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Vol. I. FEBRUARY, 1909. No. 2

EDITORIALS.

NATURAL RESOURCES AND MANUFACTURE.

We have, as a nation, acquired the habit of being vastly satisfied with what we have accomplished. We marvel at our enterprise in scraping iron ore from the earth's surface by steam shovels, in growing wheat on virgin soil, in stripping great areas of primeval forest, in burning natural gas and allowing petroleum to spout from the ground. Even Germany acknowledges that she cannot compete with us in raising cotton, and we cut more ice in a month in the single state of Maine than all the Pictet machines in France can turn out in a year. We control the copper market of the world—because we have the copper. If you want cheap sulphur, you must come to us, we pump it from the ground. We develop great centres of power distribution because our rivers run so fast down hill.

To these vast resources we have, indeed, brought a native energy, an unusual capacity for organization, and a genius for mechanical affairs. What we do, we do on a great scale, but we often do it very badly. It is quite time for us to pause in our self-congratulation long enough to inquire whether the things we are doing cannot be better done, whether, in fact, other nations have not developed and put to use much better methods, which, given equal opportunity, would put our own performance to the blush.

Although the resources of a country form the basis of its prosperity, this is, nevertheless, determined in the long run by the manner in which these resources are utilized, or, in other words, by the industrial efficiency of the means and methods of production. We have developed great transportation systems, we handle raw material on a titanic scale, we have applied machinery to the addressing of our letters and the sticking of the stamps, but it remains true, none the less, that with a few conspicuous exceptions, our manufacturing operations are carried forward in trustful ignorance and disregard of many of the factors upon which real industrial efficiency depends. This is shown in the stupendous waste which accompanies the first crude preparation of the raw material; it is shown in the general absence of a true selective economy in the apportionment of that raw material among the different industries, and it is shown again, and yet again, in the losses which attend nearly every step in the progress of the raw material toward the finished product. One need only refer to the wastes which attend lumbering, or to the growing of flax for seed, the making of coke in bee-hive ovens, and the failure to utilize the casein of skim milk as a high-grade food product, to realize vaguely something of what these initial losses are. The absence of proper selective economy in the adaptation of raw material to use is everywhere, as when our railroads use untreated ties and poles, when coal-tar is burned as fuel, crystal alum used for purifying water, or valuable publications printed on ground-wood

papers. We are still polluting our streams with wool grease, still wondering whether we can make alcohol from waste molasses, still buying coal without reference to heating power, and paying 65 cents a gallon for cylinder oil.

When wastes so obvious and so easily remedied are everywhere taking heavy toll of our manufacturers, it is not surprising that in all lines of productive effort subtle and elusive problems present themselves and still further lower our industrial efficiency. Steel rails break by thousands, trolley wires snap, boilers corrode, milk-cans rust, unsightly bloom appears on leather, cloth is stained or tendered, paints fail to protect the metal underneath. In a large proportion of cases, those who are confronted by the problem, have neither the time, the training, nor the equipment required for its solution, and yet such problems and thousands of others far more complex upon their face must be solved if our industrial efficiency is to be brought to its proper level.

No one at all conversant with the facts can doubt that our industrial salvation must be found in a closer alliance and co-operation between the scientific worker and the actual agencies of production. Such co-operation exists, as we are all beginning to learn, in Germany, and its results are evident throughout the world in the tremendous expansion of German industry.

No one at all familiar with the conditions under which thousands of American manufacturers are working can fail to realize the unique and fruitful opportunity which spreads out before the Laboratory nor can they doubt that the funds for its development will be forthcoming. Within the last few years there has opened out to the worker in applied chemistry a new horizon with a sweep so broad that it is seen to include far more than the mere material gains which come from more efficient effort. It has come to be recognized that the lives of great masses of the community are constricted and confined because our industrial efficiency as a people is still far below what it ought to be. In this stage of our industrial development no agency is more directly available for increasing this efficiency than that afforded by chemistry as applied to industry. Every waste that is prevented or turned to profit, every specification which gives a better control of raw material, every problem solved, and every more effective process which is developed, makes for better living in the material sense and for more wholesome living in the higher sense. It means much to the material and more to the higher well-being of German workmen that their nation now controls the coal-tar industries, the manufacture of fine chemicals, and the markets of the world in many other lines, chiefly as the result of the application of the scientific method to the problems of production. The general application of these methods will mean even more to our own country.

ARTHUR D. LITTLE.

STANDARDIZED SAMPLES.

IT is not at all surprising that the subject of accuracy in chemical analysis is constantly undergoing discussion. As so much depends upon the analysts' work, both in settlements for materials bought and sold and in the control of works' processes, it would seem that almost anything within reason ought to be done to insure the accuracy of his results. At frequent intervals, papers appear dealing with the preparatory and technical training of the chemist, the formation of an Institute of Chemistry, the use of reagents of guaranteed composition, the calibration of weights and measuring instruments, and the development of new or improved analytical processes. In addition, much effort is expended in the endeavor to obtain uniform or official methods of analysis for use in certain industries.

Experience has shown that the coöperative analysis of a given sample by a number of chemists serves only to call attention to the probable variations in results obtainable by men who strive for accuracy, but who work under such different conditions that non-concordant determinations are almost inevitable. As a means for developing better, and perhaps uniform, methods of analysis, it would be desirable to have a limited number of well trained men work out what they consider the best method available, with one or more optional methods in addition, and then put their analytical "scheme," together with carefully standardized samples, in the hands of chemists at large. An analyst of any degree of experience or skill could then practice with the standardized sample until he had perfected himself in the proposed method. This way of working brings splendid results with students of quantitative analysis—why should it not be equally successful with chemists of greater maturity?

The use of a standardized sample as a check

upon different chemists using the same method of analysis, upon the same chemist using different methods of analysis, and as an umpire sample in cases of dispute between analysts, will suggest themselves immediately. As a check upon original methods of analysis, enabling the operator to determine with comparatively little effort the applicability and accuracy of his method, standardized samples are well worth their cost. In cases of disputed analytical results it would doubtless lead to a better agreement were both parties to analyze a standardized sample of the same kind of material and discover which is at fault, rather than go through the usual process of submitting the original sample to a third party—who may be no more capable than the contending analysts—for an umpire analysis.

Some ten or more years ago, a foundrymen's association did a real service to the chemists of the iron and steel industry by preparing, with great care, a set of iron samples, having them analyzed by three or four chemists of recognized ability, and then selling these standardized samples at a reasonable price. In a recent number of *Science* (October 2, 1908), Launcelot Andrews proposed that similar work, but on a much larger scale, be undertaken by the Bureau of Standards. He would have the Bureau furnish both substances used in the preparation of standard solutions and samples of raw materials or finished products.

The Bureau of Standards has already prepared a number of standardized iron and steel samples which it sells at fixed prices (THIS JOURNAL, page 41) and has under consideration the preparation of special steel samples. The National Fertilizer Association has prepared and distributed four samples of phosphate rock which may now be considered to be standardized. The Committee on Analysis of Fats, Soaps and Glycerine, of the American Chemical Society, has prepared and distributed samples of the products which it has under consideration and these after analysis by experts may be considered as standardized. Thus, even up to the present time, some work on the preparation of standardized samples has been done.

Even before Andrews' paper appeared, the thought had occurred to some members of the American Chemical Society that this was a field which the Division of Industrial and Engineering Chemistry might do yeomen's service. With a membership made up of representatives of almost all the chemical industries carried on in this country, with three publications of large circulation at its disposal, and with the enthusiasm of youth to enable it to carry out successfully big undertakings, the Division ought to be in a position to prepare, standardize and distribute samples of materials for which there may be a demand. The expenses involved could be defrayed from the sale of the samples. Is the suggestion not well worth considering?

W. C. EBAUGH.

THE ROAD PROBLEM AND THE CHEMICAL ENGINEER.

ONE of the most important problems of the day, which requires the consideration of those who are in charge of the construction and maintenance of our highways, is that of how to meet the destructive effect of modern motor traffic. It has become one of such prominence that an International Road Congress was held in Paris in October last, on the initiative of the French Government, to consider the subject, at which twenty-five nations were represented by nearly twenty-three hundred delegates and individuals. It was surprising to find, as a result of the Congress, how little the chemical engineer and chemist have been utilized abroad in solving the problem, and that America is far in advance of other nations in this respect. Nothing has been done abroad which in any way corresponds to the investigations carried out in the laboratory of the Office of Public Roads of the United States Department of Agriculture, and it is rumored that an effort has been made to interest it in an examination of the stone in use in the construction of roads in Great Britain. For at least twenty years, American chemists have been engaged in the study of the native bitumens in the light of their application to the construction of pavements and roads, and it is interesting to note that the appreciation of their usefulness in this work has grown to such an extent that the services of a very considerable number are employed by municipalities and others in regulating the construction of pavements and the materials employed therein, as well as in investigating the character of the bitumens available for rendering macadam roadways more resistant to the attacks of motor travel. The field of usefulness is constantly increasing and widening, and the opportunity for accomplishing something by chemists in aiding to solve the road problem is large.

CLIFFORD RICHARDSON.

THE ELECTRIC STEEL FURNACE.

It is probable that portions of the testimony recently given by Mr. Charles M. Schwab before the Ways and Means Committee at Washington will be very generally considered by those interested in iron and steel. Mr. Schwab pointed out the superiority of open-hearth steel, but affirmed the certainty of his opinion that within ten years, these furnaces would become practically useless because of the development of the electric steel furnaces. Naturally, much weight is attached to the evidence of such a witness before such a tribunal. The conditions may well warrant his conclusion. It has been recognized that there is sufficient difference between the value of the iron and other components of a high-grade tool steel and the prevailing price of the combination plus necessary labor, to pay for any electrical energy likely to be used in the electrical processes. There seems to be an essential disparity between the prices of a cent a pound for 97 per cent. iron and fifty cents a pound for 99 per cent. iron. Probably this difference, which is due to conditions inherent in crucible-steel manufacture, will be the first to attract the attention of the steel producers to the electric furnace. It was pointed out by Mr. Schwab that the electric furnace could be used for improving the quality of the lower grades of steel, such as rails. Here there does not seem to be a great chance for saving in cost of production of identical product. The refining of already molten open-hearth steel by treatment under the conditions supplied by the electric furnace (high temperature, slag- and composition-control, and reducing atmosphere) will doubtless not exceed a very few dollars a ton, if indeed it is not less than one dollar per ton.

Judging by the magnitude of the past fluctuations in prices of rail and structural steel, it would seem a small matter if the cost of production were even doubled, provided the quality was essentially improved. No one of our useful metals seems likely to be soon reduced cent per cent. by any conceivable change in the cost of production. Therefore, the premonitions of great reduction in cost of tool steel are particularly interesting. The open-hearth production is nearly one hundred times as great as the crucible steel production, so that any proportionately smaller ripple of improvement on the surface will represent a much greater real commercial wave of advance.

W. R. Whitney.

AN AMERICAN INSTITUTE OF CHEMISTRY.

There is a real need for an American Institute of Chemistry. In any given trade or profession, it is impossible for any body to accurately estimate the fitness or capability of any particular member of it, except another and experienced member of that same trade or profession. All through human effort it is the case that it takes a fellow craftsman to judge intelligently a worker's efforts. Therefore, to safeguard employers, and at the same time help deserving workers, the principle of the proposed Institute of Chemistry is a principle that should be applied in every department of industrial activity, and doubtless in time will be so applied.

As a matter of fact, it is to-day applied in many lines: medicine, law, dentistry, civil and mining engineering, etc. The doctor's diploma for instance gives from experienced and able men in the same profession, assurance to the public that the holder has a good knowledge of the rudiments of his business. It serves exactly the same purpose and is based on exactly the same principle as a certificate later on from an Institute of Medicine, supposing there was such a thing. And it would be well if there were such a thing, because the original diploma certifies merely to the rudiments and gives no hint with regard to after-standing. Take two graduates in medicine and in ten years the one may far outstrip the other in knowledge and efficiency. Yet, as far as the information conveyed by the original diploma is concerned, they are still on the same level. But if an "Institute" would be a good thing in the medical profession, where there is always a diploma to start with, far more would it be a good thing in the chemical profession where there is no diploma at all—or rather, no diploma that corresponds to the medical diploma. The medical diploma certifies that the holder has spent a certain number of years in the exclusive study of his profession. The chemist's, on the contrary, testifies that he has not spent these years in the exclusive study of his profession, but has spent them in the study of science in general with chemistry merely as an appropriate (though it is true, an exaggerated) incident.

In other words, the chemist's degree certifies to employers merely that the holder is not a chemist but a general scientist who has made a specialty of chemistry. This is much but leaves plenty of room for the Ph.D. degree and the M.S. degree, and these latter in their turn, though far above the proposed Institute certificate in dignity and

importance, still leave room for the Institute certificate. Although the need for something like the proposed Institute is a need that exists in every business high and low, yet there is no business where it is needed so badly as in analytical chemistry, for there is no other business where the employer is more absolutely incapable of judging for himself whether or not his employee is capable and deserving, and as matters now stand, the analytical chemist must rely for advancement and appreciation rather upon his engaging personal qualities, if he has any, than upon his professional capabilities. In fact, it is hardly too much to say that in the iron trade at least, the latter are a bar rather than a help to advancement, if the employer has no outside sources of information about his chemist, for if the chemist has professional capability, and professional pride, in his work, he will rarely succeed in satisfying his employers in the matters of speed and output of work. A far closer approach to the steel man's ideal in these respects would be made by a laboratory boy ignorant of chemistry, and innocent of conscientiousness, and it is by no means a reckless or random statement that in the iron trade, the better the chemist, the lower his employer's opinion of him, if the employer has nothing to guide him but his own impressions. With conditions as they are to-day, with employers almost unanimous in the conviction that chemical analysis is quick and easy work, and with the gr at majority of chemists seeking to humor and adapt themselves to this foolish misconception, rather than to combat and correct it, the lot of the conscientious analyst would be hard indeed without the testimony and the support of college degrees and other honors that he may succeed in gaining. Let us have more on the same principle as the College degree!

The College degree is the first thing; it is most important but it is not enough. It certifies to college study. But study does not end with the closing exercises of college. At that point it may be said to begin. What have we now to certify to this real serious life study that begins only as college ends? We have the Ph.D. degree and it is a glorious thing. But that it leaves nothing more to be desired, and that its testimony represents the acme of human effort, and human achievement, we have to deny. Admirable as is the Ph.D. degree, and of more dignity and importance than anything else in the same line, still there is room for more in the same line. The question confronting the chemical profession in America is this: Since the College degree is a good thing, shall we develop the underlying principle of it further, or shall we stop there and be content?

GEORGE AUCHY.

ORIGINAL ARTICLES.

[CONTRIBUTION FROM THE LABORATORY OF THE FUEL ENGINEERING COMPANY, CHICAGO, ILLINOIS.]

THE AMOUNT OF INERT VOLATILE MATTER IN THE MINERAL CONSTITUENTS OF COAL.

BY W. BRINSMAID.

Received November 5, 1908.

Chemists working on the analysis of coal have long known that the non-volatile mineral matter that they weighed and called ash did not truly represent the weight of the inorganic matter, when in its original form in the unburned coal.

They have also known some, if not all, of the sources of error but have not been able to calculate the amount.

The combined water in fire clay and gypsum, and the presence of carbonates that give off carbon dioxide on heating may be mentioned as probably the principal sources of error in weighing an ash correctly.

Pyrite also loses weight when burned to ferric oxide and is thus a source of loss. In case the amount of pyrite present in the coal were known, then the addition of five-eighths the weight of its sulphur content to the ash would correct for this loss. Unlike the other cases, however, this loss is accompanied by combustion and develops some heat. We might, therefore, call the iron in the pyrite inert matter and the sulphur a combustible and deduct the weight of the oxygen that unites with the iron, when the pyrite is burned to iron oxide. However, the determination of the amount of pyrite in coal is attended with some difficulty. The usual method has been to calculate the pyrite from either the iron present or from the total sulphur.

As coal may have iron present in other forms than pyrite, and generally has organic sulphur and sometimes gypsum present, it can readily be seen that any determination of pyrite in coal that is based on total iron or total sulphur may be the reverse of accurate.

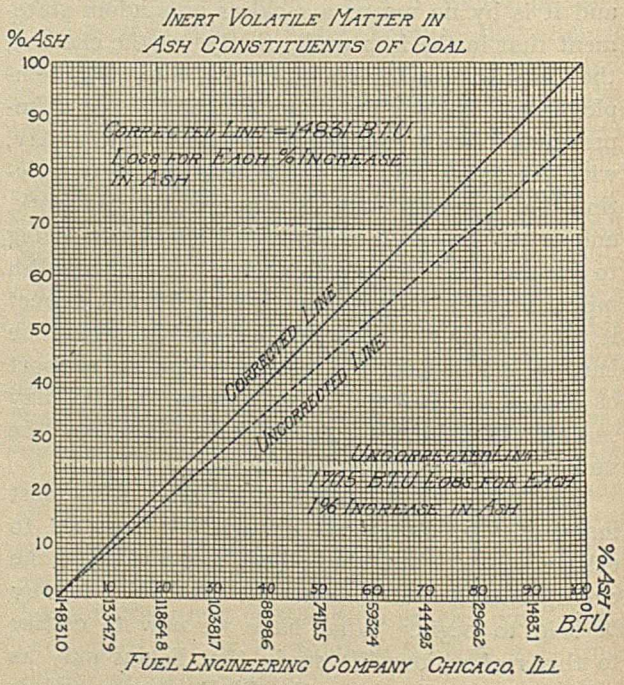
In speaking of the determination of oxygen by difference in ultimate analysis of coal, Prof. Lord

says:[1] "The result so obtained is always inaccurate, the error increasing with the percentages of the ash and sulphur. The weight of the ash does not represent that of the mineral matter in the coal, the pyrite in the coal being burned to Fe_2O_3, and the sulphur passing off as SO_2. Thus 4 of sulphur in 2 FeS_2 (pyrite) is replaced by 3 of oxygen in Fe_2O_3, and the loss of weight is equal to five-eighths of the sulphur. For this reason many chemists use five-eighths sulphur instead of sulphur in the determination of oxygen by difference. As coals contain sulphur in other forms than FeS_2 and also frequently other compounds that lose weight on burning, such as $FeCO_3$ and $CaCO_3$, it is doubtful whether the results obtained in this way are any better than those given by the simple formula first given."

In fact a calculated correction made in this way may be a greater source of error than the absence of any such correction. These various losses in weight, of the mineral constitutents of coal, are an important factor in some calculations and vary considerably in different coals. I will endeavor to show how we can arrive at the sum total of these losses, although we cannot tell of what they consist or at present make any special corrections.

Sometime ago it became necessary for the laboratory of the Fuel Engineering Company to get out a set of tables for various coals, by which one coal could be compared with another, and their relative value shown in heat units. Under the advice of Mr. E. H. Taylor the following method was worked out in the laboratory. A sample of coal was taken and the whole sample (which was usually about thirty pounds) was turned on to a clean table.

There were then picked out by hand some of the very best pieces in the whole sample. These were laid aside and another sample was picked by hand in such a way that it would have about a 20 per cent. ash. Care was used to see that this sample had all its ash constituents present in the proportion natural to the sample. This could be done by proper crushing and mixing and was necessary for the reason that there were often present pieces of both the roof and bottom which varied widely in their character. We had then two samples of coal, one of which was the very best coal that could possibly be gotten from that mine in a commercial way, and the other representing the same coal but very high in ash. These two samples were then ground to pass a 100-mesh sieve and the ash was determined on each sample. From the ash of these two samples there were calculated four more coals to make a series having per cents. of ash increasing in regular order, and these were then made up from mixtures of the low and high ash samples. For instance, if we found our low ash coal to be 5 per cent. ash and the high ash coal to be 20 per cent. ash, four mixtures of these two coals would be made having respectively 8, 11, 14 and 17 per cent. ash. Thus we got samples of a certain coal having 5, 8, 11, 14, 17 and 20 per cent. ash. There is nothing artificial about this set of coals but on the contrary it is all the natural coal and everything is in its proper proportion. This set of six coals was then carefully run in duplicate in a Mahler oxygen calorimeter and the ash was also run in duplicate. These results were then plotted on cross-section paper, and if the work had been carefully done the result was a straight line. The line, however, showed a peculiarity.



As an example I will use an Illinois coal that is quite common in the Chicago market. The line of this coal shows that it has at

5 per cent. Ash	—13978.5	British	Thermal	Units
4 per cent. "	—14149.0	"	"	"
3 per cent. "	—14319.5	"	"	"
2 per cent. "	—14490.0	"	"	"
1 per cent. "	—14660.5	"	"	"
0 per cent. "	—14831.0	"	"	" or pure coal.

[1] "Notes on Metallurgical Analysis," N. W. Lord, page 170.

This shows that each addition of 1 per cent. ash means a loss of 170.5 British Thermal Units. If the line is carried along through increasing per cents. of ash it will be found that at 87 per cent. ash the B. T. U. are used up. We can also say that if each 1 per cent. of ash represents the loss of 170.5 B. T. U. (which the line shows to be the case), then the loss of 100 per cent. ash or the pure coal would be 100 × 170.5 B. T. U. or 17050 B. T. U. However, we have already seen that our pure coal is 14831 B. T. U. Now as to this difference. We rely much on the accuracy of the Mahler oxygen calorimeter.

Much use of the instrument has proved that it is accurate and reliable, and if properly handled will give close and concordant results. We, therefore, conclude from this that our determinations of the B. T. U. are correct and that the discrepancy is caused by the ash, and as our line shows that we have not sufficient B. T. U. to carry the line to 100 per cent. ash, then also we conclude that what we have been weighing and calling 1 per cent. ash represents matter that in the original coal weighed more than 1 per cent.

This being so, then the only correct figure we have in our table is 0 per cent. ash or pure coal, as this is the only figure in which all errors, due to loss of volatile non-combustible matter in the ash constituents, are eliminated. Our figure for pure coal then being 14,831 B. T. U., our factor of loss for each 1 per cent. ash will be 148.31 B. T. U. instead of 170.5. Our line having used up all B. T. U. at 87 per cent. ash shows us that we have lost 13/100 of our total ash, or in other words, each 1 per cent. ash that we have weighed as such really represented 1.13 per cent. inert matter in the original coal, of which amount 0.13 per cent. was volatile and non-combustible. While we have good grounds for stating the amount by weight of this volatile non-combustible matter in the ash constituents, we have nothing to show of what it is composed, and so we are as far as ever from this very desirable and interesting point.

This loss, of which no account has been taken, throws some light on certain discrepancies which have never been explained. Much work has been done on certain lines in the endeavor to get a basis on which coals might be compared. The pure coal basis has been used, with and without a correction for pyrite.

Ash- and moisture-free coal has usually been called pure coal. Now pure coal must necessarily be only one thing for a certain coal and should be the same figure when calculated from any per cent. of ash. The fact is, however, that it varied with each difference in the ash, and the same coal from the same mine would give as many different figures for pure coal as there were different per cents. of ash in the different samples.

As stated in the first part of this article, the correction for pyrite in the coal based on the total iron or total sulphur may in some cases be misleading. A glance at our original line with uncorrected ash will show the cause of some errors. As the error in the ash increases regularly for each 1 per cent. of ash as weighed, then the error in a 10 per cent. ash would be twice as great as in a 5 per cent. ash, and the error not being recognized as present, they would never figure back to a common basis.

This method has never proved of any practical value and has been generally discarded. It has also been stated that in calculating this pure coal, a correction should be made for water of constitution in the mineral constituents. While this is a fact commonly recognized, still we are as yet unable to make such a determination. Until we are able to determine this point with a reasonable degree of accuracy, there is not much that we can say in regard to it.

In the ultimate analysis of coal there are three of the principal determinations that we know may be in error. Carbonates if present will give up carbon dioxide and this will be calculated as carbon, so this determination will be too high. Combined water will be calculated to hydrogen and cause this determination to be also too high. Oxygen being determined by difference is a very uncertain figure as all the errors may effect it. Having no ultimate analysis on this particular coal that I am using as an example, I will take the average oxygen and ash of thirty Illinois coals on which the ultimate analysis has been run, to show what difference a corrected ash would make in the oxygen figure alone. The average ash is 14 per cent. and the average oxygen figure is 8.75 per cent.

In the coal taken as an example when 1 per cent. of ash as weighed equals 1.13 per cent. ash in the coal, the corrected ash would be 15.82, thus lowering the oxygen figure from 8.75 per cent. to 6.93 per cent. Such corrections as this show why at times heat values calculated by Dulong and Pettit's formula vary so widely from the heat values as determined by the oxygen calorimeter.

We find that coals vary, as we would naturally expect, in the amount of this volatile non-combustible that is included in the mineral constituents. On the few coals that have been calculated in this manner we find the largest amount to be as in the table used as an example. In this table each 1 per cent. of ash as weighed equals 1.13 per cent. of ash in the coal unburned. The smallest amount found, 1 per cent. ash as weighed, equals 1.067 per cent. ash in the unburned coal.

It is very probable that further work along this line will show a wider variation than is given above.

THE STABILITY OF ROSIN AT SLIGHTLY ELEVATED TEMPERATURES.

By Chas. H. Herty and W. S. Dickson.

Received November 16, 1908.

On heating American rosin to 120°–140° C. in a current of air freed from carbon dioxide, Schwalbe[1] obtained a copious precipitate of barium carbonate by conducting the gases from the flask in which the rosin was heated into a solution of barium hydroxide. He interpreted this as evidence of the decomposition of the abietic acid in the rosin with consequent formation of the hydrocarbon abietene, and pointed out the effect such a decomposition must have upon the melting point and saponification number of rosin.

From evidence obtained during the course of another investigation we were inclined to doubt the accuracy of Schwalbe's interpretation. Accordingly the following investigation was undertaken, the results of which show that rosin which has not been long exposed to the oxygen of the atmosphere can be heated indefinitely at 140° without showing any evidence of the formation of carbon dioxide, provided oxygen and moisture are excluded from the flask in which the rosin is heated.

EXPERIMENTAL.

At the outset Schwalbe's experiment was repeated. For heating the rosin a 200 cc. Erlenmeyer flask was placed in a beaker containing cottonseed oil. The air entering the flask was freed from carbon dioxide by being drawn through three wash bottles filled with a strong solution of sodium hydroxide. After leaving the flask the air was passed through a test tube half filled with freshly filtered barium hydroxide solution. Another wash bottle containing a solution of sodium hydroxide was placed between the barium hydroxide tube and the aspirator, a suction pump. A blank experiment with this apparatus showed no precipitation of barium carbonate after drawing air through for one hour. A repetition of Schwalbe's experiment showed a copious precipitation of barium carbonate.

The possibility that this evolution of carbon dioxide might be due to the action of the oxygen of the air upon the heated rosin, aided by the presence of slight traces of spirits of turpentine in the rosin, led to a repetition of the experiment using spirits of turpentine alone instead of rosin. With a specimen of old spirits of turpentine an even heavier precipitation of barium carbonate occurred than with rosin. No question of the splitting off of a carboxyl group could arise here. A specimen of freshly distilled turpentine showed also a precipitation of barium carbonate, but not so marked as with the old specimen.

Having proved that the spirits of turpentine alone was capable of giving the precipitation observed by Schwalbe, a current of steam was passed through molten rosin for eight hours in order to completely remove all spirits of turpentine. Repeating Schwalbe's experiment with this rosin the precipitation was still observed. Evidently the presence of slight traces of spirits of turpentine was not alone responsible for the precipitation observed.

It remained therefore to determine the possible influence of oxygen and of moisture on the formation of carbon dioxide from the molten rosin. Accordingly, the current of air drawn through the flask was freed first from carbon dioxide by sodium hydroxide, then dried by passing through sulphuric acid. A marked precipitation of barium carbonate was again observed. Then moist nitrogen was substituted for air. The nitrogen was prepared by drawing air through three wash bottles filled with an alkaline solution of pyrogallic acid. Again a precipitation of barium carbonate occurred. Finally a current of dry nitrogen was drawn through the flask and after all air had been expelled the rosin was heated to 140° and kept at this temperature for seven hours without the slightest precipitation in the tube containing barium hydroxide.

The above experiments were carried out on a specimen of freshly distilled rosin from the oleoresin of *Pinus heterophylla* (Cuban Pine). This

[1] Zeit. angew. Chem. 18, 1852.

suggested the possibility that Schwalbe had used a rosin from the oleoresin of *Pinus palustris* (Longleaf Pine). Accordingly a fresh specimen of rosin was prepared from the oleoresin collected from a single tree of this species. On heating the rosin in dry nitrogen to 140°, again no trace of precipitation was noticeable.

Finally Schwalbe states that his experiment was made upon a sample of commercial American rosin. On heating such a sample in dry nitrogen we found an abundant precipitation of barium carbonate.

Four factors therefore may have entered into the formation of the carbon dioxide observed in Schwalbe's experiment: first, traces of spirits of turpentine in the rosin; second, moisture; third, oxygen in the air conducted through the flask; and, fourth, oxygen absorbed either by the oleoresin previous to distillation or by the rosin on standing in the air. The explanation of a probable splitting off of a carboxyl group is demonstrated to be erroneous by using a sample of rosin recently distilled from a fresh specimen of the oleoresin and heating in a current of dry nitrogen.

And yet, paradoxical as it may at first appear, Schwalbe's explanation is even more than true; not in regard to "American rosin," but as applied to the acids of the oleoresin from which rosin is prepared. In order to avoid any elevation of temperature in the preparation of these acids, freed from the other constituents of the oleoresin, a specimen of the oleoresin of *Pinus heterophylla* was dissolved in ether. From this ethereal solution the potassium salts of the acids were precipitated by addition of a saturated water solution of potassium hydroxide. The crystal broth was mixed with glass wool to render it more permeable to an extractive, then thoroughly extracted with ether in a Soxhlet extractor. After removal of the last traces of ether the salts were dissolved in water, the solution acidified with dilute hydrochloric acid, and the precipitated acids washed and dried. On heating a specimen of these acids in the apparatus described above in a current of dry nitrogen the mass melted at 65°–70°, immediately evolution of carbon dioxide began as shown by the escape of gas bubbles from the molten mass, and the heavy precipitation of barium carbonate.

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SOIL ACIDITY IN ITS RELATION TO LACK OF AVAILABLE PHOSPHATES.

SECOND PAPER.

By C. W. Stoddart.

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In a previous article upon this subject[1] it was shown that acid soils need a phosphate fertilizer. This fact was noted not only in the work of other men on acid soils but also from field and plant-house fertilizer tests on numerous Wisconsin soils. Since the publication of the preliminary paper further tests have confirmed that statement, with one exception, and that, where a test was made on an acid virgin soil in which no fertilizer need was indicated, as might be expected.

Although there is phosphoric acid present in these soils in sufficient quantity for many crops, it is not available, and hence the soils need phosphate fertilizers. That acid soils do lack available phosphates is a fact, but the question now arises as to a causal relation, if any, between the two conditions; that is, whether lack of available phosphates is due to the acid condition of the soil. If this is true, it may be explained as follows: The soil acids act upon the readily available phosphates, such as the calcium phosphates, at a more rapid rate than the normal, neutral, or alkaline soil moisture, and when once in solution these phosphates are readily washed out by heavy rains, or are fixed by iron and aluminum compounds—that is, are precipitated and rendered unavailable as insoluble iron and aluminum phosphates. When there is sufficient lime in the soil to maintain the phosphoric acid in the form of calcium phosphate, the plant is able to obtain enough phosphorus for its use, since calcium phosphate is soluble enough to supply the needs of the growing crop. If it can be shown by chemical analysis that acid soils contain more iron and aluminum phosphates and less calcium phosphate than do non-acid soils, and particularly if they contain a greater ratio of iron and aluminum phosphates to calcium phosphate, there is evidence in favor of causal relationship between acid soils and lack of available phosphates.

In order to test this matter it is necessary to find some solvent which will extract the iron and aluminum phosphates and not the calcium phosphate, and vice versa. In selecting solvents which will extract these minerals separately from the soil

[1] Whitson and Stoddart, *J. Am. Chem. Soc.*, **29**, 757.

it is advisable to try them first on pure minerals, although it is well recognized that the action of any solvent on a pure mineral may not be the same on that mineral when it occurs in the soil, since the soil is a complex mixture of various minerals in different stages of weathering, and of organic matter in all forms of decomposition. It is commonly assumed that the principal phosphate minerals occurring in the soils are apatite (calcium phosphate), dufrenite (basic iron phosphate), and wavellite (basic aluminum phosphate). The mineral samples tested with the various solvents contained, in the case of apatite, 39.7 per cent. phosphoric anhydride; of dufrenite, 19.6 per cent.; of wavellite, 19.5 per cent. Since precipitated aluminum phosphate is soluble in a solution of sodium hydroxide, this solvent was tried in varying strengths, *viz.*, one, five, and ten per cent. Three portions of wavellite (0.2 gram each) were treated with 100 cc. of the different strengths of solvent in an Erlenmeyer flask carrying a condenser. The flask was kept in a boiling water bath for five hours. The solution was filtered and the phosphoric anhydride determined in the usual manner. One per cent. solution extracted 99.4 per cent. of the phosphoric anhydride in the mineral; five per cent. solution extracted 94.3 per cent., as did the ten per cent. solution. Accordingly, one per cent. solution of sodium hydroxide was chosen for the experiments. Sodium hydroxide free from phosphates was used. To try the effect of the one per cent. caustic soda on the other minerals, 0.2 gram each of dufrenite and apatite were treated with 100 cc. of the one per cent. sodium hydroxide solution. Dufrenite yielded 90.4 per cent. of its phosphoric anhydride to this solvent, and apatite 8.4 per cent.

Fraps[1] gives the result of the action of various acid solvents on several kinds of phosphates, mineral and precipitated. From his table it is to be noted that fifth-normal nitric acid acting at a temperature of 40° for five hours extracts 100 per cent. of phosphoric anhydride in apatite, 1.5 per cent. in dufrenite, and 3.6 per cent. in wavellite.

The minerals used by the writer, taking as before 0.2 gram of mineral and 100 cc. of solvent at 40°, gave for apatite 95.4 per cent. of phosphoric anhydride extracted, for the dufrenite 4.4 per cent., and for the wavellite 9.4 per cent. Using this same strength of acid in a boiling water bath for five hours, 100 per cent. of the phosphoric anhydride in the apatite was extracted, 58.9 per cent. in the wavellite, and 8.2 per cent. in the dufrenite. From these results it can be seen that it is necessary to keep the temperature at 40° during the five hours of extraction.

In using these solvents on the soil it was assumed that there would be extracted, in the case of sodium hydroxide solution, the iron and aluminum phosphates, but not the calcium phosphate, and in the case of the fifth-normal nitric acid, the calcium phosphate but not the iron and aluminum phosphates; or at least that there would be extracted proportional parts of the phosphates wherever there might be inclusion of the minerals within the soil grains. In every case, samples which had been passed through a 100-mesh sieve were used.

Twelve soils were selected, six of them not acid and six of them acid, as shown by the usual litmus paper test. The history of the various soils follows:

No. 523, from Mayville.—Cropped sixty years to grains; during the later years a four-year rotation has been practiced with clover and timothy, and manure has been applied. Yields good, and fertility is maintained. Not acid.

No. 270, from Blue Mounds.—Cropped sixty years to grains. Clover now raised and manure applied so that the yields are good and fertility is maintained. Not acid.

No. 865, from Evansville.—Cropped fifty-seven years to grains and considerable tobacco. Manured heavily for tobacco, and crops are good. Not acid.

No. 127, from Superior.—Raised but few crops of wild hay. Fertilizer tests in the plant house showed a slight lack of available phosphates. Not acid.

No. 61, from Stanley.—Cropped about ten years; very poor yields, differing from rest of this region. Plant house test showed need of phosphates. Not acid.

No. 618, Plot 6, Station Farm at Madison. Cropped about 25 years in rotation to corn, oats, seeded to clover, clover and potatoes, and manured on clover sod. Not acid.

No. 293, from Twin Bluffs.—Cropped forty years; some wheat, corn, and oats; crops sold off; the soil has been kept up for twenty years. Field test showed need of phosphates. Acid.

No. 736, from Afton.—Cropped sixty-one years to grains, a little manure has been applied; oats poor; responded to phosphate in the field. Acid.

No. 852, from Evansville.—Cropped fifty years

[1] "Availability of Phosphoric Acid of the Soil," *J. Am. Chem. Soc.*, **28**, 824.

to grains, some tobacco; manured for tobacco. Has been badly exhausted; not quite so bad now. Responded to phosphate in the field. Acid.

No. 246, from Onalaska.—Cropped about forty years to corn and oats; little or no clover; badly exhausted. Field test showed need of phosphates. Acid.

No. 277, from South Wayne.—Cropped sixty years, first to wheat, then mixed farm crops with some stock kept on the place; crops all removed; is badly depleted. Field test showed need of phosphates. Acid.

No. 297, from Black Earth.—Cropped twenty years in rotation and fairly well managed; it is in fair state of fertility. Field tests showed need of phosphates. Acid.

It is to be noted that all of these soils have been cropped from ten to sixty years, except one which has been under cultivation but a few years. One soil, No. 127, is a heavy red clay; the others are loams with varying amounts of sand and clay.

Twenty-five grams of soil were treated with 250 cc. of solvent in a flask fitted with a ground glass stopper carrying a condenser. For the sodium hydroxide solvent the flask was set in a boiling water bath and shaken every hour. At the end of five hours it was allowed to cool, filtered through a dry filter, twice if necessary, to remove the clay. The sodium hydroxide extracted some of the humus in the soil, so that the liquid was black in color. It was necessary to remove this organic matter before determining the phosphoric anhydride extracted by sodium hydroxide from the minerals. Accordingly, duplicate portions of 100 cc. each were placed in test tubes, made slightly acid with 2 cc. of concentrated hydrochloric acid, placed in a centrifuge and whirled at a speed of about 1,200 revolutions per minute for 15 minutes. The slimy organic matter, or "humic acid," as it is popularly called, was firmly packed in the bottom of the test tube by this treatment, and the clear, supernatant liquid could be readily decanted through a dry filter. Aliquot portions of the filtrate were then oxidized with bromine to remove some organic matter which remained in solution. It is unfortunately not possible to throw all of the organic matter out of the solution by acid, but the amount left is very small, and the phosphoric anhydride in this organic matter would be practically nil, since, as we shall see later, the phosphoric acid, even in a true humic extract, is very small in amount. After the extraction of the organic matter the solution is acidified with nitric acid, and evaporated to dryness, silica dehydrated, then taken up with nitric acid and water and filtered. From this point the usual methods for determining phosphoric acid were employed. It may be well to state that, although the gravimetric method was used wherever possible, in many cases the amount of phosphoric acid was so small that this method was not feasible. In such cases the yellow precipitate was dissolved in standard caustic soda and the excess titrated with standard nitric acid. While it is well to note that the volumetric method is apt to give high results, it has been found that working with very small amounts the method is reasonably accurate, certainly much more so than the gravimetric method where it would be necessary to weigh a precipitate of considerably less than one milligram in weight.

Where the soils were treated with fifth-normal nitric acid the temperature of the water bath was kept at 40° for five hours, the flasks being shaken every hour. After filtering off the soil the clear filtrate was found to contain a very small amount of organic matter dissolved by the nitric acid. The solution, accordingly, was made slightly alkaline with sodium hydroxide, oxidized with bromine, acidified with nitric acid, evaporated to dryness, silica dehydrated, etc., as before.

In addition to the above-mentioned determinations, it was thought advisable to make a brief study of the phosphoric acid combined with the humus. It is well known that the humus of the soil—that black, waxy coating of the soil grains—contains in chemical combination some phosphoric acid, as well as other inorganic compounds. The question had arisen as to the composition and amount of humus in well-drained, cropped, acid soils as compared with non-acid soils. The only method available for this work was the usual extraction with ammonia after treatment with hydrochloric acid, but in this method there are certain weak points to be avoided. The determination of humus by loss on ignition of the dried extract is open to two objections: First, the heating causes loss of zeolitic water from the clay which remains in suspension in the solution and cannot be removed by ordinary filtering; and, second, even if the clay were removed, the result would represent only the volatile compounds in the humus and not the important ash constituents which are certainly a part of the humus.

It has been possible to remove the clay by filtering through an unglazed porcelain filter, but this removes some of the organic matter with the clay, since the humus extract is not in all respects a true solution but has some properties of a colloid.

After the clay is removed the humus must be precipitated by an acid, since the ammonia extracts some of the phosphoric acid from the minerals in the soil. Fraps[1] shows that samples of apatite, dufrenite, and wavellite extracted by ammonia yielded 3 per cent., 13 per cent., and 44 per cent. of phosphoric anhydride, respectively. He used material corresponding to 0.1 gram phosphoric anhydride, shaken with 2,000 cc. of 4 per cent. ammonia at intervals for 24 hours and then filtered. The minerals used by the writer, when treated in 0.2-gram portions with 500 cc. of 4 per cent. ammonia for 24 hours with shaking and then 12 hours at rest, yielded approximately 1 per cent. for apatite, 3 per cent. for dufrenite, and 55 per cent. for wavellite. These results show that it is unsafe to determine the phosphoric acid in the ammoniacal extract and call it all humic phosphate.

Fraps, in the article just referred to, makes use of ammonium sulphate to flocculate the clay in the ammoniacal extract of the soil, and this method was pursued in the following experiments. Hydrochloric acid was used to precipitate the humus, since this acid has been found[2] to give better results than nitric acid; the filtrate is less colored by soluble organic matter. The humus is not completely precipitated even by hydrochloric acid, but is practically so.

The method as used in this work was to treat 25 grams of soil in a glass stoppered bottle with 400 cc. of 1 per cent. hydrochloric acid for 1 hour, shaking constantly in a mechanical shaker. Then it was filtered through a Büchner funnel on which had been placed a hardened filter paper. The soil was washed free from chlorides and transferred to a liter Erlenmeyer flask with 500 cc. of 4 per cent. ammonia and shaken at intervals for 24 hours. At this point 25 cc. of ammonium sulphate solution, containing 10 grams of the solid, were added, the mixture shaken and allowed to stand over night. When filtered through ordinary filter paper the filtrate was absolutely free from clay, the latter containing little, if any, gelatinous organic matter, and the filtration proceeded rapidly. For the determination of humus, 50 cc. were placed in a heavy test tube, acidified with 8 cc. concentrated hydrochloric acid and whirled in the centrifuge. The supernatant liquid was decanted through a weighed Gooch crucible, the humus transferred to the Gooch, washed with 1 per cent. hydrochloric acid, dried at 105° and weighed.

The humic phosphate was determined on an aliquot portion of the extract, treated in test tubes with hydrochloric acid, whirled in the centrifuge, filtered, and washed. The precipitate was dissolved through the paper with 1 per cent. sodium hydroxide solution, the paper washed clean with sodium hydroxide solution, and the organic matter oxidized with bromine, then acidified with nitric acid, evaporated dry, the silica dehydrated, and the phosphoric anhydride determined volumetrically, since the amount of yellow precipitate was very small.

Some preliminary trials of this method where the filtrate from the humus, as well as the humus, was analyzed for phosphoric anhydride, showed that there was about four times as much phosphoric anhydride in the filtrate as in the precipitate. This represents for the most part the phosphoric anhydride extracted by ammonia from the minerals of the soil.

Table I gives the percentages of the total phosphoric anhydride, phosphoric anhydride extracted by 1 per cent. sodium hydroxide solution, phosphoric anhydride extracted by fifth-normal nitric acid, phosphoric anhydride in the humus, and the humus, all expressed on the basis of dry soil; then the average percentages of phosphoric anhydride extracted by sodium hydroxide, by nitric acid, and in humus expressed on the basis of total phosphoric anhydride, and finally the humic phosphoric anhydride expressed on the basis of humus, besides the arithmetical averages of the different results.

The figures given in the averages show that, while acid soils do not contain more iron and aluminum phosphates than do non-acid soils, they do contain a larger percentage of their total phosphoric acid in the iron and aluminum form, and in addition a considerably greater ratio of iron and aluminum phosphates to calcium phosphate—three to one for acid soils, and about one and one-half to one for non-acid soils.

Turning now to the phosphoric acid soluble in

[1] "The Ammonia-soluble Phosphoric Acid of the Soil," *Am. Chem. J.*, **39**, 580.

[2] Unpublished results of H. L. Walster, Soils Dept., Univ. of Wis.

fifth-normal nitric acid, it is seen that considerably more is extracted from the non-acid soils than from the acid soils. In other words, the non-acid soils contain more calcium phosphate than do the acid soils. This is to be expected if soil acidity is the cause of the lack of available phosphates.

But, aside from this consideration, the figures show another interesting fact. It will be remembered that two of the non-acid soils responded to a phosphate fertilizer, Nos. 61 and 127. All of the acid soils have shown the lack of available phosphates. The average per cent. of phosphoric acid soluble in nitric acid of the four soils which do not need phosphate, is 0.036 per cent., and of the eight soils which do need phosphate is 0.012 per cent.—three times as much calcium phosphate in non-responsive soils as in responsive soils.

nutrition of the crop depends on the rate at which the fertilizing elements become available, and not upon the total amount present at any one time; but from these figures, it appears that the amount of calcium phosphate in a soil gives a very excellent idea as to the requirements of a soil with reference to phosphoric acid. In other words, there is a strong probability that the value of the fifth-normal nitric acid as an indicator of soil needs lies in the fact that it determines the amount of calcium phosphate in the soil, and does not merely imitate the solvent action of plant roots. From the results obtained in this work, it can be stated that, for Wisconsin at least, if a soil contains less than 0.015 per cent. of phosphoric acid soluble in fifth-normal nitric acid, it will respond to a phosphate fertilizer.[1]

Considering Table I again, it is to be noted that the humus content of the two sets of soils

TABLE I.

PERCENTAGES OF PHOSPHORIC ACID AND HUMUS.

No. of sample.	Total P_2O_5.	P_2O_5 by NaOH.	Per cent. of total.	P_2O_5 by HNO_3.	Per cent. of total.	Humic P_2O_5.	Per cent of total.	Humus.	Humic P_2O_5 per cent. of humus.	Years cropped.	Locality.
SOILS NOT ACID.											
523	0.14	0.039	27.86	0.025	17.86	0.013	9.28	0.72	1.81	60	Mayville
270	0.18	0.054	30.00	0.023	12.78	0.007	3.89	1.10	0.64	60	Blue Mounds
865	0.08	0.046	57.50	0.035	43.75	0.011	13.75	0.51	2.16	57	Evansville
127	0.15	0.039	26.00	0.010	6.67	0.009	6.00	0.19	4.74	3	Superior
61	0.19	0.080	42.10	0.010	5.26	0.022	11.58	0.69	3.19	10	Stanley
618	0.18	0.030	16.67	0.063	35.00	0.014	7.78	0.63	2.22	25	Madison
Average.....	0.153	0.048	33.35	0.027	20.22	0.012	8.71	0.64	2.46	35	
SOILS ACID											
293	0.12	0.038	31.67	0.012	10.00	0.007	5.83	0.28	2.50	40	Twin Bluffs
736	0.07	0.053	75.21	0.027	38.57	0.008	11.43	0.67	1.19	61	Afton
852	0.13	0.073	56.15	0.009	6.92	0.011	8.46	1.19	0.92	50	Evansville
246	0.12	0.014	11.67	0.011	9.17	0.011	9.17	0.30	3.67	40	Onalaska
277	0.14	0.018	12.86	0.007	5.00	0.009	6.43	0.17	5.29	60	South Wayne
297	0.17	0.054	31.76	0.014	8.24	0.019	11.18	1.51	1.25	20	Black Earth
Average.....	0.125	0.041	36.63	0.013	12.98	0.011	8.74	0.68	2.47	45	

TABLE II.

HUMUS AND PHOSPHORIC ACID IN HUMUS.

Humus.....	1.51	1.19	1.10	0.72	0.69	0.67	0.63	0.51	0.30	0.28	0.19	0.17
P_2O_5 in humus.....	1.25	0.92	0.64	1.81	3.19	1.19	2.22	2.16	3.67	2.50	4.74	5.29

Dilute nitric acid has been used by investigators with more or less success as an indicator of soil needs and the results here given show the value of fifth-normal nitric acid in determining the availability of phosphates in the soil. It seems reasonable to suppose that this is because the nitric acid dissolves out but little more than the calcium phosphate, and if calcium phosphate exists in too small amount the plant suffers for lack of phosphorus. Ordinarily a chemical analysis of the soil is of very little value in ascertaining the fertilizer requirements of a soil, inasmuch as the

is about the same, and the phosphoric anhydride in the humus is also about the same in both acid and non-acid soils, indicating that the reaction of the soil does not affect the accumulation of phosphoric acid in the humus. If, however, the soils are arranged in the order of their humus content from highest to lowest, together with the corresponding percentages of phosphoric an-

[1] In this connection it is interesting to note the following statement by Harry Snyder in *Minn. Bull.*, **102**, p. 36: "In all the trials, with the one exception noted, where the phosphate fertilizer failed to give an increase, the soil contained over 150 parts per million of phosphoric acid soluble in fifth-normal nitric acid." The two similar conclusions were drawn independently, and from work done on different soils.

hydride expressed on the basis of humus, it will be seen in a general way that as the amount of humus decreases the percentage of phosphoric anhydride in the humus increases (see Table II). Arranging the soils in groups of three, and averaging the percentages just expressed, this fact is brought out more strikingly (Table III). These results suggest the possibility that phosphorus in humus is not made available to plants as the humus decomposes, but rather that the phosphorus once in the humic form is unavailable to plants. This point is to be tested on comparable virgin and cropped samples of soils, and in addition further work is contemplated on the changes taking place in a soil during its period of cropping. Acid and non-acid soils will be used, these same soils so far as possible, but the study will be made on the differences that exist in the phosphate compounds between the virgin and the cropped samples.

TABLE III.

HUMUS AND PHOSPHORIC ACID IN HUMUS. AVERAGED IN GROUPS OF THREE.

Humus	1.27	0.69	0.48	0.21
P_2O_5 in humus	0.93	2.06	2.68	4.17

Summarizing the results, it is to be observed:

First, that acid soils lack available phosphates.

Second, that soil acidity seems to cause a lack of available phosphates, since acid soils contain a higher percentage of their total phosphoric acid in iron and aluminum form and less calcium phosphate than do non-acid soils, and particularly a greater ratio of iron and aluminum phosphates to calcium phosphate. This does not mean that non-acid soils may not be lacking in available phosphates, due to some other cause.

Third, that the figures on humus and phosphoric anhydride in humus show an interesting variation, suggesting that humic phosphorus may be unavailable to plants.

Fourth, that fifth-normal nitric acid is an excellent indicator of soil needs with respect to phosphates, due to the fact that it determines the amount of calcium phosphate in soils. If a soil, in Wisconsin at least, falls below 0.015 per cent. of phosphoric acid soluble in fifth-normal nitric acid, it will respond to phosphate fertilizer.

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A REMARKABLE ACCUMULATION OF NITROGEN, CARBON AND HUMUS IN A PRAIRIE SOIL.

BY F. J. ALWAY AND C. E. VAIL.

Received November 14, 1908.

Lawes and Gilbert[1] in 1885 reported the analysis of a sample of soil from Selkirk, Manitoba, showing 0.618 per cent. of nitrogen in the first foot. This has since been commonly used by writers as an illustration of the extent to which nitrogen may accumulate in virgin prairie soils. "The soil from Selkirk was taken from a farm which had been in cultivation for 25 years, but from a portion near the buildings which had never been broken up." Lawes and Gilbert, neither of whom had seen the farm, recognized the remarkable composition of the sample but evidently considered it representative of the soil area. The explanation of the composition of the sample may, however, be the same as that of the still higher percentages in the case of some samples taken by the senior author of this article from near Indian Head, Saskatchewan, in October, 1907.

Selkirk is situated in the bed of glacial Lake Agassiz,[2] now the so-called Red River Valley, the soil being a lacustral clay. The contemporaneous glacial Lake Saskatchewan[3] covered a considerable area in what is now the province of Saskatchewan, which adjoins Manitoba on the west. The characteristic soil covering its bed is likewise a lacustral clay. The soils of these two areas, being formed from similar sources and at the same time as well as having since been subjected to more or less similar climatic conditions, may reasonably be expected to have many characteristics in common.

A considerable number of individual samples were taken from a tract of virgin prairie adjacent to the Indian Head experimental farm in order to be able to form some idea as to the differences in composition between the surface soils of the two types before they were brought under cultivation. All the samples were taken with a brass tube 1-3/8 inches in diameter, provided with a wide collar six inches from the end. The tube was driven into the ground until the collar rested firmly on the surface of the soil.

Two types of soil occur both in the prairie tract and on the experimental farm, *viz.*, boulder clay

[1] *Jour. Chem. Soc.*, 1885, Proc., pp. 380-422.
[2] Upham, *Lake Agassiz, U. S. Geol. Survey*, 1895.
[3] *Ibid.*

or till as well as lacustral clay. Both types have previously been described.[1] The most clayey parts of the latter, which also form the lowest lying portions, are characterized by small hummocks two or three feet in diameter and about six inches high. These hummocks are often not noticed until one drives over the prairie in a carriage. They are probably similar in origin to the "hog-wallows"[2] of the southwestern United States. Three individual samples were taken from the till, three from the summits of the hummocks and three from the rifts at the edges of the latter. Besides these nine samples, four were taken from modified rifts and divided into one-inch sections, in order to determine the vertical distribution of humus and nitrogen. The rifts from which these four cores were taken differed from those from which 720, 721 and 722 were secured in that they were rounded and entirely grassed over, as though considerable time had elapsed since they had been active rifts, *i. e.*, opening in time of drouth and closing when the soil was thoroughly moistened. These were selected for the inch sections on the supposition that they would be intermediate in humus and nitrogen between the active rifts and the summits of the hummocks, it not being suspected that there would be found the great differences between the two which the analyses have since revealed.

The humus was determined by Grandeau's method as modified by Hilgard. To determine the humus nitrogen the ammoniacal solution of humus was evaporated to a small volume, magnesium oxide added, the solution boiled again and the nitrogen in the residue finally determined by the usual Kjeldahl method. The organic carbon was determined by combustion with copper oxide.

There are no marked differences in composition between the till and the soil from the summits of the hummocks. The rifts, however, show an entirely different character, being more than twice as rich in humus, humus nitrogen, total nitrogen and carbon. The humus of the rifts is somewhat richer in nitrogen while the percentage of total nitrogen in the form of humus nitrogen is decidedly higher. The proportion of organic matter in the form of humus, as indicated by the last column, is practically the same in all samples. The percentage of nitrogen in the humus is about the same in all and, as elsewhere[1] pointed out, is similar to that in soils from humid regions.

RESULTS OF THE ANALYSES.

Soil No.		Humus. Per cent.	Humus ash. Per cent.	Humus nitrogen. Per cent.	Total nitrogen. Per cent.	Nitrogen in humus. Per cent.	Total nitrogen in form of humus nitrogen. Per cent.	Ratio of humus to total nitrogen.	Organic carbon. Per cent.	Ratio of organic carbon to total nitrogen.	Ratio of organic carbon to humus.
714	Boulder clay	2.73	1.04	0.153	0.341	5.6	44.8	8.0	3.94	11.6	1.4
715	" "	3.08	1.55	0.165	0.433	5.4	38.1	7.1	4.86	11.2	1.6
716	" "	2.71	1.13	0.137	0.374	5.1	36.6	7.2	4.22	11.4	1.6
Average	" "	2.84	1.24	0.152	0.383	5.4	39.8	7.4	4.34	11.4	1.5
717	Lacustral clay, summits of hummocks	3.25	0.86	0.176	0.440	5.4	40.0	7.4	4.64	10.5	1.4
718	" " " " "	2.77	0.68	0.160	0.363	5.8	44.1	7.6	3.93	10.8	1.4
719	" " " " "	2.53	0.64	0.130	0.352	5.1	36.9	7.2	3.65	10.4	1.4
Average	" " " " "	2.85	0.73	0.155	0.385	5.4	40.3	7.4	4.07	10.5	1.4
720	Lacustral clay, rifts between hummocks	7.16	1.23	0.488	0.836	6.8	58.4	8.6	11.13	13.3	1.6
721	" " " " "	7.60	1.56	0.479	0.867	6.3	55.2	8.8	10.54	12.2	1.4
722	" " " " "	6.77	1.13	0.414	0.809	6.1	51.2	8.4	10.11	12.5	1.5
Average	" " " " "	7.18	1.31	0.460	0.837	6.4	54.9	8.6	10.59	12.7	1.5
	Section of four cores from modified rifts.										
723	First inch	9.15	1.73	0.606	1.264	6.6	48.0	7.2	16.46	13.0	1.8
724	Second "	8.38	1.86	0.469	0.948	5.6	49.5	8.8	10.88	11.5	1.3
725	Third "	5.98	1.52	0.385	0.870	6.4	44.3	6.9	10.38	11.9	1.7
726	Fourth "	5.45	1.64	0.294	0.710	5.4	41.4	7.7	8.16	11.5	1.5
727	Fifth "	4.82	1.54	0.257	0.611	5.3	42.1	7.9	6.95	11.3	1.4
728	Sixth "	4.22	1.38	0.216	0.532	5.1	40.6	8.0	6.27	11.8	1.5
Average		6.33	1.61	0.371	0.822	5.7	44.3	7.7	9.85	11.8	1.5

[1] Alway, *Am. Chem. Jour.*, **31**, 580 (1906). Alway and McDole, *Ibid.*, **32**, 275 (1907). Alway, *Jour. Agr. Sci.*, **2**, 333 (1908).

[2] Hilgard, "Soils," p. 114.

[1] Alway and Trumbull, *Am. Chem. Jour.*, **40**, 147 (1908).

It is generally recognized that the extent to which a composite sample of soil is truly representative of a given area depends in a considerable degree upon the number of individual samples of which it is composed. In the case of virgin soils that are being investigated for the first time it is, however, important to make analyses of a number of individual samples as well as of composites. Had this not been done in the present instance, a composite made up of individual samples from the modified rifts would have been used as a representative of the lacustral clay. Another person in sampling the prairie might have omitted the rifts entirely. Both methods would have given very misleading results.

The analyses show that the soil of the modified rifts is very similar in composition to that of the active rifts. The probable explanation of the very high content of humus nitrogen and carbon is as follows: The wet season at Indian Head is usually of short duration, extending from about the middle of May to the middle of July.[1] During the dry weather the heavy clay cracks. The winds of late summer, autumn and spring fill the rifts with vegetable débris. With the return of the wet weather the clay expands, the rifts close, and the organic matter protected from eremacausis, undergoes partial humification. At the end of summer the dry weather causes the opening of the rifts, which then begin anew to collect plant débris.

It is apparent from the above data that it will be extremely difficult to estimate the original content of humus and of nitrogen in a field of lacustral clay that has long been under cultivation. The portion of the surface originally occupied by hummocks and rifts, which offer no serious obstacle to the plow, would need to be known, as well as the relation between the distribution of hummocks and the average composition of the soil.

During the past month four samples of prairie soil have been received from Mr. B. J. Reynolds, of Indian Head. They were six-inch samples taken with a wood auger, three being from rifts and one from an adjacent summit. The percentages of total nitrogen were, respectively, 0.85, 0.73, 0.65 and 0.26.

The similarity of the Selkirk soil described by Lawes and Gilbert to the rift soil from Indian Head is shown in the following:

	Total N.	Carbon	Ratio of carbon to nitrogen.
Selkirk soil (Lawes and Gilbert).....	0.618	7.58	12.3
Indian Head soil from rifts.....	0.837	10.59	12.7
Indian Head soil from modified rifts....	0.822	9.85	11.8

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[1] *Bull.* **130**, *Bureau of Plant Industry*, p. 22 (1908).

RAPID DETERMINATION OF OIL IN COTTONSEED PRODUCTS.

BY CHAS. H. HERTY, F. B. STEM AND M. ORR.

Received November 10, 1908.

In the manufacture of cottonseed oil the delinted seeds are cut and the meats or kernels separated mechanically from the hulls. The meats are then mashed between heavy rollers, cooked in steam-jacketed vessels, and the oil expressed by hydraulic pressure. The determination of oil in the hulls gives a measure of the completeness of the separation of the meats from the hulls, while the oil content of the meal affords a control of the character of the press work. It would seem, therefore, that a prompt and constant knowledge of the oil content of the hulls and meal would be indispensable in the operation of a mill. However desirable, this is by no means the case in actual practice. Two explanations suggest themselves: first, the organization of the industry; second, the method of analysis employed. The erection of many small mills is made necessary by the bulky character of the cottonseed, with its consequent cost of transportation, and by the impossibility of storing large quantities of seed for any great length of time without marked deterioration. The output of many of these small mills is not sufficient to justify the employment of a trained chemist, and the time required for obtaining an analysis from a distant laboratory largely discounts the value of the knowledge gained, except in extreme cases. But even if a laboratory is close at hand, or in the mill itself, five or six hours must elapse before the result of the analysis reaches the mill from the laboratory, this on account of the method of analysis employed. A more general resort to chemical control could be expected, therefore, if a simple, and rapid method of analysis were available. The present investigation has been undertaken in the hope of filling this need. Furthermore, the purchase of cottonseed by the mills is conducted regardless of the oil content of the seed. It is hoped that the rapid method

here described may be of some service in this important matter.

OBJECTIONS TO PRESENT METHOD.

The method of analysis usually employed consists in extracting the oil from a sample in a Soxhlet extractor with redistilled, low-boiling petroleum ether, evaporating the extractive and weighing the residual oil.

The objections to the method are:

First, the extraction must be conducted at a relatively low temperature, this being determined by the temperature of the condensed extractive dropping upon the sample from the condenser above.

Second, in the Soxhlet apparatus it is necessary not only to completely extract the oil but to transfer it completely to the flask below by repeated siphonings.

Third, considerable time is consumed in the complete evaporation of the extractive.

Fourth, the limitation to a comparatively small sample of the substance.

Fifth, the danger of fire.

Sixth, the necessity of using rather expensive apparatus and the need of a supply of running water for the condensers.

Seventh, the necessity of employing a trained chemist to conduct the operations.

PROPOSED METHOD.

It is believed that all of these objections are met in the method here proposed. It consists in digesting a definite and relatively large quantity of the substance with a definite volume of carbon tetrachloride in a loosely stoppered Erlenmeyer flask for 15 minutes at a temperature of approximately 60° C., cooling to room temperature, shaking thoroughly, filtering, and determining the specific gravity and temperature of the filtered extract. From these observations the per cent. of oil is calculated. Detailed directions for the analysis of the several products follow:

Meal.—Forty grams of the thoroughly mixed sample of meal are transferred to a 250 cc. Erlenmeyer flask, which is then placed in a water bath previously heated to approximately 60° C. After heating the flask about 2 minutes 100 cc. of carbon tetrachloride are poured gently upon the meal through a funnel reaching almost to the surface of the meal. The flask is lowered in the water until the surface of the carbon tetrachloride is lower than that of the water, a cork loosely inserted and the extraction allowed to proceed. Slight variations in the temperature of the water bath do not affect the accuracy of the determination. The minimum time for the extraction is 15 minutes, it may be longer. When the extraction is completed the flask is cooled to room temperature, thoroughly shaken and the contents strained through wire gauze on to a folded filter, the gauze being squeezed to insure sufficient filtrate. The clear extract is caught in a specific gravity tube (5 inches by 1 inch test-tube on foot). This is filled to within 1 inch of the top, then tightly corked and placed in a vessel containing water at room temperature. After standing at least 10 minutes the tube containing the extract is placed in a 500 cc. plain beaker containing sufficient clear water, at room temperature, to reach the level of the extract in the tube. By this means too rapid change in the temperature of the extract is avoided. The cork is then removed, the specific gravity of the extract determined by the Westphal balance, and the temperature accurately noted from the thermometer placed within the plummet of the balance. Add or subtract the constant for the carbon tetrachloride in use and subtract the corrected specific gravity of the extract from that of the carbon tetrachloride shown in Table 6 at the same temperature. This difference is divided by 0.00286. The result is the per cent. of oil.

Hulls.—After removing whole seed from the sample of hulls, 40 grams are placed in an Erlenmeyer flask and slightly packed with a glass rod. This is necessary to insure complete covering of the bulky hulls by the extractive. 100 cc. of carbon tetrachloride are added. The rest of the determination is identical with that for meal, except that the gauze strainer is not necessary in filtering.

Meats.—The method is the same as for meal except that 10 grams of meats are used with 100 cc. of carbon tetrachloride. The per cent. of oil found is multiplied by four.

Seed.—The sample of seed is thoroughly pounded in an iron mortar and the mass passed through a meat chopper. Forty grams are used with 200 cc. of carbon tetrachloride. The method is the same as for meal except that the per cent. of oil found is multiplied by two.

Experimental.

THE EXTRACTION.

The well-known solvent power of carbon tetrachloride for fats, its relatively high boiling point,

its low heat of vaporization and its non-combustibility, naturally suggested this substance as a substitute for gasoline and ether as the extractive. A. P. Bryant,[1] using a Knorr extractor, demonstrated the rapidity of extraction of fats in foods and feedstuffs by carbon tetrachloride. But he proposed merely the substitution of carbon tetrachloride for ether or carbon bisulphide in the usual method of extraction and evaporation of the extractive.

In the following experiments the determinations were carried out as follows: five grams of a substance whose oil content had been determined by the Soxhlet method were transferred to a 500 cc. Erlenmeyer flask, 50 cc. of gasoline or carbon tetrachloride added, and the extraction allowed to proceed at room temperature and without shaking, in order to avoid the sticking of the solid particles to the walls of the flask above the extractive. After standing a definite time the flask was thoroughly shaken in order to insure uniformity of the extract. This was then filtered through a dry filter paper. The amount of dissolved oil was determined in 25 cc. of the filtrate by distilling off the extract in a hot air bath, the temperature being quickly raised to 140° C. during the last stages of the evaporation. The last traces of the extractive were removed from the distillation flask by blowing quickly into it through a glass tube inserted almost to the oily layer. From the weight of the oil the per cent. was calculated by multiplying by one hundred and dividing the product by two and a half.

Cottonseed Meal.—7.29 per cent. oil by Soxhlet extraction with gasoline.

TABLE 1.

Time of extraction.	Per cent. oil with gasoline.	Per cent. oil with carbon tetrachloride.
7½ minutes	6.72	2.84
15 "	7.32	7.10
30 "	7.34	7.22
60 "	7.28	7.30
120 "	7.32	7.46

To determine the influence of temperature upon the rate of extraction by carbon tetrachloride, a series of experiments was carried out as above except that the Erlenmeyer flasks were placed in a water bath heated to 50° C. The results follow:

TABLE 2.

Time.	Per cent. of oil.
3¾ minutes	7.08
7½ "	7.44
15 "	7.38

The marked increase in the extraction at 50° in 7½ minutes as compared with that at room temperature for the same time led to the hope that the time for complete extraction could be still further diminished by carrying out the determination near the boiling point of carbon tetrachloride. This hope was fully justified, for by making an extraction at 70° for 3¾ minutes, the percentage of oil was found to be 7.30.

Cottonseed Meats.—30.27 per cent. oil by Soxhlet extraction.

TABLE 3.

Time.	Temperature.	Per cent. of oil.
30 minutes	25°	29.22
30 "	35°	29.50
30 "	50°	30.38
30 "	70°	30.62

From these determinations it is evident that the time-consuming extraction with the Soxhlet apparatus is unnecessary and that an accurate determination of the oil in these products can be made with simple apparatus, in a much shorter time, and with no danger from fire.

RAPID DETERMINATION OF OIL IN EXTRACT.

In order to simplify and hasten the determination of oil in the extract experiments were begun on the lowering of the specific gravity of the carbon tetrachloride by the dissolved oil. The great difference in the specific gravity of carbon tetrachloride (1.62) and of cottonseed oil (0.92) gave a reasonable hope for success. In order to avoid possible difference in the specific gravity of the crude oil obtained from the presses and that left in the meal the experiments were carried out on samples of meal whose oil content was determined in the usual manner by the Soxhlet extractor. The simplicity of the Westphal balance suggested this method of determining the specific gravity. Comparison of the thermometer on the plummet of the balance used in the investigation with a standard thermometer showed it to be accurate between 15° and 30°.

A preliminary test, using 5 grams of meal and 50 cc. of carbon tetrachloride, as in the above experiments on the rate of extraction, showed that the method would probably be accurate to 1 per cent. The first step, therefore, was to determine the minimum time and temperature for extractions of larger proportions of meal, the volume of carbon tetrachloride being kept constant. A sample of cottonseed meal was passed through a 20-mesh sieve, and thoroughly mixed. In two portions of 10 grams each the oil was determined by the Soxhlet method, using redistilled gasoline

[1] *J. Am. Chem. Soc.*, 26, 568.

as the extractive in one case and chemically pure carbon tetrachloride in the other. With the former the percentage was found to be 8.68; with the latter 8.67.

With this meal extractions were made according to the method outlined above. In each case allowance was made for the constantly increasing volume of oil in the extract. The results follow:

TABLE 4.

No.	Grams of meal.	cc. of carbon tetrachloride.	Time (minutes).	Temperature.	Per cent. of oil. I.	II.
1	10	50	5	70°	8.67	....
2	15	50	5	70°	8.58	....
3	20	50	5	70°	8.60	8.63
4	25	50	5	70°	8.18	....
5	30	50	5	70°	8.56	....

From the per cent. found in No. 5 it is evident that the low result in No. 4 was due to analytical error.

The use of 30 or even 25 grams of meal was found to be impracticable, as it was found to be impossible to secure sufficient filtrate for use with the Westphal balance. But with 20 grams sufficient filtrate could be obtained by using a strainer of brass or iron gauze and squeezing the extract on to a crimped filter. In order to avoid possible error in the specific gravity determinations due to the small amount of filtrate the proportions were increased to 30 grams of meal and 75 cc. of carbon tetrachloride. For the specific gravity determination a test-tube-on-foot 5 inches long and 1 inch in diameter was used. A magnifying glass was used in reading the thermometer on the plummet of the balance.

TABLE 5.

SPECIFIC GRAVITY OF CARBON TETRACHLORIDE.

Temperature.	Specific gravity.
15°	1.6041
20°	1.5951
25°	1.5861
30°	1.5771

Since these figures show a regular variation of 0.0090 for each 5 degrees the figures for intermediate temperatures are interpolated as follows:

TABLE 6.

Temperature.	Sp. gr.	Temperature.	Sp. gr.	Temperature.	Sp. gr.
15.0°	1.6041	17.2°	1.6001	19.4°	962
.2°	037	.4°	1.5998	.6°	958
.4°	034	.6°	994	.8°	1.5955
.6°	030	.8°	991	20.0°	1.5951
.8°	027	18.0°	987	.2°	947
16.0°	023	.2°	983	.4°	944
.2°	019	.4°	980	.6°	940
.4°	016	.6°	976	.8°	937
.6°	012	.8°	973	21.0°	933
.8°	009	19.0°	969	.2°	929
17.0°	005	.2°	965	.4°	926

TABLE 6—(*Continued*).

Temperature.	Sp. gr.	Temperature.	Sp. gr.	Temperature.	Sp. gr.
21.6°	922	24.4°	872	27.2°	821
.8°	919	.6°	868	.4°	818
22.0°	915	.8°	1.5865	.6°	814
.2°	911	25.0°	1.5861	.8°	811
.4°	908	.2°	857	28.0°	807
.6°	904	.4°	854	.2°	803
.8°	901	.6°	850	.4°	800
23.0°	897	.8°	847	.6°	796
.2°	893	26.0°	843	.8°	793
.4°	890	.2°	839	29.0°	789
.6°	886	.4°	836	.2°	785
.8°	883	.6°	832	.4°	782
24.0°	879	.8°	829	.6°	778
.2°	875	27.0°	825	.8°	1.5775

Meal.—To determine the lowering of the specific gravity of the carbon tetrachloride by increasing quantities of extracted oil a meal of known oil content (8.82 per cent.) was used. Quantities of this meal were used representing 30-gram portions each of 5.0, 7.5, and 10.0 per cent. meals; thus for

5.0 per cent. used 17.0068 grams;
7.5 per cent. used 25.5102 grams;
10.0 per cent. used 34.0135 grams.

These portions were extracted, each with 75 cc. of carbon tetrachloride in Erlenmeyer flasks for 5 minutes at 70°. The filtered extracts showed the following specific gravities:

TABLE 7.

Temperatures.	5 per cent.	7.5 per cent.	10 per cent.
15°	1.5900	1.5830	1.5758
20°	1.5810	1.5740	1.5668
25°	1.5720	1.5650	1.5578

The same difference in specific gravity of 0.0018 for each degree of temperature is observed here as was found with the carbon tetrachloride alone, but the decrease in specific gravity due to the increasing quantities of oil is not quite so regular. Thus the decrease due to the oil in 30 grams of a 5 per cent. meal is 0.0141, but in a 10 per cent. meal 0.0283. To test this more carefully a new and more delicate Westphal balance was obtainedl The meal used contained 8.20 per cent. of oi.. Quantities of this meal were used, representing 40 gram portions each of 5 and 10 per cent. meals, the volume of carbon tetrachloride being increased accordingly to 100 cc. In order to avoid too rapid change in the temperature of the extract during the determination of the specific gravity, the tube containing the extract was placed in a 600 cc. plain beaker filled with clear water to the level of the extract. Different specimens of carbon tetrachloride were used in the two sets of experiments. Corrections for temperature were made on the basis of 1° = 0.0018 difference in specific gravity.

TABLE 8.

FIVE PER CENT. MEAL.

SPECIFIC GRAVITY OF CARBON TETRACHLORIDE AT 26.4° = 1.5866.

Extract.	Temperature.	Specific gravity.	Calculated to 26.4°.	Difference.
No. 1	26.7°	1.5718	1.5723	0.0143
No. 2	26.8°	1.5716	1.5723	0.0143

TEN PER CENT. MEAL.

SPECIFIC GRAVITY OF CARBON TETRACHLORIDE AT 24° = 1.5918.

Extract.	Temperature.	Specific gravity.	Calculated to 24°.	Difference.
No. 1	24.0°	1.5632	1.5632	0.0286
No. 2	24.2°	1.5628	1.5632	0.0286

The decrease in specific gravity is, therefore, regular and each per cent. of oil lowers the specific gravity 0.00286 when 40 grams of meal and 100 cc. of carbon tetrachloride are used. The per cent. of oil in a meal is obtained, therefore, by determining at the same temperature the difference between the specific gravity of the carbon tetrachloride and the filtered extract from 40 grams of the meal digested with 100 cc. of the carbon tetrachloride, and dividing this difference by 0.00286.

CONSTANT FOR CARBON TETRACHLORIDE.

The difficulty in securing different lots of commercial carbon tetrachloride of exactly the same specific gravity led to experiments with a grade differing widely from that used in the earlier part of the investigation. The following results were obtained:

TABLE 9.

Temperature.	Sp. gr. original carbon tetrachloride.	Sp. gr. new carbon tetrachloride.	Difference.
15°	1.6041	1.5903	0.0138
20°	1.5951	1.5813	0.0138
25°	1.5861	1.5723	0.0138

Having determined this difference, 0.0138, in all operations with this new lot, corrections should be made by adding the constant to the observed specific gravity and immediate reference could be made to the table of specific gravity of the original carbon tetrachloride. Such a procedure would obviate the necessity of using a thermostat. To test this, 40 grams of meal containing 8.50 per cent. of oil were digested with 100 cc. of the new lot of carbon tetrachloride for 5 minutes at 70°. The extract showed a specific gravity of 1.5572 at 20°; adding the constant 0.0138 this becomes 1.5710, a difference of 0.0241 from the original carbon tetrachloride. Dividing by 0.00286 the per cent. was found to be 8.43.

TIME AND TEMPERATURE EXPERIMENTS.

Some specimens of commercial carbon tetrachloride boil at lower temperatures than that of pure carbon tetrachloride (76.7°). It was deemed advisable, therefore, to determine the rate and completeness of extraction at lower temperatures. In the determinations the per cents. were obtained by the specific gravity method, using a meal whose oil content had previously been found to be 8.82 per cent. The results follow:

TABLE 10.

Temperature.	5 min.	10 min.	15 min.	20 min.	30 min.	Standing over night.
25°	7.32	7.50	7.32	7.50	8.00	8.16 8.19
25° [1]	7.50	....	8.00 7.81	..	7.93	..
40°	7.72	7.72	7.88	..	..	..
50°	7.72	8.35	8.72	..	..	..
60°	8.06	8.06	8.75	..	..	..
	8.08	8.06	8.81	..	..	..

From this table it is evidently impracticable to work at room temperatures. The results show, however, that a complete extraction is obtained in 15 minutes at temperatures 50°–60° C. At such temperatures there is no risk of the commercial carbon tetrachloride boiling.

Confirmation of the accuracy of the method for cottonseed meal is furnished by the following check analyses, for many of which we are indebted to Mr. Jas. B. Pratt, chemist of the Southern Cotton Oil Co., Charlotte, N. C., and Mr. O. L. Spurlin, chemist of the Georgia Cotton Oil Co., Atlanta, Ga.

TABLE 11.

No.	Soxhlet extraction.	Specific gravity method. I.	Specific gravity method. II.
1	8.81	8.72	..
2	6.47	6.44	..
3	6.49	6.59	6.56
4	7.17	7.25	7.27
5	6.47	6.47	..
6	7.17	7.22	7.28
7	7.13	7.19	7.11
8	6.69	6.78	6.69
9	6.27	6.31	6.22
10	6.47	6.56	..
11	7.17	7.06	..
12	6.27	6.35	6.34
13	7.13	7.06	..
14	6.69	6.59	..
15	7.20	7.17	7.23
16	6.70	6.71	..
17	5.96	5.94	..
18	6.40	6.43	..

Meats.—Since the per cent. of oil in meats is high and varies between approximately 28 and 36 per cent., it was decided to use 10 grams of meats, instead of 40 as in the case of meal, with 100 cc. of carbon tetrachloride, in order to bring the specific gravity within the limit already worked out for meal, multiplying the per cent.

[1] Shaking at intervals.

found by four for the actual per cent. of oil in the meats. The use of a wire gauze strainer was unnecessary with meats.

TABLE 12.

PER CENT. OF OIL IN UNCOOKED MEATS.

Temperature.	Time.	I.	II.
70°	5	27.12	27.40
70°	15	29.12	29.28
70°	30	29.44	...
60°	15	29.28	...

From these results it is evident that the standard time and temperature of extraction of meal is suited also for extraction of the one-fourth quantity of meats. Experiments using a larger proportion of meats showed low results. Whether this was due to incomplete extraction or to a possible change of the specific gravity curve for such high per cents. was not determined.

TABLE 13.

PER CENT. OF OIL IN COOKED MEATS.

Temperature.	5 min.		10 min.		15 min.	
	I.	II.	I.	II.	I.	II.
50°	29.84	...	29.72	...	29.60	29.72
60°	29.60	...	29.88	...	30.34	...
70°	29.40	29.88	30.22	30.76	...	...

Again the extraction at 60° for 15 minutes is sufficient.

That the presence of moisture does not affect the extraction was demonstrated by extractions on both raw and cooked meats, at 60° C., for 15 minutes, thus:

TABLE 14.

	I.	II.
RAW MEATS (moisture 8.95):		
Hydrous	29.28	...
Anhydrous	29.40	29.12
COOKED MEATS (moisture 5.73):		
Hydrous	30.84	...
Anhydrous	31.40	31.00

Hulls.—After transferring 40 grams of hulls to the Erlenmeyer flask it was found necessary to pack the hulls with a glass rod in order to insure complete covering with 100 cc. of carbon tetrachloride. No wire gauze strainer was necessary in filtering the extract.

TABLE 15.

Temperature.	Time. Min.	Per cent. of oil. Soxhlet extraction.	Sp. gr. method.
60°	5	0.30	0.25
60°	10	..	0.32
60°	15	..	0.25
60°	30	..	0.25

It is proposed to extend the investigation to the determination of oil in linseed products, and of fats in feedstuffs, tankage, etc.

UNIVERSITY OF NORTH CAROLINA,
CHAPEL HILL, N. C., OCTOBER 29, 1908.

DETERMINATION OF SULPHUROUS ACID OR SULPHITES OR SULPHUR DIOXIDE IN FOOD PRODUCTS.[1]

BY EDWARD GUDEMAN.

Received October 27, 1908.

The method for the determination of sulphurous acid or sulphites or sulphur dioxide in food products as given in the Official and Provisional Methods of Analysis, Bulletin **107**, revised, Bureau of Chemistry, Dept. of Agriculture, having been accepted by the Association, is the standard or official method of analysis.

The essential part of the method calls for a direct distillation of 100 grams of the food product (with or without addition of water) acidified with 5 cc. of 20 per cent. glacial phosphoric acid, distilling into an excess of a standard iodine solution and titrating back the excess of iodine; the distillation is considered complete when 50 cc. have distilled over. In the analysis of meats and meat products the phosphoric acid is increased to 20 cc. (page 113), and in the examination of wines, direct titration of the sulphurous acid, without distillation, is permissible (page 188).

A large number of analyses of natural and prepared food products have shown that often weighable amounts of sulphur dioxide (or its compounds) will be obtained from food products that actually contain none at all, and when sulphur dioxide is present, the amount obtained by the official method will be in excess of the amount actually present, using the official method without modification. Many animal and vegetable food products normally contain sulphur compounds, which on distillation with acids give off volatile sulphur compounds, which react with the iodine solution and reducing the excess of standard iodine solution used, are thereby calculated to and considered and reported as sulphur dioxide present in the food product examined. Recognition of this source of error is taken when applying the method to the examination of meat and meat products, it being stated "mere traces should be ignored" (page 113). A like statement should be added to the official method itself, applying in the examination of all food products, ignoring the presence of "mere traces" of sulphur dioxide.

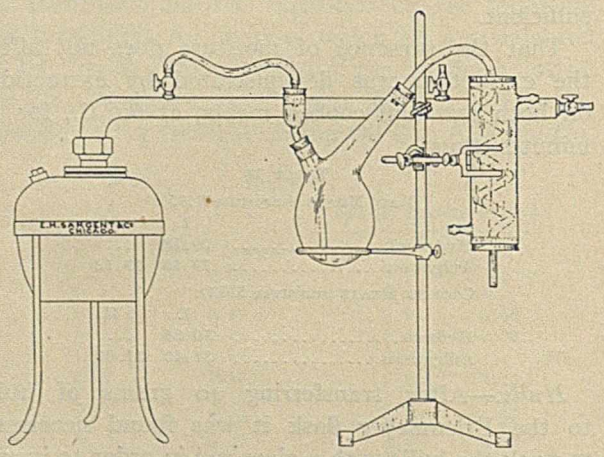
Sulphuretted hydrogen often liberated by the action of the acid, will react on the iodine solution and be reported to that extent as sulphur dioxide,

[1] Read before the Association of Official Agricultural Chemists, Nov., 1908.

unless the sulphides are separated by first passing the distillate through some solution of a metallic salt, as recommended by W. D. Horne and A. L. Winton. In many cases the sulphides can be held back by adding some metallic salt (cadmium, copper, silver, lead, etc.) directly to the product before beginning the distillation, thereby simplifying the process and not requiring the additional apparatus. Volatile sulphur compounds which do not appear as sulphuretted hydrogen and are not held back by the metallic salts, will react on the iodine solution and be calculated to and reported as sulphur dioxide present in the food product, and such volatile sulphur compounds will erroneously increase the amount of sulphur dioxide, free or combined, actually contained in the product.[1]

In the direct distillation of an acidified food product a high temperature of distillation is caused by the food product itself. During the direct distillation, concentration of the mass takes place and the acidity increases in nearly direct proportion to this concentration. With food products to which no water need be added for distillation, the original acidity (due to the added phosphoric acid) is 1 to 4 per cent. based on the product used (100 grams), and after distillation taking off 50 cc., the acidity will be nearly double 2 to 8 per cent., decomposition of the product increasing with the increasing acidity, giving rise to the peculiar volatile sulphur compounds. The decomposition of the food product due to the concentration, with increased acidity and high temperature, is often intensified by the previous treatment such prepared food product undergoes during its method of preparation, the "processing" of the product, especially noticeable with canned goods, which have been cooked or sterilized at high temperature. The analytical figures show that some fresh products, containing only traces or no sulphur dioxide in the raw state, after processing give strong reaction for sulphur compounds (see table). It was often found that a distillate of 50 cc. from a 100-gram sample was not sufficient to carry over all the sulphur dioxide and the other volatile sulphur compounds. Accidentally correct results may be obtained for sulphur dioxide, the distilled volatile sulphur compounds counterbalancing the loss of sulphur dioxide due to incomplete distillation, especially with such products that on heating become thick and gelatinous, causing foaming and bumping. Increasing the amount of the distillate produces greater decomposition, especially noticeable with food products, to which no water need be added for distillation, such as fruit juices, cordials, extracts, wine, beer, etc., all of which contain not over 20 per cent. non-volatile matter.

The following simple modification of the official method has been found to eliminate many of these sources of error and to overcome most of the other objections raised. Instead of a direct distillation, the volatile products are driven over with low pressure steam. The steam is generated from distilled water and directly led into the mass. A good Bunsen burner will generate enough steam to make from 6 to 8 distillations at one time, within one hour, giving 4 to 500 cc. of distillate per sample. Any of the well-known water stills can be used, replacing the condensing coil with a copper tube, having sufficient stop-cock openings from which the steam is led through rubber tubing, into the retorts or distilling flasks containing the products to be analyzed. The accompanying sketch is self-explanatory.[1]



On account of condensation of steam during distillation, no possible concentration can take place, in fact dilution takes place and the retort or other distilling flask must be sufficiently large to accommodate this increase in bulk. Excessive condensation can be overcome by increasing the steam pressure or by using a small burner under the sand bath on which the distilling flask rests. The temperature of distillation can never increase above that of the steam used. Acidity cannot increase, in fact the acidity will decrease in nearly

[1] Reports by W. D. Bigelow, F. Zerban and W. P. Naquin, *Proceedings A. O. A. C., Bull.* **116**, Bureau of Chemistry, issued Oct. 27, 1908.

[1] Continous water stills can be used if feed water is free from volatile sulphur compounds. Complete apparatus can be obtained from E. H. Sargent & Co., Chicago, to whom my thanks are due for the above illustration.

exact proportion as dilution takes place, due to the condensation on the sides of the retort. The steam entering below the surface of the mass acts as a mechanical stirrer and prevents foaming and bumping. Decomposition of the mass due to concentration, high temperature and increased acidity are eliminated and the distillate contains no volatile sulphur compounds due to such decomposition as found in the method with direct distillation. Using steam the amount of the distillate can be regulated and always can be sufficiently large (3–500 cc.) and of such character, that the sulphur dioxide can be determined by direct titration with iodine solution, as per method 2, page 168, Bull **107**, revised, thereby doing away with any error due to loss of iodine. For such direct titration of the distillate the method is modified by replacing the iodine in the absorption tube, beaker or flask, by a weak alkaline solution, a large beaker being most convenient. When distillation is complete, the starch indicator is added, distillate slightly acidified with hydrochloric, sulphuric or phosphoric acid and quickly titrated with a standard iodine solution.

The use of steam for distillation was found to have extended application, especially in the determination of sulphurous acid and its compounds and in the examination and determination of glycerin, ammonia, volatile fatty acids, alcoholic beverages, ether, aldehydes, extracts, turpentine, cereals, etc. and in obtaining distillates from substances that foam or bump.

Some comparative results in the determination of sulphur dioxide in food products are as follows:

SULPHUR DIOXIDE FOUND.
Parts per 100.

Prepared samples.[1]	Official method. Direct titration.	Official method. After oxidation.	Modified method. Direct titration.	Modified method. After oxidation.
Sugar and added sulphites	0.0045	0.0045	0.0050	0.0050
Flour and added sulphites	0.0037	0.0040	0.0048	0.0048
Starch and added sulphites	0.0022	0.0022	0.0025	0.0025
Regular samples				
Gelatine (home-made)	0.0002	0.0006	0.0000	0.0000
Gelatine (French)	0.0270	0.0276	0.0260	0.0260
Gelatine (American)	0.0130	0.0142	0.0120	0.0121
Meat—fresh	0.0001	0.0004	0.0000	0.0000
Eggs—fresh	0.0003	0.0005	0.0000	0.0000
Eggs—age unknown	0.0003	0.0008	0.0000	0.0000
Dried fruit	0.0083	0.0087	0.0078	0.0077
Dried fruit	0.0240	0.0251	0.0228	0.0228
Molasses—bleached	0.0456	0.0476	0.0410	0.0414
Molasses—bleached	0.0300	0.0307	0.0288	0.0287
Molasses	0.0084	0.0088	0.0079	0.0079
Candy	0.0042	0.0042	0.0047	0.0047
Candy	0.0017	0.0019	0.0017	0.0017

[1] Sulphur dioxide added as sulphite: Sugar, 0.0050 part per 100
Flour, 0.0050 part per 100
Starch, 0.0025 part per 100

SULPHUR DIOXIDE FOUND.
Parts per 100.

Prepared samples.	Official method. Direct titration.	Official method. After oxidation.	Modified method. Direct titration.	Modified method. After oxidation.
Candy	0.0120	0.0120	0.0126	0.0126
Peas, fresh, average 5	0.0002	0.0002	0.0000	0.0000
Peas, same processed	0.0002	0.0008	0.0000	0.0000
Peas, canned, average 5	0.0003	0.0008	0.0000	0.0002
Peas, canned, average 5	0.0004	0.0011	0.0002	0.0004
String beans, white, fresh	0.0000	0.0003	0.0000	0.0000
String beans, same processed	0.0002	0.0007	0.0000	0.0002
String beans, yellow, fresh	0.0001	0.0002	0.0000	0.0000
String beans, same processed	0.0001	0.0005	0.0000	0.0002
Cabbage, fresh	0.0002	0.0003	0.0000	0.0000
Cabbage, same processed	0.0002	0.0011	0.0000	0.0003
Green peppers, fresh	0.0000	0.0002	0.0000	0.0002
Green peppers, same processed	0.0002	0.0006	0.0000	0.0003
Cauliflower, fresh	0.0000	0.0000	0.0000	0.0000
Cauliflower, same processed	0.0000	0.0003	0.0000	0.0002
Rhubarb, fresh	0.0000	0.0000	0.0000	0.0000
Rhubarb, same processed	0.0001	0.0002	0.0000	0.0000
Onions, fresh	0.0000	0.0004	0.0000	0.0002
Onions, same processed	0.0002	0.0008	0.0000	0.0006
Cucumbers, fresh	0.0000	0.0000	0.0000	0.0000
Cucumbers, same processed	0.0000	0.0001	0.0000	0.0000
Okra, fresh	0.0000	0.0000	0.0000	0.0000
Okra, same processed	0.0000	0.0000	0.0000	0.0000
Processing solution[1]	0.0000	0.0000	0.0000	0.0000

The figures obtained on the prepared samples, indicate that carrying out exactly the Official method of distillation is not sufficient to drive over all the sulphur dioxide contained in the samples. The character of the products influencing the amount of sulphur dioxide held back, from 10 per cent. for sugar to 26 per cent. for flour, the loss due to incomplete distillation in the modified method being 4 per cent. for flour and none for the other two products. With the exception of candies, the modified method gives lower results, although the distillation is more complete.

When examining cereals or other products very high in starch, both methods must be modified by either increasing the amount of acid and water or decreasing the amount of the material used, as otherwise the mass becomes semi-solid or pasty. The vegetables enumerated were obtained in the open market. The processing consisted in taking the washed vegetables, breaking or crushing into small pieces, when necessary, and then placing 500 grams into Mason jars, pouring on 500 cc. of the hot processing solution, hermetically sealing the jars, and then placing into a boiling water bath for one hour. After cooling, the whole mass was pulped and of this 200 grams, representing 100 grams of the vegetable, taken for each determination, adding in each case 100 cc. water using the official method and 200 cc. water using the modified method, the amount of distillate being

[1] Processing solution: sugar 5 parts, salt 1 part, and water 100 parts.

exactly 50 cc. for the official method and not less than 400 cc. for the modified method. In making the direct titration tests for both methods, the distillates were caught in an alkaline solution, and after acidifying with phosphoric acid, titrated with 1/100 normal iodine solution and starch paste as the indicator. When sulphur dioxide was determined by oxidation, including all other volatile sulphur compounds, the distillate was caught in an iodine solution to which a few drops of bromine had been added. This was boiled over a direct flame until colorless and total sulphur compounds precipitated with barium chloride and weighed as barium sulphate. The apparatus was always filled with carbonic acid gas, generating same in the steam boiler from bicarbonate of soda and phosphoric acid.

It was found that results obtained in the direct distillation, official method, differed, as the amount of the distillate was increased above 5 cc. Up to a certain, not constant point, a slight increase was obtained by both direct titration and by oxidation. Going beyond that point, the amounts found by direct titration of the excess of iodine, were always above those found by oxidation, showing conclusively that non-sulphur decomposition products were distilled, due to further decomposition and that these materially affected the back titration of the excess of iodine. This was further confirmed by the fact that the results obtained when distilling directly into an iodine solution were greater than the actual sulphur dioxide calculated from the barium sulphate precipitate of the same solution and the results obtained by direct distillation into iodine were always higher than those obtained when distilling into an alkaline solution and titrating the sulphur dioxide liberated on acidifying. The amount of sulphur dioxide found by oxidation of a larger quantity of distillate being less than the amount found on titrating the distillate direct, using the larger amount for the determination of sulphur dioxide by precipitation.

Volatile compounds, such as essential oils, ethers and oxides, which combine with iodine, effect the accuracy of both methods, but are partly eliminated in the modified method, depending upon their solubility in the alkaline solution into which they are distilled. Such volatile compounds often can be separated by boiling the alkaline distillate, then cooling, diluting to original bulk and, after acidifying, titrating the sulphur dioxide remaining in the solution.

No appreciable error is introduced by boiling the alkaline distillate.

The comparative results obtained in the determination of sulphur dioxide and its compounds in food products by the official and modified methods, justifies the conclusions that:

I—The Official method for sulphur dioxide, sulphites or sulphurous acid should be changed so as to eliminate the possibility, as has occurred in actual practice, of food products being condemned and rejected by food officials and private persons as not meeting the requirements of Federal and State Food Acts, on account of the presence of sulphur dioxide or its compounds, examined according to the Official method, but containing no added sulphur compounds.

II—Traces of sulphur dioxide or its compounds, or what is found as such, should be ignored as well for all food products as specifically only for meat and meat products.

THE DETERMINATION OF ESSENTIAL OIL AND ALCOHOL IN FLAVORING EXTRACTS.

By Julius Hortvet and Rodney Mott West.

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Little is to be found in chemical literature relating to the determination of either essential oil or alcohol in flavoring extracts. With the exception of extracts of lemon and orange, this field of investigation[1] appears to have been in great measure neglected, and it is doubtful whether prior to 1899 any successful attempts had been made to devise methods of making these determinations. In a paper entitled "Lemon Flavoring Extract and its Substitutes,"[2] published about ten years ago, A. S. Mitchell gave two methods for the determination of lemon oil. These methods, somewhat revised, were later adopted as provisional by the Association of Official Agricultural Chemists.[3] In 1901, Winton and Ogden[4] applied the polariscopic method, as described by Mitchell for lemon extract, to extract of orange, establishing the factor 5.3 for the calculation of the per cent. of oil by volume from the reading on the Ventzke sugar scale. With reference to miscellaneous flavor-

[1] Only those flavoring extracts which consist chiefly of an alcoholic solution of an essential oil are considered in this paper.

[2] *Jour. Am. Chem. Soc.*, **21**, 1132 (1899).

[3] Bull. **107**, Bureau of Chemistry, U. S. Dept. of Agr., pp. 156–161.

[4] Report Conn. Agr. Expt. Sta., 1901, Part II, p. 176.

ing extracts, the following general statement by A. E. Leach[1] appears to have been accepted without serious question: "In the case of extracts of oils, it is generally practicable to separate the oil for examination by the centrifuge, as in the precipitation method of Mitchell, and to estimate the same directly as described on page 742 (Mitchell's method *b*), when the specific gravity of the oil in question is less than water. In the case of such oils as clove, wintergreen and cassia, where the specific gravity is greater than water, it is necessary to add salt to the extract before centrifuging to cause separation of the oil." The same author also describes an extraction method for the determination of oil of almond in almond extract.[2] An article entitled "The Precipitation Method for the Estimation of Oils in Flavoring Extracts and Pharmaceutical Preparations,"[3] published during the present year by C. D. Howard, so far as known, completes the literature on this subject up to the present time. A discussion of the general method described by Howard will be given at a later stage in the course of this article. In the case of extracts other than those of orange and lemon, many food chemists appear to have satisfied themselves with results obtained from the estimation of alcohol and the detection of essential oil by means of dilution with water. The following statement fairly illustrates some of the prevailing methods: "Extracts of clove, peppermint, orange and almond, which are of standard strength, will on dilution with water become turbid, due to the precipitation of the oil. The true extract will contain enough alcohol so that it will burn, but those below standard will not do so." It appears from published analyses, chiefly in the reports of several state food laboratories,[4] that the solids and ash of extracts have been relied upon to some extent as criteria of purity, but it is uncertain from the analyses themselves whether such results were obtained merely for the purpose of detecting the addition of sugar or glycerine. However, since the solids were determined by drying to constant weight at 100° C., it is readily seen that the results could be of little or no practical value in connection with the determination of either the oil or alcohol content of an extract.

The adoption of standards for flavoring extracts by the joint Committee on Food Standards of the Association of Official Agricultural Chemists and the Association of State and National Food and Dairy Departments, followed by the legal adoption of these standards by several of the states, has rendered it imperative that further attempts be made to perfect methods whereby the amount of essential oil in any of the common extracts used for food flavoring purposes may be readily and accurately determined. After numerous preliminary trials in this laboratory with methods which have heretofore been proposed, it became very evident that it would be difficult if not impossible to devise a single method that would be applicable to all extracts. Very few of the volatile oils used in making the common flavoring extracts are optically active. Only in the case of the oils of lemon and orange peel and celery seed are the rotations sufficiently high and constant to render possible a practical method based on the polarization of the extract. All of the oils, with the exception of oil of bitter almond, may be more or less completely precipitated by the addition of water to the acidified alcoholic solution, but only about half of them can be separated sufficiently to afford a method of any practical value. The reasons for the failure of the precipitation method in so many instances are at once apparent when the differences in chemical composition and physical properties of the various oils are considered. For instance, oil of bitter almond, which is essentially benzaldehyde, has a solubility in water of one part in three hundred, while a one per cent. solution can be made with 20 per cent. alcohol. The addition of water in any amount to such a solution will not cause the least cloudiness. In both cinnamon and cassia oils the principal constituent is cinnamic aldehyde, also fairly soluble in dilute alcohol, and in extracts where either of these oils is present in amount above two per cent. the precipitation method gives only about fifty per cent. of the total oil. Oils of lemon, orange and nutmeg, on the other hand, consisting very largely of terpenes insoluble in dilute alcohol and chemically passive in respect to the dilute acid used in the precipitation method, are all very easily precipitated. It has therefore been deemed necessary to devise for each kind of extract, or for each class of extracts, a special method for the determination of the essential oil. Any such method should be applicable not only in the presence of very low as well as very high percentages of oil, but also

[1] Food Inspection and Analysis, A. E. Leach, p. 747.
[2] Food Inspection and Analysis, A. E. Leach, p. 750.
[3] *Jour. Am. Chem. Soc.*, **30**, 1908, 608.
[4] State Dairy and Food Commission Reports: Ohio, 1896; Michigan, 1903; Illinois, 1905, 1906, 1907; Inland Revenue Department, Ottawa, Canada, Bull. **89**, p. 13 (1903).

in the presence of excessive amounts of alcohol, sugar or glycerine, and the results should not be vitiated by the presence of any of the common adulterants. Also, it is very desirable that the oil should be separated in such condition that tests for impurities may be carried out directly upon it. In many cases it has been found that the condition necessary to a satisfactory method are most nearly reached by precipitation; hence this method has been tried wherever possible. A modification of the present Official precipitation method has been found to work acceptably with extracts of lemon, orange, peppermint, nutmeg and wintergreen. Two other general methods have also been found satisfactory: one consists in the extraction of the oil by means of a volatile immiscible solvent, the other in the determination of the principal constituent of the oil; the first has been found to work acceptably with extracts of clove, cinnamon, cassia and rose, the second with extracts of almond and wintergreen.

For the purpose of preliminary work, alcoholic solutions were prepared containing in each instance an amount of oil sufficient to make an extract which would comply with the standard found in Circular No. 19 of the United States Department of Agriculture. These solutions were then subjected to examination according to the various methods for determining volatile oil.

Almond Extract.—An extract of almond was prepared, containing one per cent. by volume of oil of bitter almond, free from hydrocyanic acid, in approximately 25 per cent. alcohol. It was found that any amount of water, whether acid or neutral, or saturated with salt, could be added to the extract without causing the precipitation of even the slightest trace of the oil. Attempts at extraction with an immiscible solvent proved equally fruitless, owing principally to the rapid partial oxidation of the oil to benzoic acid during the evaporation of the solvent. All attempts to conduct the evaporation in such a manner as to complete the oxidation quantitatively resulted in loss, spontaneous evaporation giving rise to loss of aldehyde while evaporation at higher temperatures caused an additional loss of benzoic acid. The method given by Leach is open to the same objections, the long and tedious process of removing the alcohol holding a doubtful advantage over the method in which alcohol can easily be removed by washing the ether with a few changes of water. Bitter almond oil, free of hydrocyanic acid, is substantially benzaldehyde, the amount of the latter ingredient ranging ordinarily from 90 to 95 per cent. Hence it occurred that a determination of the benzaldehyde content of an extract would afford the most practicable means of ascertaining the amount of oil present. The customary aldehyde methods were tried, both the bisulphite of soda method[1] and the neutral sulphite method[2] resulting in indifferent success. Any reasonable concordance in results seemed to be impossible and approximately only from 85 to 90 per cent. of the actual oil present could be obtained. Such results were without doubt at least partially due to the presence of alcohol since in the neutral sulphite method, alcohol, even in small amounts, prevents the formation of starch-iodide, while the high acetaldehyde content of commercial alcohol would in any case vitiate results obtained by a method based on the determination of the total aldehyde. Considering also the ease with which benzaldehyde is oxidized, it is obviously inaccurate as well as unfair to estimate the oil merely from the aldehyde, when a considerable amount of the oil which was originally added may have been converted to benzoic acid subsequent to the manufacture of the extract. Furthermore, a correction based on the acidity of such an extract would be too superficial to be of value, since commercial alcohol is always decidedly acid in reaction. It has become necessary, then, for factory control work, as well as for general analytical purposes, that a method be devised whereby the benzaldehyde content of an extract at the time of manufacture may be fairly determined. It appeared to be practicable to base a calculation of the original oil on the benzoic acid obtained after complete oxidation of the aldehyde. With this plan in mind, various methods were tried, aiming at a complete oxidation of the oil without loss by volatilization. After several trials with various oxidizing agents, including oxygen, potassium bichromate and potassium permanganate, hydrogen peroxide in alkaline solution was found to be the most satisfactory. Attempts were made to oxidize in a standard alkaline solution and titrate the excess of alkali, but owing to the indeterminate increase of acidity, the results were far too high and non-concordant. Such results were in all probability due to the oxidation of the ethyl alcohol in the extract. The hydrogen peroxide was submitted to a blank titra-

[1] Sutton, "Volumetric Analysis," ninth edition, p. 371.
[2] S. S. Sadtler, *Jour. Soc. Chem. Ind.*, **23**, 303 (1904).

tion, but even with the correction thus afforded, the results were still unsatisfactory. Recourse was then had to extraction with ether as a means of obtaining the benzoic acid in a pure condition. The method finally adopted is as follows:

Ten cc. of the extract are measured out by means of a pipette into a 100 cc. flask, ten cc. of approximately a 10 per cent. solution of sodium hydroxide and 20 cc. of a 3 per cent. solution of hydrogen peroxide added, the flask covered with a watch-glass and placed on a water oven. Oxidation begins almost immediately and should be continued from five to ten minutes after all odor of benzaldehyde has disappeared. Usually from twenty to thirty minutes will be found sufficient. If nitrobenzol be present, it will be indicated at this point by its odor. When the oxidation of the aldehyde is complete, the flask is removed from the water-oven, the contents transferred to a separatory funnel, rinsing off the watch-glass into the funnel, 10 cc. of a 20 per cent. solution of sulphuric acid added, and the contents of the funnel cooled to room temperature under the water tap. The acid solution is extracted with three portions of ether, using in succession 50, 30 and 20 cc. A fourth extraction showed no trace of benzoic acid. The extracts are combined in another separatory funnel and washed with distilled water until the wash water shows no trace of sulphate on acidifying with hydrochloric acid and testing with barium chloride. Two portions of water of from 25 to 30 cc. each should be sufficient to remove all of the sulphuric acid. The ether extract is transferred to a wide-necked flask and the ether evaporated by placing the flask on a water oven. When the volume of the ether has been reduced to about 5 cc., the flask is removed from the oven and the evaporation completed spontaneously. When all of the ether has evaporated from the benzoic acid, the sides of the flask are washed down and the crystals dissolved by means of 95 per cent. alcohol which has been made neutral to phenolphthalein with tenth-normal sodium hydroxide. The solution of benzoic acid is diluted with about an equal volume of water and the acid titrated with tenth-normal sodium hydroxide, using phenolphthalein as indicator.

$$\frac{\text{cc. of N/10 NaOH} \times 0.01061 \times 10}{1.045} = \text{per cent.}$$

of benzaldehyde by volume. If desired, the ether extract may be filtered through a dry filter into a tared flask, the ether evaporated as before, the residue dried in a desiccator over night and weighed as benzoic acid.

$$\frac{\text{Weight of acid} \times 0.869 \times 10}{1.045} = \text{per cent. of}$$

benzaldehyde by volume. The volumetric and gravimetric results are not found to differ by more than a few hundredths of one per cent. The method is satisfactory,[1] giving concordant results irrespective of the per cent. of alcohol or oil which may be present. Also, results by this method are not in the least vitiated by the presence of hydrocyanic acid, glycerine or sugar. If nitrobenzol be found to be present, it may be removed by the method given by Leach[2] before attempting the determination of the aldehyde. The fact that benzaldehyde constitutes not more than from about 90 to 95 per cent. of almond oil somewhat detracts from the value of the method, but, on the other hand, as the necessary correction will be seen to be fairly constant, this objection is of far less moment than would at first appear.

Cinnamon, Cassia and Clove Extracts.—A two per cent. by volume solution of each of these oils in approximately 65 per cent. alcohol was prepared for the preliminary work. As the specific gravity of each oil averages about 1.050 at 25° C., the precipitation method recommended by Leach was attempted. Other heavy liquids, such as dilute sulphuric acid (1 : 1) and a saturated solution of copper sulphate, were also tried. With none of these precipitants, however, were the results satisfactory. This was true especially in the case of clove oil which seemed to be acted upon chemically and dissolved by the salt as well as by the acid. Only very small percentages of the total oil were found to be recoverable under the best conditions. A method similar to the one described for the determination of benzaldehyde was then tried with cinnamon and cassia extracts. By this method the cinnamic aldehyde was oxidized to the corresponding acid by means of hydrogen peroxide in alkaline solution, the solution acidified, extracted with ether, the ether evaporated and the residue weighed as cinnamic acid and calculated to cinnamic

[1] This method may be used for the determination of benzaldehyde in almond oil by weighing out 0.1 to 0.2 gram of oil, dissolving in about 10 cc. of alcohol and continuing as in the method for an extract. To determine the amount of the aldehyde actually present as such in the oil, a few cc. of oil should be dissolved in a little carefully neutralized alcohol and titrated with standard alkali. The acidity thus obtained calculated to benzoic acid is subtracted from the total acid before calculating to benzaldehyde.

[2] Food Inspection and Analysis, A. E. Leach, p. 750.

aldehyde.[1] Very concordant results were obtained, but the aldehyde content of these oils is too small to make the method practicable. There remained then the method of extraction of the volatile oil, using ether as the immiscible solvent. Ten cc. of the sample were diluted with water and extracted with three portions of ether. In the earlier experiments the ether was evaporated without previous washing in a tared dish and the residue weighed. It was found, however, after driving off the ether that there still remained a considerable amount of alcohol which could only be removed with difficulty and with considerable loss of oil. To surmount this difficulty, it was found sufficient to wash the combined ether extracts with two portions of distilled water of from 25 to 30 cc. each. By this means practically all of the alcohol was removed and the loss of volatile oil was found to be negligible. The greatest difficulty, however, consisted in finding a method for the evaporation of the solvent which would cause the least possible loss of oil. Heating on the water oven until only a few cc. of ether remained, then finishing the evaporation spontaneously, resulted in too great a loss of oil, while, strangely enough, spontaneous evaporation from the start caused a still greater loss. Rapid evaporation of the ether under diminished pressure was found to lower the temperature of the water to the congealing point, thus making it possible to obtain a weighable residue only after a prolonged drying which resulted in an apparent loss of volatile oil. Yet fairly good results were obtained by this method providing the ether extract was previously dried. Shaking for a few minutes with calcium chloride and filtering through a dry filter was found to dry the ether sufficiently in the case of extracts of cinnamon and cassia. Clove oil, however, was found to combine with the calcium chloride, as well as to react chemically with all of the common dehydrating agents, thus making reliable results impossible. Since the best results had been so far obtained in those determinations in which the ether was boiled off as rapidly as possible under diminished pressure, it appeared possible that good results might be obtained by rapid evaporation at higher temperatures in the open air, with the additional advantage of removing the water remaining in the ether. That is to say, the loss in volatile oil seemed to be somewhat in proportion to the time taken for the evaporation of the solvent, irrespective of temperatures well below the point at which the oil itself would distil. Surprising as it may seem, the best results were obtained by evaporating the ether from a wide mouth flask placed in direct contact with the live steam from a boiling water bath. The boiling ether kept the temperature of the solution below 40 to 45° C., while the few minutes required for the complete removal of the solvent gave little time for the evaporation of the essential oil. In the case of oil of cinnamon, better results were obtained when the ether extract was first dried by shaking with a little calcium chloride. With cassia and clove extracts, however, the results were exceedingly good when the evaporation was carried out without previous drying of the ether. This method requires considerable care in manipulation and the following details should be closely adhered to.

Ten cc. of the extract are transferred to a separatory funnel, 50 cc. of water added, and the mixture extracted with three portions of ether, using in succession 50, 30 and 20 cc. The combined ether extracts are washed with two changes of distilled water, using each time 25 to 30 cc., and filtered through a dry filter into a wide mouth flask, washing out the funnel and filter with a little ether. In the case of an extract of cinnamon, the ether extract before filtering is transferred to a 150 cc. flask, shaken for a few minutes with some granulated calcium chloride, then filtered through a dry filter in the manner described above. The ether is evaporated off as rapidly as possible on a boiling water bath until only a few drops remain. At this point the flask is removed from the water bath and rotated rapidly for a few minutes, spreading the residue over the sides of the flask. The rapid evaporation of the remaining ether cools the flask to near room temperature. When the odor of ether has disappeared, the flask is stoppered and weighed. In the case of cassia and clove oils, where the ether extract is not first dried with calcium chloride, a slight cloudiness gathers on the flask as the last traces of ether disappear, due to the presence of a little moisture. In such case, the flask is allowed to stand on the balance pan until the film of moisture disappears (requiring not longer than two or three minutes), then stoppered and weighed.

$$\frac{\text{Weight of oil} \times 100}{10 \times 1.050} = \text{per cent. of oil by volume.}$$

The oil thus extracted may be used for examination

[1] This method appears to be a practicable one for the determination of cinnamic aldehyde in oils of cinnamon and cassia, but as yet no data have been obtained to establish its reliability.

as to purity by means of the refractometer. It may also be tested by dissolving a portion in a little alcohol and adding a drop of ferric chloride. Cinnamon oil will give a green coloration, cassia a brown and clove a deep blue.

Rose Extract.—On account of the small percentage of oil of rose which is commonly employed in making the extract, as well as the fact that the oil is not readily separated from solution by dilution with water, no attempts were made to apply the precipitation method. Furthermore, oil of rose is comparatively optically inactive and of such composition that the choice of a method appeared to be narrowed to that of extraction by means of an immiscible solvent. Accordingly, an extract was prepared by dissolving 0.4 cc. of oil of rose in 100 cc. of approximately 80 per cent. alcohol. A number of determinations were attempted, resulting finally in a method in some respects similar to the foregoing described for extracts of cinnamon and clove. It was, however, not found advisable to wash the ether solution with water or to apply the method of rapid evaporation over a water-bath. The method is as follows:

Twenty cc. of the extract are measured into a separatory funnel, 50 cc. of water added, the solution thoroughly mixed, acidified with 1 cc. of hydrochloric acid (1 : 1), and extracted with three portions of ether, using each time 20 cc. The combined ether extracts are transferred to a 150 cc. flask, shaken for a few minutes with some granulated calcium chloride, allowed time to settle to a clear solution, then decanted through a dry filter into a flat bottom glass dish previously weighed together with a cover glass. The calcium chloride and filter are twice washed with 10 cc. of ether and the washings added to the contents of the dish. The dish is covered, placed in a vacuum desiccator over sulphuric acid and allowed to remain until all traces of ether and alcohol are removed, then weighed, again placed in the desiccator at ordinary pressure for one hour and weighed again. The last operation is repeated if necessary and the weight recorded when practically constant. The final weight divided by 0.86 and multiplied by 5 gives the per cent. of oil of rose by volume.

Lemon, Orange, Peppermint and Nutmeg Extracts.—According to chemical literature, menthol, the chief constituent of oil of peppermint, is found in the oil in amounts varying from approximately 40 to 90 per cent. Hence, any attempts to base a calculation of the amount of oil in extract of peppermint on the determination of the menthol content would be likely to yield unsatisfactory results. With oil of nutmeg the case is similar, the chief constituent of the oil being pinene which could not readily be determined in a solution containing the oil in amounts commonly employed in making the extract. It is apparent, also, from published data that the optical rotation of neither oil of peppermint nor oil of nutmeg could afford a successful polarimetric method. Preliminary trials, however, with the Official precipitation method for lemon extract were found to give satisfactory results. In the case of a peppermint extract made to contain 3 per cent. of the oil a correction of only 0.2 per cent. was found necessary, while in the case of a nutmeg extract made to contain 2 per cent. of oil scarcely an appreciable correction was required. It was considered, however, that for the purpose of further examination of the separated oil as well as for the alcohol determination, the amount of sample taken in the Official method is inadequate; hence it was thought that it might be desirable to devise an apparatus whereby there might be employed a larger sample, thus avoiding some of the usual difficulties and at the same time possibly lessening the percentages of error in the results. The larger tube, shown in Fig. 1, is similar in shape to the Babcock milk test bottle, but is of 150 cc. capacity to the beginning of the graduations on the stem. The stem is graduated to read to tenths of one per cent., the divisions being based on the use of 50 cc. of sample in the determination. As the common form of centrifuge is obviously not suitable for carrying the larger test bottles, recourse was had to the Bausch and Lomb precision centrifuge which was found to be admirably adapted to the requirements. A pair of special holders, suitable for carrying the bottles, is provided with the machine. The following method was found to work satisfactorily with extracts of lemon, orange, peppermint and nutmeg.

Fifty cc. of the extract are measured into the test bottle, the temperature noted, 1 cc. of hydrochloric acid (1 : 1) added, then water (previously heated to 60° C.) poured in to near the beginning of the stem. The bottle, closely stoppered with a small cork, is allowed to stand in water at 60° C. for about 15 minutes, occasionally giving it a twisting motion to facilitate the separation of the oil, then run 10 minutes in the centrifuge at a speed of about 800 revolutions per minute. A little hot water is added cautiously until the oil rises

above the 150 cc. mark and the bottle again run in the centrifuge 10 minutes. In case the separation does not appear to be satisfactory, or if the liquor is not clear, good results are obtained by cooling the bottle and contents to about 10° C.,

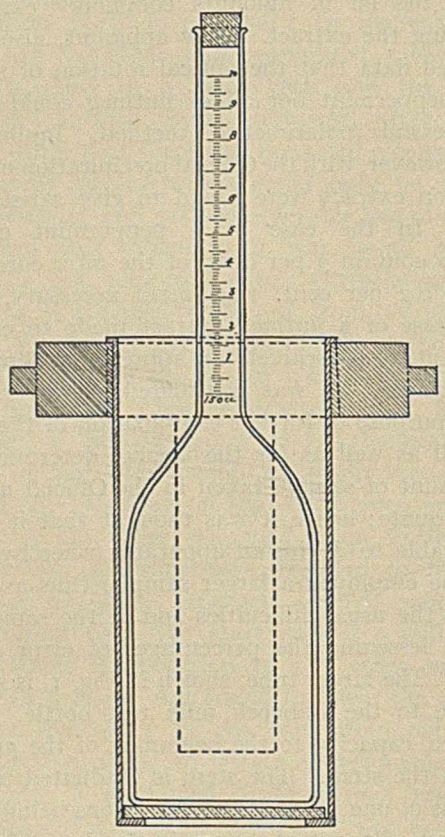

Fig. 1.

then running in the centrifuge an additional 10 minutes. Occasionally a light oil will be found which does not rise completely into the stem of the bottle. In such a case good results are secured by using salt solution (1 : 1), previously heated to 60° C., instead of water. The per cent. of oil is measured by means of a pair of dividers, from the extreme bottom to the top of the column. The oil may then be removed by means of a small pipette and a piece of absorbent paper and the remaining liquor used in the alcohol determination.

Wintergreen Extract.—(*a*) The precipitation method was also found to be applicable to extract of wintergreen, but, owing to the fact that oil of wintergreen has a specific gravity of about 1.18, the tube described in the foregoing method could obviously not be used. A tube was therefore constructed for heavy oils, as shown in Fig. 2. The capacity of the tube is about the same as that of the tube for light oils and the stem is also graduated to tenths of one per cent., based on a 50 cc. sample. This tube may also be fitted by means of a cork cushion into one of the special holders provided with the precision centrifuge. The method is essentially the same as the one given for light oils, omitting the heating in the water-bath previous to running in the centrifuge. At the close of the first 10 minutes' run the tube and contents are cooled to 10° C. and the remainder of the operation is completed as described. The alcohol-water liquor is blown out into a test bottle made for light oils, the residual liquor rinsed out with a little water and the solution set aside for the alcohol determination. The remaining oil is then removed and examined for purity.

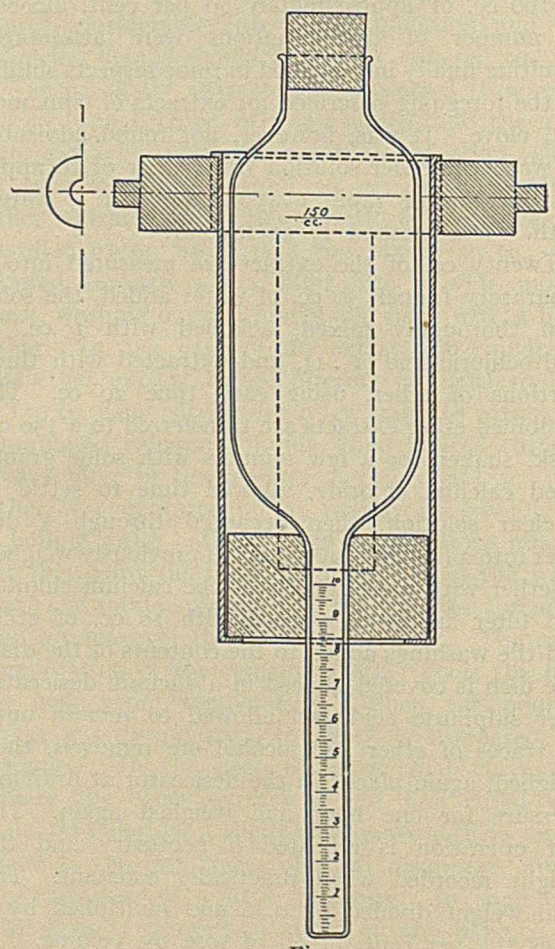

Fig. 2.

(*b*) Methyl salicylate constitutes about 99 per cent. of oil of wintergreen; hence a determination of this constituent in a wintergreen extract would give substantially the amount of oil. The following method was found to give good results.

Ten cc. of the extract are measured into a 100 cc. beaker, 10 cc. of a 10 per cent. solution of potassium hydroxide added, and the mixture heated over a boiling water-bath until the odor of oil of wintergreen has disappeared and the liquor reduced to about one-half its original volume. A 10 per cent. solution of hydrochloric acid is added to distinct excess and the liquor cooled. The salicylic acid is extracted with three portions of ether, using in succession 40, 30 and 20 cc., the combined ether extracts poured through a dry filter into a weighed dish, the filter washed with an additional 10 cc. of ether, and the combined filtrate evaporated slowly at about 50° C. The crystals are dried one hour in a desiccator and weighed.

$$\frac{\text{Weight of salicylic acid} \times 1.101 \times 10}{1.18} = \text{per cent. of oil of wintergreen by volume.}$$

The preliminary work having been completed, three extracts were prepared from each kind of essential oil. One was made to conform to the standard given in Circular No. 19 of the United States Department of Agriculture and contained approximately the per cent. of alcohol required to dissolve the oil, one was made deficient in oil, and one was made to contain oil in excess of the standard and much higher than the necessary per cent. of alcohol. These solutions were then submitted to analyses according to the methods which have been described. The results are shown in Table I.

TABLE I.—RESULTS OF THE DETERMINATION OF VOLATILE OILS IN FLAVORING EXTRACTS.

(Results expressed as per cent. by volume.)

	Composition of extract.		By extraction.		By precipitation.		By chief constituent.	
Kind of extract.	Per cent. alcohol used.	Per cent. oil.	Per cent. oil found.	Per cent. total oil found.	Per cent. oil found.	Per cent. total oil found.	Per cent. oil found.	Per cent. total oil found.
Almond	26.40	0.50	.....	.....	....	.....	0.468	93.60
	26.40	1.00	.....	.....	....	.....	0.937	93.70
	80.12	1.50	.....	.....	....	.....	1.416	94.40
Cinnamon	68.72	1.00	0.982	98.20	....	.....	.....	.....
	68.72	2.00	1.945	97.25	....	.....	.....	.....
	94.92	3.00	2.941	98.03	....	.....	.....	.....
Clove	84.67	1.00	0.991	99.10	....	.....	.....	.....
	84.67	2.00	2.004	100.20	....	.....	.....	.....
	94.92	3.00	3.005	100.17	....	.....	.....	.....
Cassia	68.72	1.00	0.995	99.50	....	.....	.....	.....
	84.67	2.00	1.998	99.80	....	.....	.....	.....
	94.92	3.00	2.962	98.73	....	.....	.....	.....
Lemon	84.67	3.00	.....	.....	2.90	96.67	.....	.....
	94.92	5.00	.....	.....	4.90	98.00	.....	.....
	94.92	7.00	.....	.....	6.80	97.14	.....	.....
Orange	84.67	2.00	.....	.....	2.00	100.00	.....	.....
	94.92	5.00	.....	.....	5.00	100.00	.....	.....
	94.92	7.00	.....	.....	6.85	97.86	.....	.....
Nutmeg	84.67	1.00	.....	.....	0.90	90.00	.....	.....
	84.67	2.00	.....	.....	1.85	92.50	.....	.....
	94.92	4.00	.....	.....	3.75	93.75	.....	.....
Wintergreen	68.72	1.50	.....	.....	1.15	76.67	1.460	97.53
	68.72	3.00	.....	.....	2.65	88.33	2.930	97.67
	94.92	5.00	.....	.....	4.60	92.00	4.836	96.72
Peppermint	84.67	1.00	.....	.....	1.15	76.67	.....	.....
	84.67	3.00	.....	.....	2.78	92.67	.....	.....
	94.92	5.00	.....	.....	4.75	95.00	.....	.....
Rose	80.00	0.40	0.360	90.00	....	.....	.....	.....
	85.00	0.45	0.410	91.11	....	.....	.....	.....

On examination of the table it will be seen that under the best conditions small corrections should be applied to the results in the case of most of the extracts. This is particularly true with extracts of cinnamon, peppermint and nutmeg. However, the errors will be found to be fairly constant, provided that the conditions under which the methods are carried out are kept as uniform as possible. It would be well for each operator to work on one or two known solutions according to each method and in that way determine for himself and for the conditions under which he works just what per cent. of oil he is able to recover. In thus establishing his own factors for the corrections, the personal

equation inherent in each method will be largely eliminated.

The precipitation method described by Howard for the "estimation of oils in flavoring extracts and pharmaceutical preparations" was subjected to a number of preliminary trials on extracts made in this laboratory and was found to give results which in several cases were apparently satisfactory. Further experience, however, disclosed serious defects in the method. This occurred especially with almond extracts, in which ten determinations made strictly according to the prescribed conditions gave at best only a small proportion (never exceeding 0.15 per cent.) of the actual oil present. In some cases the results were nil, the oil having been apparently completely lost during the process. When the method was applied to some of the other extracts the results were more satisfactory, the reading of the oil column giving the exact per cent. of oil in several instances. At times, however, the results were either too high or too low, the variations occurring under seemingly identical conditions. A little consideration led to the conclusion that the method is faulty in several particulars. It seemed incomprehensible that a one-half cubic centimeter of chloroform could be sufficient to remove from a 10 cc. sample of an extract all of the essential oil, even after the oil had been partially precipitated by means of water. Dilution of 10 cc. of a 90 per cent. alcohol to at most 40 cc. (the average capacity of a Babcock bottle) still leaves a solution containing approximately 20 per cent. of alcohol. Particularly in the case of extracts whose oils are fairly soluble in dilute alcohol, a 20 per cent. alcohol solution might be expected to retain a considerable amount of the oil. In arguing the complete extraction of the oil by his method, Howard states: "All the oil is dissolved by the chloroform, while the latter in saturating the water, apparently serves to displace any appreciable trace of oil, otherwise retainable by the alcohol-water mixture." The fact that the water becomes saturated with the chloroform arouses the suspicion that some of the oil may be retained in solution by the chloroform water mixture. During the evaporation of the chloroform the odor of the oil is very easily detected, and at the end of the prescribed one minute period of heating in contact with steam it is seldom that the odor of chloroform is not plainly evident. It is also conceivable that in certain instances a small amount of the oil may be taken up whenever dilute sulphuric acid is used for the purpose of raising the oil into the graduated neck of the bottle. Furthermore, experience has led to the belief that there is a greater loss of volatile oil during the evaporation of the chloroform than might be suspected, particularly in the case of such volatile oils as almond and cinnamon.

In order to decide some of these questions the various portions obtained in the Howard process were assayed separately for the per cent. of essential oil which they might contain. For this purpose such methods were used as have been found to be best suited to the oils in question. The results of these determinations are shown in Table II. Referring to the Table, "A" is the solution which was drawn off with the aspirator from the Babcock bottle after the first run in the centrifuge. After the operation was finished, the contents of the bottle were poured into a small separatory funnel, the oil allowed time to separate and the lower liquor run out as completely as possible and assayed as "B." The bottle was then rinsed out with ether and the rinsings added to the oil in the separatory funnel, constituting portion "C." The results under "D" were obtained by difference and represent the loss of oil during the evaporation of the chloroform. In the determinations made on the almond extracts the amounts of oil obtained were so very small that it became necessary to determine the proportions of oil present in portions "B" and "C" in one operation. In all case where deemed necessary, qualitative tests were applied for the purpose of identifying the oil obtained.

The results shown in the table are significant. While in general the results by the Howard method are apparently correct in many instances, a closer examination will reveal the fact that the actual essential oil obtained seldom approximates to the amount contained in the sample. It is evident that a considerable amount of the oil is retained by the alcohol-water solution, also that a small amount is lost in the solution added to raise the oil into the graduated neck of the test bottle. The fact that in all cases only a portion of the column read off is shown to be essential oil is adequate evidence that various amounts of chloroform are retained in the process and erroneously included in the results. In other words, the method is one involving oppositely occurring errors that may or may not compensate in the results obtained.

The determination of alcohol in flavoring ex-

tracts involves some peculiar difficulties which can best be referred to by giving a description of some of the methods which have heretofore been accepted. The method given by Allen[1] for the determination of alcohol in tinctures, modified so as to apply also in the presence of essential oils, is substantially as follows:

Fifty cc. are made up to 350 cc. by the addition of water, the liquid clarified by the addition of a few drops of a strong solution of calcium chloride followed by some sodium phosphate, made up to 400 cc., filtered through a dry filter, and 250 cc. of the filtrate distilled. The distillate is made up to 250 cc. by the addition of water and the density observed. The corresponding percentage of alcohol is multiplied by 8 to obtain the per cent. of alcohol in the original tincture.

The present Official method[2] involves the same principle, differing only in the amount of dilution and in the use of magnesium carbonate in place of calcium phosphate for removing the precipitated oil. In these methods, as in others which are essentially similar in all respects, the oil is at best only more or less imperfectly removed, a serious defect which has apparently failed to attract general attention. Thorp and Holmes[1] remove the oil by extraction with an immiscible solvent, proceeding as follows:

Twenty-five cc. of the sample are taken, 100 cc. of water added, the mixture saturated with common salt and shaken for 5 minutes with 70 cc. of petroleum ether (b. p. under 60° C.). The aqueous layer is then drawn off and the petroleum ether washed twice with 25 cc. of saturated salt solution. These washings are added to the original aqueous solution and the whole distilled slowly until 100 cc. of distillate is obtained. The alcoholic content of the distillate is multiplied by four to obtain the per cent. of alcohol in the extract.

TABLE II.—RESULTS OF THE EXAMINATION OF HOWARD'S METHOD.

(Results expressed by volume.)

Kind of extract.	Composition of extract.		Per cent. oil by Howard's method.	Assay of "A."		Assay of "B."		Assay of "C."				"D."	
	Per cent. alcohol.	Per cent oil.		Oil found.	Per cent. total oil found.	Oil found.	Per cent. total oil found.	Oil found.	Per cent. total oil found.	Per cent. oil in column.	Per cent chloroform in column.	Oil.	Per cent. total oil.
Almond.....	26.14	1.00	0.15	0.0278	27.80	.....	.....	0.0596	59.60	.....	.....	0.0126	12.60
	78.92	1.50	0.15	0.0397	26.47	.....	.....	0.0795	53.00	.....	.....	0.0308	20.53
Cassia.....	68.03	1.00	1.20	0.0153	15.30	0.0209	20.90	0.0632	63.20	52.67	47.33	0.0006	0.60
	82.98	2.00	2.00	0.0322	16.10	0.0238	11.90	0.1380	69.00	69.00	31.00	0.0060	3.00
	92.07	3.00	2.40	0.0571	19.03	0.0306	10.20	0.1992	66.40	83.00	17.00	0.0131	4.37
Cinnamon.....	68.03	1.00	1.20	0.0136	13.60	0.0262	26.20	0.0491	49.10	40.92	59.08	0.0111	11.10
	67.34	2.00	2.40	0.0255	12.75	0.0524	26.20	0.1035	51.75	43.15	56.85	0.0186	9.30
	92.07	3.00	2.80	0.0737	24.57	0.0379	12.63	0.1749	58.30	62.48	37.52	0.0135	4.50
Clove.....	83.82	1.00	1.10	0.0162	16.20	0.0095	9.50	0.0670	67.00	60.91	39.09	0.0073	7.30
	82.98	2.00	2.00	0.0377	18.85	0.0057	2.85	0.1409	70.45	70.45	29.55	0.0157	7.85
	92.07	3.00	2.70	0.0699	23.30	0.0115	3.83	0.2075	69.17	76.85	23.15	0.0111	3.70
Lemon.....	82.13	3.00	3.40	0.0133	4.44	0.0020	0.67	0.2406	80.20	70.76	29.24	0.0441	14.69
	90.17	5.00	4.80	0.0253	5.06	0.0023	0.46	0.4434	88.68	92.38	7.62	0.0290	5.80
	88.28	7.00	5.60	0.0100	1.43	0.0011	0.16	0.6683	95.47	92.82	7.18	0.0206	2.94
Orange.....	82.98	2.00	2.00	0.0110	5.50	0.0021	1.05	0.1548	77.40	77.40	22.60	0.0321	16.05
	90.17	5.00	4.80	0.0453	9.06	0.0063	1.26	0.3995	79.90	83.23	16.77	0.0489	9.78
	88.28	7.00	5.60	0.1040	14.86	0.0047	0.67	0.5145	73.50	91.88	8.12	0.0768	10.97
Wintergreen...	90.17	5.00	5.20	0.0030	0.60	0.0360	7.20	0.4439	88.78	85.36	14.64	0.0171	3.42
Nutmeg.....	83.82	1.00	0.80	0.0195	19.50	0.0102	10.20	0.0536	53.60	67.00	33.00	0.0167	16.70
	91.12	4.00	3.60	0.0479	11.98	0.0187	4.67	0.3204	80.10	89.00	11.00	0.0130	3.25

NOTE.—Two extracts of wintergreen, one of nutmeg and the three peppermint extracts made in the laboratory were exhausted before this investigation of Howard's method was begun.

In all of these methods the fact is apparently ignored that in every case the volume that was originally occupied by the essential oil in the aliquot portion taken for the specific gravity determination is replaced by the alcohol-water mixture, thus making the results for alcohol too high. In other words, a true aliquot portion of the sample after removal of the oil is never taken. The error is increased in direct proportion to the amount of alcohol in the extract. Winton and Ogden[2] recognize the latter fault in the Official method in their modification which is as follows:

[1] "Commercial Organic Analysis," 3rd Ed., Vol. I, p. 162.
[2] Bull. 107, U. S. Dept. of Agr., Bureau of Chemistry, p. 160.

[1] *Jour. Soc. Chem. Ind.*, **22**, 232 (1903).
[2] Report Conn. Agr. Expt. Sta., 1901, Part II, p. 172.

Thirty grams of the sample are diluted with water to 200 cc., the mixture poured into a dry Erlenmeyer flask containing 3.5 grams of light carbonate of magnesia, then well shaken and filtered quickly through a large dry plaited filter. The alcohol is determined in 150 cc. of the filtrate, the per cent. by weight in the extract being calculated by the following formula:

$$x = \frac{100ab\,(200 - cd/86)}{150c}, \text{ in which}$$

x = per cent. of alcohol by weight in the extract.
a = weight of the distillate.
b = per cent. of alcohol by weight in the distillate.
c = weight of the extract.
d = per cent. of oil by weight in the extract.

T. H. Harvey[1] also realized the need of a modification of the method and proposed the following:

TABLE III. RESULTS OF THE DETERMINATION OF ALCOHOL IN FLAVORING EXTRACTS BY THE PRESENT OFFICIAL METHOD FOR LEMON EXTRACTS.

(Results expressed as per cent. by volume.)

Kind of extract.	Composition of extract. Oil.	Composition of extract. Alcohol.	Alcohol found.	Per cent. of total oil in distillate.
Almond	0.50	26.27	26.30	...
	1.00	26.14	25.36	98.20
	1.50	78.92	78.94	99.33
Cinnamon	1.00	68.03	68.32	66.72
	2.00	67.34	67.20	45.77
	3.00	92.07	92.40	43.60
Clove	1.00	83.82	84.36	63.72
	2.00	82.98	83.96	49.23
	3.00	92.07	92.04	43.06
Cassia	1.00	68.03	68.32	64.74
	2.00	82.98	82.44	43.89
	3.00	92.07	92.04	39.10
Lemon	3.00	82.13	83.20	6.55
	5.00	90.17	91.68	3.58
	7.00	88.28	91.68	1.55
Orange	2.00	82.98	83.96	4.26
	5.00	90.17	91.68	3.08
	7.00	88.28	91.68	4.35
Peppermint	1.50	83.40	83.20	...
	3.00	82.13	82.44	...
	5.00	90.17	90.56	...
Wintergreen	1.50	67.69	67.70	21.40
	3.00	66.66	67.52	12.00
	5.00	90.17	90.56	9.24
Nutmeg	1.00	83.82	83.56	27.08
	2.00	82.98	82.84	20.15
	4.00	91.12	92.04	6.16

The extract is diluted to not more than three times its original volume, the liquor distilled and the distillate treated with about 0.5 gram of previously washed and dried kaolin to remove the undissolved oil. The specific gravity of the distillate is determined, and from the corresponding alcohol found the per cent. of alcohol in the sample is calculated.

In attempting to apply these methods to extracts in general, the fact should not be overlooked that dilution and filtering with magnesia or kaolin fails in many instances to effect the complete removal of the oil. It has been found in the case of extracts prepared in the laboratory that after diluting from 50 cc. to 200 cc. and filtering through magnesia, a clear solution was obtained, but, particularly in the case of oils which are more readily soluble in dilute alcohol, the distillate is cloudy when the first 60 or 70 cc. have passed over. On cooling the distillate to 15.6° C. for the specific gravity determination, a small amount of oil separates from the milky liquid. In order to determine the amount of oil carried over in the distillation, an assay of essential oil was made in each of the distillates obtained in applying the Official method for lemon extract to the extracts prepared in the laboratory. The results are shown in Table III.

In an extract composed only of alcohol, essential oil and water, the problem of determining the alcohol content may not present any serious difficulties. In such a case the determination of the oil and the specific gravity would afford sufficient data for the exact calculation of the per cent. of alcohol, as in the following:

Let S represent the specific gravity of the extract at 15.6° C., O, the specific gravity of the oil, and p the per cent. of oil found. Then $100 - p$ will be the per cent. of the water-alcohol solution, the specific gravity of which, represented by P, is calculated as follows:

$$S = \frac{Op + P(100 - p)}{100}, \text{ whence } P = \frac{100S - Op}{100 - p}.$$

The value of E, the alcohol equivalent of P, is obtained by means of an alcohol table and gives the per cent. of alcohol in the alcohol-water solution. To find the per cent. of alcohol in the extract, it is necessary to apply the following formula:

Per cent. by volume of alcohol in the extract =

$$E\left(1 - \frac{p}{100}\right).$$

In extracts containing other substances such as glycerine and sugar, recourse may be had to distillation in order to obtain a mixture consisting only of alcohol, water and essential oil. Having made the distillate up to the volume of the sample

[1] *Chemist and Druggist*, 1904, p. 178.

of extract taken, the per cent. of oil and the specific gravity are determined and the alcohol content of the original extract obtained according to the above formula. There may, however, be some hesitation in accepting a method which requires a distillation and a second determination of essential oil, since for ordinary purposes in routine work the per cent. of alcohol can safely be derived from the specific gravity of the extract, providing the character of the sample be ascertained by a qualitative examination of the solids. In all cases where the oil can be separated by means of the centrifuge the alcohol may be determined as follows:

The alcohol-water liquor, freed as completely as possible from essential oil, is made up to 150 cc. in the test bottle for light oils and the temperature noted. In case the liquor happens to measure beyond the 150 cc. mark, the exact volume is read off on the stem. The liquor is poured into a distilling flask, the residue washed into the flask with about 30 cc. of water, the mixture distilled into the test bottle to near 150 cc. and the distillate made up to the original volume at the temperature previously noted. After thorough mixing, the liquor is cooled to 15.6° C., the specific gravity determined and the alcohol equivalent found. The result multiplied by the factor obtained by dividing the exact volume of the distillate by 50 gives the per cent. of alcohol in the original extract.

The extracts prepared in the laboratory were finally subjected to alcohol determinations, by the Official provisional method, by the method based on the determination of the specific gravity of the extract, by the application of the formulae given above and by the method applicable after the centrifugal separation of the essential oil. The results are shown in Table IV.

TABLE IV. RESULTS OF THE DETERMINATION OF ALCOHOL BY VARIOUS METHODS.

(Results expressed as per cent. by volume.)

Kind of extract.	Alcohol in the extract. Strength used for solution of the oil.	Per cent. by calculation.	Per cent. by provisional method.	Per cent. by specific gravity without distillation.	Per cent. by calculation from specific gravity by formulae.	Per cent. by centrifugal method.
Almond	26.40	26.27	26.30	25.67	25.91	...
	26.40	26.14	25.36	25.27	25.78	...
	80.12	78.92	78.94	79.00	78.88	...
Cinnamon	68.72	68.03	68.32	68.09	68.03	...
	68.72	67.34	67.20	67.61	67.45	...
	94.92	92.07	92.40	93.17	92.13	...
Clove	84.67	83.82	84.36	83.94	83.75	...
	84.67	82.98	83.96	83.25	82.91	...
	94.92	92.07	92.04	93.00	92.02	...
Cassia	68.72	68.03	68.32	68.05	67.99	...
	84.67	82.98	82.44	83.28	82.95	...
	94.92	92.07	92.04	98.86	91.85	...
Lemon	84.67	82.13	83.20	85.00	82.55	82.26
	94.92	90.17	91.68	94.66	90.47	89.69
	94.92	88.28	91.68	94.48	88.65	88.42
Orange	84.67	82.98	83.96	84.97	83.27	83.06
	94.92	90.17	91.68	94.61	90.21	90.11
	94.92	88.28	91.68	94.46	88.45	88.55
Peppermint	84.67	83.40	83.20	84.41	83.47	83.58
	84.67	82.13	82.44	84.18	82.23	82.20
	94.92	90.17	90.56	93.67	90.13	89.58
Nutmeg	84.67	83.82	83.56	84.60	83.89	83.85
	84.67	82.98	82.84	84.54	83.10	82.20
	94.92	91.12	92.04	94.15	91.12	91.29
Wintergreen	68.72	67.69	67.70	66.86	67.57	6[illegible].31
	68.72	66.66	67.52	65.29	66.80	.97
	94.92	90.17	90.56	89.52	89.93	[illegible].01

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THE DETERIORATION AND COMMERCIAL PRESERVATION OF FLESH FOODS. SECOND PAPER—THE STORAGE OF BEEF AT TEMPERATURES ABOVE THE FREEZING POINT.

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In a previous paper[1] we developed certain methods of analysis adapted to the detection of degradation changes in proteins and applied them to the question of determining whether or not changes of a chemical nature occur in leaf beef stored for considerable periods of time in the frozen condition, or in what is commonly called "cold storage" beef. The average of a number of analyses of fresh beef compared with a similar average in the case of frozen beef showed results so nearly identical that the conclusion was reached that no changes of a chemical nature occur in lean beef at temperatures below —9° C. for a period of 554 days and probably for a longer time; that is, changes which can be detected by the methods employed. It should be stated that the samples of beef used in the previous experiments when properly stored for over two years gave no indications to the senses of smell and taste of deterioration.

It remains to be demonstrated that the methods employed in the previous investigation were suitable to the purpose in hand; that is, that they were capable of detecting changes of a chemical

[1] *J. Am. Chem. Soc.*, **30**, 1515-64.

or biochemical kind which might occur in flesh foods under the influence of water, salts, acids, and such agencies as enzymes and bacteria. The way to determine the suitability of the methods would be to store meats under conditions wherein obvious changes ensue and to make analyses of samples at intervals. The purpose of the present paper is first to show that the methods are capable of detecting deterioration and second to follow by means of them the changes which occur when beef is stored in ordinary cooler temperature; that is, a few degrees above the freezing point—say 2–4° C. These temperatures—just above the freezing point—are the ones used by the large packing establishments for storing fresh meats, for shipping them in refrigerator cars, and for curing salt meats. In the butcher-shop and the home where meats are kept in ice-cooled refrigerators there is an approximation to the same temperatures, but for the most part in such storage the temperatures are higher and consequently deterioration proceeds more rapidly.

A sharp distinction must be made between the temperatures now being considered and those which were discussed in the first paper on frozen meats (—9 to —12° C.), for although with progressive lowering of the temperature the conditions for bacterial multiplication and chemical activity become steadily less, still down to the freezing point of water and many degrees below, suitable forms of bacteria develop and multiply very well in suitable media, provided only that the media are liquid. It is the solid condition which limits bacterial growth absolutely. In solutions which, because of the concentration of salts or other substances not decidedly antiseptic in nature, do not freeze at 0° C., suitable bacteria are capable of development at as low a temperature as —20° C. and possibly lower. Yet in solid ice at 0° to —1° C. they have no power of growth, although in the same medium, liquid, at 0° to +1° C. they would grow readily. Thus in frozen meats below —9° C. bacteria do not develop and cannot penetrate from the surface, while they grow readily in liquids at this temperature or below. Not that the juice of meat is solid at —9° C., but the solution which remains liquid is too concentrated and viscous to permit bacterial multiplication; and further, this solution in frozen meats remains at segregated points (mostly within the contracted muscle fibres) which are isolated by solid barriers of ice.

There is no doubt that the bacterial flora which causes the deterioration of meat (for this is mainly a bacterial process) at temperatures near the freezing point differs more or less from that which produces similar changes at higher temperatures. This is a subject which has not yet been accorded the thoroughgoing investigation which it deserves (because of its great importance from the hygienic as well as the economic standpoint) but each year adds some data to that available. The demonstration of the general principle stated, namely that a characteristic bacterial flora excites the deterioration of meats at temperatures near the freezing point, particularly in large establishments whose storage rooms are maintained at fixed temperatures for years, is not difficult. But on the other hand the work of developing a technique for the proper cultivation and study of these forms (in the case of salt meats as well as fresh) has not yet been carried out.

A number of experiments to try out the methods of analysis discussed in the first paper have been made during the past four years. Of these a few will be selected for presentation here. Those in which the tannic-acid-salt method of Bigelow and Cook for determining proteose nitrogen were used, will not be reported since the results obtained are too irregular and inconsistent. In place of this method the zinc sulphate precipitation has been adopted in all cases, although this determination (of proteose nitrogen) has not proved to be so important as some of the others.

Turning to the experiments, Table I shows the analytical results obtained by applying the methods referred to above, to meat undergoing bacterial decomposition at room temperature in summer, under the following conditions: A quantity of the lean meat from beef knuckles was hashed and well mixed. 100-gram portions were then weighed into liter flasks and 250 cc. of distilled water and 1 cc. of a putrefying meat infusion added.

The flasks were loosely stoppered and allowed to stand for different lengths of time (1 to 26 days) whereupon the samples were extracted with water, the extract made up to 1000 cc. and the analyses conducted.

The total solids show no essential deviation from the normal in one day, but thereafter an increase is indicated up to a maximum at five days and afterwards a decline.

The ash figures are approximately constant

except at the last, when a slight rise is shown. This increase has been observed in other cases but the determination is so liable to error that we are in doubt whether to ascribe the increase to the error of method or to actual augmentation during the decomposition.

The total nitrogen figures increase consistently and steadily from 0 to 26 days, beginning with a minimum of 0.819 per cent. and ending with a maximum of 2.744 per cent.

With some variations and irregularities the coagulable nitrogen reaches a maximum of 0.631 per cent. at 5 days and then drops to a point (0.206 per cent.) which is nearly one-half the normal amount.

The albumose nitrogen figures starting at near the normal reach a maximum in 5 days of about 9 times the normal and then fall off to three times the normal.

simpler forms. The insoluble tissues at first apparently become soluble and coagulable; or the containing membranes become disintegrated and allow their soluble contents to pass into solution in water. The albumoses precipitated by zinc sulphate may rise somewhat but only transiently while the main tendency lies in the direction of the formation of simple soluble basic substances volatile with steam and consisting principally of ammonia.

A series of experiments, which are, however, not altogether satisfactory, were carried out on hashed lean meat from beef knuckles, mixed with various preservative substances and placed in closed Mason jars at a temperature of 2° to 4° C. Chronologically these experiments preceded that just described, but logically they follow it. The results are shown in Table II. With the exception of a few irregularities, the tendency of the sub-

TABLE I.

PUTREFACTION TEST ON HASHED BEEF KNUCKLE

100 grams meat, 250 cc. distilled water and 1 cc. putrefying meat infusion held at room temperature 21–33° C. All figures on basis of meat. For methods used, see *J. Am. Chem. Soc.*, **30**, 1527-30.

Date.	Age, days.	Moisture, per cent.	Ash, per cent.	Fat, per cent.	Total N, per cent.	Amm. N (I), per cent.	Amm. N (II), per cent.
6/3/08	0 (analysis meat)....	74.84	1.25	2.52	3.57	0.034	0.010

Cold water extract.

		Solids, per cent.	Ash, per cent.	Total N, per cent.	Coag. N, per cent.	Alb. N, per cent.	Meat base N, per cent.	Acidity, per cent. lactic.
6/3/08	0.....	6.11	1.25	0.819	0.417	0.027	0.370	0.83
6/4/08	1.....	5.93	1.19	0.840	0.453	.....	.....	0.83
6/5/08	2.....	6.96	1.19	1.078	0.549	0.085	0.034	0.60
6/6/08	3.....	7.14	1.29	1.428	0.476	0.143	0.809	0.57
6/8/08	5.....	9.36	1.31	2.093	0.631	0.196	1.166	0.47
6/12/08	9.....	8.86	1.33	2.121	0.527	0.137	1.425	0.75
6/29/08	26.....	6.64	1.31	2.744	0.206	0.087	2.506	1.40

Ammoniacal N (Method I) in extract at 8 days, 1.135 per cent.; 26 days, 2.055 per cent. Alkalinity to litmus 5 days equivalent to 12 per cent. N/10 acid; 26 days, equivalent to 26 per cent. N/1 acid.

The meat base nitrogen like the total nitrogen shows a continuous steady rise from 0.370 per cent. in the fresh sample to 2.506 per cent. in the oldest.

The ammoniacal nitrogen also increases steadily so that at the end about three-fourths the total nitrogen is in a form which can be distilled with water in the presence of a mild alkali, caught in acid and titrated.

The acidity figures, on account of the presence of weak bases and weak acids, do not afford much information. With an indicator sensitive to weak bases the extract at the last is strongly alkaline; with one sensitive to weak acids it is noticeably acid.

In general the application of the methods to the extract from putrefying meat shows a continuous transformation of the nitrogenous compounds into

stances added (salt, saltpeter and boric acid) to arrest decomposition is well shown. It might be thought that the differences between unpreserved and preserved samples would be greater, but it should be remembered that the greatest influence against the growth of microörganisms in the experiment was the low temperature of storage. Small quantities of preservative substances are not to be compared with low temperatures as effective means of preservation of meats. Combined with low temperatures, they are very effective.

The next series of analyses shown in Table III indicates the changes which occur in pieces of firm lean beef stored at 2°–4° C. for considerable periods of time. This is the ordinary temperature used in storing fresh meats (and in shipping them)

in large establishments. The carcasses of the animals immediately after slaughter are run into the chill rooms where the temperature is brought down as quickly as possible from about 38° C. to near the freezing point. In some chill rooms the open brine system is used and in others the closed system. In the former system the circulating brine, previously chilled in the refrigerating plant, is allowed to overflow a notched trough or perforated pipe and to drizzle down sheets of cloth in the rooms to collecting troughs on the floor. In the closed system the cold brine merely circulates through overhead pipes. In either case, but chiefly in the open system, there is a considerable desiccating action exerted by the cold brine on the meats. There is, since the meats are warmer than the brine—and also, in the open system, since the vapor pressure of the brine is much less than that of the moisture in the meat at the same temperature, a continuous distillation of water vapor from the surface of the meat to the cold brine or brine pipes. This has the effect of desiccating and hardening to a certain extent the surface of the meat and thus making it more resistant to bacterial invasion. This surface desiccation is analogous in its results to the effects of the dry hard coverings enclosing natural fruits and other products and serving to protect them from decay.

It is true that even after the surfaces of meats have become more or less dry in the chill room as described above, if the air becomes saturated with moisture or nearly so, the meat surface will again absorb moisture, swell and soften and a

TABLE II.

Progress of decomposition in hashed beef knuckles with various preservative substances added. Storage 2-4° C. added.

		Determined by analysis.								Cold water extract.							
Test No.	Age, days.	Salt, per cent.	Saltpeter, per cent.	Boric acid, per cent.	Moisture, per cent.	Ash, per cent.	Fat, per cent.	Total N, per cent.	Amm. N, per cent.	Solids, per cent.	Ash, per cent.	Total N, per cent.	Coag. N, per cent.	Alb. N, per cent.	Meat base N, per cent.	Acidity, per cent. lactic.	
1	14	..	..	..	75.57	1.30	2.30	3.34	0.049	5.82	1.03	0.782	0.378	0.053	0.362	0.83	..
1	30	..	..	..	75.14	1.33	2.35	3.30	0.060	5.96	1.10	0.800	0.320	0.077	0.430	0.74	..
1	51	..	..	..	74.00	1.29	2.33	3.31	0.066	6.28	1.06	0.841	0.327	0.019	0.430	0.79	..
1	92	..	..	..	76.29	1.39	2.20	3.28	0.078	6.47	1.05	0.952	0.341	0.096	0.523	0.84	..
1	400	..	..	..	75.74	1.30	2.25	3.31	0.235	7.90	1.08	1.104	0.396	0.086	0.673	0.86	..
2	14	..	..	0.20	74.82	..	..	3.36	0.036	5.74	1.09	0.759	0.358	0.036	0.372	0.81	..
2	30	..	..	0.20	75.06	1.48	2.31	3.42	0.038	5.87	1.00	0.779	0.361	0.067	0.378	0.82	..
2	51	..	..	0.20	74.12	1.45	2.37	3.32	0.061	6.35	1.18	0.841	0.345	0.085	0.418	0.86	..
2	94	..	..	0.20	74.75	1.37	2.43	3.50	0.062	6.23	1.07	0.893	0.358	0.089	0.459	0.74	..
2	400	..	..	0.20	75.53	1.57	2.25	3.46	0.194	7.33	1.10	1.131	0.392	0.101	0.581	1.07	..
3	14	4.70	..	..	71.53	6.01	2.00	3.26	0.028	10.38	5.45	0.759	0.355	0.054	0.352	0.74	..
3	30	4.70	..	..	71.74	5.87	1.93	3.12	0.057	10.60	5.49	0.786	0.348	0.075	0.358	0.79	..
3	60	4.70	..	..	70.14	5.81	2.17	3.20	0.050	10.79	5.62	0.855	0.334	0.097	0.414	0.77	..
3	97	4.70	..	..	69.86	6.08	2.03	3.20	0.063	10.58	5.63	0.859	0.310	0.106	0.456	0.69	..
3	405	4.70	..	..	71.29	5.95	2.01	3.26	0.174	12.79	5.76	1.219	0.438	0.106	0.722	0.77	..
																	KNO_2
4	14	4.80	0.21	..	71.01	6.18	2.14	3.17	0.034	10.29	5.51	0.779	0.366	0.055	0.362	0.71	0.00
4	30	4.80	0.20	..	71.36	6.18	2.00	3.19	0.051	10.71	5.61	...	0.333	0.081	0.400	0.74	0.00
4	60	4.80	0.20	..	71.25	6.24	2.12	3.16	0.050	10.84	5.74	0.835	0.300	0.102	0.466	0.72	0.0035
4	99	4.80	0.17	..	71.33	6.05	2.14	3.14	0.059	10.76	5.62	0.897	0.284	0.168	0.463	0.61	0.0035
4	146	4.80	0.15	..	72.32	6.18	2.12	3.09	0.083	10.80	5.63	0.924	0.264	0.142	0.485	0.81	0.0026
4	405	4.80	0.003	..	74.30	6.18	2.10	3.14	0.193	11.82	5.93	1.310	0.456	0.124	0.720	0.48	trace
5	14	4.80	0.44	..	70.60	6.22	2.00	3.19	0.029	10.82	5.87	0.762	0.354	0.050	0.367	0.74	0.0012
5	30	4.80	0.44	..	70.04	6.24	1.98	3.06	0.050	10.65	5.60	0.755	0.313	0.091	0.400	0.73	0.00
5	69	4.80	0.39	..	71.67	6.21	2.21	3.16	0.053	10.95	5.90	0.828	0.280	0.133	0.399	0.61	0.0025
5	105	4.80	0.36	..	72.06	6.31	2.14	3.15	0.062	11.28	5.87	0.876	0.278	0.117	0.504	0.65	0.0026
5	181	4.80	0.37	..	72.34	6.18	1.91	3.19	0.099	11.61	5.90	0.913	0.256	0.093	0.560	0.75	0.0036
5	410	4.80	0.26	..	71.56	6.23	2.00	3.19	0.120	12.45	6.21	1.035	0.238	0.147	0.651	0.78	present
6	14	4.72	0.23	0.20	70.31	6.30	2.00	3.25	0.030	10.11	5.52	0.741	0.319	0.044	0.348	0.70	0.00
6	30	4.72	0.21	0.20	70.80	6.34	1.98	3.23	0.037	10.63	5.72	0.779	0.321	0.094	0.369	0.79	0.00
6	69	4.72	0.20	0.20	71.37	6.40	2.13	3.15	0.051	10.65	5.65	0.831	0.303	0.107	0.411	0.61	0.0055
6	111	4.72	0.19	0.20	71.92	6.34	1.91	3.13	0.053	10.66	5.56	0.869	0.289	0.105	0.474	0.74	0.0007
6	184	4.72	0.16	0.20	71.13	6.30	2.05	3.18	0.073	10.58	5.86	0.840	0.299	0.126	...	0.68	0.0009
6	410	4.72	0.13	0.20	71.90	6.32	2.05	3.17	0.107	11.18	5.92	1.004	0.250	0.136	0.615	0.83	present
7	14	..	..	0.45	74.82	1.70	2.24	3.43	0.040	6.12	1.38	0.765	0.357	0.054	0.352	0.81	0.0013
7	30	..	..	0.45	75.54	1.65	2.25	3.30	0.052	6.38	1.47	0.772	0.294	0.090	0.373	0.81	none
7	69	..	..	0.41	74.27	1.64	2.23	3.40	0.068	6.61	1.45	0.845	0.313	0.102	0.425	0.56	0.0055
7	110	..	..	0.40	75.27	1.59	2.23	3.33	0.073	7.51	1.46	0.931	0.307	0.090	0.544	0.78	trace
7	183	..	..	0.33	76.18	1.55	2.13	3.31	0.091	7.40	1.42	0.930	0.302	0.104	0.524	0.63	0.0052
7	410	..	..	0.20	75.52	1.64	2.10	3.34	0.123	7.68	1.41	1.053	0.281	0.121	0.622	0.76	present

"slime" consisting of bacteria will develop and in time form a fairly thick layer on the surface of the meat. In a well ventilated, dry chill room this will not occur, even after decomposition has commenced.

Starting with recently slaughtered meat free from bacteria there are two ways by which micro-organisms enter the flesh and excite decomposition: first, by direct extension of surface colonies into the firm tissue; second, by extension and locomotion along surfaces such as those afforded by blood and lymph vessels, and nerve and connective tissue sheaths.

By the first method, penetration is slow and very regular. The progress of the extension of the innumerable bacteria from the surface toward the interior can be easily observed. If a section be cut across a lean piece of beef which has been stored in the cooler for about 20 days or more, a dark narrow sharply defined border can be seen extending all around the cut. The line separating this border from the interior marks the limit of bacterial penetration by the first method. It will readily be seen that motility can not play a great part in this process; rather the advance occurs by the actual extension of the limits of those confluent colonies which have spread over the surface and later penetrated the meat. It will also be evident that the harder and drier the surface the less readily will penetration occur at the start, although after the surface layer is pierced progress will be easier. Hence the ad-

TABLE III.

Progress of decomposition in beef at 2 ° to 4 ° C. Beef knuckles held at 2° to 4° C. and analyzed from time to time. Killed 3/2/08; cut 3/5/08. All figures on basis of meat. For methods used see *J. Am. Chem. Soc.*, **30**, 1527-30.

									Cold water extract.							
Lab. No.	Rec'd.	Age, days.	Moisture, per cent.	Ash, per cent.	Fat, per cent.	Total N, per cent.	Amm. N, Method I, per cent.	Amm. N, Method II, per cent.	Total solids, per cent.	Ash, per cent.	Total N, per cent.	Coag. N, per cent.	Alb. N, per cent.	Meat base N, per cent.	Acidity, per cent. lactic.	Remarks.
Av. anal. thirteen fresh beef knuckles			76.35	1.23	1.43	3.49	0.029	0.010	6.01	1.14	0.806	0.413	0.024	0.355	0.68	Average of 13 samples
2326	3/9/08	7	77.04	1.28	1.06	3.43	0.029	0.010	5.94	1.25	0.840	0.423	0.019	0.378	0.65	Meat sweet, venous color
2545	3/16/08	14	76.47	1.25	1.14	3.71	0.031	0.015	6.32	1.14	0.931	0.494	0.025	0.406	0.74	Ditto, surface slightly slimy
2761	3/23/08	21	76.87	1.20	1.44	3.62	0.030	0.015	5.67	1.14	0.791	0.362	0.028	0.378	0.77	Exterior shows distinct layer. Interior dull, turning purplish
2762	3/23/08	21	77.55	1.18	1.45	3.55	0.029	0.015	5.68	1.10	0.763	0.368	0.020	0.364	0.72	Sliminess increasing; odor of interior not bad
3182	4/2/08	31	77.46	1.15	1.94	3.42	0.065	0.017	5.59	0.93	0.805	0.381	0.038	0.374	0.61	Exterior layer increasing (10 mm.). Interior color dull as above. Odor interior not bad
3415	4/10/08	39	78.15	1.18	0.90	3.34	0.049	0.025	6.12	1.14	0.854	0.463	0.034	0.350	0.60	Exterior layer 15 mm. thick. Interior color brighter red.
Same, putrefying exterior portion							0.100	0.047								
3653	4/17/08	46	76.99	1.17	1.19	3.48	0.059	0.031	5.86	1.04	0.847	0.427	0.036	0.405	0.59	Very slimy and beginning to mould. Interior bright red. Bacteria localized throughout
Same, putrefying exterior portion							0.108	0.051								
3877	4/24/08	53	77.27	1.11	1.47	3.45	0.061	0.034	6.00	1.09	0.886	0.431	0.039	0.407	0.83	Ditto, but bacteria generally present throughout
4310	5/6/08	65	78.30	1.18	0.60	3.44	0.096	0.033	7.31	1.11	1.108	0.681	0.085	0.328	0.62	Ditto, Color duller but still red
5413	6/10/08	100	77.71	1.18	1.13	3.31	0.171	0.104	6.23	1.15	0.968	0.505	0.049	0.414	0.61	Ditto
8464	10/1/08	121	78.33	1.29	0.86	3.25	0.478	0.274	7.14	1.23	1.240	0.483	0.075	0.474	0.28	Ditto
Effect of freezing on putrefying beef: on 4/25/08 samples from the above lot were frozen and placed in closed tinned pails; later they were analyzed at intervals; for comparison the analysis of sample No. 3877 is repeated here.																
3877	4/24/08	53	77.27	1.11	1.47	3.45	0.061	0.034	6.00	1.09	0.886	0.431	0.039	0.407	0.83	
7101	8/12/08	132	77.30	1.19	1.38	3.41	0.071	0.036	6.69	1.14	0.987	0.488	0.042	0.474	0.47	
9107	10/22/08	203	77.31	1.15	1.87	3.37	0.098	0.039	5.96	1.01	0.883	0.470	0.036	0.378	0.52	
9108	10/22/08	203	77.79	1.19	1.09	3.35	0.094	0.032	7.33[1]	1.03	1.123[1]	0.728[1]	0.054	0.327	0.43	
10538	12/3/08	245	78.91	1.12	1.30	3.20	0.091	0.041	6.64	1.00	0.974	0.506	0.055	0.413	0.39	
10539	12/3/08	245	78.11	1.17	0.89	3.34	0.088	0.041	6.41	1.04	0.916	0.479	0.060	0.348	0.44	

[1] For some unknown reason these figures appear to be abnormal.

vantage of a certain amount of desiccation in the cooler in the preservation of meats. Again the connective tissue which surrounds muscles and with which the ordinary large cuts of beef, mutton and pork are more or less covered is especially difficult of penetration by bacteria when it is dry. In this condition it resembles parchment.

The width of the bacterial border shown in section increases with the time of storage. Under favorable conditions at 2°–4° C. it will be about 1 cm. in 30 days; under unfavorable conditions (good desiccation) it may be only two or three millimeters in the same lengths of time. Fresh beef shipped abroad from Chicago reaches the ports of England three or four weeks after the death of the animal. It is needless to say that it arrives in a satisfactory condition of preservation.

It is characteristic of this method of bacterial penetration that the line of demarcation between the decomposing surface and undecomposed interior remains always sharp and distinct like the line marking the desiccation of the surface of frozen meats or soap.

The second method of penetration wherein bacteria follow various moist surfaces and tubes including arteries, veins, nerve sheaths, lymph vessels and connective tissue, allows them to reach at a relatively early period the interior of the meat, where they set up foci of decomposition which spread and finally merge with each other and the surface decomposition until the meat contains bacteria throughout and is in an active state of putrefaction. The extension of the interior foci can be roughly followed by making smear or impression preparation from various places on the surface of the sectioned meat, on cover glasses or slides. In the earlier stages of decomposition, infected and uninfected areas will be found side by side, the infected spots will be few and small, the untainted areas large. Gradually the former increase in number and size, the latter diminish until with the encroachment of the surface layer of bacteria no uncontaminated spot remains.

So far as the external surface is concerned if the air be dry no slime will form. If the air (and hence the surface of the meat) is moist a thick layer of slime will form and later an abundant growth of mould will appear. The slime consists of quantities of bacteria.

In the experiments about to be described the air was moist, thus preventing the formation of a dry surface layer and favoring decomposition and the development of slime and mould.

After what has been said it will be seen that the method of sampling of meat which is undergoing decomposition has an important influence on the analytical results. In general, the surface layer should be separated from the sharply differentiated interior, and analyzed separately. If slime or mould has formed on the surface, it should be washed, wiped or scraped off. In the early stages the decomposing surface layer is so thin that it is difficult of separation, but later the separation is easier.

In examining Table III it will be noticed that Laboratory No. 3182 was analyzed without separation of the surface layer—that it should have been separated is shown by the ammoniacal nitrogen being higher than that of the succeeding sample (No. 3415). In fact, it requires more than thirty-two days more of storage for the ammoniacal nitrogen figure determined on the interior portion, to surpass that shown in the table at thirty-one days. Later on there is no necessity for separating exterior and interior, inasmuch as the whole piece is in a condition of active decomposition.

It must not be supposed that in tests conducted upon a number of pieces of meat, which are undergoing decomposition, that the course of the process will be the same in each piece and that on a given date two separate pieces will yield the same analytical figures. Even though the pieces are as nearly uniform as selection will make them, there are still individual differences, of structure and of composition (differences of fat content for instance) which will modify the rate of penetration and multiplication of bacteria. It may even happen that similar pieces of meat exposed to apparently similar condition will yet be invaded by bacteria, the predominating races of which are different. Slight differences in the nutrient substratum will sometimes determine the preponderating variety. And so not only the relative abundance of bacteria, but also the relative abundance of different kinds with different chemical activities affects the course of chemical change in the decomposing material. Within limits, then the putrefaction of meat may assume very different aspects. The reaction may be acid or alkaline to litmus or other indicators, although it usually becomes alkaline to litmus and remains throughout acid to phenolphthalein. A great variety of acid and basic bodies may be produced under various conditions, aerobic and

anaerobic, and at various temperatures. Under anaerobic conditions we speak of putrefaction, under aerobic conditions of decay. Recent work indicates that characteristic stinking putrefaction can be excited only by anaerobes. In spite of the different courses followed in different cases of putrefaction its characteristics are sufficiently well defined to distinguish it from all other chemical processes. The same may be said of decay.

The meat used in the present experiments was the part known as the "knuckle" from the hind quarter of cattle. The piece is of very uniform lean composition, weighs usually seven to eight pounds and is somewhat conical in shape, seven inches in diameter at the base by fourteen inches high.

In one series of experiments, the results of which are shown in Table III, the knuckles were hung in a temperature of 2°–4° C. immediately after slaughter and were allowed to remain there during the period when analyses were made, that is for 121 days. When the knuckles had been in storage 54 days several were removed from the place of storage, frozen and held at —9° to 12° C. for some time longer. This was done in order to determine whether freezing arrests decomposition promptly or whether once started it continues in the frozen state.

In the other series of experiments (Table IV) knuckles which had been held in the frozen condition for 33 days were removed to storage of 2°–4° C. and held there for 120 days.

Moisture.—The moisture determinations are fairly constant throughout and require no special comment.

Ash, Fat, Total Nitrogen.—The variations which occur in these determinations are only such as might be expected in meat from different animals. They are not especially significant.

Ammoniacal Nitrogen.—The ammoniacal nitrogen determined by Method I remains nearly constant for 21 days in the case of the unfrozen samples and for 8 days in the case of the frozen ones. Had the exterior been separated, the ammoniacal nitrogen determined by Method I in the interior would undoubtedly have remained constant for a much longer time but this was not done until the samples were 39 and 23 days in storage, respectively, on account of the thinness of the bacterial border and the difficulty of its exact separation. The ammoniacal nitrogen determined by Method II remains constant for 7 and 8 days, respectively. As the meat ages there is a fairly regular and consistent increase in the amount of ammoniacal nitrogen by both methods. The apparent exceptions to the regular increase are

TABLE IV.

Progress of decomposition in beef which had been frozen for 33 days and was then transferred to a temperature of 2° to 4° C. Beef knuckles used in the experiment. All figures on basis of meat. For methods see *J. Am. Chem. Soc.*, **30**, 1527-30.

Lab. No.	Rec'd.	Age, days.	Days in 2°-4°C temp.	Moisture, per cent.	Ash, per cent.	Fat, per cent.	Total N, per cent.	Amm. N, Method I, per cent.	Amm. N, Method II, per cent.	Cold water extract. Total solids.	Ash.	Total N, per cent.	Coag. N, per cent.	Alb. N, per cent.	Meat base N, per cent.	Acidity, per cent. lactic.	Remarks.
Average analysis thirteen beef knuckles				76.35	1.23	1.43	3.49	0.029	0.010	6.01	1.14	0.806	0.413	0.024	0.355	0.68	
2546	3/16/08	33	0	76.86	1.20	1.17	3.31	0.026	0.009	5.56	1.16	0.766	0.373	0.023	0.359	0.72	
2810	3/24/08	41	8	75.67	1.28	1.10	3.63	0.030	0.010	6.02	1.15	0.805	0.398	0.028	0.372	0.72	
3061	3/31/08	48	15	76.05	1.21	1.50	3.51	0.042	0.014	6.86	1.07	0.907	0.449	0.043	0.392	0.59	
3363	4/8/08	56	23	74.91	1.14	2.00	3.79	0.048	0.024	5.98	1.06	0.861	0.408	0.047	0.388	0.73	Bacteria at isolated points in interior
Same, putrefying exterior portion								0.092	0.040								
3369	4/9/08	57	24	76.42	1.15	1.41	3.55	0.051	0.024	6.11	1.08	0.872	0.449	0.049	0.353	0.72	Bacteria at isolated points in interior
Same, putrefying exterior portion								0.111	0.047								
3635	4/16/08	64	31	76.14	1.16	1.55	3.53	0.049	0.029	5.83	1.10	0.819	0.426	0.041	0.372	0.61	Bacteria at isolated points in interior
Same, putrefying exterior portion								0.122	0.046								
3878	4/24/08	72	39	76.18	1.30	2.01	3.63	0.058	0.032	6.60	1.11	0.931	0.516	0.041	0.380	0.73	Bacteria generally distributed throughout
Same, putrefying exterior portion								0.106	0.051								
4311	5/6/08	84	51	77.34	1.17	1.11	3.48	0.092	0.039	6.18	1.04	0.917	0.504	0.074	0.329	0.57	No distinction between exterior and interior
5456	6/11/08	120	87	76.70	1.24	1.89	3.56	0.198	0.120	6.97	1.18	1.074	0.534	0.056	0.467	0.39	No distinction between exterior and interior

to be explained as before stated, by the inclusion of the bacterial surface border with the undecomposed interior. It appears that the ammoniacal nitrogen, determined by any one of several methods, increases regularly with bacterial decomposition. Yet it cannot be said positively that it is characteristic of bacterial hydrolysis only, since the ammoniacal nitrogen increases to some extent in acid and alkali hydrolysis and peptic and pancreatic digestion of proteins. The relationship between the ammoniacal nitrogen produced by the agents mentioned requires further study. In the absence of the other hydrolytic agents, the production of ammoniacal nitrogen can be definitely ascribed to bacteria.

Total Solids in Cold Water Extract.—On the whole the total solids in the cold water extract appear to increase with the age of the meat and hence with the increase of decomposition. This increase is not marked except in the oldest samples, and even then does not reach a very high figure.

Ash in Cold Water Extract.—The figures as would be expected are nearly constant throughout.

Total Nitrogen in Cold Water Extract.—There is a fairly regular increase in the nitrogen in the cold water extract from the beginning to the end of both experiments. The increase is altogether greater (as in other experiments also) than the increase in total solids.

Coagulable Nitrogen in Cold Water Extract.—The coagulable nitrogen shows, on the whole, a tendency to increase, although this is not regular and never reaches a larger amount. It is most marked in the oldest samples.

Albumose Nitrogen in the Cold Water Extract.—The albumose nitrogen determined by the zinc sulphate method increases, although somewhat irregularly, with the age of the samples. The total amount is never large and this would indicate that the albumoses are rather quickly transformed into simpler substances—those we have grouped under the term "meat bases."

"Meat Base" Nitrogen.—The "meat-base" nitrogen gradually increases with increasing age and decomposition.

Acidity.—It is doubtful whether in a product like meat, the lean of which is composed almost entirely of proteins, any fermentation could result in decidedly altering the reaction provided the indicator used is sensitive to weak acids as well as to ammonia. The fermentations of carbohydrates and fats produce acids, but these compounds contain no nitrogen or basic groups.

While there are some contradictory figures in the analyses of the samples which were frozen after being stored at 2–4° C., from the results the conclusion may fairly be drawn that freezing of meats at —9° to —12° C. arrests bacterial decomposition but can not in any degree restore the product to its original condition.

ADDRESSES.

THE EFFICIENCY AND DEFICIENCIES OF THE COLLEGE-TRAINED CHEMIST, WHEN TESTED IN THE TECHNICAL FIELD.[1]

By William H. Nichols.

The subject which has been assigned to me as part of this symposium is one which I would hardly have dared to bring forward of my own motion. It must involve us occasionally in statements which are not complimentary to either student or professor, but which are nevertheless true. Of course there is much that is of a more agreeable character, and this is naturally the more attractive side; but if it were the whole question, we would have reached perfection, and instead of a discussion we might satisfy ourselves with a summing up of our accomplishments, ending with a genial benediction, and go away with a snug feeling of thankfulness that we were not as other men. I cannot, however, forget my own experience while a student of chemistry and its applications, and the painful failures which ensued while subsequently endeavoring to put my learning into practice; and it is conceivable that these recollections may have had an undue influence on the result of my observations of others for a period of nearly forty years ensuing, during which time I have come in familiar contact with many college-bred chemists. The gradual though steady improvement in processes and methods to be noted, indicates the constant tendency to greater efficiency, while the comparative slowness of this improvement is doubtless partly due to some of the deficiencies in the preparation for the work. When I compare the attainments of recent graduates with my own when I went out into the world in 1870, I readily notice how much farther along the college work has brought them, and how much better prepared even the dullest or laziest of them is, than any of the students of my day. This applies to analytical and research work, as well as to chemical engineering, which then was extremely rudimentary and commonplace, usually given over to men who had been brought up in the works and without any scientific training.

The college course, of necessity, must be more or less general. With a hundred or more students of varying attainments and ability, it is impracticable to treat them as units, although I am aware that frequently all that is possible is done in this direction, particularly in the upper classes. From the technical point of view, there are three great fields in chemistry, and the student should himself decide which of these he intends to occupy. If he does not

[1] Read at Baltimore meeting, American Chemical Society, December 29, 1908.

do this of his own accord, his instructor should, I think, do it for him, after he has become acquainted with the student's measure. If during the ordinary course, the student or professor has not been able to make this selection, a post-graduate year should follow, always provided that the student's work shows that he has not been mistaken in selecting chemistry as a profession. In many cases it would be a kindness to suggest the taking up of some less exacting science, and some of the deficiencies are doubtless due to this effort to make synthetically a chemist out of impossible elements.

The three fields alluded to are those of analysis, research and technology or engineering. The first is of course essential to both of the others, and yet it is a pursuit by itself, and many follow it all their lives, either in works laboratories or as public analysts. As a rule, in this department, originality is not essential, the work being one of detail constantly repeated. Naturally, there are exceptions, and many of the best analytical processes have been worked out by men of this class. While this is not the highest branch of the science, it is at least the foundation, or more properly speaking, the root. There is no reason why any college should be deficient here, as it is only a question of professor and laboratory. Of course the institution cannot be held responsible for the student's inability or unwillingness to absorb what is generally so well given, although it may be criticized for not weeding out the stupid or lazy, and thus make needed room for others who hold out more promise. But certainly, it must be the fault of the college or professor, if a man be allowed to graduate who is not a good analyst, fitted to go on with routine work even while admitting that some institutions give a far better preparation than others. The science is open to all, but some men have a much better ability than others to impart knowledge that will be comprehended and absorbed.

The second of the fields alluded to as being open to the chemical student, while embracing the first, is of far greater importance and extent. I allude to the field of research. If during the college course, the student has not received some inspiration in this direction, there is a "screw loose," somewhere. If it be approximately true that if a man finishing the chemical course be not a good analyst, it is probably his own fault; it is, I fear, equally true that if he be not imbued with a determination to put his knowledge to uses that will produce new results, it is because his instructor has not been a man filled with an enthusiasm which he must communicate. A teacher of chemistry who imparts the dry facts of the science without holding out alluring glimpses of its future possibilities, is robbing his charges of their rights, for chemistry is the great science of the future, on which the welfare of the world, if not the very lives of its inhabitants, will depend more than on any other. But it must not be forgotten that research in chemistry must be conducted on common sense principles, and these include fixed purposes and objectives. Research on general principles is apt to produce general haziness and absolute lack of concrete useful results. But here we must make two subdivisions, lest we lose sight of the value of the magnificent work done in abstract research. It is very easy for us to understand the value of research which immediately results in the production of a new article, or an improved method of making an old one; but it is not so easy to appreciate the bearing of an abstract research which simply adds certain knowledge to our store, or opens to us some new law, which in our present state of ignorance has no bearing that we can discern, but the knowledge of which will be essential to some great discovery of the future, which in its turn will lead to others and still others *ad infinitum*. The purpose of this paper is not to discuss the importance of chemical research, which is one of the most attractive subjects I can imagine. Hence, I must not dwell too long upon it. I think I have said enough, however, to remind us of the responsibility under which instructors labor in this regard. The man who for four or five years each has a number of receptive minds looking to him for light, falls far short of his duty and opportunity if among those minds he has not stirred up, according to the measure, an intense desire for working into the future. The field of chemical research, both abstract and concrete, being one of the most important that the human mind can engage in, it is gratifying to feel, as I do, that there has been a steady improvement in this regard, in the training of men fresh from college, which indicates that instructors in many cases are fully alive to the importance of the subject, even if they appreciate that much remains to be done.

The third field alluded to for the chemical student, is that including applied chemistry, so called, and chemical engineering. The former of these two subdivisions covers a much larger territory than the latter. While thirty years ago, the use of a chemist in the ordinary manufacturing establishment was the exception, it is at present, I am happy to say, the rule. This is now so well known, and the reasons or rather necessities for it are so well understood, that nothing more than the allusion is needed. There is no reason, so far as I can see, why college and technical schools are not pretty well equipped to furnish men trained to be of great service in this field, not only for the careful watching and control of the work, but for research in the special lines looking to improvements and economies. Almost all kinds of manufacturing establishments are open to the teaching staff and students alike, and the best practice employed in nearly all industries is well understood within the college walls. Details of cost and production are generally ascertainable, and the professor's lectures may be kept fairly well up to date. Therefore, there is little reason why the graduate chemist who has had this line of work in mind, should not soon become useful, and thereafter steadily improve. In addition to his knowledge of chemistry, the more he knows of engineering the better, particularly if it be of a nature to enable him to apply his chemical knowledge to the needs of the particular establishment. The general manufacturer need have no hesitation in adding to his staff, graduates in applied chemistry of many of our institutions.

With chemical engineering, the case is somewhat different. As I said at the beginning, thirty-five or forty years ago, work coming under this head, was in this country performed almost exclusively by practical men brought up in the works, whose deductions were the result of experience unenlightened by theory. It was, of necessity, somewhat crude, but good enough for practical purposes while every chemical manufacturer was in the same boat. The advent of the college-trained man changed all this, and the uneducated engineer has been gradually relegated to the background and for obvious reasons. The advantage the college-trained man had

was in his knowledge of the theory of chemistry. His disadvantage frequently, was his utter lack of engineering knowledge to enable him to apply it practically. The theory was of more value than the rough engineering without it, for engineering ability of high order has long been obtainable, and thus, theory could have a respectable application. There have been brilliant cases where both existed in the same individual, but I am compelled to say, that as far as my observation goes, this has not been general or even common. Now it must not be forgotten that the converse of this proposition may be true, though I do not think equally so. If the chemist may look to the engineer for assistance, why may not the relation be reversed? My answer to that is that it may be and generally is, in manufacturing establishments of a general character. In the chemical works, however, where the highest application of the theory is aimed at, chemistry should be the guide, and engineering the servant. If the chemist can also be the engineer, so much the better, and so much more likely will he be to bring his theory to fruition. And just here is where a great difficulty presents itself. The theory of chemistry is open to any who desire it, and who have the capacity to absorb it. This is hardly true of its application in the manufacture of chemicals. The student of Electrical Engineering has not only the theory at his command, but his text books have the latest practice, and he may visit the newest plants, and study them to his heart's content. So also with the student in Civil, Mining or Mechanical Engineering. These may in addition have their Summer Schools and field work, with the opportunity thus afforded for actual work under the most favorable conditions. In addition, the instructors are frequently employed in consulting capacity, and are thus up to date in many of the finer details of what has been accomplished, and what is being considered for future accomplishment.

It is not so with the student of Chemical Engineering. His text books contain, frequently, old processes and sketches of antiquated plants, or details accessible only after the process itself has been discarded or the plant abandoned for something better. His instructor is frequently in no better situation; and his lectures are sometimes, I fear, records of what used to be. I do not see how this sate of affairs can be greatly changed, until chemical manufacturers open their works to both professor and student, a course which the exigencies of competition would seem to be likely to postpone indefinitely.

What, then, shall we teach our students who are preparing for this field? First of all—chemistry, all they can absorb. After this, as much Electrical and Mechanical engineering as possible, for both branches have come far to the front in modern chemical works. The chemical manufacturer who takes the graduate into his works will thus be able to make his instruction partake more of the nature of post-graduate work than is now so frequently the case.

A discussion of this question would fall far short of completeness if we did not take into consideration other matters than simply chemistry and engineering. The whole question of education in general has merited and received an immense amount of attention, particularly during recent years. While naturally, there have been many views, differing in detail, and sometimes widely divergent, it is, I think generally admitted as the consensus of opinion, that the object of education is to make the most that is possible out of the material in hand, on the lines for which the student is more especially adapted. This education received at college or elsewhere, is the foundation upon which the real education of the student is to be based; which real education can only be acquired in after life, as the result of study, experience and association with others. The college has done its work when it has provided the student with the best possible foundation, and naturally his future depends to a large extent upon himself.

This preliminary education, if it may be so termed, would lack what the student has a right to expect, if it ceases when it has made him a good chemist with ambition for research and even with a good knowledge of engineering, which have been cited in what precedes, as the three necessities of the college curriculum for the production of chemists.

There is no reason, after all, why a chemist should not be qualified to assume the general duties of citizenship as well as the members of any other profession; and therefore, during his college course he must have something more than pure science. No man can fulfil the possibilities of his life if he be altogether one-sided or lop-sided; the educator should endeavor, so far as possible, to round out all of the faculties of the man. To do this, culture studies must not be omitted; particularly such culture studies as would tend to elevate the literary, the artistic or even the imaginative side of the student.

In preparing a man for life in the chemical field, he should be filled with the idea that any position in that field is open to him if he have capacity, industry and honesty, and he should look for attaining no position that is lower than the highest. This he cannot hope to do unless by the absorption of culture studies, he has prepared his aesthetic side as well as his scientific, although, perhaps, not to so great an extent.

I am well aware of the limitations imposed upon by the necessarily short time the student has in college—usually four years, rarely more than five—in which to acquire all that a man needs in the scientific line to become useful in his chosen profession of chemistry. I am aware that a good deal of work will have to be added if he is to have the engineering knowledge which I have alluded to, and have his mind cultivated by literature and kindred branches; and I imagine that the criticism will be made that it is impossible for any man to do the amount of work necessary to accomplish this result. It seems to me that the answer to this is, that with the modern idea that education is only begun in college, there is no reason why such tendencies should not be produced in the mind of the student, so that all of the necessaries of a rounded education should follow by his own later work. It is only necessary to allude to that honored man who has recently announced his intention of retiring from the presidency of Harvard University, and who, although a chemist, rose to a position of great power in other fields, as instanced by his latest office; or to that other college president, who, while a great chemist, has presided with such dignity and force over the University which has been such an honor to this City of Baltimore. There are many other instances which will occur to you, but these two will suffice to show the possibilities to which men educated as chemists have a right to expect to attain.

To sum up briefly what I have so imperfectly expressed;

there is no reason why any college should not turn out good analysts and research chemists. If the graduates fail in these particulars, it is largely due to inefficient work of the professor or the college itself. There is sufficient reason why colleges should fail in turning out good chemical engineers, and for this failure I do not think they should be held altogether accountable, provided they do what they can in the direction of furnishing engineering attainments to the chemists. I believe that nearly all colleges are more or less deficient in the culture work which is given to their chemical students, and I see no reason why this deficiency should continue. They have the means at their command and should insist upon the student availing himself of them.

But after all, the greatest deficiency will be found to reside in the student himself, who either wilfully or from lack of ability, does not take full advantage of the opportunities afforded. In this respect the chemical student is no exception to the general rule the world over, and I suppose that to the end of time, brains, carefully cultivated and backed by initiative, will be at a premium.

THE UNTILLED FIELD OF CHEMISTRY.

Address of the Chairman of the Division of Industrial Chemists and Chemical Engineers, Baltimore, December 29, 1908.

By Arthur D. Little.

Two years ago, upon a similar occasion, it was my privilege to address many of you and to point out some of the relationships existing between the chemist and the community. In so doing, there naturally devolved upon me the pleasant duty of recalling to your minds a few of the great and more significant achievements of members of our profession by way of indicating, although briefly and most inadequately, something of the extent to which the chemist has already placed the community in his debt. It is good for any body of men, animated by a common purpose, to take, from time to time, mental stock of what they have accomplished and of the relation in which they really stand to their environment. Whereas in our own case, the record is one of which we all may well be proud, its contemplation brings a new sense of the dignity of the work itself, a pride of fellowship and an incentive to increased endeavor.

We move, however, in a world where it is easy to take much for granted, where symbols and conventions quickly come to take the place of the realities they represent. Our mental processes are apt to run along the line of least resistance and the apparent and the obvious obscure the fundamental truths. This being so, we can well afford to leave our achievements in the security of the past while we consider for the moment the things we have left undone.

The volume of chemical literature has become so great, so many compounds have been described and classified, so many methods have been laid down, so much of detail confronts the student, and his field of study has been so subdivided as to suggest and foster the delusion that the total sum of chemical knowledge must be vast indeed. Vast in its detail perhaps it is, but lacking in those fundamental unities which out of the confusion of detail bring wisdom. Chemistry still awaits its Newton. It still justifies the estimate of Kant who said of it more than 100 years ago:

"Chemistry is a science, but not a science in the highest sense of that word; that is, a knowledge of nature reduced to mathematical mechanics."

Despite the immense amount of dry detail which we have accumulated and in some measure correlated, chemistry is still in the imaginative era where generalizations are more the result of happy inspiration than of close mathematical analysis.

Chemistry concerns itself with the changes which matter undergoes in its varying relations to certain forms of energy and yet we do not know what matter is nor have we any conception of the real nature of energy. One has only to state in their ultimate terms the problems confronting us to bring a realization of how very far from their solution we still stand. They are, for instance, thus summarized by Karl Pearson:

What is it that moves?
Why does it move?
How does it move?

Where, yet, I ask you is their answer to be found in chemistry?

We have built our science upon the assumption that matter, whatever that may be, is composed of indivisible atoms, of a comfortable and ultimate simplicity, only to find that the atom is in fact divisible and that its structure is undoubtedly complex beyond imagination. As to those phases of energy which are concerned with chemical change, even so great a philosophical chemist as Otwald says: "Chemical energy is to us the least known of all the various forms, as we can measure neither it nor any of its factors directly. The only means of obtaining information regarding it is to transform it into another species of energy."

So we have gone on for a hundred and fifty years transforming chemical energy into electrical energy or into heat, making minutely refined measurements of the relatively small amounts of energy appearing in our processes, while wholly unconscious all this time of the stupendous stores of potential energy which we now vaguely begin to realize are bound up in matter.

Our study of matter has led us to teach that it manifests itself in some seventy distinct and separate forms which we call elements, and yet our very definition of an element is a confession of our failure. An element is something which we have not been able to decompose into anything simpler. We have discovered some curious and interesting relationships between the elements which point to their common origin. In his heart each one of us believes that they must have had a common origin and that the cycles of development which they exhibit can have resulted only from the action of a periodic variable upon a constant and yet we very rarely even consider the question of their genesis or why their properties are what they are. We are content to regard them as so many distinct creations. The discovery of a new element is hailed as marking an epoch in the history of our science when our real business should be the elimination of the elements as such.

In their interactions, the elements, as we know them, manifest valences and selective affinities which determine the course of all chemical change and yet we are without an acceptable working hypothesis of the cause and nature of either valence or chemical affinity. Our ideas regarding the constitution of the molecules of many compounds have been developed in great detail and have led us to so many happy conclusions which the facts have verified as to justify our belief that these ideas must rest upon a substantial

basis of truth. This sometimes leads us to forget that the graphic formulas which we build up and write on a plane surface are an attempt to represent in terms of two dimensions actualities which exist in three. Moreover, these formulas depict the molecule as something fixed and rigid although everything tells us that the atoms within the molecule are in rapid and ceaseless motion. A new chemistry will dawn when we take proper cognizance of these motions and their influence upon the properties and relations of the compound. We state molecular weights with a finality of assurance, forgetting that we know very little of the molecular weights of liquids and nothing of the molecular weights of solids. We write cellulose as $(C_6H_{10}O_5)_n$ but the unknown n is probably the most significant part of the entire formula.

Sulphur passes before our eyes from the crystalline to the amorphous variety, phosphorus assumes the red or yellow form and an almost complete change of properties attends the transformation. Carbon exists in several markedly different states, and yet as to the meaning and mechanism of these molecular changes we remain in complete ignorance. Fortunately for the comfort or even the very fact of our existence upon the planet water is denser at 4° than it is at zero. Had it not been so, our lakes and oceans would be simply so many solid ice masses upon which the summer sun could make only a superficial impression, but, in spite of the paramount importance of the fact itself no one of us can say why the water molecule presents this curious anomaly. We write the water molecule as H_2O and commonly regard it as a relatively simple compound. How then shall we account for the fact that the dielectric constant or specific inductive capacity of water is about fifty times that of air, or perhaps ten times that of glass. As the dielectric constant is in a sense a direct measure of the massiveness of the molecule, are we not forced to the conclusion that the water molecule really is built up of many of these H_2O groups? How else indeed shall we explain the power of water to knock asunder the molecules of electrolytes which it dissolves and does not this complexity of the water molecule bear some relation to the essential part which water plays in the ultimate phenomena of living matter?

And this brings me to the main point of my thesis. A great German chemical company tells us in an attractive book just issued that it employs 218 chemists, 142 civil engineers, 918 officials, and nearly 8,000 workmen. It covers an area of 220 hectares, has 386 steam engines, 472 electric motors, and 411 telephone sub-stations. The plant represents the highest development which industrial chemistry has reached, but none the less it cannot produce an ounce of starch which a potato growing in the ground fabricates from water and carbonic acid gas under the influence of sunshine.

True it is that this great aggregation of engines and dynamos, furnaces, retorts, and stills can, under the direction of its highly trained and specialized chemical staff, produce certain natural products in condition so available and pure as to even improve upon nature, but by what monstrous effort is it accomplished? In the spring the tender grass and the delicate unfolding leaves cover the whole earth with the green of chlorophyll. There is no noise, no smoke, no stench. The grass is cool and grateful to the touch and clean. In similar manner vegetation everywhere is fabricating cellulose to the extent of several billion tons each year, and not only cellulose, but all the countless other complex products of the vegetable cell. What shall we say of our own chemistry in the face of facts like these, except that we have gone far enough to encourage a faint hope that we may some day be able to approach such methods. Professor Wheeler has defined so clearly a thought which has been in my own mind for years that I cannot do better than quote his words. He says:

"The vegetable cell is a laboratory in which are carried out a most remarkable series of chemical reactions. As we contemplate the immense number of organic compounds of all degrees of complexity which are formed within this wall of the plant cell we are convinced that this is the chemical laboratory *par excellence*. Two features impress us particularly: first the silence in which the operations are carried on; second, the narrow range of medium temperatures required. Notwithstanding this apparent simplicity of conditions, the products are of the most varied kind. Some of these men are able to synthesize in their own crude way; others are still the secrets of nature. It is utterly impossible for man to prepare certain naturally occurring compounds except at a temperature which would burn the plant tissue. We are led to wonder whether forces exist with which we are unacquainted or whether we are merely unable to control the forces already familiar to us. It would be difficult to say which supposition is the more probable. It will be granted that investigation into the activities of the cell is of profound importance. In fact, it has been said that "it is in the plant cell where synthetical operations are predominant, that we have to look for the foundation of the 'New Chemistry' which may be expressed broadly as the relation of matter to life."

I expressed two years ago my own belief that the distinctions which we now regard as fundamental between living matter and dead matter would soon break down. This break will in my opinion come through the study of the colloids which are the link between matter which we regard as living and that which we regard as dead. At this point, I cannot refrain from volunteering a suggestion. We know that the atoms within the molecule are in rotation. It must follow that as the complexity of the molecule increases, more and more of its motion of translation must be converted into rotary motion. In the colloidal molecule we know that many simpler molecules are linked together, and in the molecule of living matter, what? May it not be merely that the more or less haphazard and confined movements of the molecules which together build up the colloid are in the molecule of living matter coördinated and controlled in a manner which suggests the vortex? Dead matter drawn within this vortex would partake of this movement and exhibit the phenomena of life. Matter thrown off tangentially would resume its rectilinear course and become for the moment dead.

When we consider that in theory at least, in which accidents are barred, a tiny bit of living jelly, an amoeba for example can endow with life an ocean of its proper pabulum, it seems obvious that the forces which are to manifest themselves in the phenomena of life are already existent in the pabulum and that what the living jelly does is to induce a coördination and direction of the atomic movements which then take on the vital aspect. Do we not have something

roughly analogous to this in the magnetization of successive pieces of steel drawn across a lodestone? A certain coördination of movement in the molecules of the steel has been induced and magnetism results. So in some manner far more complex, life I believe may be epitomized as coördinated motion.

The subject-matter of such speculations lies so far outside our present-day chemistry as to almost require apology for their presentation, but they are well within the subject-matter of the chemistry of the future, for, to again quote the words of Pearson:

"The goal of science is clear, it is nothing short of the complete interpretation of the universe."

Or as Muir has put it:

"The great business of chemistry is to force men into close contact with some aspects of external realities and, with the help of her sister sciences, to remove everything that prevents the full vision of nature."

NOTES.

THE METHODS OF THE UNITED STATES STEEL CORPORATION FOR THE COMMERCIAL SAMPLING AND ANALYSIS OF IRON ORES.

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IRON ORE SAMPLING IN GENERAL.

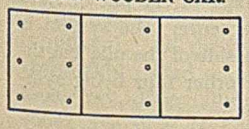
Owing to the varying conditions under which iron ores must be sampled, both by the producer and the consumer, variations from any uniform procedure of sampling are necessitated. The elements of time, size of shipment, kind of ore and other considerations are determining factors in the details of procuring the sample. Hence it is the purpose to render the following methods general in their scope, promulgating those ideas applicable in the broader sense to ore sampling, without attempting to prescribe for ever varying and unforeseen contingencies in the details.

CAR SAMPLING AT PRODUCER'S END.

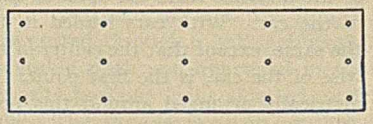
All samples must be taken uniformly over the surface of the cars after being loaded by taking a minimum of twelve places for wooden, and fifteen places for steel cars, by either the parallel or the zigzag system. The following diagrams illustrate these two systems and show the minimum number of points where samples are to be taken.

PARALLEL SYSTEM.

25 TON WOODEN CAR.

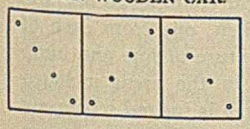


50 TON STEEL CAR.

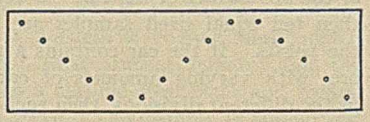


ZIGZAG SYSTEM.

25 TON WOODEN CAR.



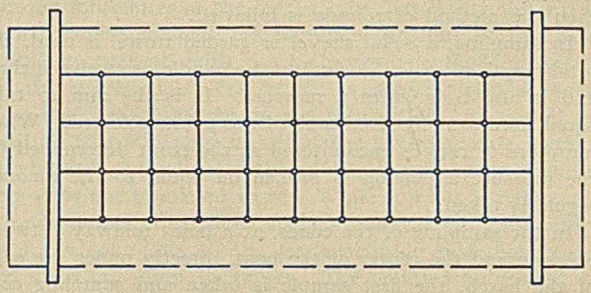
50 TON STEEL CAR.



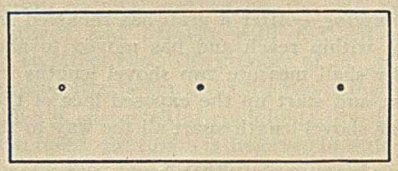
When lumps of ore are encountered at the designated points where samples are to be taken, small portions of each lump must be chipped off, about equal in size to the first joint of the thumb. When rock occurs it must also be sampled as ore, that is, a proportionate amount of the rock or off grade material is to be taken, not equal in amount to the regular sample for the area covered by that point, unless the entire area is such off grade material. Then the same amount of adjacent material must be taken, ore or rock, equal in amount to the portion taken at each regular point. This also applies to field boulders, unless thrown from cars.

The samples are taken with a garden trowel. Each ten cars, either steel or wooden, must be combined, as a rule, into one sample; but less than ten cars may be grouped. The weight of the sample must not run under fifteen pounds for ten wooden, and twenty pounds for ten steel cars.

Whenever very lumpy ore is to be sampled in the cars, the rope net system is used as shown below, which gives about thirty-two places on each car, the knots being eighteen inches apart. In using the net system, if a lump of ore or rock comes directly under the knot, a piece should be taken about the size of the first joint of the thumb. If fine ore occurs under a knot, an equal amount is taken with the trowel. The rope net system is used at the Marquette Range on the hard, lump ore; also at the Soudan Mine, Vermilion Range.



A moisture sample must be taken for each train load from all open pit mines; a train consists of forty to forty-five of the fifty ton steel cars, or sixty to sixty-eight of the twenty-five ton wooden cars. A moisture sample must also be taken for all the cars loaded at a shaft or stockpile during each ten hour shift. Samples must be taken from three places on top of each car as shown by the following diagram.



Care should be taken to secure the sample from well underneath the surface as soon as practicable after loading, maintaining the true proportion of lump and fine ore. The sample as taken must be immediately placed in a can with a tightly fitting lid, and brought to the crusher house. It is optional to take a moisture sample from the regular sample, provided it has been taken from well underneath the surface.

CARGO SAMPLING AT CONSUMER'S END.

Cargo ores present the most serious obstacles to a uniform method of sampling. The boats vary in size from three thousand to twelve thousand tons with one or two decks, and in the number of hatches from six to thirty-six with widths varying from twelve to twenty-four feet. The

grabs at the different unloading points vary in number, kind, size, and the rapidity of their operation. The ores vary from the very fine to the all lump, from the so-called mixed ores such as the groups to the mixed cargoes, consisting of different ores in the same boat, and with different ways of loading the boats.

Grab Sampling.—An excellent method of cargo sampling where the entire cargo will be represented, and particularly adaptable for fine ores is called grab sampling. A sampler with a small scoop attached to a handle of suitable length and holding a definite amount of ore, a quarter, a half, or a whole pound, takes a scoopful from each grab as it rises above the deck. The disadvantage of this system is its increased cost due to the extra number of samplers, one being required for each grab during the entire time of unloading.

The general plan for the sampling of all cargoes is to first sample the tops of the piles, before the grabs have started to unload; this is called sampling of the cones. After the grab has removed from the hatch all the ore within reach, the exposed faces standing on each side are sampled; this method is known as face sampling. Or when the latter practice is impracticable owing to the operation of the grabs, then the method of rounds is followed.

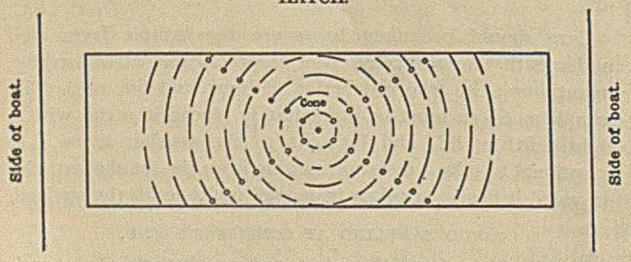
In sampling, a small shovel or garden trowel is used, the total length of which, including the handle, is twelve inches, and it also constitutes a measure. It is the aim to take equal sized samples from each of the points selected. When lump ore or rock is encountered at the point determined by the measure a portion is broken off equal to the amount regularly taken.

In the sampling of the cones, at a point midway between the side and the center of the boat, directly under the edge of the hatch, the first sample is taken and sampling continued one shovel length apart up the surface of the cone, over its apex and down the opposite surface to a corresponding point under the other edge of the hatch. This line is crossed from corresponding opposite points under the hatch as shown in the following sketch. Not more than one-tenth of the total sample is to be taken in the sampling of the cones.

CONE SAMPLING.

Face Sampling.—After a grab has removed from a hatch all the ore within reach and has moved to another hatch, the sampler shall measure two shovel lengths from the side of the boat and start up the exposed face of the ore taking samples one shovel length apart all the way to the top, using

HATCH.

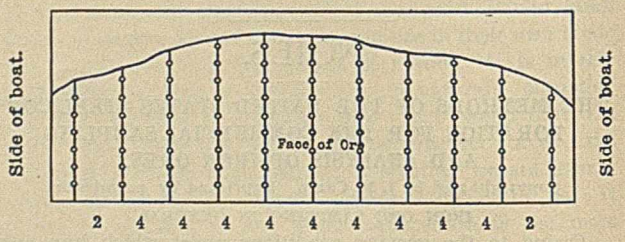


a ladder if necessary. The next vertical line is measured four shovel lengths from the first and the samples taken each shovel length apart on this line as before, and so on for each succeeding line across the boat. This is repeated on the opposite ore face, and the entire procedure continued until the ore faces of all the hatches are sampled that the character of the boat and the operation of the grabs will permit. When a bulkhead occurs, only the face opposite to it is to be sampled.

FACE SAMPLING.

Round Sampling.—When the operation of the grabs makes sampling by the face method impracticable, or with boats having twenty-four foot hatch centers, and decks furnishing protection to the samplers, then sampling shall be done while the ore is being removed by the grabs. When five or six feet of the face of the ore have been exposed, the sampler shall enter the hatch and measuring two shovel lengths from the side of the boat or edge of the face, take successive samples up the face one shovel length apart. The next vertical line is measured four shovel lengths from the first,

DECK.



and samples are taken all the way to the top as before, and so on across the entire face of the ore. This procedure is repeated on the opposite face, one-third of the total weight of the sample to be taken in the first round. When all the ore within reach of the grab is removed, the second round is taken, using the measurements as above, and the remaining two-thirds of the sample are secured.

Part of the regular sample is to be taken for the moisture sample, and for the fineness sample when such is desired.

CAR SAMPLING AT CONSUMER'S END.

When the ore is received in cars the greatest possible number are represented in the samples, and not less than ten equal sized samples are taken from each car. When cars are loaded with fine ore with the piles in opposite ends, at least five samples are taken from each pile, the first one at the apex of the pile, and the other four at points symmetrically arranged around the sides of the pile, two-thirds of the distance from the apex to the base of the pile or sides of the car. With cars loaded in the center, the system is the same, except that the center of the side of the pile lengthwise of the car, is the first point, the other four being symmetrically arranged around this point.

When the ten points are located in a car, each of them is supposed to represent a definite area, equal to one-tenth of the ore surface of the car. If the car contains all fine ore, then ten equal sized samples are taken, one from each of the points. If the car contains a mixture of fine and lump ore, with varying amounts of each in the areas included in the different divisions, then each area is judged separately and sampled accordingly. The fine and lump ore are taken each in its proper proportion, the former with the trowel, the latter being chipped, or selected small pieces being taken, each about the size of the first joint of the thumb. The combined sample of fine, chipped and selected pieces from

each area, equals the amount taken were it all fine ore. If the contents of the car are all lump ore, the proper sized pieces are chipped from four or five of the lumps in each of the ten areas making forty or fifty pieces from each car, the total amount of chipped pieces from each of the areas equaling the amount that would be taken were it all fine ore. All samples of fine ore are taken from well underneath the surface to obtain the ore in its natural state. A proportionate amount of the main sample is retained in a tightly closed can for the moisture determination.

PREPARATION OF SAMPLES IN GENERAL.

In the preparation of the sample for analysis, the ideal practice is to crush and quarter alternately until the desired quantity with the requisite degree of fineness is attained. A more expeditious and equally efficient method, is to crush the entire sample to the desired degree of comminution, then reduce the quantity by successive quartering as before, until the desired amount remains. It should ever be our purpose to approach as closely as possible to either of these two methods in the preparation of all samples for analysis.

PREPARATION OF SAMPLE AT PRODUCER'S END.

Samples before being quartered are brought into the crusher house where they are dried, if necessary, at 100° C. until the ore can be well mixed. Care must be taken to prevent overheating when other than low pressure steam is used for the purpose, especially with ores containing a large quantity of limonite. When sufficiently dry the larger lumps are crushed, if necessary, so that the entire sample will pass through a half-inch mesh sieve; a finer sieve may be used if desired. The sample is thoroughly mixed on an iron top table, then spread out evenly about three-fourths of an inch in depth and alternately quartered and mixed until one-fourth of the original sample remains. It is now crushed until fine enough to pass a quarter-inch mesh sieve, then mixed and quartered as before until about two pounds remain. It is again crushed until fine enough to pass a twenty mesh sieve, and spread out evenly about three-eighths of an inch in depth. About three ounces of this are taken from all over the pile, with a small spatula, dried in a small pan at 100° C. and crushed on a chrome steel plate, until it will all pass through a hundred mesh sieve. After being thoroughly mixed this is transferred to a bottle, and constitutes the sample for analysis.

The following method for the preparation of the sample is optional. All the ore is passed through a half-inch sieve, thoroughly mixed on a suitable cloth and quartered in the usual way, diagonally opposite portions being rejected until about five pounds remain. The sample is again crushed, if need be, to pass a quarter-inch mesh sieve, and mixed and quartered as before until one and one-half to two pounds remain. After being dried at 100° C. for twenty to thirty minutes, it is crushed to a fineness of twenty mesh, mixed and quartered until about three ounces remain, and the entire sample is crushed on a chrome steel plate and passed through a hundred mesh sieve. This sample is spread out in a shallow pan, dried at 100° C. for thirty minutes, again mixed and transferred to a three-ounce bottle or can for the analysis.

PREPARATION OF SAMPLE AT CONSUMER'S END.

The aggregate sample is dried at 100° C. and crushed before any quartering whatsoever, so that the entire mass will pass through a half-inch mesh sieve. This is reduced by successive quartering and crushing until its weight is from four to eight ounces, and it will then all pass through an eighth-inch-mesh sieve. Or the entire sample is crushed so it will pass through an eighth-inch-mesh sieve, and then quartered as before until from four to eight ounces remain. And this final quantity is then further crushed with a chrome steel bucking board and muller and all passed through a hundred-mesh sieve. A sufficient amount of this powder for all the needs of the analyst is placed in a small air-tight container, dried for one hour at 100° C. and when cool it constitutes the sample for analysis. A separate, larger portion of the same sample is retained for further needs.

IRON.

By the Bichromate or Permanganate Methods.

One or more portions of one-half to one gram of the ore are placed in a beaker, and 5 to 10 cc. of the solution of stannous chloride and 5 to 25 cc. of strong hydrochloric acid are added. This variation in the amount of acid is due, aside from the variations in the weight of sample and the varying solubilities of the ores, to the fact that while in the permanganate method the minimum amount of free acid should be present, with the bichromate method an excess of acid is essential. The beaker is covered with a watch glass and heated gently on the steam bath, or hot plate, until all iron is dissolved, and if the permanganate method is to be used, the volume of the solution is reduced by evaporation so as not to exceed 10 cc. While still hot the stannous chloride solution is added from a burette, drop by drop, until the color due to the ferric chloride just disappears, and then one drop in excess. The solution is diluted to about 350 cc. with cold water, 5 cc. of mercuric chloride solution are added and the solution is thoroughly stirred. From this point the bichromate or permanganate titrations are optional. In the former case, the potassium bichromate solution is added from a burette until a drop of the solution being titrated, added to a drop of the potassium ferricyanide solution on a paraffined plate shows no ferrous iron. In the latter case, 10 cc. of the titrating solution are added for a one-half gram sample, and 20 cc. for one gram. The solution is stirred and titrated with the potassium permanganate. In either case, the number of cc. used multiplied by the respective factor gives the percentage of iron.

If the residue is suspected of containing iron it is filtered off, ignited and fused; the fusion is dissolved in water, acidified with hydrochloric acid, and the solution reduced and titrated as above; or the solution before being acidified is filtered, the ferric oxide on the filter is dissolved in hydrochloric acid and added to the main solution or titrated separately.

SOLUTIONS FOR IRON TITRATIONS.

The stannous chloride solution is made by dissolving 250 grams of the salt in 500 cc. of strong hydrochloric acid, and diluting to 1000 cc.

The mercuric chloride solution is saturated in the cold.

The potassium ferricyanide solution is made fresh each day by dissolving in the proportion of 0.100 gram of the salt in 100 cc. of water.

The potassium bichromate solution is made by dissolving the salt in the proportion of about 8.9 grams to one liter of water, and standardizing with an iron ore, iron wire or

steel, of known iron content. One cc. equals approximately 1 per cent. iron with a one gram sample taken for analysis.

The titrating solution is made up in the following proportion to the liter: 90 grams of manganous sulphate are dissolved in 650 cc. of water, 175 cc. of strong sulphuric acid are added, then 175 cc. of phosphoric acid of 1.75 sp. gr., and the solution is filtered if necessary.

The permanganate solution is made by dissolving the salt in the proportion of about 5.7 grams to 1 liter of water and standardizing in the same manner as the bichromate solution. One cc. equals approximately 1 per cent. iron with a one gram sample taken for analysis.

SILICA.

Using One Gram with Hydrofluoric Evaporation.

One gram of the sample is transferred to a porcelain dish, or a beaker, with watch glass cover, and 20 cc. of strong hydrochloric acid are added. A gentle heat is applied until the ore is dissolved and the solution is then evaporated to dryness, unless it has been conclusively shown to be unnecessary.[1] To the residue, which should not be heated above 120° C., 15 cc. of dilute hydrochloric acid (1 part acid to 1 part water) are added, heat is applied until the salts are dissolved and the solution is diluted with hot water. The precipitate is filtered off, washed, ignited at the highest temperature of the blast lamp or muffle furnace for at least five minutes, cooled and weighed. To the residue in the crucible one or two drops of strong sulphuric acid and 5 cc. of hydrofluoric acid are added, and the solution is evaporated to the expulsion of sulphuric acid. The residue is ignited as before and again weighed; the difference between the two weights is silica.

SILICA.

Using One to Five Grams when All or Part of the Filtrate Is Used for the Phosphorus or Other Determinations.

One to five grams of the sample are transferred to a dish (beaker optional) with a watch glass cover, 20 to 50 cc. of strong hydrochloric acid are added, and a gentle heat is applied, without boiling, for about one-half hour. The solution is diluted with an equal volume of water, filtered into another dish of the same size, and the visible iron stain is washed out of the filter paper. This solution containing the bulk of the iron is allowed to go to dryness on the steam bath. The residue is ignited in a platinum crucible, and then fused with about six times its weight of sodium carbonate. The fusion may be leached out of the crucible, but it is found more expeditious to let it cool around the end of a platinum rod, and on again heating the crucible the entire mass adhering to the rod is removed. It is then dissolved in the original filtrate, or placed while still hot in the covered dish, in which the original solution was made and to which about 25 cc. of hot water have previously been added. The small amount remaining in the crucible is dissolved in hot water, and finally in hot dilute hydrochloric acid (1 part acid to 1 part water), and added to the dish containing the fusion, which by this time has been disintegrated by the hot water and acidified with hydrochloric acid; this dish is then placed with the other on the steam bath. When the contents of both dishes are dry, 10 to 15 cc. of strong hydrochloric acid are added to the dish containing the residue of the original filtrate. After heating until the ferric salt is dissolved, the solution is evaporated until the excess of hydrochloric acid is expelled, as is indicated by the first appearance of insoluble ferric chloride on the bottom or sides of the dish. Seven cc. of strong nitric acid are added and heat is applied for about one minute, or until the violent reaction is over. The solution is diluted with cold water and filtered into a suitable flask. This is the procedure if all the filtrate is to be used for the phosphorus determination; but the evaporation of the excess hydrochloric acid and the addition of the nitric acid are dispensed with, if the united filtrates are to be received in a volumetric flask and aliquot portions taken for the other determinations.

To the dish containing the fusion, and which should not be heated above 120° C., just enough dilute hydrochloric acid is added to moisten the residue. Hot water is then added and heat applied until all the salts are dissolved, when the solution is filtered into the same flask with the last filtrate; the combined filtrates should not exceed 150 cc. if the solution is to be used for the phosphorus determination. In this case, the complete washing of the silica is continued in another flask until free from chlorides, and these washings are discarded. The filter and its contents are then ignited and weighed as silica. The filtrate in the flask is reserved for the phosphorus determination, or if in a volumetric flask, for the determinations which follow.

PHOSPHORUS.

By the Fusion Method.

For this determination, five grams of the sample are taken for all ores when the phosphorus content is under 0.100 per cent., two and one-half grams when the phosphorus content is between 0.100 and 0.200 per cent., and one gram when the phosphorus exceeds 0.200 per cent.

An aliquot part of the filtrate, the volume of which should not exceed 150 cc., representing one gram, or two and a half grams of the sample, from the five-gram portion for silica in the volumetric flask, is boiled in a suitable flask for a few minutes with the addition of 10 cc. of strong nitric acid. To this solution, or to the entire filtrate in the flask from the five-gram portion, a slight excess of strong ammonia is added over the amount necessary to precipitate the iron and alumina, and after mixing enough strong nitric acid to dissolve the precipitate with from 5 to 10 cc. in excess.

The solution is heated to a temperature of 80° C., 50 cc. of the solution of ammonia molybdate are added, and the flask is shaken continuously for five minutes. The solution is allowed to stand until the precipitate has subsided. If the yellow precipitate is to be weighed, it is caught on a 9 cm. filter paper that has been dried for at least half an hour at 110° to 120° C. and weighed between watch glasses, washed thoroughly with a 2 per cent. solution of nitric acid, dried for one hour at the above temperature and weighed between glasses; 1.63 per cent of its weight is phosphorus.

If the yellow precipitate is to be titrated by the acid alkali method, the weighing of the filter is dispensed with, the washing with the 2 per cent. nitric acid is continued as before to the complete removal of the iron and molybdenum salts, and then with a 1 per cent. solution of potassium nitrate until free from acid. The filter and its contents

[1] This clause is introduced on the insistence of the mine chemists that with certain ores evaporation to dryness is not necessary to obtain all the silica.

are then transferred to a small flask or beaker and an excess of the standard alkali added. When the precipitate is all dissolved by shaking or stirring, about 150 cc. of water and a couple of drops of a saturated solution of phenolphthalein in alcohol are added, and the solution is back titrated with the standard nitric acid to the disappearance of the color. It is optional to again add the standard alkali, the end point being a faint, permanent, pink color.

The yellow precipitate may also be dissolved in dilute ammonia, and the phosphorus determined by the permanganate titration method as subsequently described. Or it may be dissolved in a hot ammoniacal citrate solution and the phosphorus precipitated as ammonium magnesium phosphate, as hereafter described.

Ammonium Molybdate Solution.—This solution is made in the following proportions: to 225 grams of pure molybdic acid in a suitable flask there is added 500 cc. of water, and then 500 cc. of strong ammonia. When in solution, this is poured into a flask containing 2500 cc. of nitric acid of 1.20 sp. gr., the solution being kept agitated, and three or four drops of ammonium or sodium phosphate solution are added to coagulate the suspended impurities. The solution is kept in a warm place over night or until the supernatant liquid is clear, when it is filtered into a bottle for use.

Stock Solution of Caustic Soda.—145 grams are dissolved in water and the solution is diluted to 2200 cc.; 200 cc. of this solution diluted to 2000 cc. will be of approximate strength.

Nitric Acid.—20 cc. of strong nitric acid are diluted to 2000 cc. These solutions are made exactly agreeing and standardized on steels or ores of known phosphorus content so that each cc. used corresponds to 0.01 per cent. phosphorus, when two grams of the sample are used. This weight has been found the most convenient for steel analysis, and a simple calculation enables it to be used for varying weights of either steel or ore.

PHOSPHORUS

Direct Determination of the Soluble Phosphorus Using the Permanganate or the Acid Alkali Titration.

This method is particularly applicable to ores wherein all or a fairly constant proportion of the phosphorus is soluble in strong acid. The insoluble phosphorus is determined at less frequent intervals, and the amount so obtained added to the soluble gives the total phosphorus.

From one to five grams of the sample, according to the phosphorus content, are placed in a beaker with a watch glass cover, 20 to 50 cc. of strong hydrochloric acid are added and a gentle heat is applied until the ore is dissolved. Two or three drops of nitric acid are added and the solution is boiled a minute or two, and then the excess acid evaporated. The solution is diluted and filtered, the filtrate being received in an 8 to 16 ounce Erlenmeyer flask; the filter and residue are washed with dilute hydrochloric acid (1 part acid to 4 parts water) and water until free from iron discoloration.

Strong ammonia is now added until a slight precipitate persists, or until the iron is completely precipitated, and in either case this is dissolved with strong nitric acid, too great an excess being avoided. The solution is heated to 80° C., 30 to 50 cc. of ammonium molybdate solution are added, the flask is shaken and placed in a warm place to allow the precipitate to subside. The greater part of the supernatant liquid is siphoned off, the remainder being filtered, or the entire solution is filtered. The precipitate is washed with ammonium sulphate solution or a 2 per cent. sulphuric acid solution, until free from iron and molybdenum salts.

The funnel containing the precipitate is transferred to the flask in which the precipitation was made, and the precipitate dissolved in dilute ammonia (1 part ammonia to 3 parts water); the resulting solution should not exceed 25 cc. Eight grams of granulated zinc (20 mesh) are added, then 75 cc. of sulphuric acid (1 part acid to 4 parts water), and the solution is boiled gently until reduction is complete. It is then filtered through glass wool or cotton, the filtrate being received in a beaker, and the flask and filter are washed thoroughly with cold water. The solution is titrated with potassium permanganate until a slight, permanent, pink tint is obtained. From the number of cc. used, the percentage of phosphorus is calculated.

If the acid alkali titration is to be used, the yellow precipitate is washed as before with a 2 per cent. nitric acid solution until free from iron and molybdenum salts, then with a 1 per cent. solution of potassium nitrate until free from acid, and the determination is completed in the regular manner.

SOLUTIONS.

Standard Permanganate.

204.5 grams potassium permanganate,
1 carboy (48 liters) distilled water.

Ammonium Molybdate.

300 grams molybdic acid dissolved in
1200 cc. ammonium hydrate, sp. gr. 0.96, this to be added slowly to
4500 cc. nitric acid, sp. gr. 1.20, keeping the solution agitated.

Ammonium Sulphate.

40000 cc. distilled water,
1100 cc. ammonium hydrate, sp. gr. 0.96,
960 cc. sulphuric acid, sp. gr. 1.84.

Phenolphthalein.

Saturated solution in alcohol.

PHOSPHORUS.

By Ignition of the Insoluble Residue.

One to five grams of the sample in a porcelain dish, with watch glass cover, are digested in a 20 to 50 cc. of strong hydrochloric acid with the aid of a gentle heat. After diluting slightly with cold water, the solution is filtered into another dish of the same size and the precipitate washed free of the visible iron discoloration. The filtrate is rapidly evaporated and while this is in progress, the residue is transferred to a platinum crucible and ignited in a muffle furnace or over a blast lamp. The residue is broken up, reignited for a few minutes, then transferred into the original filtrate and the solution is evaporated to dryness.

Fifteen cc. of strong hydrochloric acid are now added and the solution is evaporated until ferric chloride begins to separate in the solid state. To the covered dish, 7 cc. of strong nitric acid are added and heating is continued for about one minute. The solution is diluted with cold water and filtered into a suitable flask. The filter and precipitate are washed with a 2 per cent. nitric acid solution until the iron discoloration is removed.

With unknown ores, and at regular intervals with

all ores, as a check, the insoluble residue is fused with sodium carbonate. The fusion is dissolved in dilute hydrochloric acid (1 part acid to 1 part water), and the solution is evaporated to dryness. The residue is redissolved in dilute hydrochloric acid and the solution is filtered into the main filtrate, which has also been evaporated to dryness, and the residue treated as described in the previous paragraph after the evaporation to dryness. The filtrate, or the combined filtrates in the flask are made ammoniacal, and the precipitate is dissolved in strong nitric acid with 5 to 10 cc. in excess. The solution is brought to a temperature of 80° C., 50 cc. of ammonium molybdate solution are added and the flask is shaken for five minutes.

When the precipitate has subsided, it is filtered off and washed with a 2 per cent. solution of nitric acid. From this point the yellow precipitate may be weighed, or titrated by the acid alkali or permanganate methods as previously described, or dissolved, reprecipitated, and weighed as magnesium pyrophosphate in the following manner.

The yellow precipitate is dissolved on the filter with hot ammonium citrate solution (175 cc. strong ammonia to 325 cc. water containing 25 grams citric acid), the filtrate being received in a No. 0 beaker. The filter is washed several times with hot water, then 5 to 10 cc. of magnesia mixture (100 grams magnesium sulphate plus 300 grams ammonium chloride in 800 cc. hot water plus 400 cc. strong ammonia) are poured through the filter. The filtrate in the small beaker is thoroughly mixed and allowed to stand for at least three hours, and if possible over night. The precipitate is filtered off, washed with dilute ammonia (1 part ammonia to 4 parts water), ignited at a low temperature until the carbon of the filter is destroyed, then at the highest temperature of the muffle furnace or burner, cooled and weighed as magnesium pyrophosphate containing 27.84 per cent. phosphorus.

PHOSPHORUS.

When Titanium is Present.

Two grams of the sample are intimately mixed with eight to ten times its weight of sodium carbonate in a large platinum crucible and fused over a blast lamp, the fusion being maintained at the highest temperature for fifteen to twenty minutes. The fusion is then transferred to a beaker containing hot water and thoroughly disintegrated; the solution is then filtered and the residue washed with hot water, or preferably with a 1 per cent. solution of sodium carbonate. As an extra precaution, not deemed necessary in every-day work, the residue may be ignited until the filter is destroyed, crushed in the crucible, fused, disintegrated and filtered off as before, the filtrates being combined. The filtrate, or the combined filtrates are acidified with hydrochloric acid and evaporated to dryness. The residue is moistened with dilute hydrochloric acid, dissolved in hot water and the solution filtered into a suitable flask.

The filtrate is made ammoniacal, then strong nitric acid is added with from 5 to 10 cc. in excess. The solution is brought to a temperature of 80° C., 50 cc. of ammonium molybdate solution are added, the flask is shaken for five minutes, and the phosphorus determined by any of the methods previously described.

When it is desired to separate the phosphorus from the large amount of sodium salts in the filtrate after the evaporation to dryness, a weighed amount of iron, about 0.05 gram, of known phosphorus content, and in the ferric condition, is added and precipitated in the hot solution with ammonia. The precipitate is filtered off, dissolved in warm, dilute nitric acid, and if the resulting filtrate is somewhat cloudy from particles of filter paper in suspension, and it is desired to remove them, it is refiltered after the addition of a little paper pulp. This filtrate is now made ammoniacal, 5 to 10 cc. excess nitric acid are added, and the determination is completed as above described.

ALUMINA.

Phosphate Method.

For this determination the hydrochloric acid filtrate from the silica of one gram of the sample, or the hydrochloric acid solution of the precipitated iron and alumina from the gravimetric determination of manganese is used. To the cool solution diluted to about 400 cc. in a No. 5 beaker, 30 cc. of a 10 per cent. solution of ammonium phosphate are added, and then ammonia until a faint, permanent precipitate is formed. One and one-half cc. of strong hydrochloric acid are now added, the solution is stirred until the precipitate is dissolved, and 50 cc. of a 20 per cent. solution of sodium hyposulphite are added. The solution is heated just to the boiling point, at which 8 cc. of strong acetic acid and 15 cc. of a 20 per cent. solution of ammonium acetate in the same measure are added, and the boiling is continued ten minutes. The precipitate is allowed a few minutes to subside, is filtered off as quickly as possible and washed ten times with hot water. The filter and its contents are ignited at a low temperature until the carbon is destroyed, then at the highest temperature of the muffle furnace, cooled and weighed as aluminum phosphate, 41.85 per cent. of which is alumina.

ALUMINA.

Ether Method.

One to five grams of the sample are placed in an 8 ounce beaker and dissolved in 20 to 50 cc. of strong hydrochloric acid with the aid of a gentle heat. The solution is diluted and filtered, the residue washed, and the filtrate evaporated to dryness. The residue on the filter is ignited in a platinum crucible, 2 to 6 drops of sulphuric acid (1 part acid to 1 part water) and 5 to 10 cc. of hydrofluoric acid are added and the solution is evaporated to dryness. The residue in the crucible is fused with one-half gram of potassium or sodium bisulphate and dissolved in 10 cc. of dilute hydrochloric acid (1 part acid to 1 part water). The residue from the original filtrate, after its evaporation to dryness, is dissolved in 20 to 30 cc. of hydrochloric acid, 1.13 sp. gr., cooled and transferred to a separatory funnel, 40 to 100 cc. of ether are added and the funnel is shaken for one minute, the solution being kept cool. The acid layer is drawn off into an 8 ounce beaker, and to this is added the solution obtained from the treatment of the insoluble residue. The combined solutions are heated until the ether is driven off, a few crystals of sodium or potassium chlorate are added, and the heating is continued until free chlorine is expelled. The solution is diluted to about 75 cc., ammonia is added until a slight precipitate persists, then 10 cc. of a 20 per cent. solution of ammonium acetate. The solution is boiled about one minute and filtered; the precipitate is washed

with hot water, then dissolved in warm dilute hydrochloric acid. After being diluted to about 75 cc. and a slight excess of ammonia added, the solution is boiled, filtered and the precipitate washed as before. The combined filtrates are reserved for the determination of manganese, lime and magnesia. From this point, the alumina may be determined by either of the following methods:

The precipitate is ignited, and weighed as oxides of iron, alumina and phosphorus. The ignited oxides are dissolved in 3 cc. of stannous chloride and 10 cc. of strong hydrochloric acid, and the iron is titrated with a weak bichromate solution, 1 cc. of which is equal to 0.2 per cent. iron. Or the precipitate is fused with sodium carbonate, the fusion is dissolved in hydrochloric acid and the iron titrated as above. The iron calculated as the sesquioxide, plus the phosphorus as pentoxide is deducted from the total weight of the precipitate, leaving the alumina.

Or the filter and precipitate are ignited at a low temperature until the carbon is destroyed. The precipitate is crushed in the crucible and thoroughly fused with about 5 grams of sodium carbonate. The fusion is dissolved in hot water, a little paper pulp is added to reduce any manganese that may be present, the residue is filtered off and washed. The filtrate received in a small beaker is acidified with hydrochloric acid, heated to boiling, and ammonia is added in slight excess. After boiling for a minute or two, the precipitate is filtered off, washed with hot water, ignited and weighed. This weight minus the weight of the phosphorus as pentoxide is alumina.

MANGANESE.

Gravimetric Method.

The filtrate from the one-gram portion for silica, or an aliquot part of the filtrate, equal to one gram of the ore, from the five-gram portion for silica in the volumetric flask, is boiled for a few minutes in a No. 5 beaker with the addition of 10 cc. of strong nitric acid. A basic acetate separation is then made as follows. The solution is diluted with cold water to about 150 cc. and the greater part of the free acid is neutralized with strong ammonia, then cautiously with dilute ammonia (1 part ammonia to 4 parts water) with vigorous stirring until a faint precipitate persists. The solution is then diluted with hot water to about 400 cc. 25 cc. of a 20 per cent. solution of ammonium acetate are added, the solution is heated to boiling and boiled about one minute. After the precipitate has subsided, the solution is filtered through a 185 mm. filter on a 4-inch ribbed funnel into a No. 6 beaker.

When the manganese is under one per cent., only one basic acetate separation is made and the precipitate is washed several times with hot water. If over one per cent., the precipitate is washed but once, then dissolved on the filter in hot, dilute hydrochloric acid and the basic acetate separation made as before, except that the dilution before precipitating need not be so great. The filtrate and washings, or the combined filtrates in this determination, or from the iron and alumina in the ether alumina method are heated to boiling and 25 cc. of strong ammonia added, and then cautiously about 1 gram of ammonium persulphate. One gram is sufficient to precipitate the manganese in any iron ore. The solution is boiled about ten minutes and the precipitate filtered off, washed with hot water, ignited at a gentle heat and then at the highest temperature of the muffle furnace or blast lamp, and weighed as mangano-manganic oxide, containing 72.05 per cent. manganese. This precipitate may be dissolved before or after ignition, reprecipitated, and weighed as manganese pyrophosphate.

The ignited precipitate of mangano-manganic oxide is usually contaminated with a small amount of silica sufficient to cause an appreciable error in a small percentage of manganese. In this case it is dissolved in the crucible in warm, dilute hydrochloric acid; the residue, if any, is filtered off, washed, burned and the weight obtained is deducted from the previous weight.

The precipitate of iron and alumina on the filter obtained in this determination is to be retained for the determination of alumina by the phosphate method.

MANGANESE.

Volhard's Method.

One to two grams of the sample are placed in a No. 2 beaker with cover, 15 to 25 cc. of strong hydrochloric acid are added, and heat is applied until the ore is dissolved. Five cc. of nitric acid or a few crystals of potassium chlorate are now added, and the solution is boiled until nitrous fumes or free chlorine are expelled. Evaporation is continued to the expulsion of free acid, the solution is diluted to about 100 cc. and washed into a volumetric flask. Zinc oxide emulsion is added in excess, the solution is diluted to the mark and mixed thoroughly; the precipitate is allowed to subside and an aliquot portion is decanted off, equal to one-half or one gram of the ore. For ores low in manganese, the solution is made slightly turbid with zinc oxide emulsion to coagulate the manganese dioxide set free in the titration. The solution is heated to boiling and titrated with permanganate until a faint, pink color persists. Or, after the dilution to about 100 cc., the solution is washed into a half liter flask, an excess of zinc oxide emulsion is added as before, the solution is heated to boiling and titrated directly in the presence of the iron precipitate.

Strength of permanganate solution for iron ores: 1 cc. equals 0.1 per cent. manganese, standardized on iron ore of about one-half per cent. manganese content.

For manganese ores, the procedure is the same up to the removal of the free acid and the addition of zinc oxide emulsion, when the solution is diluted to the mark in a 500 cc. volumetric flask and mixed. Then 50 cc. are transferred to a flask, diluted to about 250 cc., heated to boiling, and titrated as before. No zinc oxide emulsion need be added here, the voluminous precipitate of manganese dioxide acting as coagulant.

Strength of permanganate solution for manganese ores: 1 cc. equals 0.3 per cent. manganese, standardized on manganese ore of known manganese content.

MANGANESE.

Color Comparison or Titration Method.

One gram of the sample in a porcelain dish, or beaker, is warmed after the addition of 25 cc. of strong hydrochloric acid and 5 cc. of strong sulphuric acid, and when in solution, the heating is continued until fumes of sulphuric acid appear. The solution is allowed to cool, 25 cc. of water are added, heat is applied until the salts are dissolved and the solution is transferred to a 50 cc. volumetric flask, diluted to the mark

and mixed. If the color comparison method is to be used, the solution should be poured through a dry filter after mixing; if the titration method is to be used, the filtration may be omitted. With a pipette 10 cc., equal to 0.2 gram of the ore, are transferred into a 1-in. by 8-in. test tube, if for comparison, or into a 150 cc. Erlenmeyer flask, if to be titrated. Fifteen cc. of the nitrate of silver solution are added, the solution is heated over a flame or in a water bath, and about one gram of ammonium persulphate is added. The reaction takes place equally well in a nitric or sulphuric acid solution or a mixture of the two. The essential point is the presence of a sufficient amount of silver nitrate. As soon as the formation of the permanganic acid is well under way, the test tube or flask is removed from the source of heat and placed in a cold water bath. This solution may be compared against the color obtained from an ore or a steel of known manganese content, in the latter case by dissolving 0.2 gram of the steel in 10 cc. of nitric acid of 1.20 sp. gr., adding 15 cc. of the silver nitrate solution and the persulphate as above.

If the solution in the 150 cc. Erlenmeyer flask is to be titrated, it is diluted to about 100 cc., 7 cc. of a 0.2 per cent. solution of C. P. sodium chloride are added and the sodium arsenite solution is run in until the color is just discharged.

If the insoluble residue is suspected of containing manganese, it is filtered off, burned, 2 or 3 drops of sulphuric acid (1 part acid to 1 part water) and about 5 cc. of hydrofluoric acid are added and the solution is evaporated until sulphuric acid fumes appear; 5 cc. of the same dilute sulphuric acid are added, and the crucible is warmed till the residue is in solution, when it is added to the main solution.

The stock solution of silver nitrate is made in the proportion of 66.66 grams of the salt to the liter. Twenty cc. of this solution are diluted to one liter for use; each 15 cc. contains 0.02 gram of silver nitrate.

The sodium arsenite solution is made by boiling 10 grams of arsenious acid until dissolved in an aqueous solution of 30 grams of sodium carbonate and diluting to 1 liter. About 125 cc. of the stock solution are diluted to 2000 cc., and this solution is standardized on steels or ores of known manganese content so that 1 cc. equals 0.1 per cent. manganese on a 0.2-gram sample.

MANGANESE.

Peroxide Reduction Method.

One-half to three grams of the sample in a beaker are digested in 20 to 50 cc. of strong hydrochloric acid. If the insoluble residue is suspected of containing manganese, a few drops of hydrofluoric acid are added, and the solution is evaporated almost to dryness. Manganese-free glass must be used if hydrofluoric acid is employed. Seventy-five cc. of strong nitric acid are added, the solution is boiled until clear, the manganese is precipitated as dioxide by the addition of a slight excess of potassium chlorate, and the boiling is continued for about five minutes. The solution is cooled, diluted to about 300 cc. with cold water, and a definite amount of hydrogen peroxide solution is added from a pipette. After the reduction of the dioxide, the excess of hydrogen peroxide is determined by titration with standard potassium permanganate solution. The value of the peroxide solution in terms of permanganate is determined, and from the total volume of peroxide used, less the excess determined by the permanganate titration, the percentage of manganese is calculated.

The relative value of these two solutions is determined as follows: About 60 cc. of strong nitric acid are boiled for about five minutes in a beaker, then cooled and diluted to about 300 cc. To the cold solution, a definite amount of the peroxide solution is added and titrated with the standard permanganate.

The hydrogen peroxide solution is made by diluting one pound of hydrogen peroxide to 9000 cc., containing 200 cc. of strong sulphuric acid.

The permanganate solution contains 1.7375 grams per liter and is standardized against an ore of known manganese content.

LIME.

The filtrate from the persulphate or bromine precipitation of the manganese, after the basic acetate or ammonia separation of the iron and alumina, is heated to boiling in a No. 6 beaker, 10 cc. of strong ammonia and 5 cc. of a saturated solution of ammonium oxalate are added, and the heating is continued until the volume of the solution is reduced to about 150 cc. When the lime is low it is essential that the evaporation be continued to this point to insure complete precipitation. Five cc. of strong ammonia are now added and the solution is boiled for about ten minutes. The precipitate is then filtered off, washed with hot water, burned and weighed as calcium oxide. In normal ores when the lime and magnesia are low, only the first few washings are allowed to flow into the filtrate, the object being to keep the filtrate as concentrated as possible for the magnesia determination which follows. The calcium oxide is also liable to be contaminated with a small amount of silica and when extreme accuracy is desired, the ignited and weighed precipitate is dissolved in warm, dilute hydrochloric acid and the solution is evaporated to dryness. The residue is dissolved in dilute hydrochloric acid, the silica is determined and deducted from the previous weight.

If the lime is to be titrated, it is dissolved on the filter in 25 cc. of hot, dilute sulphuric acid (1 part acid to 4 parts water), the solution is diluted with 150 cc. of hot water and titrated with permanganate of such strength that 1 cc. equals about 0.100 per cent. lime.

MAGNESIA.

The filtrate from the calcium oxalate, which should not exceed 200 cc. in volume, is made slightly acid with strong hydrochloric acid, and 5 cc. of a 10 per cent. solution of ammonium phosphate are added, then to the cool solution, 25 cc. of strong ammonia, drop by drop, with continuous stirring during the addition. The solution is stirred at intervals for two or three hours, or continuously on a machine for one hour, and if permissible it is allowed to stand in a cool place over night. The precipitate is filtered off, washed thoroughly with dilute ammonia (1 part ammonia to 4 parts water), ignited at a low temperature until the filter is destroyed, and finally at the highest temperature of the muffle furnace or blast lamp, and weighed as magnesium pyrophosphate containing 36.24 per cent. magnesia.

SULPHUR.

Fusion Method.

One gram of the sample is intimately mixed in a capacious platinum crucible with about ten grams of sulphur-free

sodium carbonate, and not more than a half gram of potassium nitrate. The crucible is heated cautiously over a blast lamp until the fusion is quiet, the last few minutes of the fusion being at the highest temperature of the blast. If sulphur bearing gas is used, the contents of the crucible must be protected from the flame gases with a suitable shield. The fusion is completely disintegrated in hot water, to which a few drops of alcohol may be added to reduce and precipitate any manganese present. The solution is filtered and the residue washed with hot water, or preferably with a 1 per cent. solution of sodium carbonate.

To the filtrate, the volume of which is about 150 cc., there is cautiously added a slight excess of hydrochloric acid. The solution is heated to the boiling point, 5 cc. of a 10 per cent. solution of barium chloride are added, and the beaker is placed on the steam bath or hot plate until the precipitate has subsided. The precipitate is then filtered off, washed with hot water till free from chlorides, burned in an open crucible and weighed; 13.73 per cent. of the weight is sulphur.

If the precipitate is suspected of containing silica, which is rarely the case, it can be purified after ignition by adding a drop of sulphuric acid and 2 or 3 cc. of hydrofluoric acid, evaporating and igniting as usual.

If sulphur-free sodium peroxide is used for the decomposition of the ore in place of the sodium carbonate and niter, the fusion is made in a nickel crucible with about 5 grams of the peroxide; the remainder of the treatment is the same.

SULPHUR.

Wet Method.

Five to ten grams of the sample in a porcelain dish or beaker, with a watch glass cover, are dissolved in 50 to 100 cc. of aqua regia (1 part nitric acid to 9 parts hydrochloric acid) and the solution is evaporated to dryness. The residue is dissolved in 20 to 40 cc. of strong hydrochloric acid and the solution evaporated until the first appearance of insoluble ferric chloride. This is dissolved by the addition of a slight excess of strong hydrochloric acid, the solution is diluted to about 75 cc., and filtered into a suitable beaker, the residue being washed with hot water and the least possible amount of dilute hydrochloric acid.

If the insoluble residue is suspected of containing sulphur compounds insoluble in aqua regia, it is fused with the least possible amount of sodium carbonate and niter, as previously described in the Fusion Method for Sulphur. The fusion is disintegrated in hot water, the solution is filtered, acidified with hydrochloric acid and added to the main filtrate, or the sulphur may be estimated separately.

The filtrate is heated nearly to boiling, and after the addition of 5 cc. of a 10 per cent. solution of barium chloride it is again evaporated until a film of ferric chloride appears. This is dissolved by the addition of a few drops of strong hydrochloric acid and, with a wash bottle, a stream of cold water is forced into the solution until its volume is from 175 to 250 cc. It is then set aside in a cool place over night. The precipitate is filtered off, washed with hot water and dilute hydrochloric acid until free from iron, burned and weighed as usual.

IGNITION LOSS.

One gram of the sample is placed in a platinum crucible with a tightly fitting cover and ignited at a bright red heat for fifteen minutes. It is then allowed to cool in a desiccator and weighed. This is repeated with five minutes' ignition until constant weight is obtained.

MOISTURE.

A portion of the entire sample reserved for the moisture determination, in either case not less than two kilograms, is transferred to a suitable pan and weighed, dried at 100° C. until constant weight is obtained and the percentage of moisture calculated.

REPORT OF THE COMMITTEE OF THE AMERICAN CHEMICAL SOCIETY APPOINTED TO CO-OPERATE WITH THE NATIONAL CONSERVATION COMMISSION.

In May, 1908, a meeting of the governors of the different states was held at the White House in Washington to consider the conservation of our rapidly wasting natural resources. Following this meeting, a commission was appointed by the President of the United States to investigate the subject, and the principal scientific societies of the United States were invited to coöperate with it. The committee of the American Chemical Society, appointed in response to this invitation, now has the honor to submit the following preliminary report.

On December 8, 9, and 10 the National Conservation Commission met in Washington in joint conference with the delegates of other organizations and the governors of more than twenty states. The commission, in its elaborate investigations, had, so to speak, taken stock of our natural resources, and its report, therefore, was essentially statistical in character. It had estimated the magnitude of each particular resource, and had studied the rate of consumption of such substances as lumber, coal, iron, etc. It discussed the wastage of the land by preventable erosion, and its effects not only upon agriculture but also in reducing the navigability of streams. Questions like these were treated at considerable length, and their general character is all that need be mentioned just here. The data of the commission were mainly classified under four headings, namely, minerals, forests, lands, and waters, and under each one the evils to be remedied were pointed out with all the emphasis and clearness which the statistical method of investigation made possible. The commission cleared the ground for study into the prevention or limitation of future waste; and the problem of conservation can now be taken up in a more intelligent manner than has been possible hitherto. We now know better than ever before what the evils and dangers really are; the next step is to discover remedies, and then, finally, to apply them. The public attention has been aroused; the people of the country are awakening to the necessity of greater prudence and economy in the use of our resources, and definite lines of action can now be laid down with a reasonable probability that they will be followed. Fortunately, the reports of the commission are neither sensational nor unduly pessimistic; the results of their conferences are presented seriously, and in such a manner as to compel consideration; they are therefore all the more likely to produce permanent effects of great benefit to the American people. The utterances of the mere alarmists rarely carry conviction, but disclosures like these made by the Conservation Commission can not be disregarded.

Up to the present moment chemistry has had little to do with the investigations of the commission. Henceforward the chemist must be called upon in many ways, for the waste of resources is often preventable by chemical agencies. Chemistry has already done enough to prove its potency, and its influence is felt in every branch of industry. Adopting the classification of the commission, we shall find the chemist active under every heading. Under minerals, we must note that metallurgy is essentially a group of chemical processes by which the metals are separated from the ores, a separation which may be either wasteful or economical. Within recent years, within the memory of members of this society, the available wealth of the world in metals has been enormously increased. By the cyanide process for extracting gold, ores are now profitably worked which were formerly worthless, and at the same time the demand for mercury has been decreased. The Bessemer process for steel-making, now also modified for use in copper smelting, is purely chemical, and its later modification, the Gilchrist-Thomas process, applies similar principles to phosphatic ores, which were previously of little value. Furthermore, in the last-named process, phosphatic slag is produced, which is useful as a fertilizer and helps to relieve the drain upon our rapidly wasting supplies of phosphate rock. Chemists are now studying, with much success, the problem of preventing corrosion in iron, a research which will prolong the life of iron structures and thereby reduce the waste of ore. The use of coal slack by briquetting methods is largely based upon chemical investigations; the salvage of by-products from coke ovens, such as tar and ammonia, is wholly due to chemical research; coal is further economized by the study of boiler waters and the consequent prevention of boiler scale. Even inferior coals, lignites, are now converted into what is known as producer gas, and so are transformed into the best kind of fuel. Petroleum is refined by chemical means, and every fraction of it is saved, either as illuminating oil, as gasoline, as a lubricant, as vaseline, or as paraffin. These are all notable achievements, but greater are yet to come. Enormous quantities of valuable substances are thrown into the atmosphere in fumes from smelters, which should, and probably can be, partly saved. Electrochemistry is rapidly developing a large group of new industries, making such metals as aluminum, magnesium, and calcium available for use, and it is reaching out into other fields of electrometallurgy in which electric heat, generated by water power, will be used for smelting other metals, thereby reducing the consumption of coal.

In forestry also, the influence of the chemist is distinctly felt. The sprays, used for destroying noxious insects, are chemical preparations. The manufacture of wood alcohol is a chemical process, which may be either wasteful or economical. Turpentine is now produced wastefully, but the waste can be diminished by careful refining, and furthermore, the chemist can aid in discovering substitutes for it. Substitutes for tan bark are also to be sought for by means of chemical investigations. Another distinctively chemical operation is the preparation of wood pulp for paper-making, a process which is now wasteful in the highest degree. It s estimated that for every ton of pulp now made by the sulphite process, more than a ton of waste material is allowed to drain away into our streams. How to make this material useful is a chemical problem, and so also, in great part, is the investigation of other, now useless fibers, which may replace the more valuable wood. The preservation of wood from decay is still another art in which chemistry is predominant.

In preserving the fertility of our land, chemistry has an important part to play. Our knowledge of fertilizers, of the food on which crops can thrive, is entirely chemical so far as accuracy is concerned, and must be applied in accordance with chemical principles. A fertilizer which is useless, and therefore wasted on one soil, may be needed on another. Certain fertilizers, like the Stassfurt salts, Peruvian guano, the Chilean nitrates, and phosphate rock are limited in quantity, and their future exhaustion must be considered now. What shall replace them in the future? Already processes have been devised for fixing the nitrogen of the atmosphere and rendering it available for plant food. Saltpetre and other nitrates can be and long have been made from waste materials such as old mortar and animal refuse. The phosphatic slags have been mentioned in connection with metallurgical processes. These sources of fertility are important, but greater still is the source found in our municipal sewage. The problem of its salvage has been worked out in some localities, but in the United States the people are only beginning to be aroused to its importance. Enormous masses of material, easily available for fertilizing purposes, now drain into our rivers or directly into the sea. Another question, now under investigation, is the possibility of using our common feldspathic rocks in fine powder, to replace the potassium withdrawn by plants from the soil.

The relations between the chemical composition of water and the conservation of natural resources are of intimate and fundamental importance and some of them have been mentioned under other headings. The rate at which the land surface of the United States is being transported to tide water has recently been estimated by means of chemical analyses of river water coupled with determinations of stream flow, and the results of the computations will doubtless assist considerably in studying soil erosion and the impoverishment of agricultural lands. In steam making the chemical quality of the water supply is an appreciable factor in fuel consumption, a subject to which reference has already been made. The scale that forms on the boiler shell and tubes, when water containing incrustants is used, is a poor conductor of heat and, consequently, causes increased expense for fuel. By detailed study of the chemical composition of available boiler waters, it is possible to select a supply having a minimum amount of incrusting, corrosive, and foaming constituents, thereby effecting appreciable economy in fuel. Chemical investigation of methods for purifying water supplies, not only for boilers but for paper manufacture, soap-making, and other great water-consuming industries, will enable manufacturers to make new and greater saving in many raw materials other than fuel.

Stream pollution by industrial refuse and by sewage is a source of enormous waste in our natural resources. The subject has been for many years a field of research for industrial, sanitary, and biological chemists in the United States, and their investigations have resulted in the improvement of manufacturing processes, the utilization of wastes, the purification of sewage, and the protection of domestic water supplies. When the presence of deleterious

substances in our river and lake waters has caused loss of fish life and the destruction of oyster beds, the chemist and the biologist have detected the harmful ingredients and have suggested methods for their removal. River silt, an important source of detriment to navigation, is also estimated by the chemist. It has been fully demonstrated that the prevention of stream pollution lies not alone through injunctions and other legal prescriptions but also in using waste materials or, when that is not possible, in rendering them harmless. The chemist has much to do in protecting and preserving the quality of our water supply. Upon that, in very great measure, depends the preservation of our highest resource, human life. Polluted waters distribute typhoid fever and other dangerous diseases, and so cause losses which should be, and really are, preventable.

The foregoing illustrations are enough to show, for present purposes, the intimate connection between chemistry and the study of conservation. They also bring out the fact that the classification adopted by the National Commission, although admirable for statistical research, is not final, and that it needs to be supplemented by a different subdivision of the data. The facts to be investigated often fall under more than one heading of the classification, and actually interlock in every conceivable manner. To operate a placer mine, for example, abundant water is needed, while a deep mine requires timber for its shafts and levels. In building and occupying a house one covers land, uses lumber, brick, stone, and iron, introduces water supply, and burns fuel. In short, every phase of the conservation question affects the interests of everybody. If the investigation of our natural resources is to be made effective, it must be applied to individual industries, and in order to do that another scheme of classification would seem to be necessary. Such a scheme we venture to outline, but very briefly.

At the outset the problem can be divided into two parts, one relating to sources of energy, the other to material substances. The two are not really separable, but may advantageously be considered separately.

In the first place, the energy available for industrial uses may be classified under three heads, as follows: First, inexhaustible energy, such as solar radiation, wind power, tidal power, and, with certain limitations, the power furnished by flowing streams. Second, reproducible or renewable energy, like the power supplied by horses and other domestic animals. Wood, regarded as fuel, also falls under this heading, for forests can be artificially grown. Third, the exhaustible energy represented by mineral fuel, like natural gas, petroleum, and coal, which, once used, is gone forever. Under this classification the practical problems are, to economize the exhaustible energy, to encourage the development of renewable energy, and to discover new methods of using the inexhaustible energy.

Exactly the same classification applies to material substances. Some, like sea salt, limestone, and clay are, humanly speaking, inexhaustible. Agricultural and forest products are reproducible, some of them year by year. The metallic ores and such useful minerals as phosphate rock are, however, exhaustible, and need to be conserved.

With the aid of this very simple classification it becomes possible to analyze a specific industrial problem in such a manner as to make evident its factors of waste or economy. For example, sea salt is inexhaustible, and may be extracted by solar evaporation, which is a use of inexhaustible energy. Agricultural products are renewable, and their production chiefly requires the renewal energy of men and animals. But the smelting of metallic ores, as now conducted, involves the use of exhaustible material both as ore and as fuel.

In most industries, however, the two sets of considerations are combined. Portland cement, for example, is made from inexhaustible substances, but is burned with exhaustible fuel. The latter factor in the industry, therefore, is the one to be carefully considered, while the first factor is negligible. Taking industry by industry we shall find that this condition of affairs is general, and that each one must be studied by itself with reference to its inexhaustible, reproducible, and exhaustible elements. In doing this a clear notion can be obtained as to the real needs of a given industry, and our attention can then be concentrated upon those features of it which particularly demand economy. We shall be able to locate evils with greater accuracy; to diagnose the industrial diseases, so to speak, and then to look intelligently for remedies. Many of the remedies must be sought for along chemical lines of research, which will develop economical processes of manufacture, utilize materials that are now wasted, or substitute cheap for costly substances. Cheap and costly, however, are words which need qualification. A substance or a process which is cheap to-day may be in reality wasteful with a temporary reduction in price at the cost of some permanent economy. For our purposes the two words imply a deeper discrimination than is carried by their ordinary use. Temporary efficiency and cheapness are to be discountenanced, while permanent economy for the benefit, not only of the nation but of the whole human race, is to be encouraged. This principle is sound, but its practical applications will involve many difficulties, and develop many conflicts with special interests. Like all ideals it can not be realized absolutely, but it represents a standard of action towards which we must move, even though the ultimate goal of perfection may never be attained. Evils can be mitigated, although they may not be entirely removed.

The American Chemical Society now numbers more than four thousand members, scattered through all the states and territories of the union and represented in every one of our great productive industries. These chemists are at the same time progressive and conservative in their work, for they are both discovering new utilities and protecting old ones from loss. We believe that every member of the organization is necessarily in sympathy with the great forward movement for economy, and that in our society the National Conservation Commission will find a most powerful and willing ally.

Signed, F. W. Clarke,
H. W. Wiley,
C. H. Hertz,
S. W. Parr,
R. B. Dole.

LIGHT NAPHTHA AS A TURPENTINE ADULTERANT.

Mr. A. E. Paul states in his very interesting article on "Turpentine and its Adulterants," in the January number

of this Journal, that very light naphtha is never employed for purposes of adulteration.

I would say that I have come across several samples of so-called turpentine—sold as "pure"—which contained a high percentage of light naphtha, otherwise "gasoline."

R. H. ACOTT.

A PECULIAR CHLORATE EXPLOSION.[1]

A short time ago Dr. McKenna read a paper before this section on the hazard of explosives in which he gave account of several explosions caused by chlorate of potash. Since then several unusual cases of this kind have come to my notice. The facts as reported to me are as follows: A company in this city, makers of small castings, has been purchasing kegs from a second-hand dealer. These were used for packing castings for shipment.

In the first case some of these kegs containing ingots of aluminum and zinc were being unloaded on a wharf when a keg took fire. The laborers supposed the kegs contained grains of powder and wanted to throw the entire lot into the river. The owners of the foundry could not account for the fire but thought some mysterious property of the zinc and aluminum might have caused it. No further investigation was made in this case.

A few weeks ago a shipment of small iron castings was made to a neighboring city. When unloading one of the kegs from a truck, a slight explosion occurred; two men were slightly burned while the keg burned rapidly enough to heat the castings quite hot. On investigation it was discovered that this keg had been obtained from a dye works and had contained chlorate of potash. The second-hand dealer claimed this lot of kegs had been thoroughly cleaned before delivery to the iron works but however that may have been either some chlorate had lodged in the crevices and escaped detection or else the wood had become saturated with it from standing in a damp place. The jolting of the castings struck a spark which fired the mixture of chlorate and wood.

Chlorate of potash is used by dyers and in this vicinity; where there are many dye houses, there must be a large number of these kegs to be disposed of in some manner.

Fortunately all the fires of this kind of which I am aware occurred in the open air and did no damage. They might just as easily have occurred in a warehouse or on shipboard. In fact the foundry using these kegs, ships castings abroad and it might be a very serious matter to have a fire of this sort in the hold of a ship.

Evidently purchasers of second-hand kegs have another danger to be guarded against, and we have another possible cause for fires of mysterious origin. GEORGE C. DAVIS.

A NEW COMBUSTION PIPETTE.

My experience of several years in gas analysis has led me to construct a new form of combustion pipette that I have found superior to any other form I have yet used. My duties as Gas Analyst of Sioux City have afforded me ample opportunity to test this apparatus, and it is with assurance that this form will be of interest to those engaged in gas analysis and of decided practical value that I bring the pipette to public notice.

[1] Read before the Philadelphia Section, American Chemical Society.

The combustion tube—see figure— resembles the familiar Hempel burette with the exception that the cylinder is open to the full diameter of the tube at the base. This opening is closed by means of a stopper through which

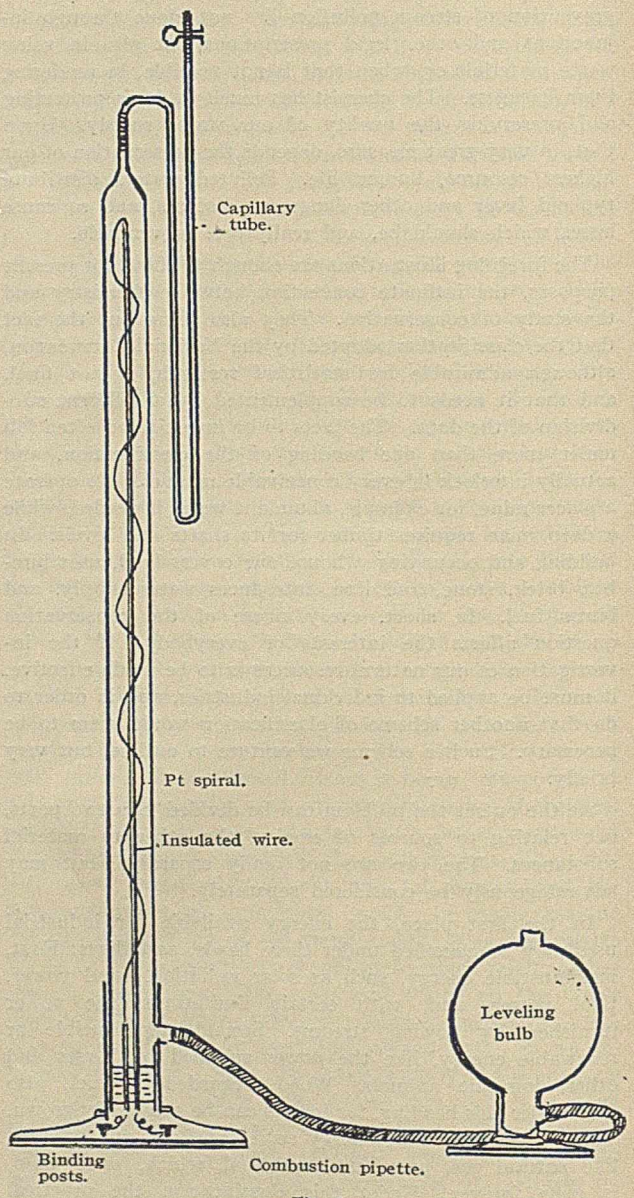


Fig. 1.

pass insulated wires connecting to a platinum spiral extending the entire length of the tube. One length of the heavy wire is insulated the full length of the cylinder to prevent a short circuit by the confining liquid, mercury. Glass insulation has been found to be the best. The lower ends of the wire pass through the weighted base to binding posts, which in turn may be easily connected to a battery. A leveling bulb connects to the tube by a heavy rubber tube.

Among the advantages of this apparatus over the old

forms may be mentioned the following: It can easily be cleaned. The platinum spiral extending the full length of the tube insures complete combustion. This spiral can readily be removed for repairs in case of injury. The apparatus is convenient to handle and owing to its tall form connects with a Hempel pipette without the use of a stand.

The apparatus can be obtained from Messrs. E. H. Sargent & Co., Chicago. WILFRED W. SCOTT.

DETERMINATION OF INSOLUBLE PHOSPHORIC ACID.[1]

Ever since the organization of the Association of Official Agricultural Chemists the Official method for the determination of insoluble phosphoric acid has been confined to the one process; that is, digestion in neutral ammonium citrate for a definite length of time at a stated temperature. From time to time some slight modifications have been proposed, but in the main, the method remains practically the same as it was twenty-five years ago. One difficulty which has been experienced, is to make an ammonium citrate which is exactly neutral, owing to the indefinite action of the solution on different indicators. The "official" method prescribes corallin as the indicator to be used in testing the neutrality of the solution. Any one who has tried this indicator knows how unsatisfactory it is, and it is safe to say that there is not one chemist in ten who depends upon it for the final test. The optional method provides for first neutralizing with corallin as the indicator, and making the final test with cochineal after precipitating out the citric acid with calcium chloride. This procedure is also unsatisfactory, and as Dr. McCandless, referee of phosphoric acid for the A. O. A. C. for the past year, truly said: "If a chemist has succeeded in getting his solution neutral or practically so, he will almost certainly get his out of joint by attempting to make it exact with calcium chloride solution." The result is that different chemists employ different methods for standardizing the ammonium citrate solution, and hardly two solutions will be found of exactly the same neutrality. Instead of prescribing two methods which may give entirely different results the A. O. A. C. should prescribe one method which can be easily worked and would prove satisfactory to a majority of the chemists who are engaged in fertilizer work. Dr. McCandless favors making the solution neutral by analysis, and calculating the exact quantity of ammonia or citric acid to be added. A citrate solution which is neutral by analysis is distinctly alkaline by the ordinary indicator, and while it would be quite a departure from the Official method, it would at least insure a greater degree of uniformity than it is possible to obtain under the present methods. It is fortunate that a slight deviation from strict neutrality has no appreciable effect in the ordinary acidulated goods, but in cases of bone, tankage, etc., it is very important that the solution should be neutral.

There is another point that seems to have been overlooked in carrying out this method which causes quite wide variations in results by different analysts; that is, the manner of filtration after digesting with ammonium citrate. I have investigated the question to some extent, and find the following methods of filtration in use in different laboratories. Through asbestos in Hirsch funnel with filter pump; through asbestos on carbon filter with filter pump; through filter paper on Hirsch funnel with filter pump; through filter paper in Buchner funnel with filter pump; through filter paper with regular funnel and platinum cone using filter pump; through folded filters without filter pump.

The filter papers used in the above vary from very thin to dense slow filtering papers. Experiments which we have made by these different methods show that the results will vary on some samples of acid phosphate having a high percentage of insoluble from ½ per cent. to 1 per cent. and on such materials as ground bone, tankage, reduced phosphate, basic slag, etc., it is probable that there would be much larger variations.

The exact wording of the "official" method with reference to the filtration is as follows: "At the expiration of exactly thirty minutes from the time that the filter and residue are introduced remove the flask from the bath, and immediately filter the contents as rapidly as possible." Now if this were amended so as to specify the exact method of filtration and the kind of paper to be used, much of the confusion in results would be avoided. Taking everything into consideration, this method if properly executed, serves the purpose for which it was intended in a very satisfactory manner, and these few comments are presented with the hope that this division will take up the question and evolve a method which will not only be acceptable to the fertilizer chemists, but will be adopted by the Association of Official Agricultural Chemists. F. B. CARPENTER.

[1] Paper read before the Division of Fertilizer Chemists at Baltimore, December 31, 1908.

COUNCIL ON PHARMACY AND CHEMISTRY, REGULATIONS RELATIVE TO NEW AND NON-OFFICIAL REMEDIES.

The last meeting of this Council of the American Medical Association was devoted to the discussion of the revision of the rules, and rearrangement of the matter contained in the "New and Non-Official Remedies." It was decided that this book shall contain descriptions of the proprietary articles accepted by the Council and of such simple non-proprietary and unofficial substances as are of sufficient importance. It was decided that proprietary mixtures unless they show originality and a marked advance over similar products, shall not be included in the main body of the book, but when they conform to the rules, they shall be included in form of an appendix to the book.

Rule 5 was so amended as to require that the actual identity of the manufacturer of a product be furnished. The Council voted to interpret Rule 8 so that after January first, 1909, pharmaceutical preparations and mixtures will be admitted only under a pharmaceutical title which shall indicate the most potent ingredients. Arbitrary coined titles will not be recognized for pharmaceutical mixtures. It was also decided that no pharmaceutical mixture shall be accepted whose name indicates its therapeutic action or is suggestive of the names of diseases or pathologic conditions in which it is to be used. After January first, 1909, this rule is to be extended to simple articles.

The Council voted to condense Rules 9 and 10 to become Rule 9 and adopted a new Rule, No. 10, under which recognition will be refused to articles which, because of their unscientific composition, are useless or inimical to the best interests of the public or of the medical profession.

These rules represent a material progress made in the gradual elimination of deception and fraud which has been in practice, without restraint, for many years. During recent years, with the great influx of newer remedies, there has appeared in the European and American markets, a large number of preparations, so named and labeled as to suggest either their therapeutic action, or similarity to other established true synthetics. These preparations, being mechanical mixtures of well-known therapeutic agents, are therefore calculated to deceive the medical profession, and considering the persistence with which they are pushed, it is high time that concerted action be taken by chemists, physicians and pharmacists on both sides of the Atlantic, in order to control this nuisance. V. COBLENTZ.

QUOTATIONS.

THE OXIDATION OF ATMOSPHERIC NITROGEN.

By DR. OTTO SCHNEIDER.

(Lecture delivered before the Verein Deutscher Chemiker, 1908; Authorized translation—Extracts.)

In the process of the Badische Anilin & Soda Fabrik, electric arcs produced by powerful currents and of a length never before attained are used. But these arcs burn very quietly, and special care is taken to displace them as little as possible by the motion of the air by causing the air to flow along the arc in the direction of its length. This way is so entirely different from the traditional one that there was no lack of dissenting voices who called in question the practical utility of our method, and curiously enough, by giving diametrically opposite reasons. Some said that this method of conducting the air did not allow it to come sufficiently in contact with the arc, while others claimed that it remained in contact too long.

Well, in the meantime we have demonstrated that our process can compete successfully with any other. The principle of the process consists, as we have already indicated, in conducting the gases to be acted upon along a quietly burning electric arc of great length, in surrounding, as it were, the electric arc with an advancing mantle of gas. The apparatus with which the operation is conducted is as simple as the principle itself. It consists essentially of a tube in the axis of which the electric arc is quietly burning surrounded by a mantle of the gases which are to be combined. The tube itself and the motion of the air in it must be so arranged that any one-sided motion in the direction of the arc is avoided as perfectly as possible, while it does no harm if the air comes from two opposite sources or is introduced towards the arc through a slit with equal pressure from all directions at once because this does not disturb the position of the arc.

Now, there is one kind of motion which satisfies this condition particularly well. For this reason we have used it almost exclusively in our method on a large scale. It enables us to obtain arcs of enormous length, which at the same time are so stable that they can burn for weeks without having to be relighted. This is a motion of the air which in spite of the greatest rapidity permits that portion of the column to remain at rest in which the arc is situated. This kind of motion is familiar to you all, it is the spiral or vortex motion. You all know with what devastating power cyclones visit certain regions of the earth with terrifying regularity. They move like a solid mass across the country for miles laying low everything before them. Nothing is able to withstand the impact of this whirling air and yet they carry in their centre a region of calm.

We have utilized this phenomenon. We generate such a vortex in miniature in our tubes and in its calm centre we maintain our electric arc. The particles of the air rush along the arc in a rapid screw-like motion and are combined in the desired way. In this way electric arcs of almost any desired length may be kept burning quietly.

The tube may be made of any material. We take for example an ordinary iron gas-pipe. An electrode insulated from the pipe is attached on the inside at one end and the air is introduced at this end. The insulated electrode is connected with one pole of a high tension current and the pipe itself with the other pole. The whirling motion of the air is produced in the simplest possible manner by tangential introduction. The arc is then lighted in any desired way. I will not mention the different ways in which this can be done, as they are well known to every electrician.

It is immaterial which way is chosen in our process, because, as we have already stated, the arc when once lighted can be maintained for weeks without relighting. In the simplest form the arc may be started by making the distance between the pipe and the electrode so small that it will be overcome by the tension of the interrupted circuit. As every electric arc must be provided with an inductive resistance which is indispensable to produce quiet burning and which is present in every ordinary alternating current arc lamp, the tension between the electrode and the pipe is diminished by a certain amount as soon as the arc is lighted and with this diminished tension the current will not jump across again as long as the arc continues to burn. If it should go out for any reason, a new arc will be formed of itself because of the renewed increase in the tension. With this method of forming the arc the air enters the tube below the electrode and pushes one end of the arc, which is quite short at first, rapidly before it along the wall of the pipe. As soon as stability is reached, which comes to pass so quickly that the eye is hardly able to follow the process, the result is an arc which burns quietly in the axis of the pipe, its end being far from the electrode attached to the centre. This end is at that point of the wall of the pipe where the gases have become so hot as to acquire sufficient conductivity for the current to jump across them. The location of this point depends entirely on the conditions adopted. Of course the place where the arc terminates must be cooled off by means of water or in some other suitable way.

The method of starting the arc which has just been described requires a very narrow space between the pipe and the electrode. A certain pressure is used to make the air which is introduced pass through the narrow space. This inconvenience is avoided by introducing the greater part of the air above the electrode, or by increasing the space, and producing in some way a brief temporary short circuit between the pipe and the electrode, as for example by means of a rod introduced from the outside, or an induction spark or a slender flame and the like. It does not matter because the arc when once lighted properly keeps on burning quietly, as has been stated, without going out. The tube may also be made of non-conducting material, such as fire-clay for example. In that case the arc must be

started by a temporary approach of the second electrode or by the use of a conductor along the wall of the tube to bridge over the space between the electrodes.

You see here three small furnaces with glass tubes constructed to illustrate the lecture. In two of these tubes there is placed a wire wound spirally and connected with the upper metallic post of the apparatus which forms the second electrode. The object of using the spiral form is to make the wire lie fimly against the wall of the tube. At the lower end this wire is separated by a very small space from the electrode situated in the axis of the tube, so that an arc may be formed there in consequence of the tension existing in the circuit. This apparatus illustrates the case where the tube is made of metal. We may imagine that the wire is all that is left of an original entire wall of metal. If the apparatus were entirely of metal you would not be able to see the rapid upward course of the electric arc. I now admit the air with a certain velocity through a tangential attachment below the spot where the electrode and the wire are separated by a short distance and connect the lower electrode with a high tension current. You will immediately see one end of the arc climb up along the wire and as a result an elongated arc burning quietly like a candle or a gas flame. And it is, in fact, a gas flame that you see, it is burning nitrogen. The arc proper, that is to say, that portion in which the electric current chiefly passes, is much slimmer than what you see—a fact which can be shown by passing a single gas, for example nitrogen alone, through the apparatus. The slight irregularity in the shape of the arc is due to the fact that the air flows through the tube with a simple screw motion. If the air were admitted through several openings or uniformly through the whole circumference, the arc would form a perfectly straight line. Please observe that the arc does not touch the wire while burning, but only when it is started.

In the other apparatus there is no wire inside the tube. Here I must start the arc by bridging the distance between the two electrodes by momentarily connecting them with a wire. The arcs in the small apparatus consume about 4 amperes at 3000 volts or in round numbers 16 h. p. of electric energy. You see the resulting product in the flasks of the apparatus in the form of a yellowish brown vapor.

As is well known, the first product in the electric arc is colorless nitrogen oxide which then oxidizes further to NO_2 on cooling in consequence of the excess of oxygen present. In maufacturing on a large scale these nitrous gases are first used for heating steam boilers and when they have been cooled in this way they are passed through percolating towers with water, where they are converted into nitric acid which is used to dissolve limestone. In this way a solution of calcium nitrate is obtained which is evaporated in a vacuum by means of steam. This lime saltpetre is equal to Chile saltpetre in every respect as a fertilizer and even superior to it for soils poor in lime.

After the details of our furnaces had been tested in the years 1905 to 1907 in Ludwigshafen, we started an experimental factory of 2000 h. p. last autumn in Christianssand, South Norway, in order to test our furnaces by long-continued operation. Three furnaces are now in continuous operation in Christianssand. The length of the flame is about 5 meters and their energy about 600 h. p. We have run one furnace temporarily with 700 kilowatts or approximately 1000 h. p. These enormous electric arcs are kept burning just as easily and surely as the much smaller ones I have shown you here. This is the more surprising when we consider that an alternating current of 50 periods is equalized in them, that is to say, that the current ceases 100 times per second when it passes zero and then flows through its long path again in the opposite direction without requiring the arc to be relighted. These manufacturing furnaces, to be sure, look rather different from the lecture apparatus exhibited here. In them the air is systematically heated by the hot discharge gases to about 500° C. before its entrance in the electrode pipe proper. The principle, however, is the same after all. To obtain a good result the pipes must have the right dimensions, the air must be supplied uniformly and the upper part must be well cooled. All this is easily accomplished in our furnaces, as you see. These furnaces are very durable. The only part subject to more rapid wear is the lower electrode and this we cool specially with water in our industrial furnaces. In its simplest form, as in the lecture apparatus before us, it may consist of an iron rod which may be moved forward for adjustment, but other materials may also be used for electrodes. Conductors of the second class, as for example zirkon oxide, may also be employed.

Our process differs from all other known methods by its excellent utilization of the electric energy and at the same time by yielding a gas of relatively high concentration which essentially facilitates absorption. The apparatus is extremely simple and cheap. There are no movable parts, expensive magnets or the like, and as the arc burns quietly the factor of electric efficiency is high.

It will not be long before saltpeter factories will spring up wherever circumstances permit. I suppose it is generally known that we have combined with the owners of the Birkeland-Eyde process in order to work together. For this purpose factories with 120,000 h. p. are in process of construction in Norway. It is true that the new industry is limited by the presence of very cheap sources of power, but for this very reason it is doing pioneer service by opening to industry regions which might have remained dormant for many decades if it had not been for the sudden demand for great quantities of cheap energy.

THE PRODUCTS OF THE BADISCHE ANILIN- AND SODA-FABRIK.

(From *The Badische Book*, 1908.)

The enormous progress and changes which have taken place in the course of the past century in industry and commerce, are due, to a great extent at least, to the utilization of the energy stored up in coal. * * * Chemical science and the art of the manufacturer have succeeded in producing from coal an unlimited series of valuable dyes, and by this meansh ave brought into existence a flourishing industry.

It is but little more than thirty years ago that dyers were entirely dependent upon the so-called natural coloring matter obtained from plants and animals, or prepared from minerals, from metals or from earths. The introduction of dyes derived from coal tar led to a complete change in the dyeing industry. * * * This brilliant success must be attributed

principally to the intimate connection between factory work and scientific investigation.

The history of the development of the coal-tar color industry is intimately associated with the history of the Badische Anilin- & Soda-Fabrik. At first this company only manufactured the few aniline dyes which were then known, such as Magenta, Aniline Blue, etc.; but in course of time a large number of the most important discoveries of science were utilized and the range of manufacture continually widened. * * *

In the manufacture of gas and coke from coal, tar is obtained as a by-product. From the tar, by distillation, benzene, toluene, xylene, naphthalene and anthracene are obtained, and these, together with gallic acid, a product of the vegetable kingdom, are used by the company as the initial materials for the production of dyes.

From the beginning, the company has worked upon the principle of itself producing all the materials used as reagents in working up and converting the initial materials into dyes. This results in the manufacture of so-called "heavy chemicals," the products of the acid and soda industries, *viz.*, sulphuric acid, sulphuric anhydride, hydrochloric acid, nitric acid, soda, chlorine, chromic acid, etc. For this purpose iron pyrites, salts, limestone, saltpeter, chrome ore, etc., are employed. The company can also claim to have enriched this branch of industry with a number of valuable achievements, especially by the method of the manufacture of sulphuric acid without lead chambers (1889), which later became of such epoch-making importance and caused a complete revolution in the sulphuric acid industry; the manufacture of liquid chlorine (1888), and recently by the industrial manufacture of stable solid sodium hydrosulphite, which is used for discharging and bleaching and also in the preparation of the indigo vat. Rongalite, which is employed in discharge printing, is derived from it.

The company has for a number of years past availed itself of the achievements of electro-chemistry in making chlorine and caustic soda by the electrolytic process.

The first important success of the Badische Anilin- & Soda-Fabrik in the manufacture of organic coloring matters was the commercial production of alizarine. In the year 1868 Graebe and Liebermann succeeded in preparing alizarine, the important coloring-matter of the madder root, from a hydrocarbon, anthracene, contained in coal-tar. The Badische Anilin- & Soda-Fabrik worked with the discoverers in order to formulate and establish a rational process for the commercial manufacture of this dye, and this object was achieved in the year 1869. By means of this process, artificial alizarine has since been manufactured. The production of this coloring-matter has increased enormously and has proven to be of very far-reaching importance to the industry.

The maufacture of alizarine soon resulted in the introduction of important derivatives therefrom; among these may be mentioned in the order of their importance: Alizarine Blue (1878), and especially the soluble form of the same dye, Alizarine Blue S (1882), Alizarine Green and Alizarine Indigo Blue (1888), Anthracene Blue (1891), Alizarine Orange (1875), and Alizarine Maroon (1885). Closely connected with these colors produced from anthracene are some derivatives of gallic acid, namely, Galleine Coeruleine (1878), Anthracene Brown (1886), and Galloflavine (1886); and in the year 1887 the series of mordant dyeing coloring agents was rounded off by the manufacture and introduction of the fastest black coloring-matter—Alizarine Black, which is a derivative of naphthalene. * * *

A new important group of anthracene colors, whose dyeing properties indicate them to be acid-coloring agents of the anthraquinone series and which are distinguished by their eminent fastness, was introduced a few years ago. To these belong Anthraquinone Blue (1901), Cyananthrol (1902), Anthraquinone Violet (1902), Anthraquinone Green (1904). * * *

The Eosines were discovered in the year 1874 and were the first dyes of the resorcine class, to which belong also the Rhodamine dyes discovered in 1887. Methylene Blue, discovered in 1877, is one of the most valuable dyes for cotton. In the same year Acid Magenta, in 1878 Naphthol Yellow S, and in 1879 the first Acid Green, Light Green S, were discovered, and they are to this day absolutely essential in wool dyeing.

In the year 1883 the industry was greatly enriched by the introduction of coloring-matter syntheses effected with the aid of phosgene. The research work in the new region thus opened up led at once to the discovery and manufacture of Crystal Violet, Victoria Blue, Acid Violet, Alkaline Violet and Wool Green, and especially of the most important dye, Auramine, which is largely used in dyeing cotton, paper and leather.

In the Induline class of dyes Acetine Blue has been manufactured since 1886 and has attained importance in calico printing, while Azocarmine, discovered in 1888, is an important color for dyeing wool.

The Badische Anilin- & Soda-Fabrik also took a leading part in opening up the great field of azo-coloring agents. Among the number of important dyes of this class discovered by its chemists, are especially to be mentioned Fast Red (1878) and Blue-Black (1882), the first black azo-dye. Brilliant Black may be regarded as a successor of this latter, as may also the more modern Palatine Black and Palatine Chrome Black, which are used in the place of logwood in dyeing wool black.

In the year 1891, the Badische Anilin- & Soda-Fabrik brought into commerce Indoine Blue, an exceedingly valuable product for dyeing cotton, and among the further successful discoveries of the company a number of important cotton dyes must be mentioned, in particular Nitrosamine Red (1894), several substantive azo-dyes: Cotton Yellow, Carbazol Yellow, Pyramine Orange, the Oxamine dyes, etc.—and the new class of sulphur dyes—Fast Black, Kyrogene Brown and Anthraquinone Black—all of which possess a high degree of fastness. Further, the introduction in 1895 of Rheonine, a valuable yellow dye for leather, deserves mention, as does also Lithol Red (1900), an azo-coloring matter which has become of the greatest importance in the lake-color industry.

Quite a special interest both on the practical and on the scientific side, attaches to the extremely fast coloring agents of the Indanthrene class introduced in 1901: Indanthrene Blue, Indanthrene Yellow (Flavanthrene), Indanthrene Gray (Melanthrene), Indanthrene Maroon (Fuscanthrene), to which have more recently been added the following products: Indanthrene Dark Blue BT and BO (Cyananthrene 1904, Violanthrene 1905), Indanthrene Green (Viridanthrene 1906), and Indanthrene Violet (1907), all derivatives of

Benzanthrone; Indanthrene Bordeaux (1908) and Indanthrene Red G and R (1908), derivatives of Dianthraquinonylamin; the derivatives of Methylanthraquinone-anthraflavone (1907), Indanthrene Orange-gold (Pyranthrene 1908), and further Indanthrene Olive (Olivanthrene 1906), Indanthrene Orange (Fulvanthrene 1908), Indanthrene Brown (Rufanthrene 1907) and Indanthrene Copper (1908).

The oldest and most important of the coloring agents obtained from the vegetable kingdom are Alizarine and Indigo. The Badische Anilin- & Soda-Fabrik having at an early date succeeded, as already mentioned, in producing the former of these commercially from anthracene. In 1881 Baeyer had solved scientifically the problem of preparing Indigo synthetically, and in 1890 Heumann succeeded in discovering a new solution of this important question. Working together with these eminent scientists, the company endeavored to make these important discoveries commercially useful, and after persevering continuously for many years and devoting to the matter costly work without stint, its labors were finally rewarded with success. The great object is achieved—indigo can be produced from coal-tar artificially in a manner that renders it capable of competing with the product obtained from the indigo plant, and since July, 1897, the Badische Anilin- & Soda-Fabrik has placed synthetic indigo on the market under the name of "Indigo Pure B. A. S. F." This marked the beginning of a new epoch in the history of the coal-tar color industry, for the manufacture of this new product constitutes an addition of quite eminent importance to its field of operations.

The substitution of artificial dyes used for ages in the dyeing industry is the goal for which the coal-tar industry has consistently striven to attain. The economic importance of this is obvious: First, the building up of a great industry, and second, the releasing of vast areas for other and more profitable purposes than the cultivation of natural dyes. To-day many million kilograms of artificial alizarine are manufactured annually, and the use of Madder has almost entirely ceased. Cochineal was formerly used in large quantities, in round figures about 600,000 kilograms were used in Germany in the year 1872, but in 1907 the quantity used was only 71,000 kilograms. Archil has had to yield to the red azo-colors and to azo-carmine. Curcuma has been displaced by fast yellow, auramine and others, and tartrazine has replaced flavine. A large proportion of logwood hitherto employed has been replaced by black azo-dyes and alizarine black, and indeed the alizarine dyes generally threaten the dye-woods, and finally, even vegetable indigo has been met by a victorious competitor in synthetic indigo. The introduction of the latter was a complete success. Founded on an installation on the largest scale, and carried on with the express intention of driving the natural product entirely from the field, the manufacture of synthetic indigo has now reached enormous proportions and is continually increasing. Corresponding to the increase in the production of synthetic indigo, that of the natural product is diminishing so that the cultivation of plant indigo, which even in 1897 showed a production of five to six million kilograms of a value of from sixty to eighty million marks, has sunk to about one-fifth. For instance, the Calcutta crop amounted in 1896 to 158,923 maunds, and 1906 to only 33,360 maunds. In 1898 there were 122 indigo plantations in Java, where there are now only 28, and the crop has sunk from 12,580 chests in that year to 2,295 in 1907. The value of the plant indigo imported into Germany in 1897 was about 12,700,000 marks, but in 1907 only 1,083,000 marks, while on the other hand the export of synthetic indigo from Germany reached in 1907 the value of 42,582,000 marks.

The issue of the struggle between the artificial and the natural product is no longer doubtful, plant indigo will in no long time share the fate of Madder, and practically disappear from commerce. * * *

The annual production in Germany of artificial organic coloring-matters represents a value of 250,000,000 marks.

The Badische Anilin- & Soda-Fabrik has recently turned its attention to one of the most important problems of modern chemistry, namely, the utilization of the nitrogen of the air. The company has succeeded, by means of an original process, in burning the nitrogen of the air in an electric arc flame, and is now about to carry out its process on the largest scale in Norway, utilizing the enormous water power which that country has at its disposal. Agriculture needs enormous quantities of saltpetre, the most important plant manure, and the demand grows from year to year so that within a short period the saltpetre deposits of Chili, up to now the sole source of this product, will be exhausted. The yearly production of Chili saltpetre amounts to 1,800,000 tons, of which the German agriculture absorbs about 500,000 tons. From these figures some idea can be obtained of the importance of the future of this industry, which is destined to supply the world's demand of saltpetre.

BOOK REVIEWS AND NOTICES.

Principles and Practice of Agricultural Analysis. Second edition, revised and enlarged. Vol. II. Fertilizers and Insecticides. By HARVEY W. WILEY, A.M., Ph.D. 8vo, 680 pp. Illustrated. Easton, Pa.: Chemical Publishing Co. 1908. Price, $4.50.

This is the standard work on agricultural analysis in the English language and is so well and favorably known in the first edition that it is hardly necessary to comment in general terms on the second. The principal addition to the present volume is that portion of the work devoted to insecticides and fungicides, amounting to some twenty-eight pages. However, the book has been in large part rewritten and new matter has been added wherever it was necessary to bring the work down to date. For instance, numerous pages have been added in Part I, devoted to phosphates and phosphatic fertilizers, under the headings of definitions, sampling and preliminary treatment, drying samples of fertilizer, mineral phosphates, direct weighing of the yellow precipitate, etc. Part II is devoted to nitrogen in fertilizers and here again extensive additions have been made so that this part is more than twice as large as in the first edition. Again, in Part III, devoted to potash in fertilizers, many new pages have been added and several new illustrations. The first edition of the work numbered 332 pages. The second numbers 680, and is correspondingly complete and up-to-date. Not the least interesting pages are those devoted to the utilization of the nitrogen in the air as a fertilizing material, which includes the fixation by soil bacteria, in the form of cyanamid, and as nitric acid or the nitrate

after oxidation to nitric oxide in the electric arc. There is so much new matter in the second edition that even fertilizer chemists who already possess the first may well add the second to their library. We shall look forward with interest to the publication of the third volume of this important work. W. D. RICHARDSON.

The Mechanical Appliances of the Chemical and Metallurgical Industries. By OSKAR NAGEL, Ph.D. 8vo. pp. vii + 307. New York. Published by the author. 1908. Price, $2.00 net.

There has been a great need for just such a work as Dr. Nagel's for some time, and on the whole, the author has satisfied this want ably and well. As an aid to the professor, college student, the works chemist or works manager, the book will be found of service, while the engineer himself may look through the work with profit. Here are gathered together in systematic form the mechanical appliances of the chemical industries and the organization alone of the great mass of material in this field and the elimination of all but the essentials is a worthy task. The instruction of the young chemist in college in the ways and means of applying his science to the practical exigencies of chemical manufacture is difficult. Dr. Nagel's book will certainly help greatly in this respect. The book is composed of fifteen chapters and is copiously illustrated with well-selected cuts.

Chemical Reagents: Their Purity and Tests. By E. MERCK. Translated by Henry Schenck. 8vo. pp. vi + 250. New York: D. Van Nostrand Company. 1907. Price, $1.50 net.

This work based on the previous one of Dr. Krauch, is of great service to the chemical profession generally and to all who have occasion to use pure chemicals. Probably no other work has had so great an influence in developing manufacturing standards for pure chemicals. The day of the C. P. chemical is past. The day of the analyzed chemical is at hand, and we must admit that in this revolution the works of Krauch and Merck have had a preëminent place. The book can be added to the library of any chemical laboratory with advantage.

Modern Power Gas Producer. By HORACE ALLEN. 12mo. pp. vii + 326. New York: D. Van Nostrand Company. 1908. Price, $2.50 net.

The literature on producer gas, gas producers and gas as a source of power, is increasing monthly, but hardly rapidly enough to keep pace with engineering progress along these lines. The present work is a valuable addition to those that have gone before, and presents in compact and useful form the essentials of gas production and utilization. There are 22 chapters, covering the different varieties of gas, the gasification of fuel, several chapters on gas producers and the application of gas to internal combustion engines. The illustrations are good and the work should prove useful to chemists and chemical engineers.

Chemiker-Kalender, 1909. By DR. RUDOLPH BIEDERMANN. Part I, cloth, pp. 384 Part II, paper, pp. 562. Berlin: Julius Springer. 1909. Price, 4 marks.

Again the chemists of the world extend their annual greeting to the Chemiker-Kalender (now in its thirtieth year) which has served them so long and so usefully. It is, of course, unnecessary to make any extensive comments on the nature and contents of a book so widely used and favorably known as this one. Four pages have been added to Part I, and six pages to Part II, for the current year. The print, paper and general make-up has not been altered. If we could make a suggestion to the publishers, we should like to ask whether or not the two parts could not be bound in one volume with advantage.

Laboratory Experiments in Metallurgy. By ALBERT SAUVEUR and H. N. BOYLSTON. Small 4to. pp. 73. Cambridge, Mass. Published by the authors. 1908. Price, $1.25 net.

It is said in the preface that this work is intended primarily for the use of students at Harvard University, who take the course in general metallurgy and metallurgy of iron and steel. The intention is also announced of extending the number of experiments, in the desire to make the book more comprehensive, in future editions. The work is divided into two parts: Part I, General Metallurgy, 30 pages; Part II, Metallurgy of Iron and Steel, 68 pages. There are ten tables and seventeen illustrations. The work is so arranged that the student can easily make notes of his experiments, being interleaved. On the whole, the book is a needed one and serves its purpose well. A number of minor criticisms might be suggested. For instance, under calorimetry, the Parr calorimeter is used in one experiment, but no mention is made of the oxygen-bomb calorimeter. The Parr calorimeter is certainly worthy a place in the book but not to the exclusion of the oxygen-bomb. Among the tables is a copyrighted one by Riehlé Bros. It would seem that the size of the type used in the tables could be increased with advantage, and the size of type in which the name of the firm is printed, could be reduced, also with advantage. Several good cooling curves for iron and steel are given and it would seem that the descriptive matter covering these experiments might be extended. No doubt it is intended to do this in the lectures accompanying the course. As stated above, on the whole, the book serves a very useful purpose and will be found valuable by teachers of metallurgy.

Laboratory Notes on Industrial Water Analysis. By ELLEN H. RICHARDS. 8vo. 49 pp. New York: John Wiley & Sons. 1908. Price, 50 cents, net.

Apparently this work is the outgrowth of teaching experience and designed for a particular class of students. It adds another to the list of many useful books from the pen of the same author. While the previous works dealt largely with sanitary science, the present one deals quite as largely with the engineering side of the subject. The special methods of analysis are principally considered and attention given only in lesser degree to general methods of analysis. On the whole, the book is good and useful and, within its scope, few criticisms are to be made. One point to be noticed is the occasional use of language which is not exactly scientific, or is at least unusual. For instance, on page 14, "incrustants *par excellence*." Definite expressions are in use among chemists for definite things and should be adhered to as much as possible. Among the tables, it would seem that, even though no great changes have been

made, a later International Atomic Weight table than that for 1905 might be given. The author says a good word for the right method of reporting when she deprecates the waste of time spent on elaborate calculations of water analysis. The method of the U. S. Geological Survey of reporting constituents as ions is certainly to be approved and in any case the actual analytical data, either as ions or oxides, should be given, along with the possible combinations reported. As the author states, the public soon learns to take results and interpret them as given. This is true and it is unfortunate that in so many industrial analyses at the present time, the data is not given while the hypothetical combinations are.

Die Fabrikation von Leim und Gelatine. By DR. LUDWIG THIELE. 16mo. pp. 158. Hannover: Dr. Max Janecke. 1907. Price, $—.

This little book is divided into nine chapters and covers in a very excellent manner, considering the limited amount of space, the manufacture of glue and gelatine under the following heads: Composition and Purposes of Glue and Gelatine; Raw Materials; Manufacture of Bone Glue; Trade Names and Uses of Various Glues; Preliminary Treatment of the Raw Material for Hide Glue and Gelatine; Gelatine; Fish Glue; The Testing of Glue; Plant for Glue Manufacture. Comprehensive works on the manufacture of glue are almost an unknown quantity. Most are of the briefer sort and too few are up-to-date. Among the briefer texts, this little book of Dr. Thiele's should find an acceptable place.

Les densities des solutions sucrées à Différentes temperatures. By D. SIDERSKY. In French and German. H. Dunod and E. Pinat, Paris. Friedr. Vieweg & Sohn, Brunswick. 1908. 48 pages with tables. Price unbound, 3.50 francs.

The importance of the determination of specific gravity in the chemical control of sugar factories has given rise in past years to a multiplicity of tables for the estimation of total solids. The various tables of Balling-Brix, Gerlach, Scheibler, and others when compared show, however, certain differences, a circumstance due partly as to whether the weight of water at 15° or 17°.5 was used as a basis and partly also to errors in observation. The efforts of the author in the present work have been directed towards evolving some order out of the chaotic condition of the literature upon this subject. As a basis of calculation he has used Plato's specific gravity table of sugar solutions adopted by the Kaiserliche Normal-Eichungskommission and now considered to be the most accurate. The tables are divided into two parts: the first part for dilute sugar solutions (0° to 5° Brix) gives the actual and relative density $D\frac{t^\circ}{4^\circ}$ and $D\frac{t^\circ}{15^\circ}$ to the fifth decimal with the corresponding percentages of sugar for each degree between 10° and 30°; the second part for more concentrated sugar solutions (5° to 30° Brix) gives the $D\frac{t^\circ}{4^\circ}$ and $D\frac{t^\circ}{15^\circ}$ to the fourth decimal with the corresponding percentages of sugar by both weight and volume at 10°, 15°, 20°, 25° and 30°.

For sugar solutions of density less than 1.13, the new specific gravity tables of Sidersky are unquestionably the most complete of their kind. Their practical value in sugar house work is, however, somewhat limited, being confined to sugar solutions below 30° Brix; for the sake of uniformity, therefore, many sugar chemists will no doubt prefer to continue using the older tables which have a range of from 0° to 95° Brix. It is to be hoped that the author may enlarge his tables so that they can be applied to syrups and sugar solutions of higher density. The text accompanying the tables is printed on opposite pages in French and German, an arrangement of great advantage to such as are familiar with but one of these languages. C. A. BROWNE.

"Effects of Formaldehyde upon Digestion and Health," Bureau of Chemistry, Circular No. 42.—In this circular, the Bureau, after describing the experiments, gives the following statement: "The final conclusion, therefore, is that the addition of formaldehyde to foods tends to derange metabolism, disturb the normal functions, and produce irritation and undue stimulation of the secretory activities, and therefore it is never justifiable."

The Decennial Index (1896–1905) of the *Journal of the Society of Chemical Industry* is now ready, price 11 s. ($2.75), and may be had by sending money order for the amount, payable to the Society of Chemical Industry, to Thomas Tyrer, Stirling Chemical Works, Stratford, London, E. The Collective Index (1881–1895) may be had at the same price.

The Bulletin of the Bureau of Standards, Volume V, No. 2, contains the following articles: Selective Radiation from Various Solids—W. W. Coblentz; Remarks on the Quartz Compensating Polariscope with Adjustable Sensibility—Frederick Bates; Methods of Obtaining Cooling Curves—George K. Burgess; Note on the Approximate Values of Bessel's Functions for Large Arguments—Louis Cohen; The Influence of Terminal Apparatus on Telephonic Transmission—Louis Cohen; The Principles Involved in the Selection and Definition of the Fundamental Electrical Units to be Proposed for International Adoption—F. A. Wolff; The Luminous Equivalent of Radiation—P. G. Nutting; The Temperature Formula of the Weston Standard Cell—F. A. Wolff.

Merck & Company have issued a pamphlet of eighty pages entitled "Prices and Uses of Tested Reagents, 1909." This work is a priced catalogue of Merck's tested reagents and contains under each item a list of tests to which the reagent has been subjected. The tests are those described in greater detail in "Chemical Reagents: Their Purity and Tests" published in 1907 by D. Van Nostrand Co., of New York, the volume which replaced Krauch's "Chemical Reagents." The booklet is useful to chemists and may be had by addressing Merck & Company, New York.

The Semi-Annual Report of Schimmel & Company, for November, 1908, contains an interesting article on Japanese Peppermint Oil, by Naojiro Inouye, of Harima, Japan,

from the laboratory of Prof. Edward Kremers, Madison, Wisconsin.

The Crane Company, Chicago, has issued an octavo pamphlet of 24 pages entitled "The Effect of Superheated Steam on Valves and Fittings" and written by the technical force of the company. This is timely and valuable. The use of superheated steam in the chemical industries and in engineering practice generally is greatly on the increase, whereas knowledge not only of its properties but of its effects, is inadequate to meet the situation. Under these circumstances, any contribution on the subject is to be welcomed. Following the introductory article which covers the effect of superheated steam on cast iron, on cast steel, on valve construction, on valve seats and stems, and on copper alloys (the effects of temperature from 70° to 950° F. upon the tensile strength of various alloys), there are a number of articles all bearing on the general subject reprinted from the *Valve World* for 1908.

Messrs. Schimmel & Company, the large essential oil distillers of Miltitz, near Leipzig, Germany, have issued a small and interesting volume on the subject of their plant and products. We note that the firm was founded in the year 1829 and erected its present plant in 1900. It is situated five miles from the center of the city of Leipzig, the total area owned by the firm being three hundred acres. The divisions of the book and subjects treated are: Power Plant, Main Factory, Chemical Department, Serum Department, Branch Distillery at Barrême, and Institutions for Social Welfare. The book is well illustrated and printed.

The Badische book, issued by the Badische Company, American representatives of the Badische Anilin & Soda-Fabrik, and distributed privately, is one of the best examples of book-making which has come to our attention from a manufacturing firm. It is bound and printed in excellent taste and contains a great deal of most instructive matter in regard to the works and products of the Company and its institutions for the benefit of its workmen. In another place we have quoted from the book, matter of considerable historical importance to chemical industry, and we note in passing that the products of the Company comprise the entire range of artificial organic coloring matters and all the intermediate and other products used in its manufacturing operations, including sulphuric acid, sulphuric anhydride, hydrochloric acid, soda, nitric acid, liquid chlorine, etc.; that the Company employs 217 manufacturing and research chemists, 142 civil engineers and 918 officials in the commercial department, besides 8000 workmen. The main plant of the Company is situated in Ludwigshafen on the Rhine. To the chemical engineer the name of the Badische will always recall with pleasure the splendid work of Rudolph Knietsch on liquid chlorine and sulphuric anhydride, as well as on many other chemical products.

Among the recent efforts by manufacturers to establish and maintain uniform methods of analysis, we note one in the form of a 12mo. pamphlet issued by the laboratories of the Pfister and Vogel Leather Company, of Milwaukee, under the direction of Dr. Louis E. Levi, chief chemist, and Carl V. Manuel, chemist in charge. The book numbers 29 pages, is well printed and put together, is interleaved for notes and contains a large number of methods. We note that the acetine method is used for glycerine. A large number of practical and skilful chemists have found this method uncertain and unreliable and prefer Hehner's bichromate method.

NEW BOOKS.

Praktischer Führer durch den Zeugdruck. By AUG. AXMACHER. Hannover: Dr. Max Jänecke. 1908. Price, geh. M. 6.60; geb. M. 7.40.

The Brewers' Analyst. By R. D. BAILEY. 8vo. Cloth. Ill. x+423 pp. New York: D. Van Nostrand Co. Price, $5.00

Chapters on Paper Making. By CLAYTON BEADLE. Vol. V. Concerning the theory and practice of beating. London: Crosby, Lockwood, and Son. 1908. Price, 5 s., net.

Analytische Chemie für Apotheker. By H. BECKURTS. Zweite neubearb. Aufl. mit 1 farbigen Tafel u. 96 abb. im Text. Stuttgart: F. Enke, 1908. Price, M. 11.60.

Chemiker-Kalender 1909. By R. BIEDERMAN. Ein Hilfsbuch für Chemiker, Physiker, Mineralogen, Industrielle, Pharmazeuten, Hüttenmänner usw. 30 Jahrung. Berlin: Verlag von Julius Springer. 1908. 1 Teil geb. in Leinwand-11 Teil. Price, geh. M. 4; in Leder, M. 4.50.

The Chemical Analysis of Iron. By A. A. BLAIR. Seventh edition. 8vo. Cloth. Ill. xix+334 pp. Philadelphia: J. B. Lippincott Co. 1908.

The Structure of the Wool Fiber, and Its Relation to the Use of Wool for Technical Purposes. By F. H. BOWMAN. New York: Macmillan & Co. 1908. Price, $2.25.

Le Parfum chez la Plante. By EUG. CHARABOT ET C. L. GATIN. Paris: Octave Doin. 1908. Price, 5 fr.

Chemisches Praktikum für Studierende der Naturwissenschaften. By MAX DITTRICH.

Die neuren Ansichten über die Erdöls. By C. ENGLER. Berlin: Verlag für Fachliteratur. W. 30.

Laboratoriumsbuch für Tinktorialchemiker, coloristen, Ingenieure, u. techn. Reisende in Färbereien, Druckereien, Farben-, Lack-, u. Papierfabriken. F. ERBAU. Mit 18 Abbild. i. Text. Halle a/S.: W. Knapp, 1908. Price, M. 2.50.

Fischer's Taschenbuch für Feuerungstechniker. By F. FISCHER. Anleitung zur Untersuchung und Beurteilung von Brennstoffen und Feuerungsanlagen. Leipzig: Verlag von Alfred Kröner. 1909. 6 neubearb. Aufl. M. 3.

Untersuchung und Nachweis organischer Farbestoffe auf spectroskopischen Wege. By J. FORMANEK. Zweite vollständig umgearb. und vermehrte Auflage. Erster

Theil. Berlin: Verlag v. Julius Springer. 1908. Price, M. 12.

Utilization of Wood Waste by Distillation. By W. B. HARPER. 4to. Cloth. Ill. 156 pp. New York: D. Van Nostrand Co. Price, $3.00.

The Mineral Industry. By W. R. INGALLS. 8vo. Cloth. Ill. xx+1,127 pp. Price, $10.00. New York: Hill Publishing Co. 1908.

Technologie der Gewebe-appretur. By BERNARD KOZLIK. Mit 161 in den Text gedruckten Figuren. Berlin: Julius Springer.

Technische Anwendung der physikalischen Chemie. By ARNDT KURT. iv+304 s. 55 Abbild. im Text. Berlin: Verlag von Meyer und Müller. Price, geb. M 8.

Technical Methods of Chemical Analysis. By GEORGE LUNGE, ED. English translation edited by Chas. A. Keane and others. Vol. I, Parts I and II. London: Gurney and Jackson. 1908. Price, 52 s. 6 d. net.

Technical Chemists' Handbook: Tables and Methods of Analysis for Manufacturers of Inorganic Chemical Products. By GEORGE LUNGE. London: Gurney and Jackson. 1908. Price, 10 s 6 d.

The Design and Equipment of Small Chemical Laboratories. By R. K. MEADE. Ill. 8vo. Cloth. Price, $2.00. iii+136 pp. Chicago: The Chemical Engineer Publishing Co. 1908.

Meat Extracts and Similar Preparations, Including Studies of the Methods of Analysis Employed. U. S. Dept. of Agriculture, Bureau of Chemistry, Bull. 114. Washington: Govt. Printing Office. 1908.

Der Dampf in der chemischen Technik. W. MOMBER. Halle a/S: Verlag von Wilhelm Knapp. 1908. 104 s. 26 fig. im Text. Price, geh. M. 3.60.

The Chemistry of Essential Oils and Artificial Perfumes. By E. J. PARRY. Second Edition, rev. and enl. London: Scott, Greenwood, and Son. 1908. 13 s.

Cement Laboratory Manual. By L. A. WATERBURY. 12mo. Cloth. Ill. vii +122 pp. New York: John Wiley and Sons. Price, $1.00.

Hevea Brasileinsis or Para Rubber. Its Botany, Cultivation, Chemistry, and Diseases. HERBERT WRIGHT. Third Edition. London: McLaren & Sons. 1908. Price, 10 s net.

Principles and Practice of Agricultural Analysis. H. W. WILEY. Vol. II, 2nd Edition, Rewritten, 8vo. Cloth. 68 pp. Easton: Chemical Publishing Co. Price, $4.50.

Royal Botanic Gardens, Kew. By W. J. BEAN. Historical and descriptive; with intro., by Sir W. Thiselton Dyer; ill with 20 reproductions in color from paintings by H. A. Oliver and 41 reproductions from photographs. New York: Cassell. Price, $7.50 net.

Electro-Metallurgy. J. B. C. KERSHAW. New York: D. Van Nostrand Co. Ill. (Westminster ser.). Cloth. Price, $2.00 net.

Laboratory Guide of Industrial Chemistry. ALLEN ROGERS. New York: D. Van Nostrand Co. 9 + 15 pp. Ill., tables, diagrams, plates. 8vo. Cloth. Price, $1.50.

Glass Manufacture. WALTER ROSENHAIN. New York: D. Van Nostrand Co. 264 pp. Ill. 8vo. (Westminster ser.). Cloth. Price, $2.00.

SCIENTIFIC SOCIETIES.

THIRTY-NINTH GENERAL MEETING OF THE AMERICAN CHEMICAL SOCIETY.

The thirty-ninth general meeting of the American Chemical Society was held at Baltimore, Maryland, December 29th to January 2nd, inclusive. The meeting was the largest and most enthusiastic in the history of the Society. There were about four hundred members registered. In other words, ten per cent. of the total membership of the Society was in attendance.

Tuesday, December 29th was devoted to general addresses. In the morning, Messrs. A. D. Little, G. N. Lewis and C. H. Herty read addresses, which were followed by F. W. Clarke with a report of the International Committee on Atomic Weights. In the afternoon there was a meeting, largely attended, of the Section of Chemical Education, and after an address by the Chairman, Dr. H. P. Talbot, and an announcement by Dr. H. W. Wiley on the International Congress of Applied Chemistry to be held in London, May 27 to June 2, 1909, three addresses were read: Wm. H. Nichols, on "The Efficiency and Deficiencies of the College-trained Chemist when Tested in the Technical Field;" Louis M. Dennis, "To What Extent Should College Training Confer Practical Efficiency along Technical Lines?" and by Wm. McMurtrie, on "The Attitude of Technical Institutions to Post-graduate Study." A general discussion followed, which was participated in by Dr. Leo Baekeland, Dr. W. R. Whitney, Dr. H. W. Wiley, Prof. Louis Kahlenberg, Dr. C. B. Dudley, Dr. L. P. Kinnicutt and others.

This was the first meeting of the Section of Chemical Education and the interest and enthusiasm aroused indicate a successful future for this branch of the Society.

On Tuesday evening, at 8.00 o'clock, a complimentary smoker was given at the Hotel Belvedere, preceded by a very interesting talk illustrated with lantern slides by Wm. Simon, on the "Lumière Process of Color Photography." Upwards of three hundred persons attended the smoker, and the good fellowship which prevailed was unbounded.

On Wednesday morning there were meetings of the Agricultural and Food Section, the Biological Section, the Division of Industrial Chemists and Chemical Engineers, the Inorganic Section, the Organic Section, the Pharmaceutical Section and the Physical Section. In the afternoon there were excursions to the Naval Academy at Annapolis, to Sharpe & Dohme's factory and to the Baltimore breweries. In the evening at 8.00 P.M., the President of the Society, Prof. M. T. Bogert, delivered his annual address, on the subject "The Function of Chemistry in the Conservation of our Natural Resources."

On Thursday morning there was a general business meeting of the Society, followed by addresses by H. J. Wheeler, on "The Future of Agricultural Chemistry;" S. F. Acree, on "The Quantitative Study of Organic Reactions;" and Edward Kremers, on "The Classification of Carbon Compounds." Following the general session, the Divisions and Sections met and continued their programs. At 4.00 P.M. there was a meeting of Associate Editors and Abstractors of Chemical Abstracts, and at 5.00 P.M. a meeting of the Board of Editors of the *Journal of Industrial and Engineering Chemistry*. In the evening at 8.00 P.M. the subscription dinner at the Belvedere Hotel was attended by about three hundred members and guests, and was one of the most enjoyable events of the meeting.

On Friday morning, January 1st, there were adjourned meetings of the Sections and Divisions, and in the afternoon an excursion to the plant of the Maryland Steel Company at Sparrow's Point. On Saturday, January 2nd, there was an excursion to Washington to visit the Bureau of Standards and the Geophysical laboratory.

Possibly the most significant event in connection with this meeting of the Society was the granting by the Council of authority to establish several Divisions. This is in line with the policy which has been developed during the past two years. Authority was granted for the establishment of a Division of Agricultural and Food Chemists, a Division of Physical and Inorganic Chemists, a Division of Organic Chemists and a Division of Fertilizer Chemists. Any member of the Society is privileged to register in one or more of these Divisions and in this way affiliate himself with the particular branch or branches of the science in which his interests chiefly lie. With the Division of Industrial Chemists and Chemical Engineers, which was organized in the summer of 1908 at New Haven, the Society now has five Divisions, and it is likely that more will follow soon.

The following officers were elected: *President*, Dr. W. R. Whitney; *Secretary*, Chas. L. Parsons; *Editors*, W. A. Noyes and W. D. Richardson; *Treasurer*, Dr. A. P. Hallock; *Librarian*, Dr. E. G. Love; *Directors*, T. J. Parker and M. T. Bogert; *Councilors-at-Large*, Dr. W. Lash Miller, Chas. H. Herty and W. H. Walker.

It was decided to hold the next meeting of the Society at Detroit during the coming summer, the date to be fixed by the President and Secretary.

DIVISION OF INDUSTRIAL CHEMISTS AND CHEMICAL ENGINEERS.

The Division of Industrial Chemists and Chemical Engineers held its second semi-annual meeting at Baltimore, in connection with the general meeting of the American Chemical Society. There were in attendance about seventy-five members, although more than this number have registered in the Division and the number is constantly increasing. The meeting was called to order by the Chairman, Dr. A. D. Little, of Boston, and the following papers were read: Walter O. Snelling, The Munroe Crucible; Percy H. Walker and H. A. Whitman, Rapid Analysis of Babbit Metal; Chas. H. Herty and W. S. Dickson, The Unsaponifiable Matter in the Oleo-Resins of Conifers; Allerton S. Cushman, Acceleration Tests of the Resistance to Corrosion of Iron and Steel; David W. Day, The Changes in Crude Petroleum Effected by Diffusion through Clay; George Auchy, Further Remarks on Vanadium and its Estimation; Paul N. Clancy, Iron from a Chemical and Commercial Standpoint; C. H. Ehrenfeld, An Unusual Incrustation on Generator Coils; H. J. Skinner, The Purchase of Material on Specification; F. A. Olmstead, Some Industrial Applications of the Ives Colorimeter; C. F. Woods, Factors Determining the Efficiency of Trolley Wires; E. B. Boughton, The Iodine Number and Some Other Values for China Wood Oil; E. G. Bailey, Accuracy in Sampling Coal; Chas. E. Munroe, The Commercial Manufacture of Amorphous Calcium Phosphide and The Distribution of Nitrate of Soda in the U. S.; L. A. Olney, Standard Methods for Determining and Recording the Relative Permanency or Resistance of Coloring Matters to the Common Color-destroying Agencies; A. G. Stillwell, Standardization of Methods for Commercial Analysis of Fats, Greases, etc., and Adoption of Rational Nomenclature for Same; Theodore Whittlesey, Guayule and Guayule Rubber; Arthur Lowenstein, The Rapid Determination of Moisture in Commercial Products of a Viscous or Semi-solid Consistency; C. F. Mabery, Lubrication and Lubricants; W. H. Walker, A Method for Testing the Continuity of Tin Plate.

Among the most interesting papers were those by Mr. E. G. Bailey, on "Accuracy in Sampling of Coal," and Wm. H. Walker, on "A Method for Testing the Continuity of Tin Plate," both of which will be published in this Journal at a later date.

Mr. Bailey called attention to the principal reasons for error in determining coal values by sample, and showed the reason why several so-called samples from the same car might vary greatly upon final analysis. Mr. Bailey's paper was illustrated by a series of elaborate charts which showed the factors of error.

Dr. Walker's paper called attention to the so-called pin-holes in the surface of tin plate and showed how he determined their exact location by flowing a gelatin solution of potassium ferricyanide over the plate. After a short interval, a blue spot appearing in the jelly located the pin-hole, and if it was desired to examine the pin-hole further, a circular punch stamp around the spot would locate it after the jelly was dissolved off.

Mr. T. A. Olmstead's paper on the Ives colorimeter showed the value of this instrument for comparing color values and also as a test for color blindness. Mr. C. F. Woods, in his paper on trolley wires, called attention to the fact that the average weight of a trolley wire is 2000 pounds to the mile; that there are many defects in the manufacture and that great trouble is caused on account of the lack of proper specifications. With a hard-drawn wire, about 0.392 inch diameter, or No. 2 B. & S. gauge would prove the most suitable; that the distance between spans averaged 100 feet. Three conditions, the pull due to its weight, the severe pounding of the trolley wheel and the wave motion

were disturbing features in the life of a trolley line. The qualities required are: homogeneity and flexibility, indicated by the elastic limit, high tensile strength, proper conductivity measured by the Wheatstone bridge, and proper torsional strength. Lake copper has proved the best for general use and next to this electrolytic copper. Practical methods of manufacture were then touched upon and an interesting discussion followed the paper.

Prof. Chas. E. Munroe spoke on the manufacture of amorphous calcium phosphide, calling attention to its use in the Whitehead torpedo.

The following officers of the Division were elected for 1909: *Chairman*, A. D. Little; *Vice-Chairman*, Geo. C. Stone; *Secretary*, B. T. B. Hyde; *Executive Committee, ex officio*, Chairman, Vice-Chairman and Secretary, and Editor of the *Journal of Industrial and Engineering Chemistry;* Wm. H. Walker; W. C. Ebaugh; J. O. Handy; A. S. Cushman; and C. P. Van Gundy.

The Chairman of the Division, Dr. A. D. Little, outlined a policy for the Division and suggested that committees be appointed to follow up the various suggestions. There were several definite undertakings which should presently interest the Division of Industrial Chemists and Chemical Engineers. For instance, the matter of trade terms; again, trade customs, both of which would require a careful and comprehensive study which would be appreciated by all present, in view of the many difficulties which arose in general business transactions, due to the lack of definitions in these two important factors. Specifically, the need of defining cylinder oils by trade terms and the decided need of specification for buyer and seller, which should receive the endorsement of the Division were mentioned. The matter of research problems to be taken up in industrial laboratories attached to the universities should arouse the Division's interest and, among other things, work on the spontaneous combustion of coal. A Committee on Publicity would serve many of the needs of the Division and should include a manufacturers' descriptive bibliography.

The following By-Laws were adopted for the government of the Division:

By-laws of the Division of Industrial Chemists and Chemical Engineers.

ARTICLE 1.

MEMBERSHIP.

Membership in the Division shall be open to all members of the American Chemical Society, and any member of the Society shall upon request to the Secretary of the Division be registered as a member of the Division.

ARTICLE 2.

OFFICERS.

1. The officers of the Division shall be a Chairman, a Vice-Chairman, a Recording Secretary and an Executive Committee.

2. The Chairman, Vice-Chairman, Secretary and Editor of the *Journal of Industrial and Engineering Chemistry* shall be members of the Executive Committee *ex officio*.

3. The Chairman of the Division shall be Chairman of the Executive Committee.

4. The Executive Committee shall consist of five registered members of the Division and the members *ex officio* provided by Section 2 of this article.

5. All officers of the Division shall be elected annually by ballot at the last session of the Division held during the annual meeting of the Society and shall take office on January 10th, following the meeting at which they were elected. They shall hold office for one year or until their successors are elected.

6. The Executive Committee shall fill any vacancies occurring through death or resignation among officers of the Division.

7. It shall be the duty of the Chairman to represent the Division in the Council of the Society, to preside at meetings of the Executive Committee, to carry into effect the decisions and recommendations of that committee, to preside at stated meetings of the Division, and to report to the Society at its regular meetings.

8. In the absence of the Chairman, the duties of the office shall devolve upon the Vice-Chairman. The Vice-Chairman shall be *ex officio* Chairman of the Committee on Research Problems.

9. It shall be the duty of the Recording Secretary to keep a record of the proceedings of the Division and of the Executive Committee, to maintain a list of registered members, to send to registered members such notices as the business of the Division may require, and to transmit to the Secretary of the Society the names of all the officers and standing committees of the Division within three weeks of their appointment; and, in like manner, to notify the Secretary of the Society of any change of officers or standing committees during the year.

10. The Recording Secretary shall be the custodian of the files of the Division. He shall have charge of the funds of the Division and shall make all disbursements, subject to the authorization of the Executive Committee. He shall report to the Division at its annual meeting.

11. The Executive Committee shall conduct the business of the Division and direct its activities. The committee shall at each general meeting of the Society and as early as may be, hold a meeting to consider the affairs of the Division and receive reports of its committees. The voting majority of the Executive Committee shall be responsible for all expenditures which it may authorize except so far as the action of the Council shall previously have provided for such expenditure.

ARTICLE 3.

MEETINGS.

There shall be a meeting of the Division at each general meeting of the Society. Business affecting the organization of the Division shall be transacted only at the meeting coincident with the annual meeting of the Society.

The order of business shall be as follows: Reading of Minutes, Report of Executive Committee, Report of Secretary, Report of Committees and Discussions, Reading of Papers and Discussions, Miscellaneous Business, and at the annual meeting, Election of Officers, Unfinished Business.

The regular order of business of the Division may be suspended at any session by the consent of three-fourths of the members of the Society present.

ARTICLE 4.

SPECIAL COMMITTEES.

The Chairman shall with the advice and approval of the Executive Committee appoint from time to time standing

committees of the Division to consider, conduct, and report upon such special matters as may be delegated to them. The following committees shall be appointed at the annual meeting or as soon thereafter as may be expedient.

1. Committee on Definition of Trade Terms; 2. Committee on Trade Customs; 3. Committee on Official Specifications; 4. Committee on Research Problems; 5. Committee on Standard Methods; 6. Committee on Descriptive Bibliographies; 7. Committee on Advertising.

ARTICLE 5.

PUBLICATIONS.

The official organ of the Division shall be the *Journal of Industrial and Engineering Chemistry.*

The Executive Committee may at its discretion provide for the issue of other non-serial publications for distribution to registered members of the Division or for sale.

ARTICLE 6.

ASSESSMENTS.

The Executive Committee may at its discretion impose dues not to exceed three dollars per year upon registered members of the Division, said dues to be collected by the Secretary and the receipts therefrom to be devoted to the purposes of the Division.

ARTICLE 7.

AMENDMENTS.

These By-laws may be amended at any annual meeting of the Division by a three-fifths vote of the registered members present, provided two weeks' notice of the proposed amendment with the text thereof has been sent to the registered members of the Division. Amendments to be effective must be approved by the Council and accepted by that body as not inconsistent with the Constitution and By-laws of the Society.

DIVISION OF FERTILIZER CHEMISTS.

The first semi-annual meeting of the Division of Fertilizer Chemists, of the American Chemical Society, was held at Baltimore, December 29th to January 2nd, in connection with the general meeting of the Society. The meeting was called to order by the Chairman, Mr. F. B. Carpenter, and the following program was presented: F. B. Carpenter, The Fertilizer Industry—an historical sketch, (Chairman's address). Arthur Lowenstein, Some Points of Interest in Connection with Present Fertilizer Laws and Proposed Fertilizer Legislation; J. E. Breckenridge, Potash Experiments in Factory Work, showing heavy loss by Official Method; M. H. Pingree, A Comparison of Various Methods in Bringing About the Solution of Potash in Mixed Fertilizers and Tobacco Stems; Geo. D. Leavens, A Discussion of Methods for Determining the Availability of Phosphoric Acid in Thomas Phosphate Powder; F. P. Veitch, A Discussion of Methods for the Determination of Iron and Alumina in Phosphate Rock; F. B. Carpenter, Note on the Determination of Insoluble Phosphoric Acid; Lucius P. Brown, Chemistry and Geology of the Inland Phosphates of the United States; F. B. Porter, Loss of Potash in Commercial Fertilizers; A. G. Stillwell, Remarks on the Gladding Method for Phosphoric Acid (weighing direct the phosphomolybdate precipitate).

The following officers were elected for the year 1909: *Chairman*, F. B. Carpenter; *Vice-Chairman*, B. H. Hite; *Secretary*, J. E. Breckenridge; *Executive Committee*, the Chairman, Vice-Chairman and Secretary *ex-officio*, and Messrs. P. Rudnick and T. S. Gladding. Two committees were appointed, one to draw up By-laws for the government of the division and the other to endeavor to draw up further specifications for the ammonium citrate solution as used in the determination of available phosphoric acid.

The *Journal of Industrial and Engineering Chemistry* hopes that the Division of Fertilizer Chemists will have a long and successful career.

CONVOCATION WEEK MEETING OF THE AMERICAN ASSOCIATION FOR THE ADVANCEMENT OF SCIENCE.

Baltimore, December 26, 1908, to January 2, 1909.

The Convocation Week meeting of the American Association for the Advancement of Science broke all previous records for scientific meetings in this country. The registration at the meeting was approximately 1200, and considering the fact that large numbers of scientists in attendance did not register, it is estimated that the attendance may have been as great as 2500. Of the 400 chemists in attendance only about 80 registered and it is likely that the ratio was nearly as small in some of the other affiliated societies. The meeting was so large, and the number of papers (about 1000) so great, that the question of organizing the next meeting is being now seriously considered. Several plans have been proposed and doubtless will be considered by those in authority before the next Convocation Week meeting.

The meetings of Section C and the American Chemical Society are reported elsewhere in this issue, and space will not permit a description of the events and meetings held by the Association. For this our readers are referred to the current numbers of *Science*.

The next Convocation Week Meeting will be held at Boston, December 27, 1909, to January 1, 1910, and after that, the following places in succession have been suggested for Convocation Week meetings: Minneapolis, Washington, Cleveland and Toronto.

In the summer of 1910 it is proposed that the Association meet in Honolulu.

The officers for the coming year are as follows: *President*, David Starr Jordan, Leland Stanford, Jr., University, California; *Vice-Presidents*, Mathematics and Astronomy, E. W. Brown, Yale University, New Haven, Conn; Physics, L. A. Bauer, Carnegie Institute, Washington, D. C.; Chemistry, Wm. C. McPherson, Ohio State University, Columbus, Ohio; Mechanical Science and Engineering, John F. Hayford, U. S. Coast & Geodetic Survey, Washington, D. C.; Geology and Geography, R. W. Brock, School of Mining, Kingston, Ontario; Zoology, W. E. Ritter, University of California, Berkeley, Cal.; Botany, D. P. Penhallow, McGill University, Montreal, Canada; Anthropology and Psychology, W. H. Holmes, Bureau of American Ethnology, Washington, D. C.; Social and Economic Science, Carroll D. Wright, Clark College, Worcester, Mass.; Physiology and Experimental Medicine, C. S. Minot, Harvard Medical School, Boston, Mass.; Education, James E. Russell, Columbia

University, New York City. *Permanent Secretary*, L. O. Howard, Smithsonian Institution, Washington, D. C. *General Secretary*, Dr. D. C. Miller, Case School of Applied Science, Cleveland, Ohio. *Secretary of the Council*, F. S. Benedict, Carnegie Institute, Washington, D. C. *Secretaries of the Sections:* A—Mathematics and Astronomy—Geo. A. Miller, University of Illinois; B—Physics—A. D. Cole, Ohio State University, Columbus, Ohio; C—Chemistry—C. H. Herty, University of North Carolina, Chapel Hill, N. C.; D—Mechanical Science and Engineering—G. W. Bissell, Michigan Agricultural College, East Lansing, Mich.; E—Geology and Geography—F. P. Gulliver, Norwich, Conn.; F—Zoology—Maurice A. Bigelow, Columbia University, New York City; G—Botany—H. C. Cowles, University of Chicago; H—Anthropology and Psychology—George Grant McCurdy, Yale University, New Haven, Conn.; I—Social and Economic Science—J. Pease Norton, Yale University, New Haven, Conn.; K—Physiology and Experimental Medicine—George T. Kemp, University of Illinois, Urbana, Ill.; L—Education—C. R. Mann, University of Chicago, Chicago, Ill. *Treasurer*, R. S. Woodward, Carnegie Institute, Washington, D. C.

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS.

Pittsburg Meeting, December 28 and 29, 1908.

The first annual meeting of the American Institute of Chemical Engineers was held at the Carnegie Technical Schools, Pittsburg, December 28 and 29, 1908. The following papers were read: Wm. M. Booth, Steam Power Plant Economics from the Standpoint of the Chemical Engineer; Edward Gudeman, Technical Coal Analysis; J. C. Wm. Greth, The Chemical Aspect of Impurities which Cause Scale and Corrosion of Steam Boilers; A. Bement, Testing and Performance of Steam Generating Apparatus; H. August Hunicke, The Examination of Flue Gases in Boiler Tests; Ernest Schmatolla, Producer Gas Installations; Richard K. Meade, Heating of Industrial Furnaces with Pulverized Fuel; Edwin T. Northrup, Modern Electrical Resistance Pyrometry; J. A. De Cew, Chemical Specifications for Sulphite Pulp; F. W. Frerichs, An Apparatus for Testing Liquefied Ammonia Gas; J. Herbert Brewster, The Sanitary Condition of the Southern End of Lake Michigan; Thorn Smith, Some Experiments with the Ferric Iron Contact Method of Making Sulphuric Acid from Smelter Fumes; Wm. M. Grosvenor, Drier Calculations and Drier Design.

The papers read and presented were referred to a Publication Committee for publication in a volume of Proceedings or Transactions of the Institute. The following officers were elected: *President*, Samuel P. Sadtler, Philadelphia, Pa.; *Vice-Presidents*, Chas. F. McKenna, New York City; H. A. Hunicke, St. Louis, Mo.; Edw. G. Acheson, Niagara Falls, N. Y.; *Secretary*, John C. Olsen, Brooklyn, N. Y.; *Treasurer*, Wm. M. Booth, Syracuse, N. Y.; *Auditor*, Richard K. Meade, Nazareth, Pa.; *Directors*, Ludwig Reuter, Berkeley, Calif.; Thorn Smith, Isabella, Tenn.; H. F. Brown, Wilmington, Del.; Eugene Haanel, Ottawa, Can.; J. M. Camp, Duquesne, Pa.; Charles A. Catlin, Providence, R. I.; George P. Adamson, Easton, Pa.; David Wesson, Montclair, N. J.; Edward Gudeman, Chicago, Ill.

The next meeting will be held in June, 1909, the exact time and place to be announced later.

UTAH SOCIETY OF ENGINEERS.

The November meeting of the Utah Society of Engineers was held in the Chemistry Lecture Room of the University of Utah on the 21st of the month. Papers were presented as follows:

"Suction Gas Producer Plants" (illustrated), by F. E. Johnson, of the Fairbanks-Morse Co.

"Short and Easy Methods for Calculating the Carrying Capacity of Pipes and Open Channels," by Richard R. Lyman, Univ. of Utah.

At the December meeting of the Society, held at the Commercial Club on the 19th, the papers were as follows:

"Smelter Smoke Treatment," by W. C. Ebaugh, Univ. of Utah.

"Lead Losses in the Blast Furnace," by Richard D. McCaffrey, consulting engineer.

SEVENTH INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

London May 27 to June 2, 1909.

The Seventh International Congress of Applied Chemistry will meet in London from May 27 to June 2, 1909. The Congress is under the patronage of His Majesty The King, and vice-patronage of His Royal Highness The Prince, of Wales. The President of Honor is Sir Henry Roscoe and the active President Sir William Ramsay, Mr. Wm. Macnab is general secretary and Messrs. Thos. Tyrer and C. Wightman are treasurers.

The Congress is divided into the following sections and sub-sections:

1. Analytical Chemistry, Dr. T. E. Thorpe, C.B., F.R.S.
2. Inorganic Chemistry and Allied Industries, Dr. Ludwig Mond, F.R.S.
3. Metallurgy and Mining Explosives:
   (*a*) Metallurgy and Mining, Sir Hugh Bell, Bart.
   (*b*) Explosives, Sir Andrew Noble, Bart., K.C.B., F.R.S.
4. Organic Chemistry and Allied Industries:
   (*a*) Organic Products, Prof. W. H. Perkin, F.R.S.
   (*b*) Coloring Substances and Their Uses, Prof. Meldola, F.R.S.
5. Industry and Chemistry of Sugar, Richard Garton, Esq.
6. Starch Industry:
   (*a*) Starch Industry, Dr. Horace T. Brown, F.R.S.
   (*b*) Fermentation, John Gretton, Esq., M.P.
7. Agricultural Chemistry, Lord Blyth.
8. Hygiene, Medical and Pharmaceutical Chemistry—Bromatology:
   (*a*) Hygiene and Medical Chemistry, Sir J. Crichton Brone, F.R.S.
   (*b*) Pharmaceutical Chemistry, N. H. Martin, Esq.
   (*c*) Bromatology, Robert R. Tatlock, Esq., F.I.C.
9. Photographic Chemistry, Sir W. de W. Abney, K.C.B., F.R.S.

10. Electrical and Physical Chemistry, Sir John Brunner, M.P.
11. Law, Political Economics and Legislation with reference to Chemical Industries, Lord Alverstone.

The American Committee is organized as follows:

Dr. Harvey W. Wiley, Chairman American Committee, Washington, D. C.

Members of the Advisory Committee of Honor.

Dr. John J. Abel, President of the American Society of Biological Chemists, Johns Hopkins University, Baltimore, Maryland, U. S. A.; Mr. Edward G. Acheson, President of the American Electrochemical Society, International Acheson Graphite Company, Niagara Falls, New York, U. S. A.; Dr. M. T. Bogert, President of the American Chemical Society, Columbia University, New York City, U. S. A.; Dr. C. F. Chandler, former President of the American Chemical Society, head of the Chemical Department of Columbia University, Columbia University, New York City, U. S. A.; Dr. Frank W. Clarke, former President of the American Chemical Society, and honorary member of the English Chemical Society, Geological Survey, Washington, D. C., U. S. A.; Dr. Wm. H. Nichols, Chairman of the General Chemical Company, 25 Broad Street, New York City, U. S. A.; Dr. Ira Remsen, President of the Johns Hopkins University, former President of the American Chemical Society, Johns Hopkins University, Baltimore, Maryland, U. S. A.

SECTION 1. ANALYTICAL CHEMISTRY.

Chas. Baskerville, Chairman, The College of the City of New York, New York City, U. S. A.; T. L. Briggs, 25 Broad Street, New York City, U. S. A.; Louis Munroe Dennis, Cornell University, Ithaca, New York, U. S. A.; Parker, C. McIlhiney, 7 East 42nd Street, New York City, U. S. A.; Henry P. Talbot, Massachusetts Institute of Technology, Boston, Massachusetts, U. S. A.; Fletcher P. Veitch, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., U. S. A.; Percy H. Walker, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., U. S. A.

SECTION 2. INORGANIC CHEMISTRY.

J. D. Pennock, Chairman, The Solvay Process Company, Syracuse, New York, U. S. A.; A. S. Cushman, Office of Public Roads, U. S. Department of Agriculture, Washington, D. C., U. S. A.; Clifford Richardson, Cortlandt Street Building, 32 Church Street, New York City, U. S. A.; Wm. D. Richardson, care Swift & Co., Chicago, Illinois, U. S. A.; W. H. Walker, Massachusetts Institute of Technology, Boston, Massachusetts, U. S. A.

SECTION 3. METALLURGY AND MINING. EXPLOSIVES.

Division a. Metallurgy and Mining.

John Hays Hammond, Chairman, 71 Broadway, New York City, U. S. A.; F. P. Dewey, 415–6 Colorado Building, Washington, D. C., U. S. A.; H. O. Hofman, Massachusetts Institute of Technology, Boston, Massachusetts, U. S. A.; N. W. Lord, Ohio State University, Columbus, Ohio, U. S. A.; J. W. Richards, Bethlehem, Pennsylvania, U. S. A.; R. H. Richards, Massachusetts Institute of Technology, Boston, Massachusetts, U. S. A.; Albert Sauveur, Cambridge, Massachusetts, U. S. A.; Thomas D. West, Sharpsville, Pennsylvania, U. S. A.

Division b. Explosives.

Chas. E. Munroe, Chairman, The George Washington University, Washington, D. C., U. S. A.; A. A. Breneman, 97 Water Street, New York City, U. S. A.; Arthur M. Comey, E. I. duPont de Nemours Powder Co., Chester, Pennsylvania, U. S. A.; Clarence Hall, U. S. Testing Station, 40th and Butler Streets, Pittsburg, Pennsylvania, U. S. A.; G. W. Patterson, U. S. Smokeless Powder Factory, Indian Head, Maryland, U. S. A.

SECTION 4. ORGANIC CHEMISTRY AND ALLIED INDUSTRIES.

Division a. Organic Chemistry.

William McMurtrie, Chairman, 480 Park Avenue, New York City, U. S. A.; F. D. Dodge, 69 Avenue A., Bayonne, New Jersey, U. S. A.; A. H. Gill, Massachusetts Institute of Technology, Boston, Massachusetts, U. S. A.; W. S. Gray, 76 William Street, New York City, U. S. A.; Arthur Lachman, Petaluma, California, U. S. A.; A. H. Sabin, 432 Sanford Avenue, Flushing, New York, U. S. A.; Homer E. Sawyer, 42 Broadway, New York City, U. S. A.

Division b. Coloring Substances and Their Uses.

Bernhard C. Hesse, Chairman, 90 William Street, New York City, U. S. A.; Herman A. Metz, Comptroller of the City of New York, New York City, U. S. A.; Hugo Schweitzer, 427 West 117th Street, New York City, U. S. A.; I. F. Stone, 100 William Street, New York City, U. S. A.; Louis I. Waldman, Box 162, Albany, New York, U. S. A.

SECTION 5. INDUSTRY AND CHEMISTRY OF SUGAR.

Charles A. Browne, Chairman, Mallery Building, 80 South Street, New York City, U. S. A.; A. Hugh Bryan, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., U. S. A.; David L. Davoll, care Guantanomo Sugar Co., Guantanomo, Cuba; Hubert Edson, Guanica Centrale, Porto Rico; Wm. D. Horne, Yonkers, New York, U. S. A.

SECTION 6. STARCH INDUSTRY.

Division a. Starch Industry.

T. B. Wagner, Chairman, Heyworth Building, Chicago, Illinois, U. S. A.; Arno Behr, Pasadena, California, U. S. A.; Henry Carmichael, Columbia University, New York City, U. S. A.; George J. Jenks, Huron Milling Company, Harbor Beach, Michigan, U. S. A.; George W. Rolfe, Institute of Technology, Boston, Massachusetts, U. S. A.

Division b. Fermentation.

Francis Wyatt, Chairman, 402 West 23rd Street, New York City, U. S. A.; Carl Glaser, 22 South Gay Street, Baltimore, Maryland, U. S. A.; Julius Liebmann, 109–111 East 15th Street, New York City, U. S. A.; Samuel C. Prescott, 739 Boylston Street, Boston, Massachusetts, U. S. A.; A. J. Schedler, Pabst Brewing Co., Milwaukee, Wisconsin, U. S. A.

SECTION 7. AGRICULTURAL CHEMISTRY.

C. G. Hopkins, Chairman, University of Illinois, Urbana, Illinois, U. S. A.; R. J. Davidson, Virginia Polytechnic Institute, Blacksburg, Virginia, U. S. A.; J. G. Lipman, New Jersey Agricultural College, Experiment Station, New Brunswick, New Jersey, U. S. A.; F. T. Shutt, Dominion Agricultural College Manhattan, Kansas, U. S. A.; Experimental Farms, Ottawa, Canada; J. T. Willard, Kansas.

SECTION 8. HYGIENE, MEDICAL AND PHARMACEUTICAL CHEMISTRY. BROMATOLOGY.

Division a. Hygiene and Medical Chemistry.

Reid Hunt, Chairman, Hygienic Laboratory, U. S. Public Health and Marine Hospital Service, Washington, D. C., U. S. A.; William J. Gies, 437 West 59th Street, New York City, U. S. A.; Walter S. Haines, Rush Medical College, University of Chicago, Chicago, Illinois, U. S. A.; M. E. Jaffa, University of California, Berkeley, California, U. S. A.; Walter Jones, Johns Hopkins University, Baltimore, Maryland, U. S. A.; John Marshall, University of Pennsylvania, Philadelphia, Pennsylvania, U. S. A.

Division b. Pharmaceutical Chemistry.

Joseph P. Remington, Chairman, 1832 Pine Street, Philadelphia, Pa., U. S. A.; Virgil Coblentz, Columbia University, New York City, U. S. A.; Edward Kremers, University of Wisconsin, Madison, Wisconsin, U. S. A.; A. B. Lyons, 72 Brainard Street, Detroit, Michigan, U. S. A.; Samuel P. Sadtler, N. E. Corner Tenth and Chestnut Streets, Philadelphia, Pennsylvania, U. S. A.

Division c. Bromatology.

W. D. Bigelow, Chairman, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., U. S. A.; E. M. Chamot, Cornell University, Ithaca, New York, U. S. A.; Chas. F. Langworthy, Office of Experiment Stations, U. S. Department of Agriculture, Washington, D. C., U. S. A.; Graham Lusk, 11 East 74th Street, New York City, U. S. A.; W. W. Skinner, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., U. S. A.

SECTION 9. PHOTOGRAPHIC CHEMISTRY.

L. H. Baekeland, Chairman, Yonkers, New York, U. S. A.; L. H. Friedburg, The College of the City of New York, New York City, U. S. A.; Frank W. Lovejoy, Eastman Kodak Company, Rochester, New York, U. S. A.; Henry M. Reichenbach, Yonkers, New York, U. S. A.; Maximilian Toch, 320 Fifth Avenue, New York City, U. S. A.

SECTION 10. ELECTRICAL AND PHYSICAL CHEMISTRY.

W. D. Bancroft, Chairman, 7 East Avenue, Ithaca, New York, U. S. A.; Chas. A. Doremus, 55 West 53rd Street, New York City, U. S. A.; F. A. J. FitzGerald, FitzGerald and Bennie Laboratories, Niagara Falls, New York, U. S. A.; L. Kahlenberg, University of Wisconsin, Madison, Wisconsin, U. S. A.; E. R. Taylor, Penn Yan, New York, U. S. A.; W. R. Whitney, General Electric Company, Schenectady, New York, U. S. A.

SECTION 11. LAW, POLITICAL ECONOMICS AND LEGISLATION WITH REFERENCE TO CHEMICAL INDUSTRIES.

Chas. B. Dudley, Chairman, Altoona, Pennsylvania, U. S. A.; David T. Day, Geological Survey, Washington, D. C., U. S. A.; Russell W. Moore, U. S. Customs Service, New York City, N. Y.; Wm. J. Schieffelin, 81 Fulton Street, New York City, U. S. A.; S. P. Sharples, 26 Broad Street, Boston, Massachusetts, U. S. A.; Geo. C. Stone, 71 Broadway, New York City; David Wesson, 24 Broad Street, New York City, U. S. A.

The price of membership is one pound for men and fifteen shillings for women. American chemists intending to become members should send check for five dollars ($5.00) either to one of the Chairmen of the Sections or directly to the Chairman of the American Committee at Washington.

Those intending to present papers should write to the Chairman of the Section to which the paper belongs. All papers should be neatly typewritten on thin paper and should be in the hands of the Chairman of the American Committee not later than April 1, 1909, in order to be forwarded to London to be translated and printed for use of the members of the Congress. It is earnestly hoped that a very large membership will be secured. Those who can not attend will be amply rewarded for the membership by receiving the publications which will be in several large volumes and which will contain the latest ideas and discoveries connected with the idea of applied chemistry.

H. W. WILEY.

At the meeting of the 25th annual convention of the Association of Official Agricultural Chemists held in Washington, D. C., November 12th to 16th inclusive, the following recommendations by the referees in charge of the work on "Soils and Fertilizers" were adopted:

POTASH.

(1) That the cobalti-nitrite method for potash be tested during the coming year.

(2) That there be a further trial of the method involving the use of ammonium hydroxide and ammonium oxalate in the preparation of the solution in the determination of the potash in potash salts, as compared with the present method of direct precipitation of the potash without the use of the reagents mentioned.

The referee stated that he had not been able to take up the extensive investigations that would be necessary in attempting to define available potash, and offered the following resolution which was passed by the association:

Resolved, That in view of the fact that practically the entire available time of the referee on potash is needed for the study of analytical methods, the investigation of the question of determining what should be designated as "available potash" provided for in a resolution adopted in 1906, be undertaken by a special referee or associate referee.

PHOSPHORIC ACID.

(1) That the recommendation of 1907 be repeated, namely, that the referee on phosphoric acid shall take up for report, at the next meeting of the association, methods applicable under American conditions to the official examination of basic slag phosphates.

(2) That the referee make a further study of methods for the preparation of neutral ammonium citrate.

(3) That the referee investigate the amount of wash water to be employed in the treatment of the residue from the ammonium citrate digestion.

This recommendation was amended to include a study of the manner of filtering, and was so adopted.

SOILS.

(1) That the modified J. L. Smith method for total potassium be adopted as a provisional method and be further studied (Cir. 32, p. 4).

This recommendation was adopted in the modified form, as presented by the committee, the referee having recommended its adoption as an optional method.

(2) That the sodium peroxide fusion for total phosphorus be continued as a provisional method and be further tested (Bull. **105**, p. 145).

This recommendation also was adopted in the form presented by the committee, the referee having recommended the adoption of the method as official.

(3) That the magnesium nitrate method for total phosphorus be adopted as a provisional method and be further tested.

(4) That the Knorr method for the determination of carbonates in soils be further studied (Wiley's "Principles and Practice of Agricultural Analysis," Vol. 1, ed. 1894, p. 338; ed. 1906, p. 380).

The magnesium nitrate method as outlined by the referee reads as follows:

Magnesium nitrate method for total phosphorus in soils—provisional.

Weigh into a 50 cc. porcelain dish 5 grams of salt.

Moisten with 5 to 7 cc. of magnesium nitrate solution (Bull. **107**, Rev., page 2, sec. (g)), bring to dryness on the water bath, burn off the organic matter at low redness, when cool moisten slightly with water, add 10 cc. of concentrated hydrochloric acid, and digest 2 hours on the water bath, keeping the dish covered with a watch glass and stirring two or three times during digestion.

Make up to 250 cc., mix well, and throw on a dry folded filter, pouring back on the filter until the solution runs through clear.

Make the determination on aliquots corresponding to 2 or 4 grams of the soil, depending upon the amount of phosphorus present. Bring to dryness, take up with hydrochloric acid and water, and filter over a pump. Filtrate and washings should not exceed 30 to 40 cc. Make alkaline with ammonium hydroxide, and dissolve the precipitate with concentrated nitric acid, using a slight excess. Add gradually, while shaking, 5 to 15 cc. of molybdate solution (Bull. **107**, Rev., page 2). Let stand a minute or two and add 15 cc. of ammonium nitrate (Bull. **107**, Rev., page 2), shaking thoroughly. Keep the solution at 40° to 50° C. for an hour, let stand over night at room temperature, filter[1] and wash well with cold water. Put filter and precipitate back into the same flask, using as little wash water as possible. Determine phosphorus volumetrically, using standard potassium hydroxide and nitric acid.

The eighth annual meeting of the Board of Trustees of the Committee of Revision of U. S. Pharmacopoeia, was held at Boston, May 23 to 25, 1908. Arrangements were made for placing the Spanish edition of the Pharmacopoeia on the market. It was also decided to publish a Digest of Comments on the eighth revision; these are to serve as aid in the next revision of 1910.

The 56th annual meeting of the American Pharmaceutical Association convened at Hot Springs, Arkansas, Sept. 7th to 12th. The able address of the President, Prof. Searby, of San Francisco, was devoted to Pharmaceutical educational subjects. Before the Scientific Section (Chairman, V. Coblentz), the more important papers presented were: The Crystalline Alkaloid of Calycanthus Glaucus—H. M. Gordin; Differences in the Structure of Belladonna and Scopola—H. Kraemer; The Estimation of Phenol—W. A. Puckner and A. H. Clark; Proteid Compounds of Heavy Metals—H. A. Dunning; Notes on the Estimation of Hydrastine—F. R. Eldred and C. M. Pence; Detection of Phenol and Cresotic Acids in Salicylic Acid—H. Engelhard and H. W. Jones; Deterioration of Hydrocyanic Acid, Volumetric Estimation of Phosphoric Acid, Quantity of Arsenic Present in Bismuth Salts—V. Coblentz and O. May; Standardization of Digitalis Preparations—C. E. Vanderkleed and E. Reed; The Superiority of Artificial Mineral Waters—E. Sander; Further Study of the Alkaloids of Gelsemium—L. E. Sayre, etc.; Officers elected for the ensuing year are: *Prest.*, Prof. O. Oldberg (Chicago); *Treas.*, Dr. H. M. Whelpley (St. Louis); *Gen. Sec'y*, Prof. Chas. Caspari, Jr. (Baltimore).

The time and place of the next meeting will be announced later. Total membership of the Association is 2,151.

[1] A Hirsch funnel, with a double qualitative filter S. and S. No 597, cut to fit and pressed down well around the edge after wetting and putting on pressure, is recommended.

COMMITTEE 24, INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS.

This committee, of which Prof. Henry M. Howe is chairman, deals with the question of Uniform Nomenclature of Iron and Steel. They have published a report in four parts:

(1) A polyglot of the names of the different varieties of iron and steel in English, French, German, Swedish, Danish and Dutch, together with the names of the apparatus in which they are usually made.

(2) Definitions of the principal classes of iron and steel in English.

(3) A glossary of special size and shapes of iron and steel.

(4) A note on the boundary between steel and cast-iron.

WM. CAMPBELL.

COMMITTEE ON THE ANALYSIS OF FATS, SOAPS AND GLYCERINE.

A meeting of the Committee on the Analysis of Fats, Soaps and Glycerine was held at the Hotel Belvedere, on Tuesday evening, December 29th. After listening to the reports of the special committees on the analysis of various products, it was decided that samples of the various products should be sent out not later than February first, to the largest list of chemists who had agreed to co-operate in the analytical work. Where the work entailed a large number of determinations, it was decided to take up the different determinations in small groups, two or three at a time, until the entire subject was covered.

It was decided to hold the next meeting of the Committee at Detroit, at the time of the summer meeting of the American Chemical Society.

At the meeting of the Verein Deutscher Chemiker (Pharm. Chem. Section) held on Nov. 20, 1908, the papers read were: "The Estimation of Iodine in Iodoform and Thymol Iodide," and "The Estimation of Metallic Iron in Ferrum Reductum."

MILWAUKEE SECTION, AMERICAN CHEMICAL SOCIETY.

The recently organized Milwaukee Section of the American Chemical Society held its first banquet and meeting at

the Plankington House, Milwaukee, January 14, 1909. The meeting was attended by 125 members and guests including a number of prominent manufacturers and superintendents. Two papers were read: the first by Mr. W. D. Richardson, of Chicago, entitled "Bernard Palissy, a Chapter from the Industrial Foundation of Chemistry;" the second by Prof. C. F. Burgess, of the University of Wisconsin, on the "Corrosion of Iron and Steel," illustrated with lantern slides. A lively and interesting discussion followed. The officers of the Section are: *Chairman*, Martin M. Rock; *Vice-chairman*, A. W. Gregg; *Secretary*, J. H. Linton; *Treasurer*, E. V. Manuel.

PERSONAL NOTES.

Mr. William J. Schieffelin was elected, on December 28th, chairman of the Citizens Union, of New York, to succeed R. Fulton Cutting. Mr. Schieffelin is president of Schieffelin & Company, of New York, and has been associated with many public movements.

F. E. Dodge, secretary of the Philadelphia Section of the American Chemical Society, has recently accepted a position as general manager of the Art Fibre Company, of Norristown, Pa.

D. W. Horn is consulting chemist to the Art Fibre Company, of Norristown, Pa.

Dr. Edward Gudeman, of Chicago, sailed on December 31st for Havana, Cuba, to look after his interests at that place.

Dr. G. W. Gray, formerly chief chemist with the Standard Oil Company, at Whiting, Ind., and recently with independent interests in Kansas, has gone to Bay View, New Jersey, to take charge of the laboratory in the new Standard Oil plant at that place.

The official announcement of the award of the Nobel prizes for the year 1908 is as follows:

Literature.—Prof. Rudolf Eucken, of Jena University, who has written much on philosophical subjects.

Physics.—Prof. Gabriel Lippman, of the University of Paris.

Chemistry.—Prof. Ernest Rutherford, director of the physical laboratory of the University of Manchester, England.

Medicine.—Divided between Dr. Paul Ehrlich, of Berlin, and Prof. Elie Metchnikoff, of the Pasteur Institute, Paris.

INDUSTRIAL AND TRADE NOTES.

The Chicago Second Annual Cement Show will be held in the Coliseum Building, February 18th to 24th. President Edw. W. Hagar expects 250 exhibitors, representing a capital of $250,000,000 to be represented. Thomas A. Edison will deliver the opening address and will also exhibit the castings by which a complete residence can be poured and cast in one day.

Following the great earthquake in Sicily, the price of lemon oil has gone from $0.80 per lb. to $3.00 per lb. and oil of bergamot from $3.00 to about $12.00 per lb.

A merger of independent fertilizer interests financed by J. P. Morgan & Co., has been incorporated under the name of the Independent Fertilizer Company. The capital will be $50,000,000, equally divided between common and preferred stock. For the present, the corporation will issue only $30,000,000 of this amount, divided in $15,000,000 common and $15,000,000 preferred. The output of the company, it is stated, will be 1,000,000 tons as contrasted with 2,000,000 when the merger was first considered. Forty companies distributed from Maryland to Florida and several in the west are interested, as well as potash interests of Waldemar Schmidtman in Germany. The main office of the company will be in New York.

Phelps, Dodge & Company, incorporated, capitalized at $50,000,000 have taken over the various copper properties of Phelps Dodge & Co., the output of which has been as high as 100,000,000 lbs. a year, including the Copper Queen Consolidated Mining Co., the Montezuma Copper Co., the Detroit Mining Co., of Arizona, and the Stag Canon Fuel Co.

It is reported that the Knapp Mining Co. will continue exploration and development in the newly discovered gold fields in Dunn County, Wisconsin, during the winter. The ore is in the form of decomposed quartz and silver; copper and platinum accompany the gold. In one vein the ore carried an average mining value of $10.00 per ton. A 150-ton cyanide-amalgamation plant will be installed.

At Minas Nuevas in the Parral district of Mexico, the Hidalgo Mining Co. will instal an extensive cyanide plant and make exhaustive tests with different classes of ore.

It is reported that a flowing well of petroleum has been struck between Goldfield, Nevada and Blair, Nevada. The oil shows a paraffine base and will be of great value in the mining region of Nevada.

On December 15th the Fink smelter, constructed at the expense of Samuel Newhouse, the Salt Lake mining operator, was blown in. Salt Lake mining interests are greatly interested in the results of the plant.

Agents of the Japanese government early in December bought from the owner the Pilgrim River (Alaska) antimony mine, the largest in America. The price reported is $100,000.

Cyanide mills are projected for the Nevada Wonder mine at Wonder, Nevada, and for the Bullfrog Diamond mine at Ladd Mountain.

The copper production for Arizona during 1908 approximated 300,000,000 lbs. The Copper Queen mine led with about 8,220,000 lbs., the Calumet and Arizona, and Superior

and Pittsburg yielding 4,940,000 lbs.; the Old Dominion, 3,685,000 lbs.; the Arizona Copper, 3,000,000 lbs.; the Shannon, 1,636,000 lbs.; the Detroit, 2,000,000 lbs.; United Verde, 3,139,000 lbs.: and Imperial, 750,000 lbs.

The coke oven interests of the American Coal Products Co. were turned over, January 1st, to the German American Gas Co., operators for German interests. The capital of the new company will be $2,000,000.

An important find of tin, thirty miles northeast of Eli, Nevada, near the Utah line, was reported early in December. The ore also carries a little tungsten and 11 per cent. to 30 per cent. of tin.

On December 21st, the first blast furnace of the new Indiana Steel Company's plant at Gary, Indiana, was blown in. The furnace is the first of sixteen planned for the plant. The general superintendent of the plant, Mr. W. B. Gleason, delivered an address to the department heads and guests.

The concrete poles along the lines of the Pennsylvania railroad west of Pittsburg are reported to be a success. The poles are 30 feet long, 14 inches in diameter at the bottom and 6 inches at the top.

The hurricane which struck Turk's Island on September 10th, besides doing other damage, flooded great quantities of salt which was stored in the open. The salt ponds were flooded and the total production for the year 1908 is probably not more than half that raked during 1907.

The U. S. Smelting, Refining and Mining Co. has successfully solved the problem of smelter fumes at its lead smelter in Salt Lake valley and has asked the courts for authority to operate its copper smelter again under the court's direction. The company's copper smelter, together with several others, was shut down over a year ago, on account of damage to agricultural interests by smelter fumes.

The representatives of the Anaconda Copper Co. met President Roosevelt at the White House, December 5th, to present their side of the controversy over the destruction of vegetation at Anaconda, against which the farmers of the region had protested. President Roosevelt agreed to make an investigation to ascertain whether a practical method is possible for the extraction of the poisonous substances from the fumes, and will send a representative to Ducktown, Tenn., to investigate sulphur and arsenic recovery plants at that plant. At Butte and Anaconda mass meetings were held to protest against the threatened federal injunction against copper smelter operators.

The now famous Standard Oil case, in which previously a fine of $29,000,000 was imposed, has been remanded for a new trial by the Supreme Court, a writ of *certiorari* petitioned for by the government having been refused.

The Supreme Court of Missouri, on December 23rd, handed down a decision ousting the Standard Oil Company of Indiana and the Republic Oil Company, from the state of Missouri, forbidding them ever again to do business in Missouri and dissolving the Waters-Pierce Oil Company of St. Louis. In addition, each company was fined $50,000.

In the suit to dissolve the Standard Oil Company, of New Jersey, before Ex-Judge Franklin Ferris, John D. Archbold of the company was on the witness stand November 25th and from November 30th to December 5th.

On December 16th the charge was made that peonage existed in connection with the construction of the $10,000,000 plant of the Corn Products Company, at Argo, Ill., fifteen miles from Chicago. The charges were investigated by the Federal Grand Jury. The representatives of the company have denied all the charges.

At the hearings before Chairman Sereno E. Payne's Ways and Means Committee of the House on the tariff, probably the most interesting figures who appeared during December were Andrew Carnegie, E. H. Gary and Chas. M. Schwab. Mr. Carnegie was of the opinion that a duty was no longer necessary on steel produced in the United States. Messrs. Schwab and Gary were quite as firmly in favor of the present tariff. In this connection a most interesting article entitled "My Experiences with, and Views upon the Tariff" by Andrew Carnegie, appeared in the December *Century Magazine*.

A new smelter organization, with New Jersey incorporation, Standard Oil backing and headed by the Cole-Ryan interests, is reported. The smelter now in course of construction for the Utah Consolidated Mining Co. will be the nucleus for several others which will be built on a smaller scale. It is understood that the American Smelting & Refining Co. refused to renew a contract with the Utah Consolidated at the old rate.

Construction work is well under way on the cyanamid plant of the American Cyanamid Company on the Canadian side at Niagara Falls. The plant is being built of concrete, and it is reported will use, when completed, between 60,000 and 70,000 kw. which is contracted for at a low rate with the Ontario Power Co. Three-phase calcium carbide furnaces of the Steffan type will be used, the furnaces being designed abroad. The plant will operate the Caro-Frank cyanamid process under a license from the Societa Italiana per la fabricazione di Produtti Azotati, of Rome, covering the sole right to manufacture and use, distribute and sell calcium cyanamid in the United States, her territories and colonies. Other plants will be established at a later date. The works either in operation or projected in Europe under license from the Societa are as follows: Northwestern Cyanamid Co., of London (ultimate capacity 55,000 tons) Odde, Norway, 13,750 tons; Ostdeutsche Kalkstickstoff Works of Berlin, Mulhthal, Prussia, 3,300 tons; Société Suiss Probera Azotati, Martigny, Switzerland, 4,400 tons; Société Français Pour Produis Azotati of Paris, Notre Dame de Briancon, Savoy, France, 13,200 tons; Cyanamid Gesellschaft of Berlin, Trosberg, Bavaria, 16,500 tons; Societa Italiana pel Carbura di Calcio of Rome, Collestatti, Italy, 13,750 tons; Societa

Italiana pel Carbura di Calcio of Rome (projected), Sebenico Dalmatia, Austria, 27,500 tons; Societa Italiana pel Carburo di Calcio, of Rome (projected), Almissa, Dalmatia, 50,000 tons; San Marcel Company, Italy, 8,250 tons; Societa Italiana per la fabricazione di Produtti Azotati of Rome, Piana d'Orta, Italy, 11,550 tons; Societa Italiana per la fabricazione di Produtti Azotati, of Rome, Fiume, Dalmatia, 4,400 tons.

The use of sulfite pitch (selpech) for briquetting coal in Rhenish Westphalia is reported by Consul George Eager, of Barmen, in *Daily Counsular Report* No. 3361.

A damage suit brought by Shoup and Jaynes *vs.* the Utah Consolidated Mining Co. and the United States Smelting, Refining & Mining Co., for alleged injury to sheep, was decided in favor of the defendants in the United States District Court at Salt Lake City on December 15, 1908. This is one of the few smelter smoke cases that have not resulted in an award of damages.

The United States Smelter at Bingham Junction, Utah, is adding an extension to its bag house to take 1200 bags. The results of bag house treatment of smoke at the two smelters in the Salt Lake valley are quite satisfactory.

The plant of the Inland Crystal Salt Co., at which the salt obtained from the water of Great Salt Lake by solar evaporation was purified, was totally destroyed by fire on the evening of November 5th. The loss is placed at $125,000.00 with $50,000.00 insurance. Plans for rebuilding on an enlarged scale are now under consideration.

The Utah Consolidated Mining Company has made a contract with W. D. Thornton and others, of Butte, for the treatment of its ores at a new smelter to be erected in Tooele Valley, southwest of the Garfield plant of the American Smelting & Refining Co. The statement given to the press by President Broughton, of the Utah Consolidated Company, is as follows:

"This company has now entered into a contract with W. D. Thornton, of Butte, for the smelting of its ores for a period of ten years, commencing April, 1910, the proper performance of which contract is guaranteed by four persons acceptable to the board of directors. Under the provisions of this contract Mr. Thornton agrees:

"(1) To erect a smelting plant in Tooele County, Utah, to be in operation by April, 1910, to treat the output of the mine up to 1200 tons per day, at a rate which is 67.20 cents per ton more favorable to our company than the contract now existing with the Garfield Smelting Company.

"(2) To acquire from this company, at cost, all the lands and options it has acquired in Tooele County.

"(3) To give this company an option upon any part of $500,000.00 par value of the capital stock of the company to be formed to operate the new smelter upon as favorable terms as may be extended to any other subscriber to stock.

"The cost to this company of transporting ore to the Tooele smelter will not exceed 15 cents per ton.

"All figures in connection with the existing contract and that entered into with Mr. Thornton have been approved by E. P. Mathewson, of Anaconda, this company's consulting engineer."

It will be remembered that the smelter of the Utah Consolidated Company near Murray, Utah, is now closed as a result of the injunction granted by the United States Court on complaint of an association of farmers.

The Association of American Portland Cement Manufacturers met at the Hotel Knickerbocker, New York, from December 7, to December 12, 1908. The companies represented have a combined output of 90,000,000 barrels or 92 per cent. of the total output in the United States. In this country there are at present 92 operating cement mills with a combined capacity of 98,534,000 bbls. About 125 new mills are being promoted. In his address, John B. Lober, the president, stated that a review of the cement industry for 1908 shows that for the first time it has reached the condition characteristic of other great industries of the United States.

In the 10 years ending with 1907, production has increased from 2,430,000 pounds to 48,785,000 pounds. The decreased percentage of growth during the last two years indicates that the business has reached a period of ordinary industrial development, and that future development must be based upon normal increase of demand.

Denatured Alcohol in France.

Present Extent of Manufacture—Prices and Influences.—Consul-General Frank H. Mason, of Paris, has prepared the following review of present and prospective conditions affecting the denatured alcohol product in France:

The total production of alcohol in France during the five years ending with 1907 has been as follows, the statistics being given in hectoliters of 100 liters, equal to 26.42 gallons:

Year.	Total production. Hectoliters.	Average wholesale price per hectoliter.
1903	2,047,000	$8.29
1904	2,257,000	8.49
1905	2,609,000	8.68
1906	2,710,000	8.10
1907	2,514,810	8.29

The substances from which the output of alcohol in 1907 were distilled, and the proportion derived from each source, were as follows:

Derivatives.	Hectoliters.	Gallons.
Grains	454,142	11,998,430
Molasses	466,608	12,327,783
Beets	1,145,433	30,262,339
Wines	239,877	6,337,550
Apples and pears	69,283	1,830,456
Grape skins and lees	122,608	3,239,303
Other fruits	16,658	440,104
Various substances	201	5,310
Total	2,514,810	66,441,275

Besides the foregoing native supply, France imported in 1907, mainly from Great Britain and Germany, 153,766 hectoliters (4,062,497 gallons) of alcohol and exported 300,532 hectoliters, being an excess of exports over imports amounting to 146,766 hectoliters, or 3,877,557 gallons. Deducting this excess from the total of production and im-

ports, there remained a supply of 2,368,044 hectoliters (62,563,722 gallons) for the various purposes of consumption.

Production and Consumption.—Alcohol for industrial and other purposes is denatured in France, in presence of a government official, by mixing with each hectoliter, or 26.42 gallons, of spirit the following standard denaturing mixture: 15 liters of methyl alcohol, ½ liter of heavy benzine, 1 gram of malachite green. The cost of these ingredients in the above quantities is about 19.86 francs, or $3.83, which adds that amount to the price of 115½ liters of denatured alcohol and imposes what many consumers consider a burdensome tax on methylated spirits. Notwithstanding this the consumption of denatured alcohol in France is steadily increasing, having grown from 146,529 hectoliters (3,871,296 gallons) in 1897 to 593,836 hectoliters (15,689,147 gallons) in 1907.

The various uses to which denatured spirit is applied and the relative proportion consumed in each during the past five years will be shown by the following table, in which the statistics of consumption are given in hectoliters:

Year.	1903.	1904.	1905.	1906.	1907.
Heating and lighting	262,036	289,748	315,079	374,506	401,230
Varnish	11,580	12,433	11,544	13,444	13,386
Cabinet-making	2,502	1,890	1,757	1,702	1,190
Celluloid, etc	20,095	18,771	21,293	14,589	17,133
Hat manufacture	365	234	228	186	297
Dyes and colors	532	391	554	1,071	632
Rennet, liquid	142	111	148	139	178
Collodion	146	272	417	697	750
Chloroform	377	174	186	146	101
Chloral	246	302	382	227	155
Tanning	798	1,549	567	484	510
Chemicals, soap, artificial silk, etc	11,381	6,905	6,770	6,865	10,400
Scientific uses	519	864	1,193	2,457	1,302
Ether, fulminates, and explosives	63,879	89,917	112,121	128,954	146,572
Total hectoliters	374,598	423,561	472,239	545,467	593,836

This shows a total denaturation in 1907 of 15,689,246 gallons. It thus appears that the increased consumption of denatured alcohol in France is due almost entirely to two groups of uses, namely, for heating and lighting purposes, and for the manufacture of ether and explosives.

The use of alcohol as fuel for motors has not yet attained in France the importance that was anticipated when the internal-revenue tax was reduced to a merely nominal rate of 5 cents per hectoliter in 1901, because of the high cost of denaturing materials, the tendency of alcohol vapor when burned to corrode the interior of cylinders, and the fact that alcohol, without a large admixture of benzol or gasoline, does not explode with sufficient rapidity to meet the requirements of automobile practice, and requires to be used in motors with valves and ignition apparatus specially adapted to that purpose.

The cost of denatured spirits fluctuates naturally in France as elsewhere with the wholesale market value of pure alcohol, which has ranged during the past year from 47.6 francs ($9.18) per hectoliter in June last, to 44 francs ($8.49), which was the mean price for first quality, 90° spirit (not including the entrepôt tax of 39 cents per hectoliter) in the wholesale market of Paris during the week ended October 17th.

The alcohol used for denaturation is generally of the second and third qualities, and the open-market values of denatured spirits in Paris on October 27th were as follows: First quality, 55 francs per hectoliter, or $10.61 per 26½ gallons; second quality, 42 francs, or $8.10. The retail price was 0.65 franc per liter, or $0.47 per gallon.

Paris imposes no *octroi* tax upon denatured alcohol, whereas alcohol of 60°, not denatured, is subject on entry into the city to a municipal tax of 2.79 francs, or 53 cents, per liter.—*Daily Consular Report*, Nov. 30, 1908.

Consul James G. Carter makes the report from Tamatave that petroleum has been recently discovered in Madagascar, the particulars being as follows:

The oil occurs in the nature of several springs which occur in the regions of the Manambolo and Manamboa rivers, on the west coast of the island, and while there has not yet been any official announcement of concessions being granted for oil prospecting, I have been reliably informed that there are several concession holders [names and addresses are obtainable from the Bureau of Manufactures]. Thus far no serious work has been done on the fields, but it is believed that extensive operations will commence shortly.

According to a local newspaper, a colonist residing in the environs of Tullear, on the southwest coast of Madagascar, has recently shipped to the governor-general at Tananarive a sample block of 60 kilos (132 pounds) of coal, said to have been taken from existing coal deposits found in the regions of Tullear.—*Daily Consular Report*, Dec. 5, 1908.

Japanese Camphor Trade.

Consul John H. Snodgrass writes from Kobe that for the period from January to September, 1908, there was an appreciable decrease in the exports of camphor from Japan, they being reduced to 1,934,000 pounds, valued at $844,500 and 760,000 pounds of camphor oil, worth $53,500. The details follow:

The following table shows the amount and value of camphor exports for the first eight months of 1906, 1907, and 1908:

	1906.	1907.	1908.
Pounds	2,453,334	3,236,667	1,938,000
Value	$1,140,000	$2,030,000	$844,500

On May 1, 1907, the market price was 140 yen per 100 kins ($70 per 132.51 pounds), and refined camphor was 220 to 230 yen ($110 to $115), but the ruling price has gone down as low as one-third of that of 1907, and yet the demand abroad is not brisk. The monopoly bureau has lowered the price to 80 yen ($40), but stocks are plentiful and the market price is gradually decreasing, causing a number of dealers to close up business. One of the reasons offered for the loss of this foreign trade is the keen competition caused by the introduction of German artificial camphor and the entrance of China into the lists for supplying the natural product.—*Daily Consular Report*, Dec. 12, 1908.

Zinc Mining in Mexico.

Working and Output of the Mines in the State of Chihuahua.—In compliance with instructions, Consul Louis A. Martin, with the coöperation of H. B. Pulsifer, mining expert, has prepared the following report covering the zinc mine industry of Chihuahua:

Zinc ore bodies are found in the following camps in the State of Chihuahua: (1) Picachos, on the Kansas City, Mexico and Orient Railroad, less than 100 miles from Presidio del Norte, opposite the Texas border, where carbonate ores are mainly produced, sulphides occurring, but not on a commercial scale; (2) Santa Eulalia, near the city of Chihuahua, produces carbonate ore exclusively; (3) near San Ysidro, on the Chihuahua and Pacific Railroad, where sulphides only are produced; (4) Almoloya, where carbonate and silicate are produced, and Parral and Santa Barbara, producing sulphides only.

The zinc ore bodies are found associated with silver-lead mines, and in this way partake of the development of the latter, although certain mines might properly be called zinc mines. In every case, however, silver-lead ores are those sought for and mined. A conservative estimate places the crude zinc-bearing ores, more or less well exposed, in the State at 1,000,000 tons, *viz.*, Picacho, 50,000 tons; Santa Eulalia, 100,000 tons; San Ysidro, 200,000 tons; Almoloya, 50,000 tons; Parral and Santa Barbara, 600,000 tons. The probable available amount is many times this, most of the present mining being at very moderate depths.

The carbonate and silicate ores are running from 32 to 45 per cent. metallic zinc, as broken off, after the merest hand sorting. Crude sulphide ores will run from 10 to 35 per cent. metallic zinc. The carbonate ores always carry a trifling amount of silicate, sometimes a little lead carbonate, calcium carbonate, and iron minerals.

Disposition of Sulphide.—The sulphide ores are always complex and must, as at present disposed of, be milled. A silver-lead concentrate, possibly carrying some iron and gold, is the marketable product always procurable at the same time as the zinc concentrate.

The mines are in all possible conditions of development, from the simplest tunnel adit, where the ore is found at or above the tunnel level and is simply broken down, run through chutes into the mine car, and pushed to the open, to where the ore is found at moderate depths, 100 to 400 feet, and reached by shafts and drifts or by tunnels and internal hoists. Ore is seldom found at considerable depths, as at 1,600 feet, but in such case it is reached by high-speed first-motion hoists. This special case is in a very large and rich silver-lead mine, where the zinc ore may be said to be a valuable by-product.

A considerable part of the ore is mined by hand drilling, but power drills, worked by compressed air from steam or gas engines, supplied with producer gas, are used in the larger mines and will supplant hand drilling as the mines become developed.

Sulphide ores present hard and complex milling difficulties. Formerly zinc ores were a detriment to sulphide ores. The ores being valued for the silver-lead content and disposed of to the smelters without removing the zinc, were not only losing their zinc value, but were penalized for its presence; but from a detriment it is now changed to a valuable product, consequent on the art of separating and utilizing the zinc portion. Mines equally rich as those worked are found idle and unequipped in developing plants, owing to local conditions and management.

Even during these times of unusual depression in the metal market, experiments on a large scale are in progress at several of the mines, and the future industrial development will surely be of great magnitude.

Cost of Production.—The question of the cost of production of zinc ore presents certain complications from the fact that the ore occurs with other valuable ores, as at Santa Eulalia, or that other valuable products are obtained at the same time, as at the mine near San Ysidro, or from the fact that nearly all the mines are at present working much under normal capacity.

At Picachos the case is simple; zinc ore alone is produced, and practically all the cost is that of labor. The total cost (mining and hauling to points in Kansas) should not exceed $12.32 (American currency, which is the unit used throughout this report) per ton.

From the mine near San Ysidro, where mining cost is very low, where 45 per cent. zinc is produced from zinc concentrate, with silver-lead concentrate, the total cost (mining, handling, freightage, etc., to points in Kansas) should not be much more than $13.57 per ton.

In Santa Eulalia the cost of production is also very low, and the total cost, laid down at points in Kansas, should not exceed $9.75 per ton.

The foregoing mines are producing almost the whole amount now being shipped from the State of Chihuahua, which for September and October was about 125 tons per day.

From Picachos comes carbonate, lead-free, 45 per cent. of zinc; from San Ysidro, sulphide concentrate, 45 per cent. of zinc; from Santa Eulalia, carbonate, lead-free, 35 per cent. of zinc.

As far as known, there are no facilities for smelting zinc ores in Mexico. Coal and wood, the chief fuels in Chihuahua, are too expensive, the wholesale prices being as follows per ton: Coal, $6 to $8; wood, $3 to $5. The production of zinc ore and concentrates is a new industrial development, and, so far as known, the product is shipped to the United States exclusively.—*Daily Consular Report*, Dec. 7, 1908.

Responding to a New York inquiry in regard to the new patent act in the United Kingdom, Consul J. N. McCunn writes from Glasgow, as follows:

With regard to the clause "to an adequate extent," I am informed that it would appear to be perfectly in order, in the meantime, to manufacture the parts of a machine in the United States and have them assembled in the United Kingdom. The ground on which this view is taken is that each part of the machine, taken separately, is not a patented article, the patent merely applying to the machine as a whole. No test case has yet been taken in the British courts, and I am informed that the above is the generally accepted reading of the law until such a test case is brought.

A company has recently been started in Glasgow having for its object the safeguarding of the property of foreign patentees selling in the British Isles, and one of their pamphlets, which gives excerpts from the patent act, is forwarded and may be obtained from the Bureau of Manufactures.—*Daily Consular Report*, Dec. 14, 1908.

(It is understood that the British Patent authorities do not agree with the opinion expressed above, but take the contrary view.—Ed.)

Consul-General Robert P. Skinner, of Hamburg, writes under date of November 16th, that very little refined and

caustic potash had been used during the previous few months and that the market was comparatively lifeless, to which he adds:

There is now some improvement, inquiries are coming in, and for 1909 contracts have been closed at higher prices. Refined potash is quoted to-day, per 100 kilos (220 pounds), as follows: 80 to 85 per cent. goods, 32 marks ($7.62); 90 to 92 per cent. goods, 34.5 marks ($8.21), including original barrels of 500 kilos (1,100 pounds), f. o. b. Hamburg. Net cash fo lots of 5,000 kilos (5 tons).

Caustic potash of non-German origin is quoted to-day, per 100 kilos: 70 to 72 per cent. goods, 45 francs ($8.68); 74 to 76 per cent. goods, 47.5 francs ($9.17); 80 to 85 per cent. goods, 51.5 francs ($9.94), including drums, containing about 250 kilos (550 pounds), f. o. b. Antwerp. Cash less 2 per cent. discount by orders of 5,000 kilos (5 tons).

The exports of potash from Hamburg in 1906 were 6,276 tons, valued at $496,449, and in 1907, 7,436 tons, valued at $588,029.

(The exports of chloride of potassium from all Germany in 1907 amounted to $5,995,934, of which a large share is shipped to the United States.—B. of M.).—*Daily Consular Report*, Dec. 19, 1908.

Niagara Power Distribution.—Consul Harry A. Conant, of Windsor, sends the following account of the present status of the distribution in Canada of electric power from Niagara Falls:

An assurance that Windsor can secure Niagara power under the Ontario government scheme for about half the cost of production by coal and for at least $5 a horse-power less than Toronto corporations are paying the Electrical Development Company for power from the falls, was given by Hon. Adam Beck, minister of power in the Ontario cabinet, in discussing the government power proposition at a public meeting held in Windsor, December 1st.

In addition to paying the actual cost of $9 or $9.40 a horse-power at the falls, municipalities will be required to pay operating expenses, besides 4 per cent. of the actual cost of construction transmission lines, and a sinking fund sufficient to wipe out the cost of construction in thirty years, each municipality being assessed its share.

One point on which Mr. Beck laid emphasis is that the rates to be charged by the city after purchasing power from the government commission are subject to regulation by the commission. The municipality can not sell for less than cost, and it is thus prevented from bonusing industries by offering free power, while a protest from any rate payer who believes the rate is too high will be sufficient to insure an arbitration by the commission.

Work on the western extension of the transmission line can be carried on while the building of the line already contracted for is under way. In that case the line to Windsor ought to be finished in little more than a year.—*Daily Consular Report*, Jan. 5, 1909.

Sulphur in Chile.—Consul Rea Hanna, of Iquique, in response to a California inquiry, has secured from a firm in Chile information in regard to the deposits and refining of sulphur in Chile. The agent of the refinery at Tacna writes as follows:

As to the advisability of securing crude native sulphur in Tacna and its neighborhood and shipping same to the United States for refinement or to be used there, the crude sulphur is never sent to Tacna, nor yet is it made up for exportation, as the danger is that in the crude state there is a foreign matter that would burn any class of sack in which it might be shipped.

The two classes of sulphur to be secured in this district are: (1) Azufre sublimado or flour sulphur of a ley of 99.82 and 75°; (2) refined sulphur in lumps of a ley of 99½. The price of the former is $6.30 Chilean (at 18 pence exchange = $2.2995 American gold) per quintal (101.61 pounds) and the refined at $4.30 ($1.5695 American gold). Both these prices are cash down for the goods placed in the railway station at Tacna. To insure a general all-round supply contracts must be made and a few commissions allowed.

There is already some scheme to secure the whole production, but we are without full details. From one source a supply of 500 quintals monthly can be contracted, and this can be easily doubled should the business appear feasible, on the terms indicated.—*Daily Consular Report*, Jan. 9, 1909.

Consul Maxwell Blake, of Dunfermline, writes that approximately 1,500 tons of acetone, valued at about half a million dollars, are annually consumed in Great Britain, practically all of which is imported from the United States. His details of this trade follow:

There seems to be but one company manufacturing this article in Great Britain. By far the larger percentage of this import is utilized as the solvent ingredient of smokeless powder, guncotton, and mine explosives; but it is also employed in the making of chloroform, for the preparation of photographic sensitive plates, and as a valuable constituent of certain dyes.

Up to the present time all efforts on the part of the British Government, as well as of various private chemical concerns in this country, have utterly failed to provide acetone in quantities sufficient to supply the commercial demand for it. It is now stated, however, that a simplified method of wood distillation lately discovered and put in practice by a certain French chemist, has gone far toward successfully cheapening its production, it being now successfully manufactured as a by-product of charcoal. In consequence of this, Great Britain expects to look more to France than formerly as a source of supply for this product. A technical description of this new French method of wood distillation, which is also likely to soon be imitated by British manufacturers of this product, can easily be procured.—*Daily Consular Report*, Jan. 15, 1909.

Recently Representative Hull introduced a bill into Congress, the purpose of which is to regulate interstate commerce of misbranded products of all kinds. As considerable opposition has developed, the bill will probably remain with the committee.

A bill for the taxation of bay rum from Porto Rico on the same basis with other products from the island has been introduced into Congress by Chairman Payne.

The Mann bill introduced into the House last spring, will

come up for action in the Committee on Interstate and Foreign Commerce shortly. This measure is strenuously opposed by the National Wholesale Drug Association, the Proprietary Association of America and the National Association of Retail Pharmacists. This bill not only prohibits the sale of all narcotics, their derivatives and preparations except on the prescription of a physician, dentist or veterinarian. It also compels that a poison label be placed on all preparations containing even an infinitesimal amount of such oils as pennyroyal, and bitter-almonds, salts of copper, lead, silver, zinc, hydroxides of the alkalies and concentrated mineral acids.

In the suit to dissolve the Standard Oil Company, of New Jersey, before Special Examiner Franklin Ferris, Mr. W. W. Tarbell testified on January 13th that the United States Pipe Line Company, a Standard competitor, is now and has for several years been operating as a common carrier under the laws of Pennsylvania Mr. Tarbell is general manager of the United States Pipe Line Co. and Pure Oil Co.

In the United States District Court at Chicago, on January 13th, one of the most important patent litigations of recent years was started by the Union Carbide Company, of New York, against the American Carbolite Company, of Duluth. The Union Carbide Company is capitalized at $14,000,000 and has factories at Niagara Falls and Sault Ste. Marie and owns numerous patents on the production of calcium carbide and processes of manufacture. The charge against the Duluth Company is an infringement of the fundamental patent, in addition to damage sustained by the Union Carbide Company in sale and use of this product and perpetual injunction is asked.

Mr. Geo. O. Knapp is president and Mr. W. P. Martin, vice-president of the Union Carbide Company. The directors are C. K. G. Billings, A. N. Brady, E. N. Dickerson and C. F. Deiterich; A. B. Proal is treasurer. The American Carbolite Company started to manufacture and sell carbide over a year ago. Suits against the company have previously been started in New York and Ohio courts.

The demands made in the bill filed in the present instance are: that the American Carbolite Company be enjoined perpetually from using crystalline calcium carbide in any way; that it be forced to appear in court and give an account of the profits derived from such use of the patent; that it be forced to pay damages to the Union Carbide Company; that the material on hand in the plants of the American Carbolite Company be destroyed; and that the defendant pay the costs of the suit.

It is set forth in the bill that calcium carbide was discovered by Thomas Willson, an electrical engineer, through experiments in his laboratory in Spray, N. C., in 1892. The manner of the discovery is set forth in a report of the department of chemistry of the University of Pennsylvania as follows:

"Mr. Willson was conducting experiments with a view to the preparation of metallic calcium, for which purpose he employed an improved Héroult electric furnace with a current of 2,000 amperes and 36 volts, operating upon a mixture of lime and coal-tar. He secured a melted mass of dark color, which on cooling became solid and brittle. This product was thrown into a neighboring stream as useless, when to the astonishment of those who saw it, there was liberated suddenly a great quantity of gas which on being kindled, burned with the now familiar bright but smoky flame, and gave off quantities of soot which showed that it was a rich hydrocarbon gas. The smelt was repeated, the product submitted to analysis, which demonstrated that the solid substance contained calcium carbide and that the gas was acetylene."

In June, 1895, it is declared in the bill, Mr. Willson obtained a patent from the United States government upon crystalline calcium carbide, the patent dating June 18, 1895, and running for seventeen years.

August 24, 1903, it is further declared this patent was sold to the Union Carbide Company. The process, however, was an open one, and a year ago the American Carbolite Company started up with this patent as a basis of its manufactures, according to the bill. Several warnings have been issued by the Union Carbide Company to all concerns engaged in the manufacture and sale of the carbide, but the American Carbolite Company, it is said, has refused to heed these warnings and has threatened to go ahead. The bill was filed by the firm of Offield, Towle and Linthicum.

The United States Geological Survey has issued the following letter and circular which are self-explanatory.

DEPARTMENT OF THE INTERIOR UNITED STATES GEOLOGICAL SURVEY, WASHINGTON.

Office of the Director. January 9, 1909.

SIR:

A series of preliminary tests of explosives in the presence of coal dust, inflammable gas, etc., has now been completed at the United States Geological Survey testing station at Pittsburg, Pa., and in the course of a few days this Survey will be ready to begin official tests of explosives which the manufacturers thereof may desire to have tested as to their permissibility for use in mines containing gas, gas and dust, or dust in dangerous quantities.

I enclose herewith a statement of the test requirements to which the various explosives manufactured for use in such mines will be subjected at this station. The conditions under which these tests are to be made are as follows:

(1) The manufacturer is to furnish 100 pounds of each explosive which he desires to have tested; he is to be responsible for the care, handling, and delivery of this material at the testing station on the United States Arsenal grounds, Fortieth and Butler Streets, Pittsburg, Pa., at the time the explosive is to be tested; and he is to have a representative present during the tests who will be responsible for the handling of the packages containing the explosives until they are opened for testing.

(2) No one is to be present at or to participate in these tests except the necessary Government officers at the testing station, their assistants, and the representative of the manufacturer of the explosives to be tested.

(3) The tests will be made in the order of the receipt of the applications for them, provided the necessary quantity of the explosive is delivered at the plant by the time assigned, of which due notice will be given by the Geological Survey.

(4) Preference will be given to the testing of explosives that are now being manufactured and that are in that sense already on the market. No test will be made of any new ex-

plosives which is not now being manufactured and marketed, until all explosives now on the market that may be offered for test have been tested.

(5) A list of the explosives which pass certain requirements satisfactorily will be furnished to the state mine inspectors, and will be made public in such further manner as may be considered desirable.

If you are manufacturing any explosive which you desire to have tested under the conditions specified above, please notify this office to that effect and, at the same time, send a copy of your communication to Mr. J. C. Roberts, U. S. Geological Survey, Fortieth and Butler Streets, Pittsburg, Pa.

It is expected that subsequent to the completion of these tests of explosives for the manufacturers, the station will also make tests of these explosives as they are supplied for use in coal mines in the various states.

Very respectfully,

GEO. OTIS SMITH,
Director.

DEPARTMENT OF THE INTERIOR, UNITED STATES GEOLOGICAL SURVEY.

Test Requirements for Explosives Used in Coal Mining.

The tests will be made by the engineers of the United States explosives testing station at Pittsburg, Pa., in gas and dust gallery No. 1. The charge of explosive, to be fired in tests 1 and 2, shall be equal in disruptive power to one-half pound of nitroglycerine dynamite in its original wrapper, of the following formula:

	Per cent.
Nitroglycerine	40
Nitrate of sodium	44
Wood pulp	15
Calcium carbonate	1
	100

Each shot shall be fired with an electric fuse of sufficient power to completely detonate or explode the charge, as recommended by the manufacturer. The explosives must be in such condition that the chemical and physical tests do not show any unfavorable results. The explosives in which the charge used is less than 100 grams will be weighed in tin foil without the original wrapper.

The dust used in tests 2, 3, 4, and 5, will be of same degree of fineness and taken from one mine.

Test 1.—Ten shots with a charge as described above, in the original wrapper, shall be fired, each with 1 pound of clay tamping, at a gallery temperature of 77° Fahrenheit, into a mixture of gas and air containing 8 per cent. of methane and ethane. An explosive will pass this test if all ten shots fail to ignite the mixture.

Test 2.—Ten shots with charge as previously noted, in its original wrapper, shall be fired, each with 1 pound of clay tamping at a gallery temperature of 77° Fahrenheit, into a mixture of gas and air containing 4 per cent. of methane and ethane and 20 pounds of coal dust, 18 pounds of which is to be placed on shelves laterally arranged along the first 20 feet of the gallery, and 2 pounds to be placed near the inlet of the mining system in such a manner that all or part of it will become suspended in the first division of the gallery. An explosive will pass this test if all ten shots fail to ignite the mixture.

Test 3.—Ten shots with charge as previously noted, in its original wrapper, shall be fired, each with 1 pound of clay tamping at a gallery temperature of 77° Fahrenheit, into 40 pounds of bituminous coal dust, 20 pounds of which is to be distributed uniformly on a horse placed in front of the cannon and 20 pounds placed on side shelves in sections 4, 5 and 6. An explosive will pass this test if all ten shots fail to ignite the mixture.

Test 4.—A limit charge will be determined within 25 grams by firing charges in their original wrappers unstemmed at a gallery temperature of 77° Fahrenheit, into a mixture of gas and air containing 4 per cent. of methane and ethane and 20 pounds of bituminous coal dust, to be arranged in the same manner as in test 2. This limit charge is to be repeated five times under the same conditions before being established.

Test 5.—A limit charge will be determined under the same conditions as in test 4, except that 2 per cent. of methane and ethane will be used instead of 4 per cent.

Note.—At least 2 pounds of clay stemming will be used with slow-burning explosives.

WASHINGTON, D. C.,
January 8, 1909.

OFFICIAL REGULATIONS AND RULINGS.

FOOD INSPECTION DECISION 98.

The Labeling of Whiskey Compounds.—The labeling of whiskey compounds, under the Food and Drugs Act of June 30, 1906, will be governed by the opinion of the Attorney-General, dated December 1, 1908, published herewith.

(Signed) JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., *December 4, 1908.*

DECEMBER 1, 1908.

The Honorable, the Secretary of Agriculture.

SIR: I am duly in receipt of your letter of this date. In this you call my attention to a passage in my opinion of April 10, 1907, addressed to the President, which passage is in the words following:

I conclude that a combination of whiskey with ethyl alcohol, supposing, of course, that there is enough whiskey in it to make it a *real* compound and not a mere semblance of one, may be fairly called "Whiskey," provided the name is accompanied by the word "Compound" or "Compounded," and provided a statement of the presence of another spirit is included in substance in the title—and you ask me how much whiskey there must be in a mixture of whiskey and neutral spirits to fairly entitle this mixture to be called a "Compound" or "Compounded" whiskey, or, as stated in your letter, "whiskey: a compound of pure grain distillates."

In the passage in question I stated that there must be, in any such a mixture, "enough whiskey * * * to make it a *real* compound and not a mere semblance of one." In the absence of any legislative provision or judicial determination on this subject, the proportion of whiskey necessary for the purpose in question can be stated only tentatively and for the time being; and a selection of any particular fraction of the whole as a necessary proportion must be, at least in appearance, somewhat arbitrary. I have, however, very carefully examined the evidence on this subject,

submitted by your department, and, after full consideration of such evidence, have reached the conclusion that, until better informed in the premises from the action of the Congress or of the courts, this department will not advise a prosecution on the ground of violation of law in using any one of the three labels above suggested or any substantial equivalent therefor when the amount of whiskey in the mixture equals or exceeds one-third in volume of the spirituous content; that is to say, in the case you mention, one-third of the whiskey and neutral spirits combined.

Very respectfully,

CHARLES J. BONAPARTE,
Attorney-General.

FOOD INSPECTION DECISION 99.

Change in Form of Guaranty Legend. (Amending Section *b* of Regulation 9).—Section 9 of the Food and Drugs Act, June 30, 1906, provides that no dealer shall be prosecuted under the provisions of the act when he can establish a guaranty signed by the wholesaler, jobber, manufacturer, or other party residing in the United States, from whom he purchases such articles, to the effect that the same are not adulterated or misbranded within the meaning of the act. There is a further provision that the guarantor shall, if the goods be adulterated or misbranded within the meaning of the act, be amenable to the prosecutions, fines, and other penalties which would attach in due course to the dealer.

Section *b* of Regulation 9 provides that a general guaranty may be filed with the Secretary of Agriculture by the manufacturer or dealer and be given a serial number, which number should appear on each and every package of goods sold under such guaranty, with the words "Guaranteed under the food and drugs act, June 30, 1906."

It is obvious from a reading of Section 9 of the act that the guaranty is in no sense a guaranty by the Government, and that it is merely an assumption of responsibility for the character or labeling of the goods by the manufacturer, jobber, or packer. Yet, notwithstanding this plain fact, attempts have been made by some unscrupulous persons to cause the public to interpret the phrase "Guaranteed under the food and drugs act, June 30, 1906," as a guaranty by the government that the goods upon which the phrase appears are pure and conform, in all respects, with the provisions of the act. This misrepresentation has been scattered broadcast in prominent advertisements in the press, and by means of circulars and billboard posters. Even in the absence of such misrepresentation there can be no doubt that the phrase, unfortunately, is misleading, and is therefore prohibited by the law and should be changed. The Commissioner of Patents has refused to register trade-marks of which the phrase formed a part, on the ground that it is misleading and under the law can not be registered. The Board of Food and Drug Inspection for some time has realized that the wording of the guaranty legend should be changed, but it has also been mindful of the fact that the manufacturers and jobbers of the United States have, in the aggregate, large sums of money invested in labels and plates, upon which appears the legend in its present form, a form indorsed by the regulations and copied therefrom in good faith by the owners of these labels and plates. Entirely apart from the expense and loss of property, it is a fact that a change in the form of the legend, without due notice, would seriously embarrass business interests, because the printing and lithographing of new labels will require considerable time.

As a solution of the question, the Board recommends that the guaranty legend be changed so as to show plainly that the guaranty is that of the manufacturer and not of the government, that the old form of labels now in use representing guaranties already filed with the Department of Agriculture shall be recognized for a term of two years, and that for all guaranties filed with the Department of Agriculture on and after January 1, 1909, the guaranty legend shall read "Guaranteed by [insert name of guarantor] under the food and drugs act, June 30, 1906."

Accordingly, the following amendment is proposed to Regulation 9 of the Rules and Regulations for the Enforcement of the Food and Drugs Act:

Section *b* of Regulation 9 is hereby amended to read as follows:

(*b*) A general guaranty may be filed with the Secretary of Agriculture by the manufacturer or dealer and be given a serial number, which number shall appear on each and every package of goods sold under such guaranty with the words "Guaranteed by [insert name of guarantor] under the food and drugs act, June 30, 1906."

This amendment shall become and be effective on and after January 1, 1909. Labels bearing the form of guaranty legend provided in the original regulations and representing guarantees now on file with the Department of Agriculture may be used for a period of two years, but it is suggested that, as new labels are prepared, the change in the form of guaranty legend should be made.

H. W. WILEY,
F. L. DUNLAP,
GEO. P. MCCABE,
Board of Food and Drug Inspection.

Approved:

GEO. B. CORTELYOU,
Secretary of the Treasury,
JAMES WILSON,
Secretary of Agriculture,
OSCAR S. STRAUS,
Secretary of Commerce and Labor.

WASHINGTON, D. C., *December 8, 1908.*

FOOD INSPECTION DECISION 100.

Bleached Flour.—Flour bleached with nitrogen peroxide, as affected by the Food and Drugs Act of June 30, 1906, has been made the subject of a careful investigation extending over several months.

A public hearing on this subject was held by the Secretary of Agriculture and the Board of Food and Drug Inspection, beginning November 18, 1908, and continuing five days. At the hearing those who favored the bleaching process and those who opposed it were given equal opportunities to be heard.

It is my opinion, based upon all the testimony given at the hearing, upon the reports of those who have investigated the subject, upon the literature, and upon the unanimous opinion of the Board of Food and Drug Inspection, that flour bleached by nitrogen peroxide is an adulterated product under the Food and Drugs Act of June 30, 1906; that the character of the adulteration is such that no statement upon the label will bring bleached flour within the law; and that such flour can not legally be made or sold in the

District of Columbia or in the Territories; or be transported or sold in interstate commerce; or be transported or sold in foreign commerce except under that portion of Section 2 of the law which reads:

* * * *Provided*, That no article shall be deemed misbranded or adulterated within the provisions of this act when intended for export to any foreign country and prepared or packed according for the specifications or directions of the foreign purchaser, when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped: * * *

In view of the extent of the bleaching process and of the immense quantity of bleached flour now on hand or in process of manufacture, no prosecutions will be recommended by this Department for manufacture and sale thereof in the District of Columbia or the Territories or for transportation or sale in interstate or foreign commerce, for a period of six months from the date thereof.

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., *December 9, 1908.*

FOOD INSPECTION DECISION 101.

Benzoate of Soda.—Frequent inquiries have been received by the Department in regard to the use of benzoate of soda in foods. The following is typical of this class of inquiries:

In F. I. D. 89, the position of the National authorities in regard to the use of benzoate of soda is to allow its use in food, pending the report of the Referee Board of Consulting Scientific Experts. Based upon Bulletin 84, Part IV, of the Bureau of Chemistry, issued subsequent to F. I. D. 89, certain manufacturers of food products are representing to the officials of the States, charged with the enforcement of food laws, and to the consuming public generally, that the U. S. Government has condemned the use of benzoate in foods. We write to ask the position of the Deparment on this subject.

The Department has not changed the position outlined in Food Inspection Decision 89. Pending the determination by the Referee Board of the wholesomeness or unwholesomeness of benzoate of soda, its use will be allowed under the following restrictions:

Benzoate of soda, in quantities not exceeding one-tenth of one per cent., may be added to those foods in which, generally heretofore, it has been used.

The addition of benzoate of soda shall be plainly stated upon the label of each package of such food.

F. L. DUNLAP,
GEO. P. MCCABE,
Board of Food and Drug Inspection.

Approved:
JAMES WILSON,
Secretary of Agriculture,

WASHINGTON, D. C., *December 18, 1908.*

FOOD INSPECTION DECISION 102.

Entry of Vegetables Greened with Copper Salts.—Until further notice, vegetables greened with copper salts, but which do not contain an excessive amount of copper and which are otherwise suitable for food, will be allowed entry into the United States, if the label bears the statement that sulphate of copper or other copper salts have been used to color the vegetables.

Food Inspection Decision No. 92 is amended accordingly.

GEO. B. CORTELYOU,
Secretary of the Treasury.
JAMES WILSON,
Secretary of Agriculture.
OSCAR S. STRAUSS,
Secretary of Commerce and Labor.

WASHINGTON, D. C., *December 23, 1908.*

Under date of October 10, 1908, the Board of Food and Drugs Inspection has announced the following judgments under the Food and Drugs Act: No. 12, Misbranding of Flour (hard spring wheat mixed with durum); No. 13, Misbranding of Flour (as to place and manner of manufacture); No. 14, Misbranding of Vanilla Extract (imitation colored with caramel); No. 15, Adulteration and Misbranding of Whiskey (neutral spirits artificially colored); No. 16, Misbranding of a Drug Product (Sartoin skin food); No. 17, Misbranding of Flour (as to place of manufacture and name of manufacturer).

Under date of October 2nd, judgments Nos. 18–21, all concerning the Misbranding and Adulteration of Honey, Glucose and Invert Sugar being present.

Under date of November 25th, judgment No. 25, Misbranding of a Drug (Harper's Cuforhedake Brane Fude or Cuforhedake Brain Food).

Under date of Nov. 16th, Nos. 26–27, Misbranding of Canned Blackberries.

Under date of December 28th, the following: No. 28, Adulteration and Misbranding of Pepper; No. 29, Misbranding of a Drug Product (Liquid Sulphur); No. 30, Misbranding of a Drug Product (Concentrated Oil of Pine Compound); No. 31, Adulteration and Misbranding of Buckwheat Flour; No. 32, Misbranding of a Drug Product (Blackburn's Cascara, Wild Lemon, Castor Oil Pills, Compound); No. 33, Misbranding of Maple Sirup; Nos. 34 and 35, Misbranding of Canned Peaches.

(T. D. 29342). Nov. 11, 1908.—Countervailing duty on wood pulp and other products of wood imported from Sweden.

(T. D. 29343). Nov. 14, 1908.—Drawback on leather manufactured by Thomas A. Kelley & Co., of West Lynn, Mass., with the use of imported bichromate of soda, logood crystals and refined glycerin.

(T. D. 1433). Nov. 13, 1908.—Bottling of spirits in bond. No foreign materials such as caramel or rock candy sirup to be added.

(T. D. 1434). Nov. 16, 1908.—Compound known as Marrofat exempt from taxation as a substitute, not being made in imitation or semblance of butter or calculated or intended to be sold as butter.

(T. D. 1435). Nov. 16, 1908.—Suspension of the provision of regulations prohibiting use of fortified wines in the manufacture or preparation of patent or proprietary medicines or compounds.

(T. D. 1436). Nov. 17, 1908.—Record of grape brandy.

(T. D. 29354). Nov. 21, 1908.—When apparatus, imported free of duty for a scientific or educational institution under paragraph 638, tariff act of 1897, is rejected for any reason by such institution, rejection should be reported to the collector of customs at port of entry in order that duties may be collected.

(T. D. 29356). Nov. 23, 1908.—Drawback on Giant Dynamite Powder, in cartridge form, manufactured by the Giant Powder Company, Consolidated, of San Francisco, Cal., in part with the use of imported wood flour. T. D. 27138 of February 20, 1906, extended.

(T. D. 1437). Nov. 21, 1908.—Rectifiers prohibited from making a so-called wine mash and using the product thereof in the production of compound liquors.

(T. D. 29365). Nov. 25, 1908.—Drawback on steel rails manufactured by the Bethlehem Steel Company, of South Bethlehem, Pa., with the use of imported iron ore. T. D. 27127 of February 19, 1906, extended.

(T. D. 29370–G. A. 6830). Nov. 25, 1908.—Gauge of oil. Dutiable quantity-method of ascertainment. In ascertaining the value of oil dutiable on an *ad valorem* basis, it is proper to ascertain in American gallons the quantity actually arriving in this country, and afterwards to reduce the unit of the invoice to English or imperial gallons in the relative ratio of 231 cubic inches for the American gallon, liquid measure, and 277.274 cubic inches for the English gallon. The proper value would thus be determined by multiplying the number of gallons by the price per gallon, as shown by the invoice, if found to be correct by the appraiser.

(T. D. 29373). Nov. 17, 1908.—Court decision adverse to the government. Petroleum products countervailing duty-Belgian paraffin. Paraffin which is derived from petroleum originating in Russia, a country that imposes a duty on petroleum products exported from the United States, but which is manufactured in Belgium, a country that imposes no such duty, is not subject to the countervailing duty provided in paragraph 626, tariff act of 1897, on "the products of crude petroleum produced in any country, which imposes a duty on petroleum or its products exported from the United States."

(T. D. 29374). Nov. 16, 1908.—Decision adverse to the government. Casein-lactarene. Casein is free of duty as "lactarene," under paragraph 594, tariff act of 1897.

(T. D. 1438). Nov. 23, 1908.—Certification, by collectors, of specially denatured alcohol used or disposed of by manufacturers or dealers during the month.

(T. D. 1439). Denatured alcohol. Authorizing formula 17 as special denaturant for use in the manufacture of chloral hydrate.

TREASURY DEPARTMENT,
OFFICE OF COMMISSIONER OF INTERNAL REVENUE,
Washington, D. C., November 24, 1908.

SIR: You will please inform the ———, in your district, that the following formula has been authorized as a special denaturant for use in the manufacture of chloral hydrate:

Formula 17.—To 100 gallons of ethyl alcohol add 0.05 gallon (6 ½ fluid ounces) of animal oil.

The animal oil must conform to the following specifications:

Color.—The color shall be a deep brown.

Boiling Point.—When 100 cc. of the animal oil are subjected to distillation in the same manner as prescribed for the determination of wood alcohol in Section 26, Part I of the Regulations, not more than 5 cc. should distil over below 90° C., and not less than 50 cc. at 180° C.

Pyrrol Reaction.—Two and one-half cc. of a 1 per cent. solution of the animal oil in 90 per cent. alcohol are diluted to 100 cc. with alcohol. A splinter of pine wood, previously moistened with concentrated hydrochloric acid, is dipped into 10 cc. of this solution, containing 0.025 per cent. of animal oil. After a few minutes, the splinter should show a distinct red coloration.

Reaction with Mercuric Chloride.—Five cc. of the 1 per cent. solution of the animal oil in 90 per cent. alcohol, when treated with 5 cc. of a 2 per cent. solution of mercuric chloride in alcohol should give immediately a voluminous flocculent precipitate. Five cc. of the 0.025 solution of the animal oil when treated with 5 cc. of the 2 per cent. solution of mercuric chloride, should show at once a distinct turbidity.

Respectfully,
ROBT. WILLIAMS, JR.,
Acting Commissioner.

MR. E. B. ALLEN, *Collector First District, St. Louis, Mo.*

(T. D. 1440). Denatured alcohol. Authorizing formula 1 as special denaturant for use in the manufacture of furniture polish.

TREASURY DEPARTMENT,
OFFICE OF COMMISSIONER OF INTERNAL REVENUE,
Washington, D. C., November 28, 1908.

SIR: You are informed that formula 1 (to 100 gallons of ethyl alcohol add 5 gallons of approved wood alcohol) has been authorized as a special denaturant for use in the manufacture of furniture polish.

Applicants for a permit for the use of this formula in this line of manufacture must forward to this office the formula for the furniture polish they propose to make, together with a sample of the same, the sample to consist of not less than 1 pint.

Respectfully,
ROBT. WILLIAMS, JR.,
Acting Commissioner.

MR. W. V. MCMAKEN, *Collector Tenth District, Toledo, O*

(T. D. 29388—G. A. 6833). Dec. 8, 1908.—Olive oil—"fit only." Olive oil which, although imported in good faith for manufacturing or mechanical purposes and actually used for such purposes, is of a grade that is suitable for human consumption as food, is not within the provision in paragraph 626 of the free list of the tariff act of 1897, for "olive oil for manufacturing or mechanical purposes fit only for such use," but is subject to duty under paragraph 40, relating to "olive oil, not specially provided for."

(T. D. 29425—G. A. 6838). Sugar—polariscopic tests—Regulations of the Secretary of the Treasury.

1. "TESTING BY POLARISCOPE" DEFINED.

The expression "testing * * * degrees by the polariscope," occurring in paragraph 209 of the present tariff act of 1897, is construed to mean the percentage of pure sucrose contained in imported sugar as actually ascertained by polariscopic estimation, and has no reference to the commercial meaning attached to the phrase as recognized in trade between the sellers and buyers of sugar prior to the adoption of said act.

2. TREASURY REGULATIONS VALID AND REASONABLE.

The regulations of the Secretary of the Treasury promulgated under the authority conferred on him by Congress, and designed to carry into effect the provisions of said paragraph, are reasonable and valid, and free from constitutional objections.

3. CONSTITUTIONAL QUESTION—HOW RAISED IN REVENUE CASES.

Direct appeals from the circuit court of the United States to the Supreme Court, under Section 5 of the act of 1891 (26 Stat., 826), can not be entertained unless the construction or application of some provision of the constitution of the United States is *bona fide* involved, so as to raise a real and substantial dispute or controversy concerning the construction of the constitution upon which the matter in question depends.

United States General Appraisers, New York, December 18, 1908.

In the matter of protests 147213, etc., of American Sugar Refining Company against the assessment of duty by the collector of customs at the port of New York.

Before Board 3 (WAITE, SOMERVILLE, and HAY, General Appraisers).

SOMERVILLE, *General Appraiser:* The importations covered by the protests contained in the schedule consists of sugar which was assessed for duty by the collector under paragraph 209 of the tariff act of July 24, 1897, according to the polariscopic test prescribed by the regulations of the Secretary of the Treasury in order to ascertain the percentage of pure sucrose. These regulations are assailed by the importers variously on the ground that they are unreasonable and invalid, and alleging in some cases that they are unconstitutional. It is further objected in many cases that the phrase used in said paragraph 209, "testing by the polariscope," must be construed to mean the usual commercial polariscopic test as recognized and accepted in the trade between buyers and sellers of sugar at and prior to the adoption of the tariff act of 1897.

Similar questions were under consideration and were decided by this Board as far back as March, 1899, in the case of Bartram Brothers, G. A. 4386 (T. D. 20850), where the protests were overruled and the legality and reasonableness of the regulations were fully sustained. On appeal being taken from that decision to the circuit court, that court reversed the judgment of the Board, holding that the term "testing by the polariscope" had a well-settled commercial meaning prior to 1897, and must be interpreted in accordance therewith. Bartram Brothers *v.* United States (123 Fed. Rep., 327). On further appeal being taken to the circuit court of appeals, that court reversed the circuit court and fully sustained the decision of the Board (131 Fed. Rep., 833; 65 C. C. A., 557; T. D. 25395). In October, 1904, a petition for a writ of certiorari was presented to the Supreme Court in that and other similar cases and was denied (195 U. S., 635; T. D. 25901).

A renewal of all this litigation has taken place recently, where similar questions were again brought under review directly by the Supreme Court of the United States in the case of the American Sugar Refining Company *v.* United States (29 Sup. Ct. Rep. ; T. D. 29411). These appeals were dismissed by the Supreme Court for want of jurisdiction; and the following observations were made by Chief Justice Fuller in delivering the opinion of the court:

By Section 5 of the act of 1891 (26 Stat., 826), the judgments or decrees of the circuit courts of appeals are made final in all cases arising under the revenue law, and can only be carried to the Supreme Court by certificate, or on a certiorari. In the aforementioned cases there was no certificate for instruction on any question or proposition of law, and the application for certiorari was denied. The present direct appeal to this court is a mere attempt to obtain a reconsideration of questions arising under the revenue laws and already determined by the circuit court of appeals in due course. Such direct appeals, under Section 5 of the act of 1891, can not be entertained unless the construction or application of the Constitution of the United States is involved.

This is conceded, and counsel for appellant attempt to sustain the jurisdiction on the ground that the regulations assumed to add something to the dutiable standard prescribed by the tariff act, and that in doing so the Secretary exercised legislative power confided by the Constitution solely to Congress. But this does not constitute a real and substantial dispute or controversy, concerning the construction or application of the Constitution, upon which the result depends.

The admitted duty of the Secretary of the Treasury was to construe as best he could the paragraph relating to collection of duty upon sugars and to promulgate regulations for carrying it into effect (Rev. Stat., Sec. 251). This and this alone he did. The only real substantial point involved is whether or not he misconstrued the statute, and that gives this court no jurisdiction upon direct appeal. Sloan *v.* United States (193 U. S., 614, 620) and cases cited; United States *ex rel.*, etc., *v.* Taft, Secretary (203 U. S., 461).

Undoubtedly, Congress, without violating any constitutional provision, could have in terms directed exactly what was prescribed by the Treasury regulations; and prior decisions have held that the statute was properly construed by the secretary.

We concur with counsel for the government, that if the construction or application of the Constitution of the United States, within the meaning of Section 5, act of 1891, is involved in every case where one claims that according to his interpretation of a statute excessive duty or tax has been demanded by executive officers, the provisions of that act making decisions of the cicuit court of appeals in revenue cases final are of very limited value, and this court must entertain direct appeals from the circuit courts in most tariff and tax controversies, which we regard as out of the question.

The contentions made in these cases are the same as those made in the decision above cited. Following the views of the Supreme Court, as expressed in said decision, we overrule the protests on all grounds and affirm the decision of the collector in each instance.