This is done by delivering the stream of molten slag to the action of a rotary feeder, driven at a high rate of speed, the feeder members of which scrape or shear off extended portions of the stream of slag and project it upward into the air. Simultaneously with the separation of the portions of slag from the main stream by the feeders, it is subjected to the action of a small stream of water, which combines with the feeders to produce the granulating action. During its flight through the air, it cools and dries, so that it is deposited in a nearly dry granulated state. As may be seen from the accompanying illustration, the lower portion of the feeder wheel runs in a suitable trough or runner, the sides of which rise nearly to the hub of the wheel. A guard bar, or shield, is arranged above the wheel. The trough, or runner, is approximately V-shaped in cross-section before it reaches the disintegrator, but broadens out to a flat-bottom trough in that portion which underlies the feeder blade. At a point approximately central to the lowermost part of the trough, a wide, relatively thin water inlet communicates with the trough. The feeder wheel comprises a number of plates supporting rods, or bars, separated by a number of face blocks.

SOME INNOVATIONS IN THE PAPER INDUSTRY

Philip B. Sadtler, of the Swenson Evaporator Company, of Chicago, Ill., has pointed out that by employing a battery of blow tanks to systematically wash the liquor out of the pulp, this may be accomplished with the least expenditure of water and the highest cleansing effect. This is, moreover, said to be one of the best methods for eliminating the objectionable odor of the sulfate pulp mill. Pulp is blown under pressure from the digesters directly into the blow tanks, the battery being used in countercurrent system for washing. The small expenditure of water leaves a minimum of water to be evaporated.

An idea which Mr. Sadtler has included in the design of the apparatus in the Chesapeake Pulp & Paper Company, at West Point, Va., is the use of waste heat gases from the rotary black ash furnaces to produce the generation of steam in the boiler; this steam is then used to carry out the evaporation of the black liquor in the evaporator. This idea of employing waste heat from special furnaces to generate steam in boilers is a novelty, it is said, in the pulp field.

THE GAS AND ELECTRIC CONSUMPTION IN GREATER NEW YORK

The Coal and Coke Operator, 21, No. 11, 231, reports that there were 1,101,174 consumers of gas in Greater New York in 1912. The 13 different gas companies which make and sell this product made and bought during the year 55,542,488 cubic feet of gas, of which 47,775,188 cubic feet were sold to consumers. The Consolidated Gas Company had a total of 483,727 consumers, while the Brooklyn Union Gas Company had 361,845.

The electric companies generated and bought 702,174,871 kilowatt hours of electricity and sold 518,294,646 kilowatt hours. The reports of the six electric companies serving Greater New York show that there were 229,758 active meters in operation; of these, the New York Edison Company had 168,814 active meters and the Edison Illuminating Company of Brooklyn 41,684.

THE CONSUMPTION OF THORIUM IN THE UNITED STATES

It is noted in the *Chemiker-Zeitung*, 37, No. 77, 776, that the only company in the United States which produces thorium compounds from monazite sand is the Welsbach Company, of Gloucester City, N. J., although there are two or three other concerns which obtain thoria from waste Welsbach mantles. The mining of the monazite deposits in the Carolinas, which supplied 1½ million pounds in 1912, has been discontinued, owing to the decrease in the import tax from 6 cents to 4 cents per pound. The number of gas mantle factories has diminished from 89, in 1909, to 50, the most of which are found in the States of Ohio, New York, Pennsylvania, and Illinois. In all, 65 million mantles are manufactured per year, of which 40 per cent are made by the Welsbach Company. The consumption of thorium compounds for this purpose amounts to about 220,000 pounds, more than half of which is imported from Germany. The duty on thorium salts, gas mantles and spent mantles is 40 per cent of their value. According to a report made by the General Gas Mantle Company, of Camden, N. J., the cost of 1,000 incandescent gas mantles is \$42.07 in New York, and the German manufacturers can sell the same for \$42.50, including the import tax.

SCIENTIFIC SOCIETIES

SOCIETY FOR PROMOTION OF ENGINEERING EDUCATION

The twenty-first annual meeting of the Society for the Promotion of Engineering Education was held at the University of Minnesota, Minneapolis, June 24 to 26. Among the conspicuous features of the program as reported by *Engineering Record*, July 12, 1913, were a series of papers on the design and construction of buildings for technical schools, papers on hydraulic engineering education presented at a joint session with the American Water Works Association, a symposium on highway engineering education, a paper by a representative of the Pennsylvania Railroad on cooperation between technical schools and the industries, and a paper by a prominent manufacturer advocating a movement in the direction of standardization of technical terms.

Last year at the Boston meeting of the society a number of experts on scientific management showed that there was a connection between this subject and education. This year Director H. S. Person, of the Tuck School of Administration and Finance, Dartmouth College, was requested to apply the principles of scientific management to college administration. In doing so he showed that by functionalizing college work greater efficiency can be obtained than by present methods. The functions involved in education are teaching, administration, advising, and guiding non-classroom educative influences. As teachers

are not equally fitted for all functions, it is advisable to assign to each only those for which he is best adapted.

At a session held jointly with the American Waterworks Association, Prof. D. W. Mead, of the University of Wisconsin, and O. L. Waller, of the State College of Washington, read papers covering the preparation of young men as hydraulic engineers. The authors showed that the best preparation for a hydraulic engineer is that which will teach him to think. As Professor Mead stated, the value of any education is not so much in the acquirement of knowledge as in the ability to acquire it. He pointed out that the tendency of educational methods is to discourage the development of judgment, for the students are apt to accept the dictum of their teacher as infallible and hence not subject to their critical inspection.

Professors Sherman and Schlafly, of the Ohio State University, explained how their students are encouraged to get into commercial work in the summer time. If this is impracticable, the instructors take commercial contracts and employ the students as assistants. These "practical" summer surveying camps take the places of "practice" camps and are giving excellent results. Under this plan the students feel and act more like engineers than under the old plan. Prof. F. P. McKibben, of Lehigh University, gave details of the experience of his students

in obtaining summer employment and of the excellent results obtained therefrom. The discussion brought out a general sentiment in favor of the use of the summer months in actual work and emphasized the duty of future employers of the men to provide such work for them.

A marked tendency to lengthen the college course to five or even six years has been conspicuous of late. A number of schools have introduced longer courses, these being either required or optional. This tendency is opposed by some leading educators, and two of them, President A. C. Humphreys, of the Stevens Institute of Technology, and Prof. G. F. Swain, of Harvard University, vigorously voiced their opposition at the Minneapolis meeting. They claim that four years cover a period long enough for a boy to remain from actual work and that the expenditure of an extra year or two does not bring commensurate results. In championing the other side the advocates of the longer course pointed out that the extra time is not wanted for more technology but for studies selected to prepare for better citizenship, and that on this basis its use is justified. Obviously no agreement was reached or could be reached as to the proper length of a course, but the discussion served to keep the topic alive and to put the authorities in charge of technical schools in a defensive attitude toward their present practice, whatever that may be.

A subject which affects all engineers, because it relates to the ease and understanding with which they can read technical literature, is the standardization of technical terms. Mr. D. M. Wright outlined the results of his experience in a paper in which he recommended the appointment of a committee to study the subject.

At the request of the society a representative of the Pennsylvania Railroad, Mr. Ivy L. Lee, gave the results of the experience of that company with the many technical graduates who have entered the company's employ. The faults which they have noted are a lack of practical experience and judgment, an idea that they are far superior to the rest of mankind, and a certain narrowness of mind, inculcated through a too exclusive attention in college to mathematics and theoretical science and to a too great neglect of those broader subjects such as political economy, history and general literature.

Mr. Lee believes that these faults can be overcome and that by directing attention to the shortcomings and limitations as well as the strong points of the students, teachers can do much to assist them in eliminating their weaknesses.

Of all the difficult concepts which students have to grasp that of the relation of force, mass, weight and acceleration is one requiring possibly the greatest mental effort. Teachers of mathematics, physics, mechanics and engineering struggle with it. The situation is aggravated by the use of many different units for these quantities. Prof. E. V. Huntington, of Harvard University, proposed a simplification of these by eliminating most of these units and substituting what he calls the "standard" weight and the "local" weight for the usual "mass" and "weight." Naturally a proposition like this stirred up much discussion and the whole subject was referred to a special committee on the teaching of mechanics to engineering students, which was appointed forthwith, with Prof. E. R. Maurer, of the University of Wisconsin, as chairman.

The cause of the much-discussed thesis, once an essential part of the engineering curriculum, was championed by Prof. H. Wade Hibbard, of the University of Missouri, and many other teachers.

In his presidential address Prof. William T. Magruder, of the Ohio State University, painted a picture of an ideal instructor. A number of committees appointed last year to study the improvement of instruction in the several branches of engineering reported progress and were continued for another year with instructions to carry out their own suggestions. The chairmen

of these are Profs. F. P. McKibben, of Lehigh University; C. F. Scott, of Yale University; A. M. Greene, Jr., of Rensselaer Polytechnic Institute; and F. W. Sperr, of the Michigan College of Mines. The committee on college administration, which made a report covering such matters as the salaries of professors, falling off in the number of technical students, leaves of absence, etc., was continued. A new committee on the teaching of physics to engineering students, with Prof. G. V. Wendell, of Columbia University, as chairman, was also appointed. Altogether the society has a large amount of committee work in progress, greater than at any other time in its history.

The following elections occurred this year: *President*, G. C. Anthony, Tufts College, Mass.; *Vice-Presidents*, H. S. Jacoby, Ithaca, N. Y. and D. C. Humphreys, Lexington, Va.; *Secretary*, H. H. Norris, Ithaca, N. Y.; *Treasurer*, W. O. Wiley, New York, N. Y.; *Councillors*, H. W. Tyler, Boston Mass.; J. F. Hayford, Evanston, Ill.; A. S. Langsdorf, St. Louis, Mo.; S. M. Woodward, Iowa City, Ia.; M. S. Ketchum, Boulder, Col.; F. P. Spalding, Columbia, Mo.; and P. F. Walker, Lawrence, Kan.

Several important actions were taken at the meeting. The council voted to take the steps necessary to bring before the membership a plan to make the secretaryship permanent. Princeton was recommended for the 1914 meeting and California for the year following. The secretary was instructed to confer with the officers of other societies with a view to reducing the conflict between convention dates.

INTERNATIONAL ENGINEERING CONGRESS, 1915

In connection with the Panama-Pacific International Exposition which will be held in San Francisco in 1915, there will be an International Engineering Congress, in which engineers throughout the world will be invited to participate. The congress is to be conducted under the auspices of the following five National Engineering Societies: American Society of Civil Engineers, American Institute of Mining Engineers, The American Society of Mechanical Engineers, American Institute of Electrical Engineers, and The Society of Naval Architects and Marine Engineers. These societies, acting in cooperation, have appointed a permanent Committee on Management, consisting of the Presidents and Secretaries of each of these Societies, and eighteen members resident in San Francisco.

Thus constituted, the personnel of the Committee is as follows:

FOR THE AMERICAN SOCIETY OF CIVIL ENGINEERS.—Geo. F. Swain, *President*; Chas. Warren Hunt, *Secretary*; Arthur L. Adams, W. A. Cattell, Chas. Derleth, Jr., Chas. D. Marx.

FOR THE AMERICAN INSTITUTE OF MINING ENGINEERS.—Charles F. Rand, *President*; Bradley Stoughton, *Secretary*; H. F. Bain, Edw. H. Benjamin, Newton Cleaveland, Wm. S. Noyes.

FOR THE AMERICAN SOCIETY OF MECHANICAL ENGINEERS.—W. F. M. Goss, *President*; Calvin W. Rice, *Secretary*; W. F. Durand, R. S. Moore, T. W. Ransom, C. R. Weymouth.

FOR THE AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS.—Ralph Davenport Mershon, *President*; F. L. Hutchinson, *Secretary*; J. F. De Rémer, A. M. Hunt.

FOR THE SOCIETY OF NAVAL ARCHITECTS AND MARINE ENGINEERS.—Robert M. Thompson, *President*; D. H. Cox, *Secretary*; Geo. W. Dickie, W. G. Dodd, Wm. R. Eckart, H. P. Frear.

The Committee has effected a permanent organization, with Prof. Wm. F. Durand as Chairman, and W. A. Cattell as Secretary-Treasurer, and has established executive offices in the Foxcroft Building, 68 Post Street, San Francisco.

The Presidents and Secretaries of the five national societies will constitute a Committee on participation, through whom all invitations to participate in the Congress will be issued to governments, engineering societies, and individuals.

The actual management of the Congress and the work of securing and publishing papers will be in charge of the members

of the Committee resident in San Francisco. The work of the Resident Members has been assigned to different sub-committees, and Chairman Durand has made the following appointments:

EXECUTIVE COMMITTEE: W. F. Durand, *Chairman, ex-officio*; W. A. Cattell, *Secretary, ex-officio*; E. H. Benjamin, W. G. Dodd, A. M. Hunt.

FINANCE COMMITTEE: W. G. Dodd, *Chairman*; Newton Cleaveland, R. S. Moore.

PAPERS COMMITTEE: A. M. Hunt, *Chairman*; A. L. Adams, H. F. Bain, G. W. Dickie, W. R. Eckart, C. D. Marx, C. R. Weymouth.

PUBLICITY COMMITTEE: W. A. Cattell, *Chairman*; C. Derleth, Jr., W. S. Noyes, T. W. Ransom.

LOCAL AFFAIRS COMMITTEE: E. H. Benjamin, *Chairman*; J. G. De Remer, H. P. Frear.

The scope of the Congress has not as yet been definitely determined, but it is hoped to make it widely representative of the best engineering practice throughout the world, and it is intended that the papers, discussions and proceedings shall constitute an adequate review of the progress made during the past decade and an authoritative presentation of the latest developments and most approved practices in the various branches of engineering work.

The Committee of Management has extended a most cordial invitation to the officers and members of the American Chemical Society to attend and to participate in the proceedings of this Congress. The formal invitation, handsomely engraved, is now in the hands of Secretary Parsons and will be presented at the next meeting of the Council.

AMERICAN ENGINEERS ENTERTAINED IN GERMANY

Upon invitation of the Verein Deutscher Ingenieure, three hundred American engineers and their lady companions attended the Fifty-fourth Meeting at Leipzig, Germany. A tour of inspection throughout Germany was arranged for the foreign guests by the society.

WILLIAM JAMES EVANS

RESOLUTIONS BY THE CHEMISTS' CLUB

On the occasion of the death of William James Evans, the Board of Trustees of the Chemists' Club of New York City passed the following resolutions in token of their esteem and respect:

The Board of Trustees of the Chemists' Club, for itself and for the members of the Club, take this opportunity of recording its sorrow and regret at the passing of William James Evans, a member of the Board and former Treasurer.

The kindly and friendly interest which Mr. Evans took in the Club and all its functions, his sincere and cordial meeting with all its members, and his efficient and unflagging coöperation in forwarding the interests of the Club made him not only a member of unusual usefulness but also a warm, personal friend of each of us.

The Board of Trustees also takes this opportunity of extending to the family of Mr. Evans its sympathy in the affliction that has befallen them.

WALKER BOWMAN

B. C. HESSE

CHAS. BASKERVILLE

L. H. BAEKELAND

E. G. LOVE

Committee.

MINE SANITATION SECTION OF U. S. BUREAU OF MINES

The United States Bureau of Mines is about to investigate the conditions under which a miner works, believing that the unsanitary conditions which exist in some of the mines as well as in some of the mining towns are a factor in the death rate among the men. It is intimated that these conditions not only unnecessarily cause the death of miners through disease, but are often responsible for accidents which might not happen if the miners were in perfect health.

The bureau has organized what is known as the Mine Sanitation Section, in charge of J. H. White, engineer.

NOTES AND CORRESPONDENCE

A CRITICISM OF CHEMICAL ABSTRACTS

Editor of the Journal of Industrial and Engineering Chemistry:

In the July number of THIS JOURNAL, in an editorial entitled "An Index to Chemical Literature," Dr. L. H. Baekeland makes a criticism of *Chemical Abstracts* which has the appearance of being so serious that I feel some reply must be made to it.

He quotes from a report on the Patent Office in which the Government indexers state that they compared 6 pages of the index of the *Journal of the (London) Chemical Society* for 1909 with 1909 index of *Chemical Abstracts* and that out of 256 articles indexed in the former they failed to find 159 in *Chemical Abstracts*.

This certainly looks bad, and might lead the reader to conclude that *Chemical Abstracts* is not half covering the field.

I have gone minutely over three of the six pages referred to and find the following: Out of 125 articles indexed in the London index, only 4 have not been found in *Chemical Abstracts*.

One cause for failure to find entries is that in 1909 *Chemical Abstracts* was behind on many journals and struggling to catch up (*Chemical Abstracts* was only in its third year and just getting started); about 50 of the missing entries are to be found in the 1910 volume and so would eventually have come into the card index of the Patent Office. But the principal discrepancy is due to different methods of indexing. For instance, the London journal indexes aminoacetanilide under acetanilide, while

Chemical Abstracts indexes it under 'Am. The same applies to nitro and many other compounds. Furthermore, the English journal takes great liberties with the names of organic compounds as they appear in the article, renaming them if it thinks desirable. This has advantages and disadvantages; *Chemical Abstracts* has never done so much renaming.

This variance in indexing between the two journals only emphasizes, of course, the difficulties in the way of preparing a general index.

A letter received by us from Mr. W. F. Woolard, Chief Clerk of the Patent Office, says:

"It is probable that no two chemists, making this comparison, would exactly agree in the number of articles covered by one index and not mentioned in the other, without consulting the original monographs. Different abstractors will treat the same article from different view points, so that it becomes at times difficult to say whether or not an article has been abstracted by one journal and omitted by the other, without reference to and study of the original articles. For this detailed study there was neither time nor necessity in our investigation.

"The object which the office had in making this comparison was not with a view to criticism of the work of the American Society, but to ascertain whether subject matter appeared in the English work, which could not be found, in the American in order to decide whether or not it was advisable to index both works."

It is evidently out of the question for *Chemical Abstracts* to index so minutely as to cover all the entries in the English index. We have already three times as many entries as it has, and it, as well as the index to *Chemisches Zentralblatt*, has, for some time, been regularly checked to see that no article is omitted. We do not pretend that of the articles in the 600 journals which we now have listed, all have been abstracted quickly. Many have been delayed and perhaps some missed altogether. Particular journals are constantly falling behind for various causes and it is a continual struggle to bring them up-to-date. Nevertheless, in 1909 the abstracting was not in the state that Dr. Baekeland's well-meant criticism might imply, and at the present time the material in nearly all departments is reaching us very promptly indeed.

AUSTIN M. PATERSON

July 25, 1913

PAINTS FOR INDICATING THE USES OF PIPES

Editor of the Journal of Industrial and Engineering Chemistry:

I note with interest, THIS JOURNAL, p. 609, July, 1913, the extract of an article from *Engineering* that in 1911 German engineers "came to a preliminary understanding that it was desirable to paint pipes so as to avoid confusion, etc."

I do not know whether this practice was original with me, but I instituted it in the late fall of 1896, and at the same time instituted a method of painting barrels so as to be able to distinguish them at a glance. In 1903, when we built our present plant, the method of painting pipes was carried out to a nicety and color charts were distributed at various places so that every workman had the key to the colors. Every year that students in industrial chemistry visit our plant, the superintendent makes it a point to show the various colored pipes and their meaning.

My object in writing is, however, not to claim priority, although I never saw it anywhere until some years later I noticed it in one of our battleships. I wish, however, to point out that certain kinds of paint are better than others for this purpose. For instance, it is stated that steam pipes should be white. This is a mistake, because almost all white paints turn yellowish brown in time when subjected to continued heat. The varnish enamels of the Demar mastic type remain white but eventually become brittle and flake. At the same time, all steam pressure pipes excepting those used for heating are usually insulated with magnesia covering. In our plant we found that aluminum paint is ideal for all steam pipes whether covered or uncovered like exhaust pipes and this aluminum paint can be bought from any reputable manufacturer. It should be composed of a hard copal with plenty of diluent in it and must not be of the collodion type.

The other paints should be enamel paints and not oil paints. Enamel paints dry with a smooth gloss and do not take the dust readily. Oil paints take dust and are difficult to clean. Nothing is more obnoxious in a factory than a fine artistic layer of dust on the top and sides of painted pipe.

In addition to the enameling of pipes, we mark each one with a key number showing the contents.

This system is carried out in our works to other apparatus. For instance, we have about twenty eccentric rotary pumps for handling volatile and other liquids. These pumps are all enameled to correspond with the pipes and the chart. I mentioned the subject of barrels, and we paint all insulating and water-proof paint barrels one color and all oil-paint another color, so that there can never be a mistake in labeling them afterwards.

The color scheme is carried out in every department. The factory orders which come from the New York office are made out in quintuplicate of different colors. All stationery and instructions to the shipping office are on bright yellow paper. All manufacturing slips are blue, and so on. In this manner,

when a yellow order sheet is found in the manufacturing department it is at once easy to see that some one from the shipping department is there investigating the date when an order was filled. I could continue this, but it would take too much space. Suffice it to say that these small matters go a great way toward making a factory "fool-proof."

MAXIMILIAN TOCH

9TH ST. AND WEST AVE.
LONG ISLAND CITY
August 5, 1913

ON METHODS OF ANALYSIS OF CRUDE GLYCERINE

Editor of the Journal of Industrial and Engineering Chemistry:

I wish to open a discussion on the present system of the analysis of crude glycerine, and would say at the outset that I believe there is an unhealthy condition existing somewhere. Let me mention a case, withholding the names. A sells two car loads of Soap Lye Crude to B, with C named as referee. A, upon careful analysis, finds 83.8 per cent Glycerol by the International Standard Method, and bills it accordingly. B reports that his chemists find 85.2 per cent Glycerol, but will split the difference. A has been very careful with his work (having been there before), and has his figures from two chemists, who are experienced in carrying out the International Standard Method. A, therefore, has a third portion of the sealed sample, taken by a public sampler, sent to C for analysis, requesting a detailed report, that he may check up the work of his chemists. In a few days the report comes in as follows:

	Per cent
Residue at 160° C.....	12.60
Water.....	5.4
Glycerol by the International Method.....	82.80
	82.85 Av. 82.87
	82.95
Correction for acetylizable impurities in the residue at 160° C.....	0.45
Anhydrous Glycerol.....	82.42

This is not a full report. According to the British Standard Specifications and Recommendations of the Executive Committee, the report should be as follows:

	Per cent
Total Acetyl Value as Glycerol.....	...
Acetyl Value(a) of Residue as Glycerol.....	...
Less Allowance.....	...
Correction for Acetyl Value of Residue as Glycerol.....	...
Glycerol (I. S. M., 1911).....	...
Ash.....	...
Organic Residue.....	...
Free Acid in terms of Na ₂ O.....	...
Alkali Hydroxide, and Carbonate, in terms of Na ₂ O.....	...
(a) Not determined if Total Organic Residue is 2.5 per cent or under in the case of Soap Lye Crude.	

Note that in C's report there is no mention of Ash, Organic Residue, or Acetyl Value of Residue as Glycerol. By the correction, we take it for granted that Organic Residue was over 2.5 per cent, and that it was acetylated.

A, wishing to compare results more fully, wrote the referee asking for the percentage of Ash, Organic Residue at 160° C., and the Acetyl Value of the Residue as Glycerol. The reply was: "It is not customary to give a detailed report." Why? We pay for it. A insisted on a detailed report and the reply was as follows: "It is our custom in making the analysis of samples of Crude Glycerine to make those determinations which are necessary to enable us to report the total glycerol figure. For this reason the only determinations we make are

Total acetylizable matter in the sample as received.
Total residue at 160° C.
Acetylizable matter in the residue non-volatile at 160° C.

"Our customary charge for the above determinations is \$15, and when additional information is desired there is an additional charge."

Now how can C tell whether the non-volatile organic residue

at 160° C. is over 2.5 per cent unless they determine it, and how can it be found unless the Ash is determined? It is worthy of note that the report gives "Correction for Acetylizable impurities in the Residue non-volatile at 160° C. No mention is made of the Acetyl Value of the Residue nor of the Allowance. Was the Allowance made? The percentage of water is given, which is one of the most unsatisfactory of all tests, and which is not called for in the regular forms. Can you blame A for wondering if the International Standard Method was carried out at all, or feeling that he is being handed a gold brick?

One "Authority" (?) on the Analysis of Soap Lye Crude by the International Standard Method refuses to give anything but the final Glycerol Content. Let us study the three different results a moment. Is it likely that B or B's chemists are going to make errors against their interests by three-quarters of a per cent? Is it likely that A's chemists would both make an error of 1.38 per cent, knowing that their figures were sure to be checked? This is not an isolated case. I can cite others like it in every detail, in which other parties were concerned. What is the Answer? Do our leading chemists as referees, relying on their having the last word and their reputation, do careless work or leave the work to incompetent men? Do they carry out the International Standard Method according to specifications, and, if they do, WHY do they object to giving the result at every step? If such a report were given, then we poor unskilful chemists could check our work and find our errors. If we have a standard, let us live up to it.

One who knows writes that in cases of samples being referred to a referee, the reports are "almost always in favor of the buyer." Why? Is there no way to obtain satisfaction in the matter? If the work is done in order to reach the final figure, what objection can be raised to giving the figures according to the regular forms? I hope other chemists and soap-makers will take up the discussion and see if we can start something in the right direction.

E. A. RAY.

[I have in my possession a pound of the sealed sample (referred to above), taken by a public sampler, from the same mixture, and at the same time as were the other three samples, and am willing to submit a portion of it to anyone wishing to examine it.—E. A. R.]

MALDEN, MASS.
July 26 1913

WHAT'S THE MATTER WITH THE AMERICAN CHEMIST?

Editor of the Journal of Industrial and Engineering Chemistry:

I feel the need of expressing myself on some of the ideas put forth in the address of Mr. Daniel M. Grosh in THIS JOURNAL, 5, 692.

Years ago I was engaged with other chemists in promoting the development of the American dyestuff industry, by sitting comfortably in the laboratory and lamenting its comparative insignificance. In the midst of our lamentations the boss walked in and started things by remarking "Quit your tin-canning and get to work."

What is the use in celebrating the ability, the energy and efficiency of the American chemist and damning the manufacturer and capitalist?

The manufacturer and capitalist won't be convinced of the error of their ways, and their lost opportunities for fortune, by talk or printers' ink. They must be shown and they won't need any more showing than their German analogues, though, of course, Rene Bohn, for instance, will have less trouble in showing a German, than John Smith, A. C., will have in showing an American.

John Smith, A. C., is up against certain conditions that he must meet, and "tin-canning" isn't the best method of approach. If John Smith, A. C., has a job and will adapt himself to Amer-

ican manufacturing conditions, he can make good if he has anything to make good on. But if he is infected with *B. research tersanctus*—God help him.

Cords of good white paper and millions of cubic feet of air are used up annually in extolling research, but the average young chemist doesn't know what the word means. As nearly as I can judge, he thinks it means from \$1,500 to \$2,000 a year, a beautiful white laboratory, and plenty of time in which to read the morning paper and wait for inspiration. If he will realize that commercial research means a dollar sign and not a halo, and go to work on the problems that present themselves with the means at his disposal, or that can be obtained by the exercise of all his tact, he may know what research means after a year or two.

As the American dyestuff industry is generally taken as the "horrible example" in these lamentations, I hope to be allowed to state that a large number of useful dyes are made in this country and they are just as good as the same dyes of German make.

Furthermore, if John Smith, A. C., has any well-developed plan by which the American Coal Tar Color industry can be extended, I know where he can find all kinds of financial support for it. It should be understood, however, that while Rene Bohn can probably get backing for a test tube experiment, John Smith, A. C., will have to show something more.

W. H. WATKINS

BUFFALO, N. Y.
Aug. 8, 1913

WHAT'S THE MATTER WITH THE AMERICAN CHEMIST?

Editor of the Journal of Industrial and Engineering Chemistry:

In Mr. J. M. Matthews' reply to Mr. Grosh's article with the above title (THIS JOURNAL, 5, 626 and 692, respectively), he states that one of the main troubles with the American Chemist is that he wants real money for his services, truly a heinous condition, while his German colleague is willing to work on "prospects." We have heard the dogma preached from Wall Street for many years: "Young man, don't work for money. Work for experience and when you are old enough we will give you a nice little pension."

The trouble is not with the chemist but with the manufacturer. Instead of regarding his research chemist as a highly trained specialist, he regards him as a day laborer, makes him punch a time clock and thinks that if favorable results are not obtained immediately, the man is not worth his hire. Would he for one minute think of putting his physician on such a basis—*No Cure, No Pay*?

In the case of the physician or lawyer, even laymen can appreciate the difficulties with which he struggles, but the chemist battles only with "the innate perversity of inanimate things," and that, as we all know, is absurdly easy!

Of course, the manufacturer has had enough experience with "has-been," "would-be" and "analytical" chemists who pretended to be "research" chemists. It is this class who have pattered away the manufacturer's time, money and patience and prejudiced him against research. These "has-been" chemists, too, are always crying that there is a vast difference between laboratory and factory research.

There is only one kind of research, and that is research that brings results to the person who commands it. The skilled research chemist must have a large modicum of "horse-sense" which is acquired neither by experience nor association.

And after all, suppose a young chemist has worked for some time on "prospects" and finally has achieved something for a manufacturer. What guarantee has he that his work will be remunerated? Will there not be hundreds of new chemists waiting eagerly in the bread line for some manufacturer to let them work on prospects?

In a great many cases, having obtained the best products of a man's brain, there would be no further inducement for retaining him.

BROADWAY AND 116TH ST.
NEW YORK CITY
August 16, 1913

SIDNEY BORN

A RAPID METHOD FOR THE DETERMINATION OF FAT IN ICE CREAM

Editor of the Journal of Industrial and Engineering Chemistry:

From time to time there have been published in various journals many methods for the rapid determination of fat in ice cream. I have tried them all. Some required special apparatus or expensive Babcock bottles with glass stopcocks attached. Others required the removal of added sugar. These usually gave results too low. None of them seemed to be entirely satisfactory. Therefore, I decided to try some experiments to meet the following requirements: Time for the complete test must not exceed 10 minutes. The Babcock centrifuge must be used because almost every ice cream factory can boast of that much of a laboratory. Babcock *milk* bottles should be used on account of finer graduations than cream bottles.

After trying various acids and mixtures in varying quantities, I finally hit upon the following simple modification of the Babcock test which does not char the sugar and meets all the requirements aforementioned.

Weigh into a 10 per cent Babcock *milk* bottle, 9 grams of melted sample. Add 20 cc. of glacial acetic acid (gravity 1.049). Mix well and add 10 cc. of sulfuric acid (gravity 1.83). Mix again and proceed as in the regular Babcock test. With the aid of a pair of dividers, read the fat column (from one extreme to the other, in other words read the highest part of the meniscus which appears as a straight line when bottle is held on the level of the eye and away from the source of light) at a temp. of 130° F. The result $\times 2 =$ per cent fat.

I have used this method for about one year and have always obtained a clear straw-colored fat column free from any char or undissolved casein.

HOEFLER ICE CREAM CO.
BUFFALO, N. Y.
July 30, 1913

H. F. LICHTENBERG

PEAT POWDER AS FUEL FOR LOCOMOTIVES

Consul Douglas Jenkins, Goteborg, Sweden, reports that the recent announcement that Von Porat, a Swedish engineer, had perfected a process for utilizing peat powder as fuel for locomotives has awakened interest in the possibility of developing the extensive peat bogs of Sweden.

The powder is manufactured by the Ekelund process. A factory

has been in operation several years at Back Moss, Sweden, under the management of Mr. Herman Ekelund, inventor of the powder process. It does not appear that the process has made much headway as yet, but it is now predicted that in connection with the discovery of Mr. Von Porat, the use of peat powder will in time become extensive.

In the Von Porat system, the peat powder is fed by an automatic process into the furnace of the locomotive, which is specially arranged to consume it. The Ekelund process is on the market in various countries, including the United States, but little has been made public concerning the Von Porat method. According to Mr. Von Porat the results obtained with peat powder may be summed up as follows:

Substantially the same results can be had from 1½ tons of peat powder that 1 ton of coal will produce. Peat powder may be burned with an admixture of about 5 per cent of coal. As to firing with peat powder, the work is almost nothing in comparison with firing with coal, because the powder is forced into the furnace by an automatic process. No change had to be made in the boiler and none in the fire-box, except installing the special apparatus. There is no difficulty in bringing the powder from the tender to the fire-box, as it passes through a conveyance pipe. Another advantage in using peat powder is that no cold air can get into the fire-box and neither smoke nor sparks escape from the smokestack.

As a result of this invention, it is reported that a number of the Swedish railways are preparing to use peat powder instead of coal.

In view of the fact that the Von Porat appliances have not yet been patented, it is impossible to obtain any detailed description of his process or appliances.

THE CHEMICAL COMPOSITION OF COOKED VEGETABLE FOODS. PART III.—CORRECTION

In the article under the above title, THIS JOURNAL, 5, 653, the following changes should be made:

Page 654, first column, 10th line from bottom should read "— 48.92 per cent of solid" and the last line should be "— 10.9 per cent of solid matter."

Page 655, Table III, the heading over the first brace should be "Proximate analysis of the water-free substance."

KATHERINE I. WILLIAMS

PAPER FROM SEAWEED

A Liverpool newspaper states that an English chemist has succeeded in making paper from seaweed. The product is said to be fireproof, waterproof and odorless and is expected to have considerable effect on the present system of wrapping perishable goods for transport.

BOOK REVIEWS

Chemical Works. Their Design, Erection and Equipment.

By S. S. DYSON AND S. S. CLARKSON. 204 pp., 80 illustrations and nine plates. London: Scott, Greenwood and Son. New York: D. Van Nostrand Co. Price, \$7.50 net.

The very attractive title of this book is rather misleading. The general make-up of the book is good, especially the illustrations, but the subject matter is presented in a decidedly elementary and popular style. The eleven chapters include: choice of site, notes on construction, first principles in laying out of works, the power house, sulfuric, hydrochloric, and nitric acid plants, high explosives, sulfate of ammonia, and artificial manure plants, and the general plant. A rather long appendix treats of the English Alkali Works Regulation Act and general welfare work.

The authors state that their aim has been to present, in each chapter, a summary of sound practice in up-to-date construction, to give practical details and to deal with the subject of designing new works as it would be dealt with by a consulting chemical engineer.

There are many general statements, such as "The weight of lead, roughly speaking, is about eleven times that of water" and "..... cast iron is a substance which, in consequence of its varied composition and uncertain properties, it is most difficult to classify," etc., etc., and furthermore there is an absolute lack of cost tables. While the general idea and aim of the authors are excellent there is not enough new material presented to make the book of very much value, particularly in this country.

The authors have omitted modern American factory practice and that, together with the high price charged for the book, will prevent its wide-spread use.

R. K. MURPHY

The Materials Used in Sizing. By W. F. A. ERMEN. Published by D. Van Nostrand Co. 126 pp., 6 illustrations. Price, \$2.00.

The author has brought together, in book form, the subject matter of a course of lectures delivered in the summer session of the Manchester School of Technology in 1911 on "The Materials used in Sizing."

The scope of the book can be perhaps best understood by naming the various chapters into which the book is divided: Introduction, Chapter I, The Starches and other Agglutinants; Chapter II, Weighting Materials; Chapter III, Softening Ingredients; Chapter IV, Antiseptics; Chapter V, Analysis of Sized Warp and Cloth; Chapter VI, The Preparation of Normal Volumetric Solutions; Chapter VII, Tables.

The various sizing agents are considered as to their use, particular physical and chemical properties, tendering effect, tendency to mildew, etc., and such methods of analysis are given as are of value for the rapid evaluation of each particular sizing agent.

The various methods of analysis are considered more from the standpoint of rapidity than great exactness and are treated in an elementary way.

The book will be of some interest to the trained chemist, but its greatest value is that the factory foreman, with limited knowledge of chemistry, will be able to use the rapid tests of identification given.

OTTO KRESS

Fatty Foods, their Practical Examination. By E. RICHARDS BOLTON AND CECIL REVIS. Philadelphia: P. Blakiston's Son & Co., 1913. Small octavo, 371 pages. \$3.50 net.

The subtitle states this to be a handbook for the use of analytical and technical chemists. The point of view from which the book is written is further indicated by the following from the preface: "To the more humble investigator in this difficult field we desire to present the results of many years' experience, with the hope that methods and ideas which have brought success to the authors may enable him also to elucidate the problems with which he may have to grapple, though we cannot lay too much stress on the fact that scientific and skilled adulteration has sounded the death-knell of rough and ready methods of examination." The reader is rarely offered more than one method for any given purpose, is not told which methods are "official" either in the food or the drug industry, is not given the range of "constants" recorded in the literature, but only what the authors consider the usual limits, and is rarely given a reference to the original publication of either the methods or the data which are included in the book.

This willingness of the authors to assume so complete a control of the reader, both as to selection and detail of methods and interpretation of results, is the distinguishing feature of the book and makes it a significant expression of the authors' experience. On the other hand, the professional chemist, or even the reasonably advanced student, should know the sources of his methods and of the data which he uses in the interpretation of his results, and should have command of a considerable range of confirmatory methods. In this country it is important that he also know which methods have received "official" sanction for use in food and drug inspection, and that he be able to interpret his data in the light of "standards of purity" of the Association of Official Agricultural Chemists and of the United States Pharmacopoeia.

Those who have to do with the examination of fatty foods

should examine this work and profit by the ripe experience of its authors, but should use it in conjunction with other books, and not rely so exclusively upon it as the authors seem to contemplate.

The book is abundantly provided with good illustrations and its scope is somewhat broader than might be expected from its title, since it includes chapters on cocoa and chocolate, feeding-stuffs and milk. It may also be noted that the chapter on vegetable oils and fats covers many substances not primarily regarded as foods. The text is not strictly confined to analytical matters, but contains considerable information as to botanical and commercial sources and yields of the fats and oils described.

H. C. SHERMAN

Gasanalytische Methoden. VON WALTHER HEMPEL. Vierte Auflage, 1913. Friedrich Vieweg & Sohn, Brunswick \$3.00.

The fourth edition of Hempel's work on gas analysis contains considerable additional material; not much has been eliminated from the older book.

The chapter on sampling is essentially the same. Hempel's well-known gas analysis apparatus and pipettes, some of which are much used in this country, are again described. A form of absorption whereby only a small quantity of liquid is brought in contact with the gas sample is an addition. Hempel's apparatus for exact gas analysis without the use of rubber tubing connections between pipettes and the burette is again described. Although capable of giving exact results, the apparatus is probably used little or not at all in this country.

The Rotameter for measuring the flow of gases is described. Its limitations are not dwelt upon. Brief mention is made of the gas interferometer. This is another instrument which as yet has been little used in this country. For small amount of carbon dioxide, methane, etc., in air, it gives very accurate results.

The chapter on the purification of mercury remains essentially the same. An additional form of pipette for gas purification has been added.

The chapter on the combustion of gases has been enlarged.

A chapter has been added on the separation of gases by means of liquid air. Ramsey's and Travers' experiments are largely drawn upon.

The determination of dust in gas mixtures has been given attention.

The chapter on the determination of different gases is enlarged. Reference is made to Paul and Hartman's method whereby hydrogen is determined by absorption in palladium solution. The convenient Orsat is again described. Two gas balances are again described. It is questionable whether they are much used in this country. Some new material has been added to the chapter on coal gas. The chapter on atmospheric air remains good as in the other book.

Two forms of bomb calorimeter for the determination of the heating value of coal are described; also the Junker calorimeter for gases, and another one for which there is required only a small quantity of gas. A scheme for the determination of sulfur in organic material by combustion is again included. Hempel's gas lantern which he describes is not used in this country.

The book constitutes the same excellent treatise on gas analysis as the former editions of the work. Some of Hempel's apparatus are probably still more used in this country than other forms, although the tendency to-day is, to install more one-piece or built-up forms of apparatus, for convenience and speed. The book is very similar to that of L. M. Dennis recently issued. The latter has eliminated some parts that deal with methods in Hempel's book that are little used in this country.

GEORGE A. BURRELL

NEW PUBLICATIONS

By D. D. BEROLZHEIMER, Librarian, Chemists' Club, New York

- Alkali Chlorides, The Electrolytic Decomposition of, with Stationary Cathodes.** Vol. II. JEAN BILLITER. 2 Vols. 8vo., pp. 180. Price, \$2.50. Wilhelm Knapp, Halle.
- American Electrochemical Society, Transactions, XXIII, 1913.** 8vo., pp. 432. American Electrochemical Society, South Bethlehem, Pa.
- Asphalts and Bitumens, Natural Rock.** A. DANBY. Cr. 8vo., pp. 254. Price, \$2.25. Constable & Co., London.
- Coal-Dust Explosion Tests, First Series of, in the Experimental Mine.** GEO. S. RICE, L. M. JONES, J. K. CLEMENT AND W. L. EGY. 8vo., pp. 115. U. S. Bureau of Mines, Bulletin 56.
- Colloidal State of Matter.** L. CASSUTO. L. 8vo., pp. 252. Price, \$2.25. Dresden. (Translation into German.)
- Disinfection and Disinfectants.** M. CHRISTIAN. 8vo., pp. 112. Price, \$1.25. Scott, Greenough & Co., London. (Translation.)
- Dyestuffs, The Synthetic, and the Intermediate Products from which they are Derived.** J. C. CAIN AND J. F. THORPE. 2nd edition. 8vo., pp. 442. Price, \$4.00. C. Griffin & Co., London.
- Ethereal Oils, The. II.** GILDEMEISTER AND HOFFMAN. 2nd edition. 8vo. Price, \$5.00. L. Staackmann, Leipzig. (German.)
- Gas, Analytical Methods for.** WALTHER HEMPEL. 4th edition. Price, \$3.00. Friedrich Vieweg & Sohn, Brunswick. (German.)
- Gas Engineers and Managers, Handbook for.** THOMAS NEWBIDDING. 8th edition. 8vo., pp. 578. Price, \$4.75. Walter King, London.
- Gas Manufacture, The Chemistry of.** H. M. RAYLE. 8vo., pp. 316. Price, \$4.50. A. M. Callender & Co., New York.
- Heat.** J. A. RANDALL. 8vo., pp. 345. Price, \$1.50. D. Van Nostrand Co., New York
- Oil, Heavy, as Fuel for Internal Combustion Engines.** IRVING C. ALLEN. 8vo., pp. 36. U. S. Bureau of Mines, Technical Paper No. 37.
- Oil Industries, Chemistry of the.** J. E. DAUTHCOMBE. 8vo., pp. 209. Price, \$3.00. D. Van Nostrand Co., New York.
- Oils and Fats, Carbohydrate, Examination of.** D. HOLDE. 4th edition. 8vo., pp. 612. Price, \$4.50. Julius Springer, Berlin. (German.)
- Oils, The Volatile. Vol. I.** E. GILDEMEISTER. 2 vols. 2nd edition. 8vo., pp. 677. John Wiley & Sons, New York. (Translation.)
- Organic Chemistry, Yearbook of. VI (for 1912).** J. SCHMIDT. 8vo. Price, \$3.75. Johannes Woehner, Leipzig. (German.)
- Organic Compounds, Chemical Technology of.** R. O. HERZOG. L. 8vo., pp. 732. Price, \$5.50. Heidelberg.
- Potassium Salts, The Occurrence of, in the Salines of the United States.** J. W. TURKENTINE. 8vo., pp. 96. U. S. Dept. of Agriculture, Bureau of Soils, Bulletin No. 94.
- Pumping and Hydraulic Machinery, Modern.** EDWARD BUTLER. 8vo., pp. 473. Price, \$4.50. Chas. Griffin & Co., London.
- Qualitative Analysis from the Ionic Standpoint.** W. BOETTGER. 3rd edition. 8vo. Wilhelm Engelmann, Leipzig.
- Rubber, The Chemistry of.** B. D. PORRITT. 8vo., pp. 96. Price, \$0.50. Gurney & Jackson, London.
- Steel, Its Selection, Annealing, Hardening and Tempering.** E. R. MARKHAM. 4th edition. Cr. 8vo. Price, \$2.75. Spon & Co., London.
- Sugar, Beet and Cane, Treatise on the Manufacture of. II.** DE GROBERT. 2 vols. 8vo. Price, \$12.00. J. Rousset, Paris.
- Water Purification and Sewage Disposal.** J. TILLMANNS AND H. S. TAYLOR. 8vo., pp. 143. Price, \$2.00. Constable & Co., London. (Translation.)
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- Sulfuric Acid Plant, Chamber Tests in a.** By E. H. ARMSTRONG. *American Fertilizer*, Vol. 38, 1913, No. 11, pp. 25-28.
- Tar and its By-products.** By S. R. CHURCH. *Gas Age*, Vol. 31, 1913, No. 10, pp. 497-501.
- Towers, Absorption and Reaction, for the Chemical Manufacturing Industry.** By RUDOLF HEINZ. *Zeitschrift fuer angewandte Chemie, Aufsatzteil*, Vol. 26, 1913, No. 57, pp. 419-422.
- Transmutation of Elements, Recent Work on the.** By A. T. CAMERON. *Radium*, Vol. 1, 1913, No. 3, pp. 3-5.
- Water Analysis for the Textile Industry, Methods of.** ANONYMOUS. *American Wool and Cotton Reporter*, Vol. 27, 1913, No. 28, pp. 857-858.
- Water Distilling Plant, Salt.** ANONYMOUS. *Louisiana Planter and Sugar Manufacturer*, Vol. 50, 1913, No. 22, pp. 347-351.
- Waterproofed Fabrics, Composition of.** By JOSEPH F. X. HAROLD. *Textile Manufacturers' Journal*, Vol. 21, 1913, No. 29, pp. 21.

RECENT JOURNAL ARTICLES

- Air Filters.** By S. WILDING COLE. *Journal of the Institute of Brewing*, Vol. 19, 1913, No. 5, pp. 336-359.
- Aluminum Carbo-Nitrid.** By S. PEACOCK. *American Fertilizer*, Vol. 39, 1913, No. 3, pp. 25-29.
- Ammonia, Oxid of Iron Purification in Sulfate of, Works.** ANONYMOUS. *Journal of Gas Lighting*, Vol. 123, 1913, No. 2619, pp. 238-245.
- Arsenic and Antimony in Converter and Electrolytic Copper, The Determination of.** By E. E. BROWNSON. *Bulletin of the American Institute of Mining Engineers*, 1913, No. 80, pp. 1489-1495.
- Arsenic Trioxid from Flue Dust.** By JAMES O. ELTON. *Bulletin of the American Institute of Mining Engineers*, 1913, No. 80, pp. 1497-1509.
- Brass Analysis.** By BERTHOLD KOCH. *Chemiker Zeitung*, Vol. 37, 1913, No. 87, pp. 873-874.
- Coke Oven Carbonization.** By W. CHANEY. *American Gas Light Journal*, Vol. 99, 1913, No. 2, pp. 17-21.
- Coke Oven Plant, the Modern By-product, Recovery of By-products in.** By C. A. MEISSNER. *Metallurgical and Chemical Engineering*, Vol. 11, 1913, No. 8, pp. 454-458.
- Combustion, Flameless Surface.** By FRITZ KRULL. *Zeitschrift fuer angewandte Chemie, Aufsatzteil*, Vol. 26, 1913, No. 55, pp. 401-404.
- Copper Mining Industry, Development of the World's, in the Years**

RECENT INVENTIONS

By C. L. PARKER, Solicitor of Chemical Patents, McGill Building, Washington, D. C.

Precipitation of Material from Cyanid Solutions. C. W. Merrill, June 3, 1913. U. S. Pat. 1,063,569. The solution is passed through a comminuting apparatus in which it is brought in contact with a precipitant of the metals in solution having an inert non-metallic abrasive substance incorporated therewith.

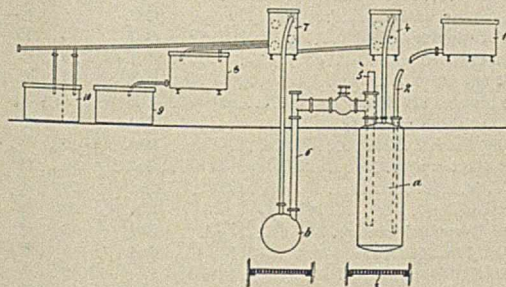
Bleaching Vegetable Fiber and Fabrics. R. Gruter, June 3, 1913. U. S. Pat. 1,063,678. The fibers or fabrics to be bleached are saturated with a hot solution of hydrogen peroxid; after removing the excess of the solution from the material it is exposed to heated ammonia vapors.

Refining Brown Sugar. Lundgren and Gierling, June 3, 1913. U. S. Pat. 1,063,708. A sugar solution of a density of 20° Baume is subjected to the action of ozone or ozonized air.

Preparing Hydrogenized Products from Unsaturated Compounds. A. Skita, June 3, 1913. U. S. Pat. 1,063,746. The hydrogenation is effected by the action of hydrogen in the presence of a small amount of a dissolved palladium salt.

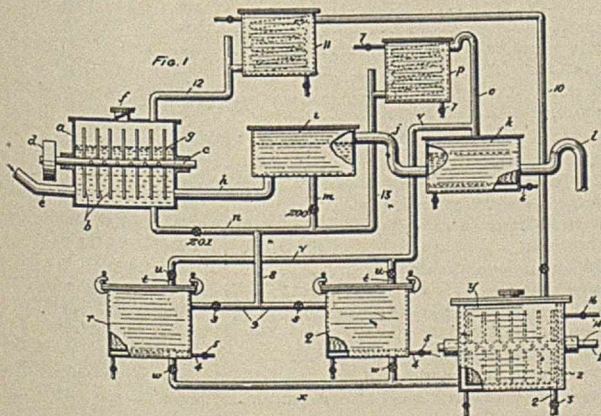
Magnetic Separation of Ores out of Slime. B. Schwerin, June 3, 1913. U. S. Pat. 1,063,893. The slimes are brought into a colloidal state and subjected to magnetic separation, with or without the addition of an electrolyte.

Production of Coke from Wood-Tar. F. W. Lefelmann, June 10, 1913. U. S. Pat. 1,064,350. The tar is first heated in a retort, *a*, to about 225° C. until constituents volatile at this temperature are driven off and condensed in condenser 4. The



residue is then heated to a high temperature and, having a tendency to foam, it boils over through siphon 6 into retort *b* where it is immediately exposed to a temperature of 400° C. or above and is coked without further foaming.

Production of Bromin or Bromids. F. I. du Pont, June 10, 1913. U. S. Pat. 1,064,460. Brine, containing the usual small content of bromin, is first electrolyzed to set free the bromin.



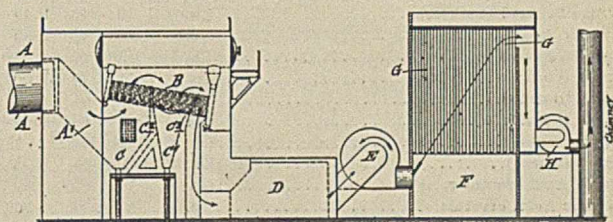
The bromin is extracted from the brine by means of a liquid

substantially insoluble in the brine and having a greater avidity for the bromin than the brine, such as carbon tetrachlorid. The mixture is allowed to stand until the carbon tetrachlorid collects in the bottom of the vessel from which it is drawn off.

The carbon tetrachlorid with its bromin content is subjected to the action of a water-absorbing reagent such as calcium chlorid which leaves the bromin and its solvent free from water.

The carbon tetrachlorid with the bromin therein is then agitated in the presence of some reagent as antimony, which will combine with the bromin. The mixture is then subjected to heat to drive off the carbon tetrachlorid.

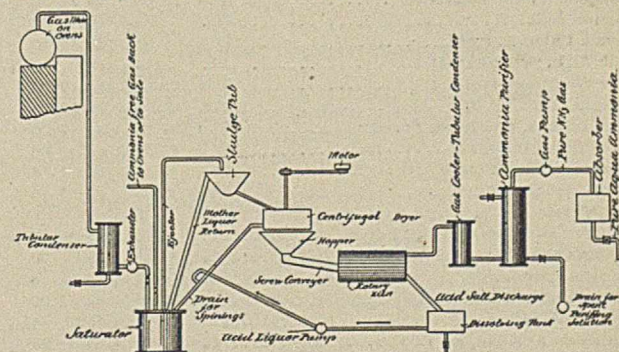
Utilizing Waste Gases of Combustion from Portland Cement, Lime and other Kilns. F. Schott, June 10, 1913. U. S. Pat. 1,064,550. The waste gases from the kiln *A* are passed through a steam boiler, *B*, and are therein cooled to a temperature of from 200° to 250° C. The coarser particles of dust are collected in receptacles *C* and *C'*.



The waste gases are then drawn by a fan or blower, *E*, through the cooling chamber *D*, where they are cooled below 100° C. and are forced through the filtering chamber *F* where the fine dust and potassium and ammonia salts are collected. The purified gases are drawn off by the fan or blower *H* and discharged into the chimney.

Steel, Iron, Etc. A. W. Machlet, June 24, 1913. U. S. Pat. 1,065,379. The steel is exposed to a temperature above 900° F. but below the melting point of steel, in an atmosphere of ammonia.

Obtaining Purified Ammonia from Coal Gas. C. G. Tufts, June 24, 1913. U. S. Pat. 1,065,566. In this process the oven



gases are directly treated to recover purified ammonia without the usual preliminary steps of washing and forming ammoniacal liquor.

The oven gases are passed through a saturator containing sulfuric acid or acid sulfate of ammonia. The ammonia combines with the acid to form ammonium sulfate.

The combined gases are drawn off leaving the ammonia, together with the remaining impurities, in the sulfate.

This is quickly heated to drive off the ammonia with the accompanying volatile impurities and the ammonia is finally separated from these impurities.

MARKET REPORT

AVERAGE WHOLESALE PRICES OF STANDARD CHEMICALS, ETC., FOR THE MONTH OF AUGUST, 1913

ORGANIC CHEMICALS

Acetanilid.....	Lb.	21	@	23
Acetic Acid (28 per cent).....	C.	2.00	@	2.15
Acetone (drums).....	Lb.	15 ³ / ₄	@	16 ³ / ₄
Alcohol, denatured (180 proof).....	Gal.	36	@	39
Alcohol, grain (188 proof).....	Gal.	2.46	@	2.48
Alcohol, wood (95 per cent).....	Gal.	45	@	47
Amyl Acetate.....	Gal.	2.20	@	2.30
Aniline Oil.....	Lb.	10 ¹ / ₈	@	10 ⁵ / ₈
Benzoic Acid.....	Lb.	23	@	27
Benzol (90 per cent).....	Gal.	21	@	23
Camphor (refined in bulk).....	Lb.	42 ¹ / ₂	@	44
Carbolic Acid (drums).....	Lb.	9 ³ / ₄	@	13
Carbon Bisulfide.....	Lb.	6 ¹ / ₂	@	8
Carbon Tetrachloride (drums).....	Lb.	7 ³ / ₄	@	8 ¹ / ₄
Chloroform.....	Lb.	25	@	35
Citric Acid (domestic), crystals.....	Lb.	46 ¹ / ₂	@	47
Dextrine (corn).....	C.	3.12	@	3.75
Dextrine (imported potato).....	Lb.	6	@	7
Ether (U. S. P., 1900).....	Lb.	14	@	20
Formaldehyde.....	Lb.	8 ¹ / ₂	@	9 ¹ / ₂
Glycerine (dynamite).....	Lb.	18 ³ / ₄	@	19
Oxalic Acid.....	Lb.	7 ⁷ / ₈	@	8
Pyrogallic Acid (bulk).....	Lb.	1.20	@	1.40
Salicylic Acid.....	Lb.	28	@	30
Starch (cassava).....	Lb.	3 ¹ / ₄	@	4
Starch (corn).....	C.	2.34	@	3.00
Starch (potato).....	Lb.	4 ¹ / ₂	@	5
Starch (rice).....	Lb.	8	@	9
Starch (sago).....	Lb.	2 ³ / ₈	@	2 ³ / ₄
Starch (wheat).....	Lb.	5	@	6 ¹ / ₂
Tannic Acid (commercial).....	Lb.	35	@	36
Tartaric Acid, crystals.....	Lb.	30 ¹ / ₄	@	30 ³ / ₄

INORGANIC CHEMICALS

Acetate of Lead (brown, broken).....	Lb.	7 ³ / ₄	@	8
Acetate of Lime (gray).....	C.	2.50	@	2.60
Alum (lump).....	C.	1.75	@	2.00
Aluminum Sulfate (high grade).....	C.	1.25	@	1.75
Ammonium Carbonate, domestic.....	Lb.	8	@	8 ¹ / ₂
Ammonium Chloride, gray.....	Lb.	6 ¹ / ₄	@	6 ³ / ₄
Aqua Ammonia (drums) 16°.....	Lb.	2 ¹ / ₄	@	2 ¹ / ₂
Arsenic, white.....	Lb.	3.35	@	3.60
Barium Chloride.....	C.	1.60	@	1.80
Barium Nitrate.....	Lb.	5 ¹ / ₄	@	5 ¹ / ₂
Barytes (prime white, foreign).....	Ton	19.00	@	23.50
Bleaching Powder (35 per cent).....	C.	1.20	@	1.25
Blue Vitriol.....	Lb.	5	@	5 ¹ / ₄
Borax, crystals (bags).....	Lb.	3 ³ / ₄	@	4 ¹ / ₄
Boric Acid, crystals (powd.).....	Lb.	7	@	7 ¹ / ₂
Brimstone (crude, domestic).....	Ton	22.00	@	22.50
Bromine, bulk.....	Lb.	30	@	35
Calcium Chloride, fused.....	C.	85	@	1.10
Chalk (light precipitated).....	Lb.	4	@	4 ¹ / ₂
China Clay (imported).....	Ton	11.50	@	18.00
Feldspar.....	Ton	8.00	@	12.00
Fuller's Earth, powdered, Foreign.....	Ton	16.00	@	17.00
Green Vitriol (bulk).....	C.	55	@	60
Hydrochloric Acid (18°).....	C.	1.15	@	1.55
Iodine (resublimed).....	Lb.	3.05	@	3.10
Lead Nitrate.....	Lb.	8 ¹ / ₄	@	8 ¹ / ₂
Litharge (American).....	Lb.	6 ¹ / ₂	@	7
Lithium Carbonate.....	Lb.	65	@	70
Magnesia (powdered).....	Lb.	5 ¹ / ₂	@	6
Magnesite (raw).....	Ton	30.00	@	31.00
Nitric Acid, 36°.....	Lb.	3 ⁷ / ₈	@	4 ¹ / ₄
Phosphoric Acid (sp. gr. 1.75).....	Lb.	21 ¹ / ₂	@	25 ¹ / ₂
Phosphorus.....	Lb.	45	@	1.00
Plaster of Paris.....	Bbl.	1.50	@	1.70
Potassium Bichromate, 50°.....	Lb.	6 ³ / ₄	@	7
Potassium Bromide.....	Lb.	39	@	40
Potassium Carbonate (calcined) 80 @ 85%.....	C.	3.40	@	3.65
Potassium Chlorate, crystals.....	Lb.	8 ¹ / ₄	@	9 ¹ / ₂
Potassium Cyanide (bulk) 98-99%.....	Lb.	19	@	24
Potassium Hydroxide.....	C.	3.00	@	4.75
Potassium Iodide (bulk).....	Lb.	2.60	@	2.65
Potassium Nitrate (crude).....	Lb.	4 ³ / ₄	@	5
Potassium Permanganate (bulk).....	Lb.	9 ¹ / ₂	@	11
Quicksilver, Flask.....	C.	40.00	@	—
Red Lead (American).....	Lb.	6 ¹ / ₂	@	7
Salt Cake (glass-makers').....	C.	55	@	65

Silver Nitrate.....	Oz.	37 ¹ / ₂	@	39 ¹ / ₂
Soapstone in bags.....	Ton	10.00	@	12.00
Soda Ash (48 per cent).....	C.	65	@	70
Sodium Acetate.....	Lb.	4	@	4 ¹ / ₂
Sodium Bicarbonate (domestic).....	Lb.	1.50	@	1.80
Sodium Bicarbonate (English).....	Lb.	2 ³ / ₄	@	3
Sodium Bichromate.....	Lb.	4 ¹ / ₂	@	4 ⁷ / ₈
Sodium Carbonate (dry).....	Lb.	60	@	80
Sodium Chlorate.....	Lb.	8 ¹ / ₄	@	9 ¹ / ₂
Sodium Hydroxide, 60 per cent.....	C.	1.57 ¹ / ₂	@	1.62 ¹ / ₂
Sodium Hyposulfite.....	C.	1.30	@	1.60
Sodium Nitrate, 95 per cent, spot.....	C.	2.35	@	2.40
Sodium Silicate (liquid).....	C.	65	@	1.50
Strontium Nitrate.....	Lb.	6 ⁷ / ₈	@	7 ⁷ / ₈
Sulfur, Flowers (sublimed).....	C.	2.20	@	2.60
Sulfur, Roll.....	C.	1.85	@	2.15
Sulfuric Acid, 60° B.....	C.	85	@	1.00
Talc (American).....	Ton	15.00	@	20.00
Terra Alba (American), No. 1.....	C.	75	@	80
Tin Bichloride (50°).....	Lb.	11 ³ / ₄	@	12
Tin Oxide.....	Lb.	44	@	46
White Lead (American, dry).....	Lb.	5 ¹ / ₄	@	6
Zinc Carbonate.....	Lb.	9	@	10
Zinc Chloride (granulated).....	Lb.	4 ¹ / ₂	@	5
Zinc Oxide (American process).....	Lb.	5 ³ / ₈	@	6 ³ / ₈
Zinc Sulfate.....	Lb.	2 ¹ / ₄	@	2 ³ / ₄

OILS, WAXES, ETC.

Beeswax (pure white).....	Lb.	42	@	45
Black Mineral Oil, 29 gravity.....	Gal.	13 ¹ / ₂	@	14
Castor Oil (No. 3).....	Lb.	8 ⁷ / ₈	@	10
Ceresin (yellow).....	Lb.	12	@	22
Corn Oil.....	C.	6.65	@	6.70
Cottonseed Oil (crude), f. o. b. mill.....	Gal.	—	@	—
Cottonseed Oil (p. s. y.).....	Lb.	8 ¹ / ₂	@	9 ¹ / ₂
Cylinder Oil (light, filtered).....	Gal.	21 ¹ / ₂	@	32
Japan Wax.....	Lb.	9	@	9 ¹ / ₂
Lard Oil (prime winter).....	Gal.	95	@	97
Linseed Oil (double-boiled).....	Gal.	54	@	55
Menhaden Oil (crude).....	Gal.	29	@	30
Neatsfoot Oil (20°).....	Gal.	98	@	1.10
Paraffine (crude, 120 & 122 m. p.).....	Lb.	3 ¹ / ₂	@	3 ³ / ₄
Paraffine Oil (high viscosity).....	Gal.	26	@	28
Rosin ("F" grade).....	Bbl.	4.40	@	4.45
Rosin Oil (first run).....	Gal.	30	@	34
Shellac, T. N.....	Lb.	23	@	23 ¹ / ₂
Spermaceti (cake).....	Lb.	30	@	35
Sperm Oil (bleached winter), 38°.....	Gal.	72	@	74
Spindle Oil, No. 200.....	Gal.	18	@	19
Stearic Acid (double-pressed).....	Lb.	9	@	11
Tallow (acidless).....	Gal.	63	@	65
Tar Oil (distilled).....	Gal.	30	@	31
Turpentine (spirits of).....	Gal.	39 ¹ / ₂	@	40

METALS

Aluminum (No. 1 ingots).....	Lb.	22 ¹ / ₂	@	23
Antimony (Hallet's).....	Lb.	7 ³ / ₄	@	8 ¹ / ₂
Bismuth (New York).....	Lb.	2.10	@	2.15
Bronze powder.....	Lb.	50	@	3.00
Copper (electrolytic).....	Lb.	15 ³ / ₈	@	16
Copper (lake).....	Lb.	15 ³ / ₄	@	16 ¹ / ₄
Lead, N. Y.....	Lb.	4.75	@	—
Nickel.....	Lb.	50	@	55
Platinum (refined).....	Oz.	46.00	@	—
Silver.....	Oz.	59 ¹ / ₈	@	60
Tin.....	C.	40.00	@	44.00
Zinc.....	Lb.	5.70	@	5.80

FERTILIZER MATERIALS

Ammonium Sulfate.....	C.	3.00	@	—
Blood, dried.....	Unit	—	@	2.80
Bone, 4 ¹ / ₂ and 50, ground, raw.....	Ton	27.50	@	28.00
Calcium Nitrate (Norwegian).....	C.	2.05	@	2.10
Castor meal.....	Unit	—	@	3.00
Fish Scrap, domestic, dried.....	Unit	2.75	@	10
Mowrah meal.....	Ton	—	@	nominal
Phosphate, acid, 16 per cent bulk.....	Ton	6.75	@	7.00
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent.....	Ton	3.00	@	5.25
Tennessee, 70-80 per cent.....	Ton	5.00	@	5.50
Potassium, "muriate," basis 80 per cent.....	Ton	38.55	@	—
Pyrites, furnace size, imported.....	Unit	—	@	0.13 ¹ / ₂
Tankage, high-grade.....	*Unit	2.72 ¹ / ₂	@	2.75 & 10