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LUBRICATION AND LUBRICANTS.

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Next to the conservation of the world's fuel supply
there is probably no subject of greater importance
in the manufacturing world than the control of waste
power caused by imperfect lubrication and needless
friction. Notwithstanding the increasing interest
in more economical methods the immense losses from
this source are scarcely appreciated. In his recent
work on lubrication and lubricants Archbutt stated
that of the 10,000,000 h. p. in use in the United King-
dom of Great Britain considerably more than half
this amount, 40 to 80 per cent. of the fuel, is spent in
overcoming friction, and that a considerable proportion
of this power is wasted by imperfect or faulty lubrica-
tion. On account of the great abundance of cheap
fuel in the United States, doubtless the conditions
here are even less desirable. It is safe to state that
losses from this source in this country are from 10 to
50 per cent. of the power employed. Not infre-
quently in factories where the annual expense for
lubrication amounts to thousands of dollars, lubrication
experts find a loss of 50 per cent. or greater.

The manufacturer often knows very little concern-
ing the economic qualities of the lubricants he receives
in using them too much is left to "rule of thumb"
methods with little knowledge of the actual conditions
of friction, the action of metallic surfaces under the
dynamic stress of the transference of power, or such
modified action as is produced by the intervention
of a lubricating film. For example, the different
effects on a journal of a soft and hard bearing may be
sufficient to cause a considerable loss of power, if
improperly selected, and yet escape attention. In
the earlier tentative study of the conditions depended
on for the results described in this paper, under such
loads as 100, or 150 lbs. per sq. in. of bearing surface,
the grades of babbitt in ordinary use were found
much too soft and yielding to sustain such work under
the necessary conditions of speed and oil feed; only
a very hard alloy of exceptional composition could
be used; the one selected of approximately the com-
position tin 90, copper 2, antimony 8 gave results
entirely satisfactory. Then since it was desired to
maintain such conditions of load and speed that any
oil could be broken down at any moment, it was
found to be necessary, not only that the journal and
bearing be milled to mechanically true surfaces, but
that by continued operation and repeated careful
milling even a higher degree of permanent evenness
be maintained. If such be the essential conditions in
precise quantitative observations, similar precautions
are evidently necessary in factory operations.

In the earlier days of machinery lubrication before
the introduction into the trade of products from
petroleum, the manufacturer had little concern about
viscosity and other physical constants of lubricants,
for dealing with simple oils or greases of definite com-
position, he could be sure of obtaining what he desired
within the capacity of the materials at his disposal.
Then, in the days of higher prices of manufactured
products and less severe competition imperfect lubri-
cation was of less consequence than in more recent
times when every detail of cost and loss should prop-
erly receive careful attention; and, furthermore,
the principles of friction and the importance of its
control were in the earlier days of lubrication only
imperfectly understood. Modern high speeds and
excessively heavy loads had not then to be provided
for in the applications of power in manufacturing
operations, in transmission, or transportation.

The discovery that the heavy hydrocarbons in
petroleum possessed the qualities requisite in lubri-
cants—viscosity, durability, stability under varying
conditions of speed and load—was the beginning of

a new era in lubrication. Methods of treatment and refining, with little or no knowledge of the hydrocarbons of which the lubricating oils were composed developed entirely along empirical lines, were slow in producing suitable products. The earlier methods have undergone no fundamental changes even to the present time, except in the introduction of heavier hydrocarbons from crude oil territory more recently developed. Crude oils of the Pennsylvania type containing a considerable proportion of the hydrocarbons C_nN_{2n+2} have always yielded excellent light spindle oils composed for the most part of the hydrocarbons C_nH_{2n} and C_nH_{2n-2}. But as we now know this type of oils include too small a proportion of the heavier hydrocarbons for the body necessary in lubricators subjected to the great stress of heavy loads, and cylinder friction. This need in heavy lubrication led to the practice of compounding oils, or mixing with the petroleum products various proportions of the vegetable oils, such as castor or rape, and the various animal oils or greases, which so fully monopolized this field, that manufacturers were often led to believe that no other products could serve an equivalent purpose. Even since the more recent introduction of heavy lubricators from Texas and California petroleum the belief still prevails that only compounded oils can be relied on for heavy work. But with care in distillation and treatment, it is certain that heavy lubricators well adapted for bearings and cylinders may be prepared from those crude oils, and large quantities of such lubricants are now widely in use.

All experimenters with lubricating oils who have given thoughtful attention to the essential needs of lubrication have been impressed by the superiority of an ideal solid lubricant, one that should embody an equivalent of the desirable qualities of the liquid products with a greatly superior wearing quality, a low coefficient of friction, and readily convertible into a form that can conveniently be applied to the various forms of journals and bearings. Soapstone, asbestos, natural graphite, etc., do not, altogether, possess these fundamental qualities of the liquid products. Greases compounded with graphite are useful on low-speed bearings and under heavy work. Natural graphite serves an excellent purpose on cast-iron bearings, acting as a surface evener of the porous metal. On finer surfaces care is necessary that it does not collect in such quantities as to seriously scratch or abrade the journal and bearing. Of all the solid bodies available for lubrication, graphite possesses the desirable unctuous quality and great durability. For general use in lubrication graphite must be in its purest condition and in a state of extreme subdivision. Whether in such a condition as the deflocculated form, the ultimate molecules or atoms have a certain freedom of movement, analogous

to that of liquid molecules under stress of friction, or whatever explanation may be suggested of its unctuous quality the fact remains that it possesses this quality in very high degree.

Such graphite is now produced by processes discovered, perfected, and placed on a manufacturing basis by Dr. Edward G. Acheson, of Niagara Falls, as a part of his great work in the development of electrochemical processes. Besides his immense output of pure graphite for general commercial use, Dr. Acheson has succeeded in converting it into a new form, a deflocculated condition, that meets the requirements of an ideal solid lubricant. This deflocculated form greatly surpasses ordinary graphite in unctuous quality, and its adaptability for prolonged suspension in water and oils render it especially applicable to frictional conditions. Furthermore, the readiness with which it forms coherent films on journals, its great wearing qualities and the ease of the application, constitute it a lubricant of extremely high efficiency.

Acheson graphite can be produced from any substance that contains carbon in a non-volatile form. Under the extreme temperature of the electric furnace any and all other elements are readily volatilized. Even carbon itself is freely vaporized and its peculiar appearance in the burning carbon-monoxide is depended on as an indicator of suitable conditions in furnace operation, much as the drop in the manganese flame which shows the disappearance of carbon in the Bessemer converter. As commercial products, two forms of graphite are produced, the unctuous and the deflocculated modifications, the first form accompanying the production of carborundum in furnaces charged with carbon and sand, the second obtained from a charge of coal or coke alone. The first form is leafy in structure, coherent, and extremely unctuous or greasy in its feel; it is segregated and not readily disintegrated. The second form is also unctuous, in a high degree, but very pulverulent and capable of extreme subdivision; it is readily converted into a deflocculated condition, and this form in water forms the commercial "Aquedag," or aqueous Acheson deflocculated graphite. In combination with oils it is known as "Oildag."

This deflocculated condition of graphite has peculiar properties; it remains suspended indefinitely in water, but is quickly precipitated by impurities. On account of its extreme subdivision, a very small amount suspended in water serves for efficient lubrication. From numerous and long-continued trials it appears that 0.35 per cent. serves an adequate purpose and that a larger proportion is superfluous. It is certainly remarkable that such a small quantity of graphite is readily distributed by water between a journal and bearing while sustaining a load of 70 lbs. per sq. in. of bearing surface, and that under high-speed con-

ditions it maintains an extremely low coefficient of friction.

Proper lubrication of bearing surfaces involves careful consideration of the metals composing the journal and bearing, since the influence of the metals employed has an effect even in the intervention of the best lubricating film. The materials in common use for the construction of bearings include cast-iron, steel, and alloys of variable composition included under the general terms bronze and babbitt. In high-speed work cast-iron bearings must be used with extreme care. In the accurate adjustment necessary in machine testing of lubricators, we have found it impossible to prevent injury to the journal when using a cast-iron bearing. Results obtained by the use of bronze have not been altogether satisfactory. Properly selected babbitt, however, on a steel journal seems to fulfil the desired conditions most satisfactorily and it possesses a wide range of applicability. As mentioned above, satisfactory lubrication is possible only when the journal and bearing are properly milled to true surfaces, kept smooth, accidental scratches worked out, and bare spots avoided. Successful lubrication demands constant skilled attention to the condition of journals and bearings, and no factory supervision affords more desirable returns. Lubrication consists in reducing friction to the lowest increment of the power in use. A lubricant is an unctuous body that readily forms a continuous, coherent, durable film capable of holding apart rolling or sliding surfaces, and itself interposing the least possible resistance by its own internal friction. The economic problem in lubrication depends on the use of such a lubricant under suitable conditions.

The lubricators in commercial use include water, oils, greases and solids. Under oils is classified the great variety of light spindle, heavy engine and cylinder products, either unmixed hydrocarbons from petroleum or compounded oils—mixture of the petroleum hydrocarbons with some one of the vegetable oils, or with animal oils—tallow, wool grease, etc. The greases may be generally classified under a few heads depending on their consistency which is derived from the proportion of lime or soda soaps or oleates mixed with a hydrocarbon oil as a carrier. The solid greases have already been referred to. Water in itself possesses no oiliness whatever, but under certain conditions in cylinders it is found to assist in imparting to the metallic surfaces an extremely smooth condition which serves to materially reduce the friction. A practical knowledge of hydrocarbon lubricants should include a knowledge of the source; that is, the crude oil from which the lubricant is prepared, since there is a wide difference in composition and properties of the hydrocarbon oils from different oil fields. Methods of refining petroleum oils have very much to do with the quality of the

products. In general terms inferior products are obtained when the process of distillation is conducted in such a way as to produce decomposition; the best products are obtained only by careful distillation and careful treatment in refining, whereby the hydrocarbons in the refined products obtained have essentially the same composition that they had in the original crude oil. An examination of various lubricating oils in the trade frequently reveals a condition of the oils indicating improper refining. For example, it does not need the application of extremely delicate tests to show the presence of free alkali, of sodium sulphate or of sodium salts of organic acids, any one or all of which may be injurious to metallic surfaces. One of the most exacting duties of the refiner is the treatment with caustic soda in such a manner as to remove all acid products and at the same time to avoid such an excess of caustic as will form an emulsion, which is one of the "terrors" in the refinery. An examination of a great variety of oils in the trade such for instance as the spindle oils in use in automobile service indicates that the best refined oils are those that contain a minute trace of alkali.

The ordinary methods of testing lubricating oils include determinations of viscosity, specific gravity, the flash and fire temperatures; another important property of these oils which is termed oiliness or greasiness is not so readily determined by analysis; in fact there seems to be no accurate method for its determination, yet it is readily distinguishable and has much to do with the efficiency of all lubricating oils. Concerning the most efficient methods of testing lubricating oils, various opinions are expressed by different authors. Redwood in his work on petroleum and its products asserted that the viscosity of an oil is the best guide to its lubricating value since it enables the consumer to select oils similar to those that have afforded him the best practical results. He alludes to the close relationship between viscosity and the laws of friction of liquids. In comparing the use of viscosity with observations on the behavior of lubricants on a frictional testing machine he states that he was unable to obtain satisfactory results with any machine at his disposal; his conclusions in general were that in the present state of our knowledge the indications afforded by testing machines are wholly misleading, and this led him to attach special importance to a good system of testing viscosity. He refers to the opinion of Thurston that any oil should be tested on a machine under the conditions of load and speed similar to those of the use for which the oil is intended. Referring to the work of Ordway and Woodbury in 1884 with an apparatus constructed to apply pressures of 40 ℔s. per sq. in., and to those of Tower carried on under what he terms great pressures, 100 to 600 ℔s. per sq. in. in an oil bath system of lubrication, and opinions of others on these results,

Redwood presents the view that the agreement between machines and actual practice is extremely slight and his final conclusion is that viscosity affords the most valuable tests of lubricating qualities at our disposal. Inasmuch as Redwood's opinion on machine testing is a result of his observations during several months on the Ingram and Stafer machine in which the speed is 1500 r. p. m., and that the friction is gaged by the number of revolutions necessary to carry the temperature to 300° F., it is not difficult to understand his conviction that in his experience testing machines do not afford results comparable with those of actual practice.

The value of viscosity as a distinguishing property of lubricating oils is recognized by all who have given attention to the subject, but all are not agreed as to the extent of its practical reliability. Archbutt suggests that the quality of oiliness or greasiness is nearly of as much importance as viscosity. Although as mentioned above there is no precise method whereby oiliness can be determined, it is not difficult to recognize it nor to distinguish the marked differences in this respect shown by different oils and greases. Archbutt calls attention to the fact that at very low speeds the friction of a cylindrical journal should be proportional to the viscosity of the oil, but at higher speeds and consequently increased temperatures the relation of friction to speed ceases, and the viscosity is diminished with a corresponding change in the carrying power of the journal. While fully appreciating the value of the information to be obtained by chemical analysis, Archbutt insists that the oiliness of a lubricant is of especial importance under heavy loads and high speeds. He suggests that it is advantageous for an engineer to test oils for himself on a machine without depending altogether on analytical data or physical tests obtained from the expert.

Hurst also mentions that a broader knowledge of the practical working of oils is necessary than can be obtained from chemical or physical tests alone. He maintains that the test of an oil from a journal under the practical conditions of its use shows conclusively its adaptability to such use.

The principal points to be observed in mechanical tests are the effects of speed, load, temperature, and the frictional effects due to viscosity and oiliness; the measurements on which depend the quality of the oil include the frictional resistance, the temperatures, and the endurance of the oil film. Doubtless the numerous machines that have been constructed for testing oils have certain merits and advantages. In the wide range of work carried on in this field during the past year, a part of the results of which are presented in this paper, the machine devised by Professor Carpenter has been used. In its sensitive adjustment, durable efficiency, and the wide range of possible tests, this machine in continuous use during this period on light and heavy oils, greases and graphite has fulfilled all requirements. Since the results to be presented are closely dependent upon the method employed, a view of this machine is here introduced.
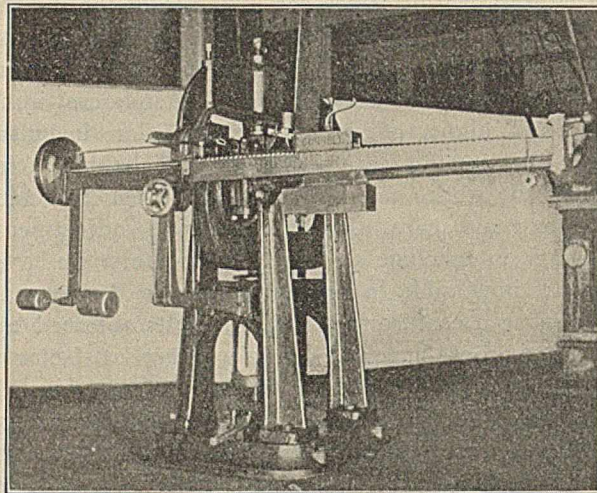
Carpenter machine.

This machine has an accurate adjustment for recording the speed, and a long lever arm with a vernier attachment graduated to tenths of a pound for recording the friction. The load is applied by a powerful spring worked by a cam and lever and the limit of the machine is 6000 lbs., total load; careful calibration of the spring showed it to be properly adjusted.

In projected area the bearing in use is approximately 8 sq. in.; the journal is about 3 in. in circumference nearly equal to 1 foot in linear extension. A cast-iron frame babbitted and milled down to a true surface was used for the most part in this work. Even after careful milling some continued frictional work was necessary on the babbitt surface to bring it to the proper conditions of constant results. The hard form of babbitt mentioned above gave satisfactory results, and there was little difficulty in keeping the surfaces in suitable condition after they were once obtained. For measuring temperatures a thermometer was inserted in a hole in the bearing which extended close to the journal. Tests made at steam temperature, 210° Fahr., were carried on with the aid of a hollow cast-iron babbitted bearing, with steam attachments by which it was found that the desired temperature could readily be maintained. The lubricant is run in from a sight-feed cup through a small hole close to one side of the bearing with careful regulation of the flow for proper adjustment of the oil feed. For delivery of the lubricant over the entire face of the bearing two channels or grooves are run diagonally across the babbitt face from the inlet hole which gives equal and even distribution; these channels must be carefully gauged for an even flow, otherwise dry spots or streaks appear on the journal accompanied by a sudden greatly increased friction

indicated on the friction bar. This detail of operation requires careful and constant attention, for on it depends the continuous regularity of the friction curve. In this respect this method of observation is extremely sensitive, and is one of the important elements in frictional tests. Partial exposure of the journal enables the operator to observe the formation

this machine may be readily ascertained on any other equally efficient machine. In duplicate tests made with the same bearing and under the same conditions, the results were closely concordant. At the outset it should be clearly understood that these tests must be performed with a scientific accuracy of exact quantitative observations, with close supervision of

CURVES OF FRICTION
WITH VARYING VISCOSITY OF LUBRICANT

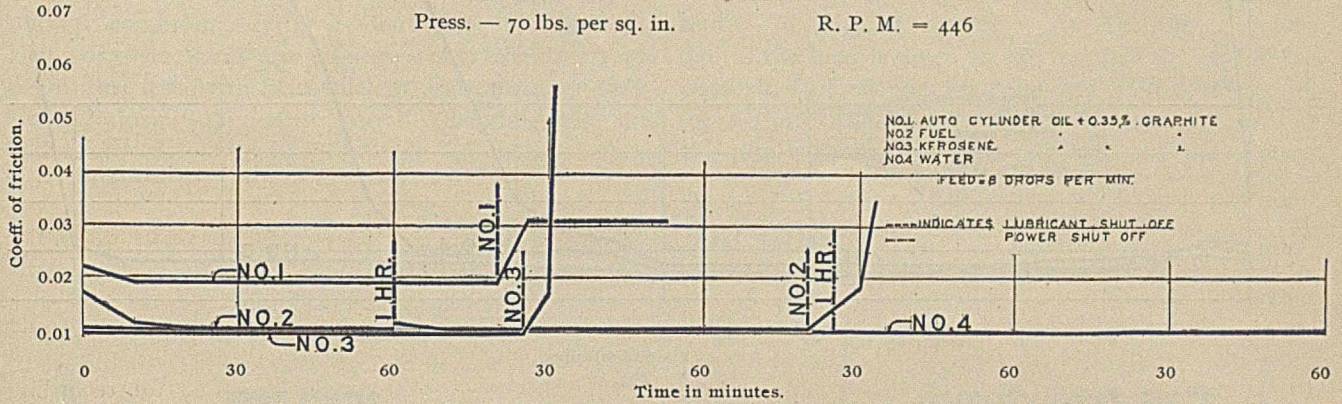Press. — 70 lbs. per sq. in.   R. P. M. = 446

Fig. 1.

of the film, its comparative thickness and any irregularity due to an imperfect condition of the journal or bearing, or improper lubrication.

Accurate testing of the mechanical efficiency of oils with the precise quantitative observations possible on the Carpenter machine, including the various classes of lubricators under consideration in this

all details; the work then becomes the regular routine of any scientific investigation which involves long series of observations; after it is ascertained by preliminary trial what conditions are necessary in testing any given oil? of course for commercial benefit these conditions should be as close as is practicable to the factory conditions of use.

CURVES OF FRICTION
WITH VARYING VISCOSITY OF LUBRICANT

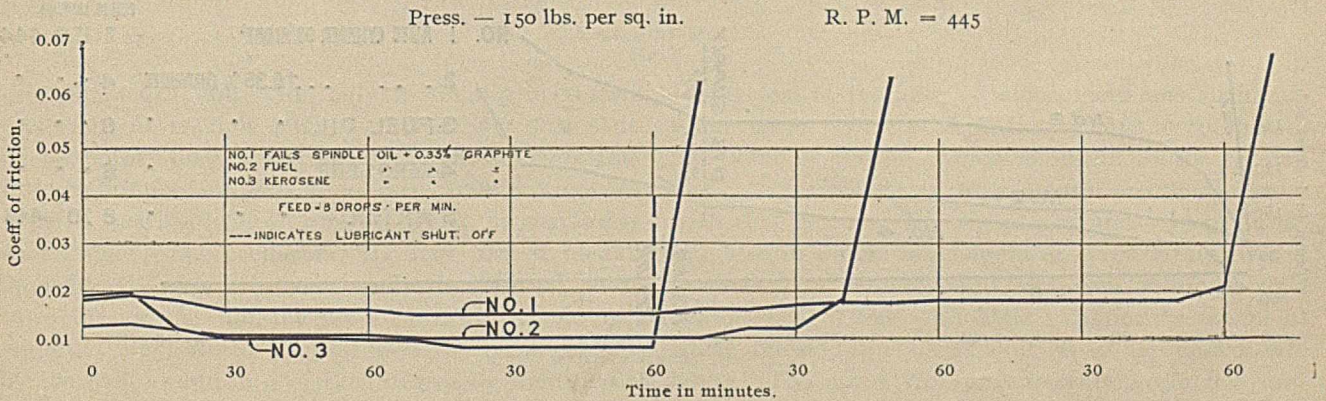Press. — 150 lbs. per sq. in.   R. P. M. = 445

Fig. 2.

paper represented an extensive field of labor, especially since there are no general standards of comparison under any conditions of operation. Such constants must of necessity be based on arbitrary data; nevertheless if they are accurately determined on a standard machine, with the conditions of the journal and bearing selected—the load and speed—the constants on

The results to be described on the use of water, kerosene, and fuel oil, as vehicles of graphite present novel and interesting features. Under certain conditions as mentioned above in steam cylinders, it is well known to engineers that water alone serves as a lubricating film. But since on journals it serves no purpose whatever, the lubricating qualities of

aqueous suspended graphite must be due wholly to the graphite. The same is true of kerosene, which alone is practically devoid of lubricating quality, and likewise of fuel oils.

For the purpose of testing the effects of varying viscosity in lubricants, and at the same time the lubricating quality of deflocculated graphite, tests

by a dotted line in the figure, and it appears that there was no change whatever in the direction of the curve by stopping and starting. Curve No. 3, representing the observations on the coefficient for kerosene oil with graphite, is also a straight line, showing a very slightly lower coefficient than water. The coefficient curve for the fuel oil and graphite

CURVES OF FRICTION
OIL AND OILDAG—VARYING FEEDS

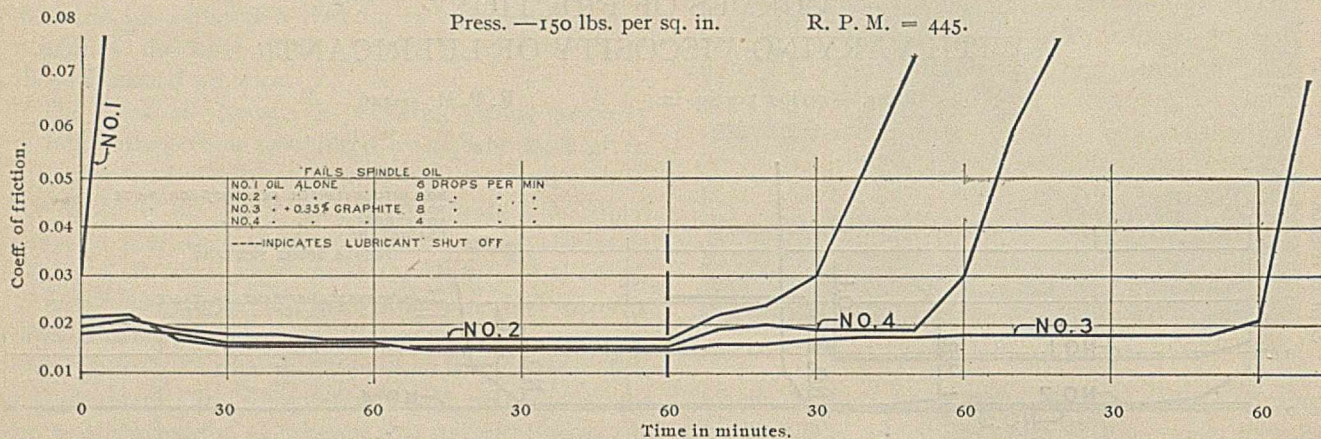Press. —150 lbs. per sq. in.   R. P. M. = 445.

Fig. 3.

were made with water, kerosene oil, a fuel oil, and an auto cylinder oil each carrying 0.35 per cent. graphite. The results obtained in these tests are shown by the curves in Fig. 1, in which the speed, r. p. m., is maintained at 446, and the load at 70 lbs. per sq. in. The observations of frictional load and temperature were made at intervals of ten minutes each, and on that basis a curve is drawn for each

is also practically a straight line, and with an endurance test extending $1\frac{1}{2}$ hours after the oil supply was shut off; here the frictional coefficient is slightly higher than that either of water or kerosene. A similar regularity appears in the curve of the auto cylinder oil with graphite, but it is to be noted that the frictional coefficient is very materially higher than those of the other lubricators shown in the figure,

TEMPERATURE CURVES FOR LUBRICANTS OF VARYING VISCOSITY
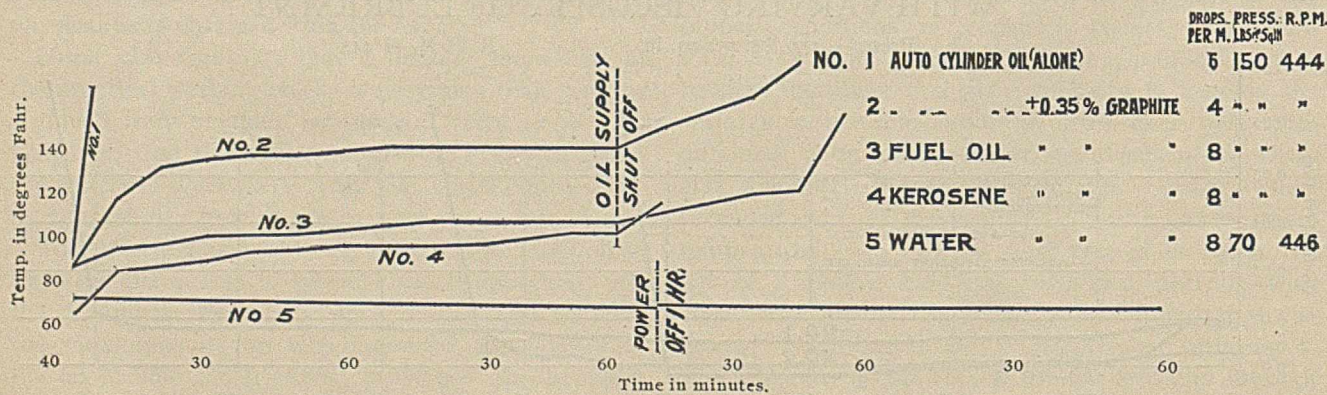## WITH AND WITHOUT GRAPHITE

Fig. 4.

of the lubricators tested; in the figure the time is given in half-hour limits and the coefficient of friction in hundredths of a unit. It will be observed that the curve for water and graphite is practically a straight line with scarcely any variation for the four hours shown on the curve; this test continued altogether for 15 hours with a precisely similar result. There were several stops which are indicated

which may be considered as a measure of comparative greater internal viscosity of the auto oil; this oil showed a much longer endurance test than appears in this figure.

The effect of varying viscosity in lubricants and the lubricating quality of the graphite under practically the same speed, 445 r. p. m., but with a load of 150 lbs. per sq. in. using kerosene, a fuel oil and a

spindle oil, with the same proportion of graphite, and the same oil supply is shown in Fig. 2. Kerosene here shows a very slight irregularity in its coefficient, which differs only slightly from that in the preceding figure. Here again the greater internal viscosity of fuel oil is shown by the increased friction which appears in this curve. No doubt the fuel oil possesses the quality of oiliness in a very slight degree which enables it in the beginning of the test to take a lower coefficient than kerosene, which maintains for a few minutes a considerably higher coefficient until the continuous film of graphite has been formed and reduced the coefficient to its normal condition. It is evident that the fuel oil also possesses a certain oiliness which enables it to begin the test with a coefficient that changes only slightly during the entire period, including also an endurance test extending through two hours before

cating quality of the oil is also shown in curves Nos. 3 and 4, curve No. 3 representing a feed of 8 drops per min., and curve No. 4 a feed of 4 drops per min. The diminished coefficient in curve No. 4 as compared with curve No. 4 represents the lubricating effect of graphite, and this effect is still further shown by the increased endurance test in curve No. 4; it will also be observed that besides diminishing friction, curve No. 4 is based on an oil supply due to the graphite, one-half that of curve No. 2 of the oil alone.

In Fig. 4 curves are shown which represent the temperatures recorded in tests of friction presented in Figs. 1 and 2. As in the previous figures the load is given as 150 lbs. per sq. in. for the auto oil, fuel oil and kerosene and 70 lbs. per sq. in. for water. The speed was 444 r. p. m. in all but the test with water where the load was 446 r. p. m.

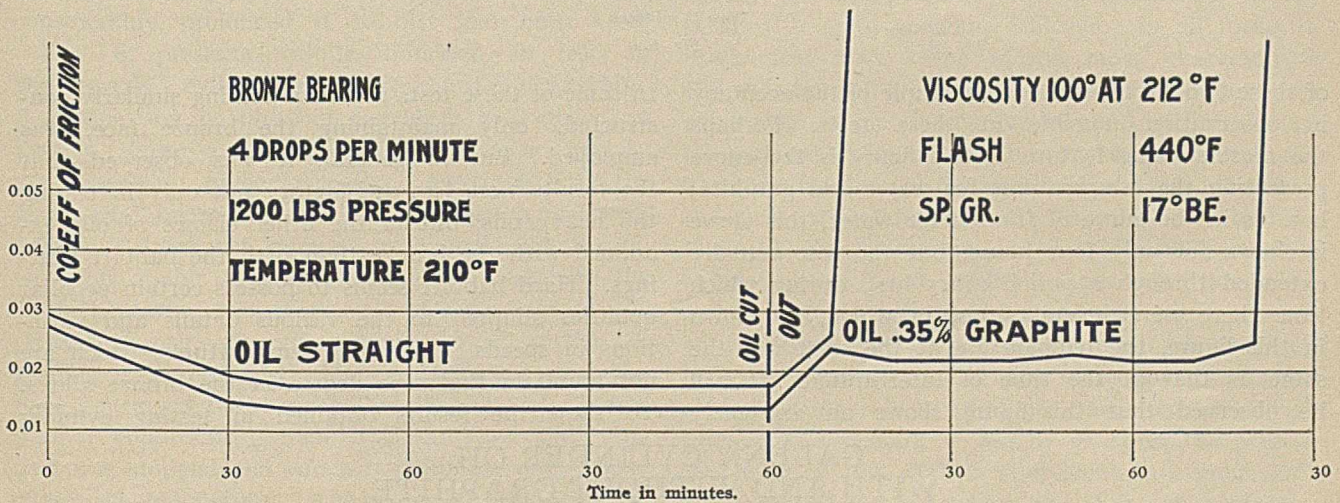## AMERICAN CYLINDER OIL
WITH AND WITHOUT GRAPHITE

Fig. 5.

the oil breaks and with only a slightly increased coefficient of friction after the oil supply was shut off. Another feature worthy of note is the comparative endurance of the three oils. While kerosene under a bearing load of 150 lbs. per sq. in. maintains an extremely low coefficient, the fact that it breaks immediately when the oil supply is shut off indicates that it has not the power to form a coherent graphite film, which is possessed to some extent by the fuel oil and in a marked degree by the spindle oil.

Fig. 3, load 150 lbs. per sq. in., r. p. m. 445, gives the effect on a spindle oil of a variable feed. In one test on the oil alone the oil supply was regulated with the object of breaking the oil at the beginning of the test and also its behavior under an oil supply that enabled it to perform its functions as a lubricant. The effect of graphite on the lubri-

In the test of the auto oil alone there was an immediate rise in temperature corresponding to the breaking point of the oil, which is shown in the friction test. It is interesting to compare this temperature with that of curve No. 2, auto oil and 0.35 per cent. graphite, in which the temperature rises within twenty minutes to a definite point and then continues in a nearly straight line with little variation to the point where the oil supply was shut off at the end of two hours. The curve No. 3, representing the temperatures of fuel oil and graphite, also shows a very slight variation after 30 min., when the stable conditions of lubrications were established. A difference in temperatures of approximately 25° is shown between the curves of the auto and fuel oils which must represent the larger escape of energy in the form of heat from the bearing due to the greater internal resistance of the auto oil. The tempera-

tures of kerosene with graphite as shown in curve No. 4, are approximately 10° lower than those in the fuel oil curve, due to the still smaller internal resistance of kerosene. Bearing in mind the small difference between the specific gravity of the fuel oil, approximately 35° Bé., and that of kerosene, approximately 45° Bé., the difference in temperatures

amined in this work, considerable attention has been given to the behavior of heavy engine and cylinder oils, both straight hydrocarbon oils and compounded oils. An especial form of bearing was constructed consisting of a cast-iron frame with a hollow chamber for introducing steam and a babbitted face using the exceptionally hard babbitt previously described.

'600 W' CYLINDER OIL
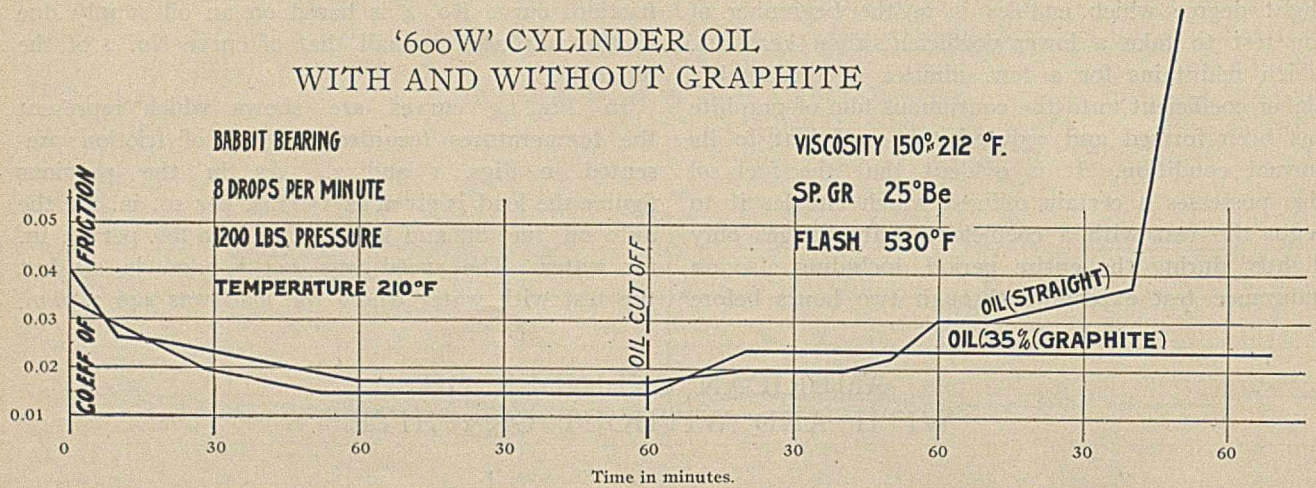### WITH AND WITHOUT GRAPHITE

Fig. 6.

of these two curves is a good example of the accuracy in observation possible in these tests. Perhaps the most striking feature in this figure is the curve presenting the temperatures for water and graphite; here as in the curve of friction for water, this curve is shown for only four hours, but the test actually extended through a period of 15 hrs., during which time there were several stops, in which, as shown in this figure, the temperature at the start was the same as that at the time of interruption. It will be observed that this figure shows an extremely

In some of these tests a bronze bearing similarly constructed, only maintaining the bronze face, was employed. But in general, it was observed that the results were less satisfactory not only in testing the heavy oils, but in the other classes of oils examined with the bronze than with the babbitt bearings. Hard babbitt seems to possess certain peculiar qualities adapted to the various details and variations in speeds, loads, and temperatures, which are not found in the same degree in the bronze alloys. To show the results obtained in testing cylinder

GALENA CYLINDER OIL
WITH AND WITHOUT GRAPHITE

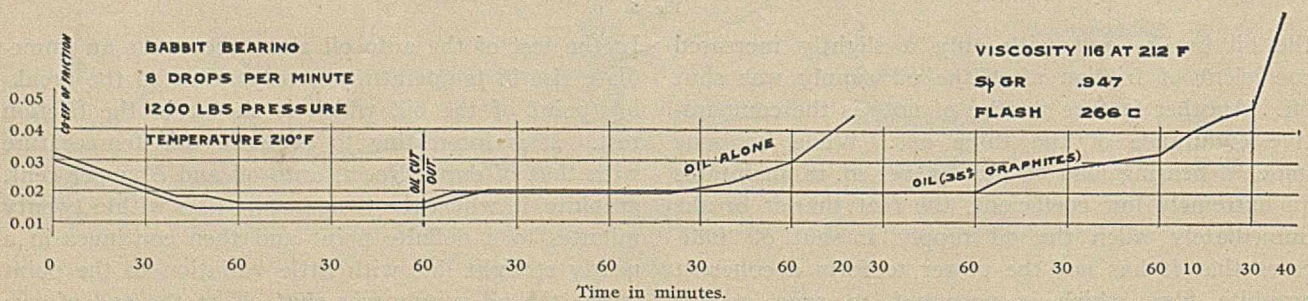
Fig. 7.

low temperature, 65°, practically the same as the room temperature, which it never exceeded by more than 5°, and that it is essentially a straight line from start to finish. In this use of water as a vehicle for the graphite there is nothing to interfere with the best work that the graphite is capable of performing.

Among the various classes of lubricating oil ex-

oils, figures are here presented on three commercial products, the American cylinder oil, Galena cylinder oil, and "600 W" cylinder oil. Tests were also made on the influence of graphite on these oils with reference to the frictional coefficient and endurance of the oils. The physical constants of the oils are also given for comparison, especially of specific gravity

and viscosity. A general procedure of the tests included a continuous run for two hours, at which time the supply of oil was shut off.

In Fig. 5 of the American cylinder oil, which is a straight hydrocarbon oil, the data of the tests includes the use of a bronze bearing, a supply of lubricant at the rate of four drops per minute, a total pressure of 1200 lbs. and a speed of 245 revolutions per minute. The curve of the oil straight begins at a somewhat higher coefficient than it maintained after the first half hour when normal conditions are established, and it then proceeds in a straight line with no variation to the point where the feed is stopped; the endurance run of this oil is doubtless considerably shorter than it would have been in the use of babbitt bearings; in fact, this was demonstrated in another test in which babbitt was used. With graphite the oil follows closely the direction of the other curve, but with a very considerable diminution in the coefficient of friction, it further appears in the endurance test that the graphite carries the load with slightly increased friction for a period of 1 hour and 20 minutes, which would have doubtless been considerably prolonged if babbitt had been used.

Fig. 6 presents results obtained in tests of the "600 W" cylinder oil with and without graphite. A comparison of physical constants with those in Fig. 5 shows a materially lower specific gravity and somewhat higher viscosity. In these tests the same total pressure, 1200 lbs., and the same speed, 245 revolutions per minute, were used, but the oil feed was double that in the preceding tests and the babbitt bearing was employed. On account of the greater viscosity the straight oil showed at the beginning a considerably higher coefficient and the tests continued one hour before the oil had reached normal conditions, which it maintained until the feed was stopped and which it doubtless would have continued indefinitely. After the oil was shut off lubrication was maintained with some slight irregularity and increased friction during 1 hour and 40 minutes, the point at which it broke. Similar conditions are observed in the curve which expresses the variation in the coefficient of friction of this oil with 0.35 per cent. graphite; it begins the test with a somewhat lower friction and reaches normal conditions sooner than the straight oil, and continues in a straight line to the point where the supply is stopped, and then still continues in a straight line with somewhat increased friction. The endurance curve would doubtless have continued for a considerably longer time but the power was shut off at the point where the curve terminates. A marked influence of graphite on the behavior of this oil is plainly apparent in a comparison of these curves.

In applying tests to the Galena cylinder oil with and without graphite, the same feed, load and pressure were used as with the preceding oil and the tests were made on a babbitt bearing. In viscosity this oil is somewhat less than the preceding oil, the specific gravity somewhat higher. Both curves begin with a slightly lower coefficient, 0.03, and this difference is maintained until the oil is shut off and for $1^1/_2$ hours on the endurance test. To reach normal conditions the straight oil ran for one hour, the oil with graphite 45 minutes. After the feed was stopped, the curves proceed regularly with slightly increased friction, the oil alone practically breaking in $1^1/_2$ hours, the oil with graphite proceeding with perfect regularity for three hours, changing slightly during the next hour and breaking at the end of $4^1/_2$ hours. The tests represented on Figs. 5, 6 and 7 are not intended to present a comparative efficiency of these particular oils but to demonstrate the application of this method of testing and also to compare the effects of deflocculated graphite.

The results presented in this paper with reference to the uses of graphite as a solid lubricator indicate that in the deflocculated form it can readily be applied with great economic efficiency in all forms of mechanical work. One of its most characteristic effects is that of a surface evener by forming a veneer equalizing the metallic depressions and projections on the surfaces of journal and bearing, and endowed with a certain freedom of motion under pressure, it affords the most perfect lubrication. In automobile lubrication the great efficiency of graphite in increasing engine power, in controlling temperatures, and wear and tear of bearings has been brought out in a series of tests conducted by the Automobile Club of America. In connection with the reduction in friction of lubricating oils by graphite the extremely small proportion necessary is worthy of note; the proportion used in this work is equivalent to a cubic inch of graphite in 3 gallons of oil. The curve of temperature for Aquadag, an increase but slightly above that of the surrounding atmosphere, demonstrates an important economic quality of controlling temperatures in factory lubrication, and thereby avoiding the danger of highly heated bearings, which are frequently the cause of fires.

In the observations described in this paper, and, in fact, in all the work that has been done in this field, there is not a more impressive example of the efficiency of graphite in lubrication than that presented in the curves of friction and temperature of water and graphite; for water serving merely as a vehicle and completely devoid of lubricating quality, the graphite is permitted to perform its work without aid and with no limiting conditions.

STUDIES ON THE CARBENES.

By Kenneth Gerard Mackenzie.

Received January 20, 1910.

In 1905, Richardson and Forrest[1] called attention to the use of carbon tetrachlorid as a solvent for differentiating bitumens. They showed that the amount of bitumen soluble in carbon disulphid, but insoluble in carbon tetrachlorid, furnished an indication of the amount of weathering to which natural asphalts had been subjected; and also revealed careless refining of the residual pitches obtained from California and Texas petroleums, having an asphaltic base. Mr. Richardson, in his "The Modern Asphalt Pavement,"[2] applied the name of "Carbenes" to this class of substances. Recently, Kirschbraun[3] has carried on extensive experiments upon the effect of overheating on the carbene content of Bermudez asphalt and cement. His "results * * * * confirm the correctness of Clifford Richardson's conclusion that carbenes are a result of overheating."

Again on the Pacific Coast, tetrachlorid has largely been used by municipal authorities and by producers as a means of controlling the character of the residual pitch, which is produced there to such a large extent for paving purposes.

Certain discrepancies having been noted between the results of determinations of carbenes in the same samples, made in the New York Testing Laboratory and in another laboratory on the Pacific Coast, an investigation was undertaken by the writer at the suggestion of Mr. Richardson with the object of discovering the cause.

The first possible explanation was a possible difference in the purity of tetrachlorid used. All tetrachlorid in use in this laboratory is distilled with a Young 18-column dephlegmator, discarding all boiling below 76°, thus removing any traces of carbon disulphid which may be present. To determine the effect of CS_2 impurities upon the amount of carbenes, samples of Durango and Texas pitch were examined.

TABLE I.—Carbenes in Texas Pitch.

	Per cent
Pure tetrachlorid	9.3
Tetrachlorid $2^1/_2$ per cent. CS_2	9.5–9.5

TABLE II.—Carbenes in Durango No. 107979.

	Per cent.
Pure tetrachlorid	3.7
Tetrachlorid 5 per cent. CS_2	3.3
Tetrachlorid water saturated	3.6

The amount of disulphid would never exceed 5 per cent., and the effect of a less amount is negligible. Likewise water has no effect on the solvent power, for tetrachlorid dried over sodium will absorb less than 1 part in 5000.

Mr. Richardson has already called attention[1] to the fact that the carbene precipitate is in a very finely divided condition, and that, after dissolving in tetrachlorid, the solution must stand over night to ensure coagulation. It at once suggested itself that we might be dealing with a colloid which gradually changed to an insoluble precipitate, the carbenes, when solution first took place, being nearly all in the form of a colloidal solution, which on standing were precipitated. If this were so, with sufficient standing and several filtrations we should be able to obtain the true carbene value.

TABLE III.—Carbenes in Durango No. 107979.

	Per cent.
Dissolved, stood over night and filtered	6.5
Refiltered next day, obtained additional	4.2
Stood over two days, refiltered, additional	2.2
Stood over one day, refiltered, additional	0.3
Total	13.2

We have thus evidently removed all the carbenes since a final standing of 48 hours gave only 0.3 per cent.

TABLE IV.—Carbenes in Durango No. 107979.

	Per cent.
Air blown one hour, stood over night	5.9
Refiltered after standing over night, with very tight asbestos pad, additional	6.9
Total	12.8
Dissolved, stood over night and filtered	3.7
Refiltered after standing one and one-half weeks, additional	10.2
Total	13.9
Dissolved in tetrachlorid with 5 per cent. CS_2 stood over night and filtered	3.3
Refiltered after $1^1/_2$ weeks, additional	10.9
Total	14.2

We see that, after long standing, we obtain thirteen to fourteen per cent. carbenes, though, with a very tight filter, two days' standing gives us almost as large an amount. One other trial gave:

TABLE V.—Carbenes in Durango No. 107979.

	Per cent.
Dissolved, etc., as usual	3.5
Refiltered after four days, additional	10.1
Total	13.6

It would thus seem that to obtain the true amount of carbenes, it is necessary to allow the solution to stand at least four days. This should give sufficient time for the complete coagulation and precipitation of these hydrocarbons.

At this time, our attention was called[2] to the possibility of the amount of carbenes being effected by exposure to light. Accordingly samples were taken, in octoplicate, of Gilsonite selects, Texas pitch and Durango No. 107979. They were treated as shown below.

Solubilities in CS_2.

	Per cent.
Gilsonite	99.8
Texas pitch	96.1
Durango	99.4

[1] *J. Soc. Chem. Ind.*, **24**, 7.
[2] First edition, p. 120.
[3] *Municipal Engineering*, **35**, 349.

[1] "Modern Asphalt Pavement," 2nd edition, p. 546.
[2] By D. B. W. Alexander. See also *J. Am. Chem. Soc.*, **31**, 1052.

Table VI.—Gilsonite.

	A.	A'.	B.	B'.	C.	C'.	D.	D'.
First Day:								
Dissolved in...	Dark	Dark	Dark	Dark	Light	Light	Light	Light
Time........	3.30 P.M.	3.30	3.30	3.30	3.30	3.30	3.30	3.30
Second Day:								
Filtered in...	Dark	Dark	Dark	Dark	Light	Light	Light	Light
Time........	8.30 A.M.	8.30	8.30	8.30	8.30	8.30	8.30	8.30
Transferred to	..	..	Light	..	..	..	Dark	..
Time........	..	..	3.30 P.M.	.	..	..	11.30A.M.	.
Third Day:								
Filtered in....	Dark	..	Light	..	Light	..	Dark	..
Time........	11.00A.M.	..	8.00A.M.	..	8.00A.M.	..	11.00A.M.	..

Table VII.—Texas Pitch.

	A.	A'.	B.	B'.	C.	C'.	D.	D'.
First Day:								
Dissolved in..	Dark	Dark	Dark	Dark	Light	Light	Light	Light
Time........	3.30P.M.	3.30	3.30	3.30	3.30	3.30	3 30	3.30
Second Day:								
Filtered in...	Dark	Dark	Dark	Dark	Light	Light	Light	Light
Time........	8.30A.M.	8.30	8.30	8.30	8.30	8.30	8.30	8.30
Transferred to	..	..	Light	Light	...	...	Dark	Dark
Time........	..	..	4.30	3.30P.M.	..	..	5.00	5.00P.M.
Third Day:								
Filtered in....	Dark	Dark	Light	Light	Light	Light	Dark	Dark
Time........	8.30	2.00	8.00	1.00	8.00	1.00	8.30	2.00
Transferred to	..	Light	Dark	..	..	Dark	Light	..
Time........	..	4.00	4.00	..	..	4.00	4.00	..
Fourth Day:								
Filtered in....	Dark	Light	Dark	Light	Light	Dark	Light	Dark
Time........	7.30	7.30	10.00	7.30	7.30	10.00	10.30	10.30

Table VIII.—"D" Grade.

	A.	A'.	B.	B'.	C.	C'.	D.	D'.
First Day:								
Dissolved in...	Dark	Dark	Dark	Dark	Light	Light	Light	Light
Time........	3.30	3.30	3.30	3.30	3.30	3.30	3.30	3.30
Second Day:								
Filtered in....	Dark	Dark	Dark	Dark	Light	Light	Light	Light
Time........	8.30[1]	8.30	8.30	8.30	8.30	8.30	8.30	8.30
Transferred to	..	..	Light	Light	..	..	Dark	Dark
Time........	..	..	3.30	3 30.	..	..	11.30	11.30
Third Day:								
Filtered in...	..	Dark	Light	Light	Light	Light	Dark	Dark
Time........	..	8.30	8.00	1.00	8.00	1.00	8.30	2.00
Transferred to	..	..	Dark	..	..	Dark	Light	..
Time........	..	..	4.00	..	..	4.00	4.00	..
Fourth Day:								
Filtered in....	Dark	Dark	Dark	Light	Light	Dark	Light	Dark
Time........	7.30	7.30	7.30	7.30	7.30	7.30	7.30	7.30

Table IX.—Carbenes Obtained.

	Second Day.	Third Day.	Fourth Day.
Gilsonite:			
A.............	0.0	0.07	...
A'............	0.0	....	...
B.............	0.0	0.07	...
B'............	0.04	....	...
C.............	0.07	0.15	...
C'............	0.00	....	...
D.............	0.07	0.13	...
D'............	0.0	....	...
Texas:			
A.............	7.3	0.2	0.1
A'............	7.4	0.2	0.9
B.............	7.3	1.5	3.1
B'............	7.2	2.6	1.7
C.............	11.4	3.2	0.6
C'............	11.2	2.8	0.3
D.............	12.2	2.0	0.6
D'............	11.5	2.4	0.1
Durango:			
A.............	4.8	....	0.4
A'............	5.1	0.2	0.1
B.............	5.0	1.9	4.4
B'............	5.0	3.0	2.0
C.............	9.0	3.2	0.5
C'............	9.2	2.8	0.2
D.............	8.5	0.2	0.5
D'............	8.4	0.3	0.1

[1] Filtering and washing two days.

The samples of Gilsonite showed practically no carbenes, and require no comment. The Texas pitch, however, gives some interesting results. "A" was carried out entirely in the dark, and showed that all the carbenes were removed by the first filtration. "C" manipulated entirely in the light gave 4 per cent. more carbenes on the initial filtration, and a further increase of 3 per cent. on the second filtration. A third filtration gave, however, practically no more. Where samples first stood in the light and then in the dark, it should be noticed that they were filtered in the light, and, after the filtration was complete, were transferred to the dark. But, after passing through the Gooch, the solution was exposed a drop at a time to light, and the filtrate remained in the light until all was filtered. In Table VII, the "time filtered" shows the time filtration commenced, "time transferred," when it was complete. Thus we see that B standing in the dark gave 7.3 per cent. Further standing in the light gave 1.5 per cent., and further standing in the dark 3.1 per cent. This very contradictory result is explained when it is noticed that before bringing back into the dark, it was light exposed from 8: 30–4: 30, while the filtration was in progress. This may be criticized as an unwise method of procedure, but on the other hand, where different fineness of filters required different lengths of time for filtration, the amount of carbenes is proportional to the length of exposure during filtration, and tends to all the more firmly establish the fact *that the amount of carbenes is very largely increased by the action of light.*

Turning now to the Durango, we find a further confirmation. "A" carried on entirely in the dark gave at first 5.1 per cent., and practically no more on further standing. "C," light-exposed, gave at first 4 per cent. more than "A," a second filtration an increase of 3.2 per cent., while a third filtration gave only an additional half per cent. The total was 12.7 per cent. in close agreement with figures obtained before on the same sample. The figures outside of the initial amounts are *strikingly* similar to the Texas pitch. Likewise B, as above, gave an exceptionally large per cent. on the final filtration. Standing and filtering in the dark, it had then stood in the light, but since there was no agitation, carbenes were precipitated only in the outer layers of the solution. On filtering in the light, the whole solution was thoroughly light-exposed after filtration, and, though it stood finally in the dark, the carbenes had already been precipitated.

"G" Grade flux, Extra Heavy flux and a blown oil, which under ordinary conditions had no carbenes, were dissolved in tetrachlorid, and the solutions exposed to sunlight for three weeks.

Solutions of Bermudez refined asphalt and Trinidad refined asphalt were allowed to stand 19 days.

TABLE X.—CARBENES FROM FLUXES ON THREE WEEKS' LIGHT EXPOSURE.

	Per cent.
"G" grade	7.6
Extra heavy	0.3
Blown oil	0.9

TABLE XI.—CARBENES FROM BERMUDEZ AND TRINIDAD REFINED ASPHALTS.

BERMUDEZ.

	Per cent.
Standing in the dark	0.0
Standing in north light	1.3
Standing in sunlight	1.3

TRINIDAD.

	Per cent.
Standing in the dark	3.0
Standing in north light	8.2
Standing in sunlight	10.7

It is seen, therefore, that the class of substances usually determined as carbenes consists of two kinds of hydrocarbons: those which are precipitated in a tetrachlorid solution, in the dark, completely on standing 12 hours; and those which are precipitated from the tetrachlorid solution by the action of light and are usually *not* completely precipitated by standing 12 hours. To the first class of substances we have given the name pure carbenes since they are evidently the true constituent of the bitumen soluble in carbon bisulphid and insoluble in carbon tetrachlorid. To the second class the name "pseudo carbenes" needs no comment. The total carbenes are the sum of these two.

The work of Kirschbraun was then repeated, determining the carbenes in the pure and pseudo forms. In each case, about 700 g. of bitumen were heated in a round-bottomed flask of twice that capacity on an air batch, with air agitation. The carbene determinations were made running parallel samples in the dark and in north light with a 12-hour stand. The pure carbenes is the amount insoluble in tetrachlorid in the dark less the amount insoluble in carbon bisulphid. The pseudo-carbenes is the difference between the CCl$_4$ insolubles in light and dark.

TABLE XII.—DUPLICATION OF EXPERIMENTS BY KIRSCHBRAUN. BERMUDEZ ASPHALT CEMENT 100 PARTS, BERMUDEZ R. A. 26 PARTS, K. CO. PARAF. FLUX.

	Original.	Heated 2 hrs. at 300° F.	Heated 2 hrs. at 400° F.	Heated 2 hrs. longer at 500° F.	600° F.	700° F.
Bitumen	97.9	97.9	97.8	98.0	97.9	95.4
Min. mat	1.2	1.3	1.2	1.2	1.5	1.9
Organic non-bitumen	0.9	0.8	1.0	0.9	0.6	2.7
Malthenes 88° Naph. in asphalt	75.1	75.0	70.0	65.1	53.8	47.1
Malthenes 88° Naph. in bitumen	76.7	76.6	71.2	66.5	54.9	49.4
Pure carbenes	0.0	0.0	0.0	0.0	0.0	10.0
Pseudo-carbenes	0.0	0.0	0.0	0.0	0.0	0.7
Total carbenes	0.0	0.0	0.0	0.0	0.0	10.7
Penetrometer	119	102	68	35	7	3
Fixed carbon	13.0	12.7	12.9	14.5	19.6	31.0

These results agree in the main with those of Kirschbraun. A larger amount of carbenes is obtained in the final stage, but conditions could not be exactly similar, and we cannot be sure, since he gave no figures, that the fluxes used were evenly approximately similar. It is especially worthy of note that the carbenes from overheated Bermudez asphalt cement come down almost completely in the dark, and that the light has very little additional precipitating action. The results on Bermudez refined asphalt follow:

TABLE XIII.—BERMUDEZ REFINED ASPHALT. DUPLICATION OF EXPERIMENTS BY KIRSCHBRAUN.

	Original.	Heated to 500° F. for 2 hrs.	Heated to 500° F. for 4 hrs.	Heated to 500° F. for 6 hrs.	Heated to 700° F. for 2 hrs. longer.
Bitumen	97.5	97.7	97.3	97.5	97.4
Min. mat	1.5	1.5	1.6	1.5	1.9
Organic non-bitumen	1.0	0.8	1.1	1.0	0.7
Malthenes 88° Naph. in asphalt	73.9	68.8	63.2	60.1	57.3
Malthenes 88° Naph. in bitumen	75.9	70.2	64.9	61.4	58.8
Pure carbenes	0.0	0.0	0.0	0.0	0.0
Pseudo-carbenes	0.0	0.0	0.0	0.0	0.0
Total carbenes	0.0	0.0	0.0	0.0	0.0
Penetrometer	16	6	4	2	5
Fixed carbon	15.2	16.3	17.8	19.2	25.6

No carbenes were found, while Kirschbraun in his final heating obtained 25.8 per cent. There seems to be no explanation available for this discrepancy. Kirschbraun worked with a smaller amount, but even if he had obtained local overheating, it would in all probability have only increased the free carbon. The absence of carbenes even from overheated Bermudez asphalt would be expected.

In order to further examine the effect of overheating upon the pure and pseudo-carbenes, a sample of Durango, California, asphalt was treated as above.

TABLE XIV.—EFFECT OF HEATING UPON THE CARBENE CONTENT OF DURANGO ASPHALT.

	Original.	Heated to 400° F. 2 hrs.	Heated to 500° F. 2 hrs. longer.	Heated to 600° F. 2 hrs. longer.	Heated to 700° F. 2 hrs. longer.
Bitumen	99.7	99.6	99.7	99.8	78.8
Min. Mat	0.2	0.1	0.1	0.1	0.1
Organic non-bitumen	0.1	0.3	0.2	0.1	22.1
Malthenes 88° in asphalt	79.2	77.0	73.2	64.8	46.2
Malthenes 88° in bitumen	79.4	77.3	73.4	64.9	58.6
Pure carbenes	2.3	1.7	2.2	4.0	15.0
Pseudo-carbenes	3.2	4.2	3.8	5.8	1.5
Total carbenes	5.5	5.9	6.0	9.8	16.5
Penetrometer	87	54	28	9	too hard.
Fixed carbon	14.7	15.5	16.9	20.2	33.2

Except in the final heating, the pseudo-carbenes were slightly larger than the pure. The fixed carbon and 88° naphtha insoluble increased gradually, but the bitumen remained constant. When heated to 700° F. 22 per cent. of free carbon was formed and the carbenes, 16.5 per cent., were almost entirely precipitated in the dark.

In order to investigate further the nature of the carbenes, 60 g. of a different Durango were dissolved in the dark in 6 l. of tetrachlorid. It was allowed to stand over night and filtered in the dark.

Yield, about 1.5 g., equivalent to 2.5 per cent. The Durango contained 0.4 per cent. mineral matter and 1.0 per cent. total insoluble in carbon bisulphid.

Thus pure carbenes were 1.5 per cent.

The solution was then placed outside (June 14, 1909) and allowed to remain 22 days. There was, during this period, continuously clear weather, part of it extremely hot. The solution was then filtered.

Yield, pseudo-carbenes 8.23 g. equivalent to 13.7 per cent.

It may be noted here *that, on opening the container after the light exposure, the odor of hydrochloric acid was plainly perceptible.*

The pure carbenes (combined, of course, with the disulphid insoluble) did not melt even at a red heat. They contained nitrogen and sulphur.

The pseudo-carbenes decomposed with effervescence on heating above 300°. They also contained nitrogen and sulphur.

They had the following properties:

	Per cent.
Soluble 88° naphtha	3.3
Soluble hot chloroform	98.5

They seemed to be sparingly soluble in disulphid, for though the insoluble(?) residue was washed continually, the washings were always colored. The results from one trial will show this.

	Per cent.
Taken 0.5000 gram	
Dissolved in 100 cc., washed with 125 cc., dissolved	16
Washed further with 200 cc., dissolved further	6.6
Washed further with 200 cc., dissolved further	4.7

This would at once suggest that those compounds called carbenes were not present as such in the original bitumen, for, as stated above, all but 1 per cent. of the Durango was very readily soluble in a small amount of bisulphid.

This conviction was strengthened when it was found that both the pure and pseudo-carbenes contained 3 to 4 per cent. of chlorine.

The original Durango, of course, contained none.

When, in recovering tetrachlorid from bitumen solutions, it is run down to dryness in the retort, hydrochloric acid is evolved. One case gave as much as 96 mg. per liter in the distillate. Thus, evidently, tetrachlorid is decomposed by bitumen when heated with it. It has already been noted that when the container with the tetrachlorid solution after sunlight exposure was opened, the odor of hydrochloric acid was quite evident.

Could it not be that by the action of light as well, hydrochloric acid is formed, which combines with certain unsaturated hydrocarbons and forms precipitates?

In order to find the effect of hydrochloric acid upon the carbene figure, 1 g. of Durango, used to prepare the pure pseudo-carbenes, was dissolved in 100 cc. tetrachlorid and dry HCl passed through for one hour. A duplicate was air-blown for a similar time to check the result. Both were allowed to stand over night.

	Per cent.
Carbenes by HCl	21.8
Carbenes by air-blowing	4.4

The Durango submitted to high temperatures above was examined in the same way, giving the following amount of carbenes:

	Per cent.
ORIGINAL	24.2
After heating up to 600° F	38.0
After heating up to 700° F	33.9

Hydrochloric acid causes thus a *large* increase in the amount of carbenes, and the hypothesis already suggested finds confirmation, namely:

That light acting upon a solution of bitumen in tetrachlorid causes the bitumen to decompose, the tetrachlorid giving hydrochloric acid, which in turn combines with unsaturated hydrocarbons and precipitates them. But only certain bitumens (namely those which have been more or less overheated) can bring about this phenomenon. Bermudez asphalt is practically unreactive.

We are thus forced to the conclusion that in overheating certain decomposition products are formed, which compounds are capable of reacting with the tetrachlorid; or, that an excess of HCl is naturally generated and by overheating, compounds capable of combining with it are formed.

We have to consider the pure carbenes which also contain chlorin and which are formed *without the aid of light.* With our present knowledge, it is useless to devise any theory to distinguish between these two classes. Much further work is necessary.

Opponents of the carbene determination will apparently find in this new ammunition for their warfare. But, after all, has the carbene lost any of its value? It is to be sure affected by light, but, if conditions are relatively the same, if the solution is allowed to stand over *one* night, as Mr. Richardson suggests, and if it is kept from direct sunlight, very concordant results may be obtained. What if by long exposure the amount may be increased? The same effect can be obtained by using hydrochloric acid gas, yet we would not think of taking the amount so obtained as the carbene figure. Like many other determinations in bitumen analysis, the carbenes are more or less arbitrary, but they do show, proportionally, when weathering or overheating has taken place, and as Kirschbraun states:[1] "The amount of carbenes allowable in asphalts must as yet be a matter of opinion, until sufficient data are available to determine such limits.

In the meantime, unless it can be conclusively shown from practical results that asphalts high in carbenes make satisfactory pavements, a discrimination against such asphalts is, at present, logical and safe.

NEW YORK TESTING LABORATORY,
    MAURER, N. J.,
        January 14, 1910.

[1] *Loc. cit.*

BACTERIAL ACTIVITY AS A CORROSIVE INFLUENCE IN THE SOIL.

By Richard H. Gaines.

One of the many subjects presented for study in connection with the construction of the Catskill Aqueduct was the corrosion of iron and steel.

In a communication to the American Electrochemical Society, in 1908,[1] the writer discussed at some length two inherent causes for corrosion of steel, namely, irregularity of structure and segregation of impurity. Experience has shown that metals which are non-homogeneous in character are far more subject to deterioration than those which have the same composition and structure throughout.

Conclusions reached from the study referred to were that although the cumulative decomposition of materials like steel may be conditioned by impurity the operation of this cause would be negligible if the process were not vastly hastened by some external impulse. Moreover, since it is probably impossible to modify greatly the composition of steel and retain the physical properties that render it useful, the solution of the corrosion problem would be found in controlling the ascertained accelerating influences.

A great deal has been published relating to the corrosion of iron and steel during the last few years, with much importance attached to the electrochemical theory of Whitney, on this side the Atlantic, while in Europe, attention has been chiefly concentrated on the effect of the presence or absence of carbon dioxide.

On taking up the present work, it was deemed best to direct attention to actual phenomena rather than to be influenced by preconceived opinion and to base theoretical conception only on observed facts. Due examination had led to the conclusion that none of the existing theories of corrosion account sufficiently for some of the observed phenomena. Only a phase of the research at the laboratory of the Board of Water Supply will be touched upon here. In connection with the series of studies in progress, analyses were recently made of about twenty-five samples of rust, collected from pits in the outside, and tubercles on the inside of steel conduits located in different parts of the country. The analyses showed, within certain limits, a remarkable difference in the character of the samples of rust, varying according to the underground conditions of exposure to which the iron or steel may have been subjected. The results appeared to indicate the operation of hitherto unconsidered influences accelerating the corrosion process.

For the purpose in view here, it will suffice to mention but two of the ingredients in the samples of rust. Attention was early attracted to the high percentages

of sulphur in combination, and the invariable presence of more or less organic matter in all the materials examined. Through the courtesy of Mr. T. Merriman, at whose instance the analyses were made, permission was granted to cite as examples results obtained from some of the samples of rust collected from different steel conduits (names must be omitted) in which combined sulphur calculated as sulphuric anhydride was found as follows:

	SO_3, per cent.
Tubercles from interior of Conduit No. 1	1.41
" " " " " No. 2	2.25
" " " " " No. 3	2.98
Rust from "pits" in exterior of Conduit No. 4	1.51
" " " " " " " No. 5	3.95
" " " " " " " No. 6	6.50

The very high sulphur content shown in the last of these examples was perhaps in part due to the peculiar soil conditions surrounding the corroded conduit, and was, no doubt, an exceptional case. The soil backfilled about this pipe gave a loss on ignition of 85 per cent., and consisted largely of decomposed organic matter. Whereas the samples of rust collected from several steel conduits gave an average of nearly 3 per cent. of sulphuric anhydride, analyses of pieces of steel cut from the same conduits showed only 0.05 per cent. or less of sulphur. In contrast with these figures, tubercles artificially formed (by connecting with a gravity cell, steel plates immersed in a solution of sodium chloride) gave on analysis less than 0.15 per cent. sulphuric anhydride. As is well known, finished steel of the character used in conduits never contains more than a minute fraction of 1 per cent. of sulphur, which is present as manganese sulphide, ferrous sulphide only appearing when the quantity of manganese is insufficient. The proportion of manganese in the samples of rust did not correspond with what would be expected if manganese sulphate were the first oxidation product. On the contrary, the percentage of manganese found in the rust was no greater than in the steel, and generally less than 0.35 per cent.

We know that iron has a tendency to form compounds with sulphur just as it has with oxygen. It is not, therefore, the mere presence of sulphur, but the occurrence of its compounds in such quantity in connection with the rusting of iron that is significant. It was believed that a satisfactory explanation of this phenomenon would shed new light on the rapid deterioration of underground iron and steel structures by corrosion.

In casting around for a theory to account for the singular presence of sulphur or its compounds in the quantities found associated with the rust materials, early consideration was given to the possible rôle played by bacteria. Many decompositions hitherto unsuspected, and chemical changes chiefly destructive in character, are no doubt accomplished by these organisms. In certain prototrophic forms a process

[1] *Transactions of the American Electrochemical Society,* **13,** 55 et sequitur.

goes on comparable to respiration, but consisting in the oxidation of inorganic compounds. Belonging to this class, some of the thiobacteria possess the unique power in organic creation of breaking up sulphur compounds and assimilating into the cells of their protoplasm pure sulphur, which is subsequently oxidized into sulphuric acid. Leaving aside tempting speculation concerning the mode in which life interacts or is associated with matter, the data at hand seemed to warrant the belief that an important connection existed between bacterial activity and underground corrosion. Whatever produces acid conditions in the soil must contribute in no small manner to the corrosive influences present. That acid compounds are formed in abundance as the result of vital activity is easily demonstrated.

Important recent advances in our knowledge of bacteria are those having reference to the agency of these organisms in the circulation of certain elements in nature. The functions of nitrogen and iron bacteria in this connection have long been known. Definite anaerobic forms also exist which have the power of fixing carbon and sulphur as well as nitrogen from inorganic sources. Owing to the peculiarities in the modes of nutrition and respiration of these bacteria (*vide infra*) they require neither free oxygen nor organic food materials for vital activity.

It is now well known that a whole series of sulphur bacteria exist of the genera thiothrix, chromatium, spirillum and monas, which play important parts both in the circulation and concentration of sulphur in nature. There exist, moreover, in the mud of marshes anaerobic bacteria which decompose cellulose, probably hydrolyzing it first and then splitting the products into carbon dioxide and marsh gas. When calcium sulphate is present, the nascent methane set free by the cellulose bacteria reduces the sulphate with the formation of calcium carbonate, sulphuretted hydrogen and water. This is the explanation of the occurrence of marsh gas and sulphuretted hydrogen in bogs, and such conditions afford favorable media for sulphur bacteria which multiply by oxidizing the sulphuretted hydrogen and storing the sulphur in their own protoplasm. Processes resulting from the bacterial decomposition of vegetable mud only take place at certain depths, and the zone of physiological activity rises and falls with the variations of partial pressures of gases due to the rate of evolution of sulphuretted hydrogen. In the deeper parts of this zone the partially anaerobic bacteria absorb the sulphuretted hydrogen and as this rises and meets atmospheric oxygen other bacteria oxidize it and store up the sulphur; then ascending into planes more highly oxygenated other bacteria further oxidize the sulphur to sulphuric acid, which combines with any calcium carbonate present to form sulphate again. These bacteria, therefore, employ sulphuretted hydrogen as their source of energy much as higher organisms employ carbohydrates—instead of liberating energy as heat by the combustion of sugars, they do it by oxidizing hydrogen sulphide. One of the anaerobic forms (*Spirillum disulphuricans*) attacks and reduces sulphates, thus undoing the work of sulphur bacteria just as certain denitrifying bacteria reverse the operations of nitro-bacteria. Again, we have sulphur taken into the higher plants as sulphates, built up into proteins, decomposed by putrefactive bacteria, and yielding sulphuretted hydrogen which the sulphur bacteria oxidize, the resulting sulphur is then oxidized to sulphuric acid and again combined with calcium to gypsum, the cycle being thus complete.

The well-known part played by iron bacteria affords an analogy to the circulation and chemical changes just described wrought by the life processes of sulphur bacteria in the widely distributed sulphur compounds in nature. Pools and marshes near iron mines abound in bacteria, some of which belong to the remarkable genera crenothrix, cladothrix and heptothrix and contain ferric oxide in their cell walls. This iron deposit is not merely mechanical, but is due to the physiological activity of the organism which liberates energy by oxidizing ferrous oxide in its protoplasm. The iron must be in certain soluble conditions, and the soluble carbonate of the protoxide seems most favorable. The hydrocarbonate absorbed by the cells is oxidized probably thus:

$$2FeCO_3 + 3H_2O + O = Fe_2(OH)_6 + 2CO_2.$$
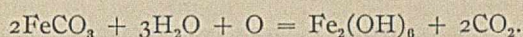
The ferric hydroxide accumulates in the sheath and gradually passes into the more insoluble ferric oxide. These actions are of great importance in nature, as their continuation results in the enormous deposits of bog iron ore.

The writer has recently been informed of a serious case of corrosion in the West, in which the evidence is apparently conclusive that the damage was due to specific bacteria. During repair work on the foundation structure of a bridge across Lake Hauser, in Montana, the attention of the engineer in charge was attracted to protuberances which occurred with more or less regularity on the steel work of the supporting tubes. The steel construction men designated this "shell rust" and said that it was often seen on steel which had been under water for some time. Mr. Wilton G. Brown, a Montana naturalist, made a careful examination of the protuberances and found that this was not a case of common rust action, but under the center of each individual an irregular pit was eaten into the steel. An inspection of some of the rust material under the microscope soon led to the conviction that some organism had played a part in the destructive corrosion. The phenomena were reported to Professor Beck, of Freiburg, Germany, who suggested that such vigorous action was probably

due to bacteria. Specimens of the rust were then submitted to the eminent bacteriologist, Dr. Shorler of Dresden, who identified *Gallionella ferruginea* in great abundance in the material. The method of attack of this organism is first the elimination of an acid product by which the iron is dissolved, and then each individual forms a pit by eating out the metal. The structural damage was reported to have been enormous for the short period of less than a year that the bridge had been in service.

The foregoing considerations apparently warrant the conclusion that the corrosion of underground iron and steel structures is in part due to bacterial activity:

(1) Directly, or by the attack of a specific microbe.

(2) Indirectly, or by creating acid conditions in the soil. Certain bacteria present, owing to peculiar modes of absorption and elimination of sulphur and its compounds convert harmless substances into corrosive acids.

The remedies proposed are:

(1) Free drainage quickly carrying off the acid solutions formed should arrest or prevent destructive action.

(2) In localities or situations where drainage is impracticable, slack lime should be packed about the metal, to neutralize acids formed as a result of bacterial activity.

147 VARICK ST., NEW YORK.

VARIATIONS IN CAR PAINTING PRACTICE.

By CARL F. WOODS.
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The past few years have witnessed a wide-spread movement in this country towards the standardization of paint products. Public attention has been called to the prevalence of adulteration and to the practice, particularly in mixed paints, of selling goods of short weight, while certain investigators ably supported by the reputable paint manufacturers have revealed many such frauds and have taken decisive steps toward the education of the consumer and the establishment of definite standards. Important data are being accumulated on the relative value of the various pigments and vehicles, but comparatively little attention is being given to the proper application of the paints themselves. The responsibility for poor results in painting rests both with the painter and the paint manufacturer, for although inferior materials cannot be made to give long life even in the hands of the most skilful painter, it is equally true that good paint improperly applied affords no better results than poor paint. There is no class of painting in which this is more clearly illustrated than in that of car finishing, for this is not a comparatively simple operation like house painting, but on the con-

trary is a complex and highly skilful procedure, requiring expert labor and involving the application of many coatings.

The object of car painting is both for protection and for decoration, although the latter consideration has exerted the greater influence on the modern practice of car finishing. It is possible to preserve the woodwork of a car body just as efficiently by frequent painting with suitable oil paints as by covering it with the ten to fifteen coats of paint and varnish customarily applied. This is well illustrated by the practice of the Interborough Rapid Transit Company of New York, who paint the bodies of the subway cars with a dark red house paint. Not a vestige of ornamentation is used and the cars are not even varnished. This is an extreme case, but the practice has been adopted by an experienced management, and indicates the simple method of car finishing which can be used where the appearance of the car is of no importance. The steam or electric car operated on the surface, however, occupies a prominent position, and the public justly demands that it present a well-kept exterior. The appearance and condition of the cars is frequently considered an indication of the liberality of the management, and as a result many companies have carried this idea to an extent which brings an unnecessary burden on the earning capacity of the system. The cost of painting the same type of car varies on different roads from $30.00 to $60.00, and in certain cases an even larger amount, while some roads are forced to repaint their cars every two years, and others with the aid of one coat of varnish each year are able to operate for ten to fifteen years before complete refinishing becomes necessary. It is particularly significant that those cars which have had the most expensive finishing are not of necessity the longest-lived. It is obvious, therefore, that there are certain underlying principles upon which the durability of the finish depends.

Car paints as a rule are mixtures of liquids and solids having widely different chemical and physical properties. Between the priming coat and the final varnish, there are not less than twenty elements intimately in contact, some of which have an affinity for each other, while others may tend to dissociate. This is particularly true of varnishes, for two different brands of equal durability may so react as to materially injure the wearing qualities of either one. While each succeeding treatment has its own specific demands, the entire paint coating must amalgamate and act as a unit to prevent separation of the various films under the physical stresses of service, produced by the expansion and contraction of the car under changes of temperature, and the wrenching and twisting incidental to operation. The importance of selecting the right composition for the successive coats is self-evident, but it is equally essential to employ a method

of finishing which will take into consideration the specific demands which are imposed upon each coat.

There are four fundamental operations in car painting which must be performed to obtain the proper finish and the desired durability:

First, the pores of the wood must be thoroughly saturated to prevent the absorption of succeeding coats and to form a cementing bond between the wood and the paint films.

Second, the natural inequalities of the surface must be corrected and a smooth, hard foundation prepared for the application of the succeeding color and varnish coats.

Third, the required color must be applied in a smooth, homogeneous film which is sufficiently thick to cover the underlying coats and which at the same time possesses proper elasticity.

Fourth, the color coat must be covered with a film of varnish, both to protect the underlying paints from the effect of the weather and to obtain the glossy, smooth finish desired. It is necessary that this final coat be hard enough to withstand the abrasive action of sand and dirt and the general deteriorating effects of sun, wind and weather, but at the same time possess the maximum amount of elasticity.

Three distinct processes for car finishing are in use, each of which, if properly applied, produces satisfactory results, but obtains the requisite qualities by essentially different methods. These three systems may be called the "Lead and Oil," the "Surfacer" and the "Color and Varnish" Processes. Other methods of finishing are employed but all of them are abbreviations or combinations of the three main types.

The "Lead and Oil" Process is the oldest system in use, and requires the application of· the greatest number of coats and the expenditure of the most labor. This process consists in thoroughly saturating the wood with a thin paint of white lead and linseed oil, followed after proper drying by thicker coats of the same paint until the woodwork is properly "primed and filled." On the foundation so prepared, several coats (usually three) of a special paint known as "rough stuff" are applied. This consists essentially of a mineral silicate of moderate fineness mixed with white lead and ground in varnish. Such a paint dries quickly and can be brought by rubbing with blocks of pumice to a smooth, slate-like finish, which affords an admirable surface for the body color. Car colors, often known as coach colors, form a special class of paints and consist of a suitable pigment ground to the last degree of fineness in "Grinding Japan," which is really a special varnish generally made of high-grade shellac, and loaded nearly to the point of saturation with lead and manganese driers. The paint so prepared thinned for application with a suitable amount of turpentine dries readily and affords a smooth homogeneous color.

After a sufficient amount of color has been applied, the entire surface is given several coats of varnish, allowing each to dry thoroughly before adding the next. Two different types of varnish are sometimes employed on the same car: the first a "Rubbing Varnish," which contains from 6 to 12 gallons of linseed oil to every 100 pounds of hard gum and which dries to a very hard, dense film capable of being rubbed to a high polish, the second, or "Finishing Varnish," contains about 25 gallons of oil to every 100 pounds of gum, and, although slow-drying, produces a very elastic, tough and durable film. Experience has demonstrated the unfitness of "Rubbing Varnish" for exterior purposes, so that in the majority of cases "Finishing Varnish" alone is employed.

The "Surfacer" Process was devised about thirty years ago to reduce the time, labor and expense of the old "Lead and Oil" System. The fundamental difference between the two processes is that the "Surfacer" System omits the lead priming and filling and the "rough stuff" coats, but builds up the surface rapidly by the application of specially prepared paints. The first, or priming, coat is usually a quick-drying varnish containing a very small amount of a mineral silicate. The second, or loading, coat is of much thicker consistency and is a mixture of yellow ochre, white lead and dolomite or other equivalent substances ground in quick-drying varnish. The third coat, frequently known as the leveling coat, is similar to the "rough stuff" of the older process, but is devised to amalgamate with the preceding coats. Three applications of this material are usually made and the entire surface rubbed with block pumice to the desired finish. From this point on, the process is identical with the "Lead and Oil" System, the "Surfacer" Process confining its efforts to the rapid preparation of a surface for the color coat.

The "Color and Varnish" Process is of very recent origin and is a radical departure from the two previously described systems. The fundamental idea of this process is that the fewer the number of coats and the more similar in composition, the more durable will be the final results obtained. With this in view, a combination of coats are applied which are so composed as to prime the wood, prepare a surface and obtain the desired color at the same time. This is accomplished by employing heavy silicate paints, containing the proper color ground in the same kind of varnish, each coat possessing suitable drying qualities for its respective demands. The best results are obtained by the use of dark colors such as green or brown, as the principal ingredient may be ochre, umber or some other natural earth pigment which not only produces the desired shade, but is well adapted for preparing a foundation. The surface so obtained is covered with a coat of the body color ground in varnish followed by one thick coat of finishing varnish.

A fourth process is used to some extent which is really an abbreviation of the "Lead and Oil" Process and consists in priming and filling with lead and oil, but instead of applying "rough stuff," which requires expensive labor in rubbing, the lead coats are brought to a reasonable smooth finish with sandpaper. This process is further improved by tinting the surfacing coats in the direction of the body color so that by the use of dark colors, which are also more permanent than the lighter and more brilliant ones, one coat of color will be sufficient.

Each of the processes above described had its specific faults and virtues, so that no one has completely superseded the other two. The "Lead and Oil" Process if properly applied requires from three to four weeks and the application of ten or more coats. The "Surfacer" Process requires about the same number of coats, but, owing to the quicker drying of the surfacers, requires but two to three weeks for application. The "Color and Varnish" Process is the simplest of all and has been applied with apparently successful results in from six to eight days with an application of four to six coats.

The "Lead and Oil" Process produces very satisfactory results both in appearance and in durability, but the lead undercoats require considerable time for drying, the rubbing of the "rough stuff" necessitates the employment of skilled labor and the number of coats applied involves the expenditure of time and money out of proportion to the value of the actual results obtained. In addition, the process is susceptible to serious faults, such as improperly made "rough stuff" paints which are very brittle and produce cracks on drying which destroy the appearance and durability of the finish. The Japan colors serve no function except coloring and yet involve the expenditure of applying one or more complete coats. The most serious drawback to the employment of this method is that few companies are willing to spend the time and money necessarily involved and attempt to cheapen or hasten the work with consequent injury. The process actually produces too fine a surface for cars, and a certain amount of handsome finish can very properly be sacrificed for greater durability.

The "Surfacer" System is capable of producing excellent results with a smaller expenditure of time, but is little cheaper than the "Lead and Oil" Process, as the specially prepared surfacers are expensive. These paints are claimed to give better results than lead and oil, but owing to their nature are dependent upon the skill and honesty of the manufacturer and afford additional opportunity for adulteration. As the process of finishing on the prepared surface is the same as in the "Lead and Oil" Process, the objection to Japan colors applies with equal force. It is true of both processes that the necessary expenditure of time and money to obtain so handsome a finish is not justified by the demands imposed.

The faults of the "Color and Varnish" Process are not as yet thoroughly understood, as the method is of very recent development and has not been subjected to the test of long-continued service. It should be understood that the aim of this shorter process is durability at the lowest cost, and that appearance is in a measure sacrificed, but it is claimed that the finish obtained is fully as durable as by the older methods, that it is free from many of their faults and that it produces a finished appearance sufficiently good for the purpose. On the other hand, the process is dependent upon specially made paints in which adulteration is difficult of detection, and which if carelessly made are not only short-lived but render more difficult the refinishing of the car. It is also claimed that the body colors employed which are in reality a sort of enamel are very apt to produce checking, but this fault can be avoided by proper manufacture. The system is only applicable to dark colors as the lighter and more brilliant pigments do not possess sufficient covering power, but this is not in itself a failing as the use of dark green and brown colors is rapidly increasing, owing to the greater stability and length of life obtained. In this connection it is of interest to note that the Pullman Company has adopted a brown body color as the most satisfactory shade available, while a large proportion of the railroads both steam and electric employ a color of similar nature.

The foregoing brief description of the methods of car finishing in use indicate the wide variation in the processes employed and the results obtained. It has been shown by actual results that a saving of $20.00 to $30.00 can be made on the painting of each car and an increase in life obtained of from 5 to 10 years by the adoption of scientific methods of finishing. It is probable that no one of the methods in use embody the maximum efficiency possible of attainment and in view of the very large amount of money involved it is desirable that the entire subject be given careful study by technical chemists. The improvements in methods up to the present time have originated largely with those concerns interested in the sale of paint materials, and the consumer has accepted the changes with no thorough understanding of the underlying principles. The development of surfacers, and body colors is particularly the work of the chemist, for it necessitates a knowledge of the behavior of the various pigments and vehicles under different conditions, and the limitations imposed by manufacturing conditions. It is the duty of the industrial chemist in this as in all other lines of work to select those portions of the older systems which have proven their value and to develop new materials and methods which will lead to a standardization along the lines of maximum efficiency.

AFTER-EFFECTS OF CERTAIN PHOSPHATES ON LIMED AND UNLIMED LAND.

By H. J. WHEELER.

Received February 17, 1910.

The chief reasons for presenting a few of the many data bearing upon this subject, which have been secured at the Rhode Island Agricultural Experiment Station, are the following: (1) It is an almost universal practice among agricultural writers to caution their readers against liming land where superphosphates are to be applied, upon the ground that the presence of the lime will hasten the reversion of the phosphoric acid, in consequence of which its efficiency will be presumably greatly lessened.

(2) Some recent writers seem to hold that the action of chemical fertilizers is exceedingly fleeting.

(3) There has not been a sufficient appreciation of the occasional importance of liming as an aid in rendering the phosphorus of the soil available to plants.

(4) It is not sufficiently recognized that the availability of roasted iron and aluminum phosphates is dependent in a high degree upon the character of the soil, as concerns its content of calcium carbonate.

(5) The recent energetic advocacy of the employment of finely ground tri-calcic phosphate rock (floats) for agricultural purposes makes it especially important that its real agricultural value for various crops, and under varying soil conditions, should be more fully known.

(6) The great difference in the requirements of individual plants and of their ability to utilize the several phosphates under varying conditions, is not properly appreciated.

(7) The tendency of late to generalize from experiments with soil extracts as to what will result in the field is fraught with danger which should be pointed out.

The results presented here were secured in field experiments which were begun in the year 1894, and which are still in progress. Twenty plats of land, each containing two-fifteenths of an acre, are employed in connection with the experiment. They are separated by three foot-paths at the sides and by wide road-ways at the ends. The entire area of each plat is fertilized uniformly, but before harvesting for the final record, the crop is discarded from a strip three feet wide on each side and six feet wide across each end. The area remaining is then exactly one-tenth acre.

The experiment was begun at the outset by others with the idea of comparing applications representing like money values of the several phosphates. However, it was decided later to fully equalize the quantities of phosphoric acid (P_2O_5) applied to each plat, which was finally accomplished in the year 1902. The phosphoric acid applied during that interval of eight years was equivalent to an average annual application of 82 pounds per acre.

Each year generous applications of nitrogen in nitrate of soda and of potash either in muriate, or in high-grade sulphate of potash, have been made annually to all of the plats of land, at a uniform rate.

The phosphate applications have been duplicated in all cases, the applications in one series having been supplemented by lime while in the other they were not.

The following phosphates have been employed:
Dissolved bone-black.
Dissolved bone.
Acid phosphate.
Fine, ground steamed bone.
Basic slag phosphate ("Thomas" phosphate).
Floats (finely ground, unacidulated, tri-calcic phosphate rock).
Raw (unroasted) iron and aluminum phosphate.
Roasted iron and aluminum phosphate.
No phosphate.
Double superphosphate.

Below are given the yields of three of the many plants which were employed in 1903. These results are selected because they illustrate so well certain features in the preceding outline which it is desired to discuss somewhat in detail.

TABLE SHOWING THE RELATIVE YIELDS, IN POUNDS, OF THREE DIFFERENT CROPS.

Kind of phosphate.	Limed.	Unlimed.
Swedish Turnip:		
Dissolved bone-black	256	119
Dissolved bone	312	175
Acid phosphate	270	107
Fine, ground steamed bone	341	245
Basic slag meal ("Thomas" phosphate)	293	232
Floats	110	58
Raw iron and aluminum phosphate	1	0
Roasted iron and aluminum phosphate	117	1
No phosphate	16	1
Double superphosphate	245	89
German Golden Millet, weighed green:		
Dissolved bone-black	162	143
Dissolved bone	177	144
Acid phosphate	139	149
Fine, ground steamed bone	165	153
Basic slag meal ("Thomas" phosphate)	148	141
Floats	163	146
Raw iron and aluminum phosphate	132	109
Roasted iron and aluminum phosphate	159	118
No phosphate	123	50
Double superphosphate	160	82
Mangel Wurzel (Norbiton Giant):		
Dissolved bone-black	328	3
Dissolved bone	380	101
Acid phosphate	340	8
Fine, ground steamed bone	447	16
Basic slag meal ("Thomas" phosphate)	362	73
Floats	223	0
Raw iron and aluminum phosphate	36	0
Roasted iron and aluminum phosphate	153	0
No phosphate	40	0
Double superphosphate	145	1

It will be seen from the foregoing results with the four superphosphates, dissolved bone-black, dissolved bone, acid phosphate and double superphosphate that, excepting with the acid phosphate, the yields of millet were invariably greater where lime had been

used than where it had been omitted. It is readily possible, if the experiments had been conducted on a soil already well supplied with lime in calcium carbonate, that the result would have been exactly the opposite. This soil contained a large amount of lime removable by extraction with hydrochloric acid, yet it lacked carbonate of lime to such an extent that, when moistened, it not only quickly and intensely reddened blue litmus paper, but it also yielded immediately to ammonium hydroxide an abundant and intensely dark extract.

The single instance where liming did not prove helpful, in connection with the acid phosphate, was in the case of the millet, but this was unquestionably a mere chance result for upon making twenty-four direct comparisons in another experiment *where more lime had been used* the same kind of millet was injured by liming in every instance. In fact in this case the amount of lime present was almost up to the limit of benefit, and even approached the limit of endurance, for millet. This was not true, however, of the turnip; and even more particularly of the mangel wurzels. These results show the absolute absurdity of discussing the effect of liming land in connection with superphosphates, or indeed other phosphates, without special reference to the character of the soil involved and to the particular variety of plant to be grown.

Since the final equalization of the phosphates was not effected until the previous spring, this being the second crop grown since its accomplishment, these results throw less light upon the duration of the effect of fertilizers than other data which are to follow.

In this case the first liming of the soil occurred in 1894 at the rate of a ton of slaked lime per acre and the second in 1903 at the same rate. It is evident that, where no phosphate was used, the interval of nine years under continuous cropping must have been sufficient to draw heavily upon the native supplies of phosphoric acid which had been rendered available by liming. Nevertheless the result with the millet, which had been limed not only nearly to its limit of maximum direct benefit, but also to its limit of actual endurance, shows a yield of 50 pounds on the unlimed plat and of 123 pounds on that which was limed. This indicates, therefore, the probability that liming had rendered the phosphorus in the soil of the limed plat more available than it would otherwise have been. In other experiments at the Station, where a great lack of lime and of available phosphoric acid existed at the outset, liming liberated sufficient phosphoric acid to render the application of phosphates unnecessary at least for two or three years thereafter.

No beneficial effect of the liming, upon the availability of the *raw* iron and aluminum phosphate, was observable in connection with the yields of the turnips,

but there was evidence of some possible benefit[1] in this connection with the mangel wurzels and the millet. When it comes to a consideration of the effect of liming in connection with the *roasted* iron and aluminum phosphate, it will be seen that it was most positive as shown by the millet, and it was striking in the highest degree in connection with each of the other crops.

In regard to the floats it will be seen that the results with the millet, on both the limed and unlimed land, were essentially on a par with those secured with the better of the phosphates. With the turnip the inefficiency of the floats on the unlimed soil was exceedingly great; and though the relative inefficiency was far less on the limed land, it was still most striking. As concerns the mangel wurzels the soil was so deficient in carbonate of lime as to practically inhibit their growth, excepting on two of the plats. On this account it is only in the case of the limed plats, that indications of the value of the floats are afforded. It will be seen here that, excepting the case of the double superphosphate, floats fell in efficiency below all of the other of the better phosphates. The reason for the exception in the case of the double superphosphate may have been its high acidic tendency.

These results show the most striking differences in the ability of the different varieties of plants to utilize the different phosphates; and also the great dependence of this ability upon the character of the soil, as affected by liming.

The great danger, and indeed the utter futility, of attempting to predicate from experiments with any given plant grown in soil extracts or indeed by way of pot culture, as to the ability of the soil to produce crops of various kinds, could hardly be more strikingly illustrated, if one bears in mind the variations with the different crops on the same plats of land.

In order to illustrate especially the duration of the effect of the several phosphates, the yields in pounds of potatoes from one-half of each of the plats (one-twentieth acre) in 1909 are given below. It must be recalled that these yields represent those of the seventh year following that in which the equalization of the applications of phosphoric acid was made, or in other words it is the eighth crop following the last application of phosphates to any of the plats.

Kind of phosphate.	Limed.			Unlimed.		
	Large.	Small.	Total.	Large.	Small.	Total.
Dissolved bone-black...............	600	80	680	217	233	450
Dissolved bone....................	607	69	676	344	185	529
Acid phosphate...................	500	54	554	333	186	519
Fine, ground steamed bone..........	570	60	630	400	156	556
Basic slag meal ("Thomas" phosphate).	530	62	592	450	130	580
Floats............................	460	45	505	330	180	510
Raw iron and aluminum phosphate....	312	35	347	64	180	244
Roasted iron and aluminum phosphate	546	48	594	40	184	224
No phosphate......................	360	45	405	20	125	145
Double superphosphate.............	560	47	607	123	210	333

[1] The better results may have fallen within the limits of natural variation in the plats.

It is evident from an examination of the *total yields* that liming was highly beneficial in connection with all of the phosphates, excepting the floats, and when the relative production of large and small tubers is considered, great benefit is shown even in that case.

These results further support the earlier observations at the Station to the effect that liming may increase the total yield of potatoes but that even if this is not the case the yield of tubers of large size is usually greatly increased on a soil like that under experiment.

The wide differences in yield shown upon the several plats, when the results are compared with those where no phosphate had been used, show that the after-effect of floats, bone meal, basic slag meal and of all of the superphosphates, is great, and long continued, both on the limed and unlimed land.

As concerns the total yields, it will be seen that where no phosphate was used the crop was nearly three times as great on the limed as on the unlimed plat thus showing a percentage gain far in excess of that in any other case. Taking account of the large tubers it will be observed that the same holds true. Therefore, both the yields of large tubers and the total yields give evidence that liming had probably placed at the disposal of the crop more phosphoric acid than would otherwise have been available.

On the unlimed land the ·inefficiency of the raw and roasted iron and aluminum phosphates is most striking, whether judged by the total yields or by the yields of large tubers. On the limed land the results with the raw iron and aluminum phosphate were poorer than where no phosphate had been used, but the roasted iron and aluminum phosphate gave results superior to those secured with acid phosphate, basic slag meal, or floats. This furnishes therefore a most striking example of the effect of applications of lime upon the continued efficiency of roasted iron and aluminum phosphate even when the last applications of each were made several years previous.

Notwithstanding that the floats had been drawn upon less heavily in previous years, by virtue of smaller crops, than most of the other phosphates, they proved far less efficient on the limed land than any of the others excepting the raw iron and aluminum phosphate which showed a negative value. On the unlimed land they gave, however, a better result than the dissolved bone-black, double superphosphate or roasted iron and aluminum phosphate, and they proved essentially as efficient as the acid phosphate. Doubtless this may be accounted for in some measure by the tendency of tri-calcic phosphate to correct the acid condition of the soil, in which it was aided by a small amount of carbonate of lime, probably present in the floats. This view is supported by the excellent results with basic slag meal, which contains a considerable excess of lime, and hence can over-

come soil acidity in a greater degree than any of the other phosphates that were employed.

These results with floats, under the conditions existing where no lime was applied, cannot be cited to show that floats are equal in value to the other phosphates, for the reasons that the conditions of the soil were not such as to produce a normal crop under any conditions, as shown by the wonderful improvement in the yields after liming. Furthermore, when more nearly optimum conditions were created, as a result of liming, the floats then·became decidedly inefficient in comparison with the other phosphates. This question of optimum conditions, in other respects, is a point in connection with comparative tests of phosphates and discussions of them which deserves great consideration if one hopes to arrive at reliable conclusions.

A CONTRIBUTION TO OUR KNOWLEDGE OF THE NITROGEN PROBLEM UNDER DRY FARMING.

By F. J. ALWAY AND R. S. TRUMBULL.

The development of so-called "dry farming" or "dry-land farming" on the semi-arid portions of the prairies of North America has introduced problems in soil fertility distinct from those of humid or of irrigated soils. The characteristic feature of dry-land farming, in so far as it relates to the soil, is the practice of bare fallowing to store water in the subsoil, a practice; well known to cause, in humid regions, a rapid decline in soil nitrogen, the portion removed in the form of crops being only a small part of the whole that is lost. While in the least arid portions of the semi-arid regions certain intertilled crops may be found capable of taking the place of the fallow to some extent, such a change in cultural methods is not likely to lessen in any considerable degree the rate of loss of nitrogen. The use of leguminous crops as green manures has been advised as a substitute for the fallow.[1] The growth of the legumes, however, exhausts more or less of the moisture which fallowing would conserve, thus increasing the supply of nitrogen at the expense of that of water and so defeating the very object of the fallow.

Investigations in dry-land farming on the Great Plains have been undertaken only so recently by experiment stations in the United States that data are not yet available to indicate how serious the nitrogen problem is, how soon it will become acute, or how best to solve it. For twenty-two years, however, an experimental farm has been maintained by the Canadian government at Indian Head, Saskatchewan, and during the whole of this time the practice of bare fallowing has been employed for the conservation of moisture, it having been introduced into the Canadian Northwest by Mr. Angus Mackay, who has been

[1] Chilcott, "Dry-land Farming in the Great Plains Area," Year-book, U. S. Dep't of Agr., 1907.

superintendent of this farm since its establishment in 1887. It has been pointed out that this farm is located in a region properly considered semi-arid and that moisture studies of the subsoil clearly show the function of the summer-fallow.[1]

It is to be expected that the soil nitrogen supply will become an immediate economic problem in some parts of the semi-arid region much sooner than in others. Considering the five widely separated points of Indian Head in Saskatchewan, North Platte in Western Nebraska, Amarillo in Northwestern Texas, Solano in Northeastern New Mexico, and Douglas in Southeastern Arizona, we have a range of 400 per cent. of nitrogen as shown by the accompanying table. The samples analyzed were taken by one of us from the heavier types of soil, and accordingly, from those richest in nitrogen and organic matter. All the samples were composites, taken to a depth of six inches from virgin prairies.

TABLE I.—NITROGEN AND ORGANIC CARBON IN SEMI-ARID SOILS.

	Nitrogen. Per cent.	Organic carbon. Per cent.
Indian Head, Saskatchewan	0.384	4.20
North Platte, Western Nebraska	0.175	2.06
Amarillo, Northwestern Texas	0.136	1.37
Solano, Northeastern New Mexico	0.161	1.28
Douglas, Southeastern Arizona	0.087	0.58

In order to be able to form some idea as to the losses of humus, of total organic matter and of total nitrogen induced by continuous cultivation under semi-arid conditions, one of us visited the Indian Head Experimental Farm in October, 1907, and secured over 70 samples of soil to a depth of six inches from various fields, plots, etc., as well as from the adjacent prairie. In the case of each sample the total nitrogen, the humus and the organic carbon were determined, but in many cases in the results reported below only the averages of series are given. The humus was determined by the Hilgard method and the organic carbon by combustion with copper oxide after the carbonates had been decomposed by treatment with phosphoric acid.

The data in regard to crop yields and the history of the fields have been secured partly from the annual reports of the Canadian Experimental Farms and partly from private information furnished by Mr. A. Mackay.

Shutt[2] had shortly before called attention to the fact that while the yield of wheat at Indian Head has not shown any marked diminution, there has been a loss of 0.118 per cent. of nitrogen to a depth of eight inches during twenty-three years of cultivation, while the amount contained in the various grain crops produced during this period he calculated to be less than one-third as much as had been lost from the soil. He did

[1] Am. Chem. Jour., 31, 580 (1906); Jour. Agr. Sci., 2, 333 (1908); Bull. 130, Bureau of Plant Industry, p. 17 (1908).

[2] Address of the chairman of the Section of Agr. Chem. at the Toronto meeting of the American Chemical Society, June, 1907. Science, N. S., 26, 661, p. 265 (1907).

not determine the organic matter, but, on the basis of his studies of the soils of humid eastern Canada, concluded that the total nitrogen and the organic matter rise and fall together.

The farm was part of the prairie until 1882 when it was all plowed and during the next five years kept in small grain. The whole was kept in clean summer-fallow in 1887, the year the government took possession, since which time the history of the different fields has not been the same.

One field which from 1889–1898 had had the following succession of crops: oats, fallow, roots, barley, fallow, roots, fallow, wheat, oats and fallow, was in 1899 divided in twenty-two one-half acre plots which have since been continuously used for rotation experiments. The character of the rotation is indicated in Table II. Lacustral clay forms the surface of the greater portion of the field, varying in depth from a few inches to three feet. The remainder of the surface consists of boulder clay, or till, which also everywhere underlies the lacustral clay. The fallowing has consisted of one plowing early in June followed by three or four cultivations during the summer, this being sufficient to keep down all grass and weeds. Where one cereal crop has been followed by another, or by a legume or by rape, the stubble has been plowed in late summer or in autumn, and in the following spring harrowed and plowed again before seeding. The rape and leguminous plants have been turned under when

TABLE II.—HUMUS, NITROGEN AND ORGANIC CARBON IN SURFACE SIX INCHES OF SOIL OF THE ROTATION PLOTS.

Soil and Plot No.	Rotation. 1899 1900 1901 1902 1903 1904 1905 1906 1907	Humus. Per cent.	Nitrogen. Per cent.	Organic carbon. Per cent.
1	Wheat, oats, soy beans	2.49	0.307	3.15
2	Wheat, wheat, peas	2.13	0.276	2.77
3	" oats, tares	1.92	0.251	2.52
4	" wheat, red clover	1.90	0.259	2.56
5	" barley, alsike and alfalfa	1.88	0.263	2.57
6	Peas, wheat, wheat	2.05	0.265	2.68
7	Tares, wheat, oats	2.07	0.264	2.83
8	Soy beans, wheat, oats	1.78	0.244	2.36
9	Red clover, wheat, wheat	1.79	0.219	2.18
10	Alsike and alfalfa, wheat, barley	2.05	0.278	2.89
11	Rape, wheat, fallow	1.92	0.262	2.59
12	Wheat, wheat, fallow	2.00	0.298	2.98
13	" oats, fallow	1.94	0.258	2.56
14	" barley, fallow	1.87	0.254	2.61
15	" wheat, oats	1.91	0.267	2.70
16	" wheat, barley, oats	2.21	0.272	2.81
17	Oats, soy beans, wheat	2.10	0.275	2.79
18	Wheat, peas, wheat	2.05	0.271	2.79
19	Oats, tares, wheat	2.03	0.263	2.69
20	Wheat, red clover, wheat	1.93	0.272	2.77
21	Barley, alsike and alfalfa, wheat	1.93	0.265	2.80
22	Rye, fallow, wheat	2.00	0.260	2.68
	Averages of above:			
1–22	All rotation plots	2.01	0.266	2.69
1, 8, 17	Wheat, oats, soy beans	2.12	0.275	2.72
2, 6, 18	Wheat, wheat, peas	2.08	0.271	2.75
3, 7, 19	Wheat, oats, tares	2.01	0.259	2.68
4, 9, 20	Wheat, red clover and wheat	1.87	0.250	2.50
5, 10, 21	Wheat, barley, alsike and alfalfa	1.95	0.269	2.75
11, 12, 13, 14, 22	Fallow every third year	1.95	0.266	2.68
15, 16	Wheat, oats or barley every year	2.06	0.269	2.75
	Fifteen plots, cereal, cereal, legume	2.01	0.265	2.69
	Seven plots, no legumes	1.98	0.267	2.70

they have appeared to have reached their heaviest growth.

The most striking result of the analyses is the lack of any apparent connection between the composition of the samples and the treatment received by the different plots during the last nine years. No. 1, the sample highest in humus, nitrogen and carbon, is from a plot on which three crops of legumes have been plowed under, while the samples showing the least of the same constituents, *viz.*, 8 and 9, are likewise from plots on which three crops of legumes had been plowed under. A comparison of the averages shows no distinct difference between the fifteen plots on which legumes had been used as green manure and the other seven.

That an explanation of the differences between the different plots is possible is evident from the data in Table III. Suspecting that distinct differences might be found between different individual samples from the same plot, according as to whether they were from the till or from the lacustral clay, ten individual samples from rotation plot 14 were kept separate and analyzed. Till forms the surface of the west third of this plot and of an isolated area near the center, while the east end has a surface of typical lacustral clay. Near the edge of this area of lacustral clay and about twenty feet within it, five samples, 24 to 28, were taken at intervals of ten feet in a north and south line, and, beginning twenty feet west of 26, five samples, 29 to 33, were taken at intervals of eight feet in an east and west line. While 29 and 30 are really typical of neither type, being intermediate between the two, 31 to 33 are quite representative of the till, being from the isolated area above mentioned, and 24 to 28 are fair samples of the lacustral clay.

TABLE III.—HUMUS, NITROGEN AND ORGANIC CARBON OF INDIVIDUAL SAMPLES FROM THE SAME PLOT.

Soil No.	Humus Per cent.	Nitrogen. Per cent.	Organic carbon. Per cent.
24 Lacustral clay	2.30	0.291	3.27
25 " "	2.26	0.268	3.19
26 " "	2.29	0.282	3.01
27 " "	2.28	0.278	3.18
28 " "	2.28	0.278	3.08
Average lacustral clay	2.28	0.279	3.15
29 Boulder clay	2.03	0.278	2.85
30 " "	1.94	0.263	2.77
31 " "	1.67	0.231	2.43
32 " "	1.62	0.227	2.29
33 " "	1.57	0.221	2.33
Average Boulder clay	1.77	0.244	2.53

The samples of lacustral clay are richer in humus, total nitrogen and carbon than are those of till. From the differences found between the two types of soil on the same plot, it seems probable that the differences found between the different plots should be attributed chiefly to the varying amounts of the two types of surface soil.

Samples were secured from other fields and also from tree rows which had for the preceding 15 years been under continuous bare cultivation. These rows

of trees were planted along the lanes in 1892 and since that time the soil for a distance of twelve feet on each side of the trees has every summer been plowed and cultivated sufficiently to prevent the growth of grass or weeds. A summary of the results are given in Table IV.

TABLE IV.—RELATION OF COMPOSITION OF SURFACE SIX INCHES OF SOIL TO CULTIVATION.

No. of samples.	Cultivation.	Humus. Per cent.	Total Nitrogen. Per cent.	Organic carbon. Per cent.
6	Virgin prairie	2.84	0.384	4.20
2	In cultivation 7 yrs., in grass 18 yrs.	2.75	0.384	4.05
2	" " 16 " " " 9 yrs.	2.46	0.367	3.82
2	" " 21 " " " 4 yrs.	2.25	0.303	2.10
22	" " 25 " (Rotation plotfield)	2.01	0.266	2.68
6	Tree strips; in ordinary cultivation 10 years, in bare cultivation 15 yrs .	2.12	0.274	2.84
6	In cultivation in fields adjacent to tree strips	2.34	0.326	3.35

It is impossible from the data on hand to satisfactorily estimate the original composition of the virgin prairie where lacustral clay occurs.[1] The numbers given in the first line of the table are the averages of samples from the boulder clay and from the lacustral clay summits. The numbers are certainly too low because samples from some fields long under cultivation give slightly higher percentages than these. On soil where grass (in this case mixed with clover) has grown for 18 years, following 7 years of cultivation, no distinct loss is to be observed. The longer the fields have been in grass the less has been the decline. Where no grass at all has been grown the loss is greatest. Continuous bare cultivation in the tree rows has caused greater losses than where bare cultivation (fallow) and cropping have alternated. The extreme loss is probably about one-third of the original amount of all three constituents.

The data showing the effect upon the yield of wheat produced by replacing the summer-fallow by a leguminous crop plowed under as a green manure have been obtained chiefly from the field of rotation plots described above (Table II). A summary of the results is given in Table V. Judged by its crop-producing power, this field was very uniform in 1899, the yield of the 12 plots of wheat ranging only from 35 bushel 20 ℔. to 36 bushel 16 ℔. per acre. The data for wheat after legumes in 1899 are from a series of half-acre plots in another field. On account of the little data on the yields after fallow in the rotation field there are given in the table the averages of all field lots of the standard varieties grown on summer-fallowed land. There being no field or half-acre plots of wheat after fallow in 1900 it has been necessary to employ the yield of a smaller plot. In 1902 the wheat after fallow on the three rotation plots was injured so seriously by rust that the yield was lower (20.3 bushel per acre) and the quality poorer than on any other plot or field on the farm. Accordingly this yield should not be used in such a comparison.

[1] Alway and Vail, THIS JOURNAL, **1**, 17 (1909).

TABLE V.—YIELD OF WHEAT PER ACRE AT INDIAN HEAD AFTER FALLOW, AFTER LEGUMES PLOWED UNDER, AND AFTER WHEAT.

| | After fallow. | | | | After leguminous green manures. | | After wheat, oats and barley. | |
| | Field lots. | | Check plots. | | | | | |
Year.	Acres.	Yield, bu.	No. of ½ acre plots.	Yield, bu.	No of ½ acre plots.	Yield, bu.	No. of ½ acre plots.	Yield, bu.
1899	16	32.7	13	35.9	6	34.5	.[1]	28.9
1900	0	....	1[2]	27.0	5	14.9	4	5.4
1901	17	49.2	1	44.7	5	43.1	2	38.4
1902	40	33.4	3	....[3]	5	24.9	4	25.5
1903	26	35.8	0	....	5	23.8	4	15.9
1904	44	39.3	1	36.0	5	34.5	2	30.2
1905	44	40.0	3	35.3	5	30.0	4	23.1
1906	45	30.5	0	....	5	20.7	4	16.5
Average,		34.6	..	....	.	28.3	.	22.9

In two years, 1899 and 1901, when the previous harvest had been followed by exceptionally wet weather, the yield after legumes was about the same as that after fallow, the autumn rainfall probably having been sufficient to fully charge with moisture the subsoil of all the fields. In the other years the legumes, by exhausting the water of the subsoil, lessened the yield of the succeeding crop. Wheat has given a lower yield when sown on wheat stubble than after legumes, chiefly because the soil has been left drier in the autumn, although partially, probably because the supply of available nitrogen has been less. Even in the case of the two wet years mentioned, the difference in yield is considerable.

The data in Table VI seems sufficient evidence that, notwithstanding a decline of 30 per cent. or more in soil nitrogen, this element has not yet become a limiting factor in the yield of wheat on fallowed land at Indian Head. The table gives the yield of Red Fife wheat both without any fertilizers and with 100 lbs. and 200 lbs., respectively, of sodium nitrate. The plots were on fallowed land in every case, soil not previously fertilized being taken each year. In 1903 all the plots in the fertilizer experiments were so badly injured by rust that the crop was not weighed. The average yields of the three plots for the remaining six years were practically the same. No trials are reported of the use of nitrates on wheat sown on stubble.

TABLE VI.—YIELDS OF WHEAT AT INDIAN HEAD WITH AND WITHOUT NITROGEN FERTILIZERS.

| | No fertilizer. | | 100 lbs. NaNO₃. | | 200 lbs. NaNO₃. | |
Year.	Straw, cwt.	Grain, bu.	Straw, cwt.	Grain, bu.	Straw, cwt.	Grain, bu.
1900	17.4	27.7	17.6	27.3	17.6	27.3
1901	66.0	62.7	59.2	61.3	54.8	58.7
1902	37.6	29.2	31.6	28.0	38.8	30.7
1904	28.6	26.3	30.4	28.7	26.4	23.0
1905	72.4	42.7	62.8	42.0	54.0	43.3
1906	48.0	35.3	62.4	38.0	52.4	38.0
Average,	45.0	37.3	44.0	37.5	40.7	36.9

There are two distinct causes of a lowering of the content of organic matter, humus, and nitrogen of cultivated soils at Indian Head, viz., the oxidation of the organic matter and the erosion of the surface soil

[1] Two 3-acre fields.
[2] A fortieth-acre plot.
[3] Badly injured by rust.

by the wind. The deeper the layer of soil is, the lower is the content of humus and nitrogen; accordingly, erosion by the wind will have an effect upon the humus and nitrogen content similar to that of cultivation and may seriously affect the value of any such soil study as this.

The seriousness of the nitrogen problem for the semi-arid region as a whole is evident when it is considered that the loss in 25 years at Indian Head has been almost equal to the total percentage in the virgin soils of northwestern Texas and to much more than that in the virgin soils of Southern Arizona. Further, the use of a theoretically good rotation with leguminous green manures over a period of nine years has failed to have any appreciable effect in checking the rapid loss of soil nitrogen, while it has greatly lessened the yield of grain.

SUMMARY.

In a comparison of 22 rotation plots no distinct relation has been found between the composition of the soil and the nature of the rotation. In a long cultivated field the till was found poorer in humus, nitrogen and organic carbon than the lacustral clay. The amounts of the above three constituents found in any of the plots depend more upon the relative proportions of the two types of soil occurring on the plot than upon the previous treatment.

The longer the fields have been kept in grasses mown for hay, the less has been the change in composition of the soil. Continuous bare cultivation along tree rows has caused greater losses than the alternation of fallow and crop in the adjacent fields. The extreme loss of nitrogen, humus and organic carbon in 25 years is about one-third of the amounts originally present in the prairie. This loss of nitrogen is not sufficient, however, to cause nitrogen to have become a limiting factor in the yield of wheat on fallowed land at Indian Head.

Attempts to substitute for the bare fallow various leguminous crops, which have been plowed under, have decreased the yields of wheat. The lowered yields are evidently due to the drying out of the soil by the leguminous crop. In the exceptional years when the rainfall of autumn has been very heavy the yields after legumes have been as high as after fallow.

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NITROGEN AND CARBON IN THE VIRGIN AND FALLOWED SOILS OF EASTERN OREGON.

By C. E. BRADLEY.

The great wheat belt of Eastern Oregon is characterized by a silt loam soil. The deposits are of volcanic origin and are remarkably uniform in structure even to considerable depths. The rainfall in this

region is light, varying from 6–15 inches in different localities.

The general composition of this soil is given in the following partial analyses reported in Table I, the results being calculated to the moisture-free sample. 99.2 per cent. of this soil passes a 0.5 mm. sieve. It reacts mildly alkaline from the carbonate of lime present.

TABLE I.

	Total Per cent.	Soluble 1.115 HCl Per cent.
SiO_2	62.85	0.34
K_2O	1.63	0.74
CaO	4.52	3.05
MgO	1.94	1.66
Mn_3O_4	0.04	....
Fe_2O_3 Al_2O_3	8.47	8.37
P_2O_5	0.30	0.21
N	0.110	....
Volatile	8.37	....

The effect of long and constant cropping with wheat by the fallowing process has been proven to be destructive to soil nitrogen[1] when carried on under humid conditions. Determination of total nitrogen on a number of these semi-arid wheat soils, however, revealed the fact that very slight differences exist between the nitrogen content of the virgin soils and those which have been farmed for a quarter of a century. In view of this fact it was thought that a comparison of the carbon content of these soils would be of interest as illustrating changes which may have taken place in their organic matter under cultivation.

TABLE II.

No.	Description	Total nitrogen. Per cent.	Nitrogen decrease. Per cent.	Organ. carbon mgs. per gram.	Organ. carbon decrease. Per cent.	Inorgan. carbon mgs. per gram.	Inorgan. carbon decrease. Per cent.
45	Virgin, surface	0.114	.....	15.270	....	0.229	....
47	Fallow, surface	0.107	—6.1	11.731	23.2	0.142	38.4
46	Virgin, subsoil, 18 inches	0.081	.....	9.860	....	0.125	....
48	Fallow, subsoil, 18 inches	0.081	—0.0	7.540	23.5	0.116	7.2
49	Virgin, surface	0.101	.....	14.133	....	0.411	....
52	Fallow, surface	0.102	+1.0	11.150	21.1	0.131	68.1
50	Virgin, subsoil, 18 inches	0.094	.....	9.730	....	0.202	....
53	Fallow, subsoil, 18 inches	0.090	—4.3	7.352	24.4	0.153	24.2
54	Virgin, surface	0.098	.....	....	....	....	....
55	Fallow, surface	0.102	+3.9	.....	.....	.....	....

In Table II are given results of nitrogen and carbon determinations on typical samples of these soils. Nos. 45 and 46 are samples of virgin surface and subsoil and Nos. 47 and 48 are the corresponding soils which have been cultivated for 25 to 30 years, wheat and fallow alternating. Nos. 49 and 50 are likewise virgin samples with 52 and 53 the corresponding soils which have been in cultivation for about 17 years. No. 55 has been farmed about 25 years. These samples were obtained from Umatilla, Morrow, and Sherman counties respectively.

The nitrogen was determined by the regular Gunning method, only traces of nitrates being present.

Total carbon was estimated by the method of Parr.[1] The soil fusion obtained by combustion with sodium peroxide and powdered magnesium in the calorimeter bomb was decomposed with sulphuric acid and the liberated carbon dioxide measured in a Parr apparatus by absorption with caustic potash. Carbon as carbonates was estimated in a separate portion of soil by direct decomposition with acid in the apparatus and measured as before.

Hilgard[2] has pointed out that oxidation in the arid soils effects the humus carbon and hydrogen particularly and that the nitrogen content of the arid humus may thus increase. Hilgard and Jaffa[3] show that the arid humus contains on the average three and a half times as much nitrogen as does the humid.

It is evident from the above results in Table II that while the percentage of nitrogen in these soils has remained practically constant under continual cropping for, in extreme cases, 25 years, there has been a marked decrease in the carbon or organic content.

With an average yield of 20 bushels of wheat approximately 30–40 pounds of nitrogen would be removed by grain alone from each acre annually. As little or no waste by leaching occurs on account of limited rainfall, it would seem that fixation of nitrogen might take place to the extent of maintaining practically a nitrogen equilibrium. The soils furthermore are warm, porous, and mildly alkaline with calcium carbonate, and when sufficient moisture is present would furnish a good medium for bacterial growth.

Credit is due Mr. B. Pilkington for the analytical determinations involved in this work.

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SPANISH PAPRIKA.

By ARTHUR LOWENSTEIN AND W. P. DUNNE.

A paper on this subject was presented by one of us at the New Haven meeting of the American Chemical Society in 1908, an abstract of which appeared in Science.[4] Coincident with the presentation of this paper was one on the "Composition of Known Samples of Paprika" by Doolittle and Ogden.[5] That this subject appears to be of considerable general interest in this country is evidenced by the fact that since this time coöperative work on this material was conducted by the referee on spices of the Association of Official Agricultural Chemists.[6] The chief point at issue in these papers has been the determination of the iodine number of the non-volatile ether extract, and its value in the detection of adulteration in paprika.

[1] Ladd, Bulletin 24, North Dakota Experiment Station. Snyder, Bulletin 70, Minnesota Experiment Station.

[1] J. A. C. S., 26, 294–1640. Bulletin 107, revised, Bureau of Chemistry, page 234.
[2] Soils, page 124.
[3] Report of Cal. Experiment Station, 1892–4.
[4] August 7, 1908.
[5] J. A. C. S., 30, 1481 (1908).
[6] Proc. A. O. A. C., Bulletin 122, 35 and 213.

The title of this paper should properly be "Spanish Pimiento" as this is the name by which it is known in its native country. The term "Paprika" is of Hungarian origin, and according to a ruling made by the U. S. Government authorities within the last year, the Hungarian product alone may be called "Paprika."

In order to obtain reliable data, for the purchase of this material, samples of known origin were obtained direct from the growers in Spain—chiefly those residing in the province of Murcia. The whole pods only were used in this investigation. The seeds were carefully separated from the shells and analyses made separately on each. The percentage of seeds was determined and found to be on an average about 40 per cent. by weight.

The forms of adulteration of Spanish pimiento encountered in the ground material are added oil (usually olive oil) seeds, stems, sweepings and artificial color. The latter can be readily detected and is not as common as formerly. In regard to the presence of seeds at the present time most all of the ground product contains seeds in varying proportions. The growers maintain that seeds are not an adulteration when present in quantities normal to the whole pods. The higher grades contain a smaller proportion of seeds than this quantity, while low grades are apt to contain a much larger proportion, in which case they unquestionably constitute adulteration.

Paprika is graded commercially by its degree of color. Seeds impart no color and consequently lower the shade. Olive oil on the other hand intensifies the color and thus makes lower grades appear better than they really are. It is claimed that the oil is added to preserve the color of the paprika, and there seems to be some merit in this claim as the coloring matter of the various species of *Capsicum annuum*, L. is very unstable to light and heat. However, it has been our experience that Spanish pimiento packed in hermetically sealed tins maintains its original color for a long period, whereas this same material shipped in burlap bags and stored loses much of its color and depreciates in value. It seems only proper that if oil is added it should be plainly stated on the label. While the addition of olive oil apparently intensifies the color of the ground material, the actual color of the paprika is in reality lowered and this can be revealed by making a tintometer reading of the alcoholic extract, a test which we find very valuable in selecting paprika prior to purchase.

The proportion of seeds may be roughly approximated from a study of the non-volatile ether extract, the ash and the iodine number of this extract. It will be noted from the appended table that the presence

TABLE I.—SPANISH PIMIENTO OF KNOWN PURITY.

Lab. No.	Description.	Loss at 105° C after deducting volatile. Per cent.	Total ash. Per cent.	Ash soluble in water. Per cent.	Ash insoluble in 10 per cent. HCl. Per cent.	Total ether extract. Per cent.	Volatile ether extract. Per cent.	Non-vol. ether extract. Per cent.	Alcoholic extract. cent.	Crude fiber. Per cent.	Butyrorefract. reading 31° C.	Tintometer reading Lovibond scale. Yel.	Red.	Method of extraction.	Iodine number. No. I.
45187a	Shells	5.87	8.77	7.57	0.24	6.39	0.67	5.72	13.50	19.60	off the scale	20	40	Doolittle-Ogden modified	136.5
45187b	Seeds	5.48	3.22	1.22	0.14	22.03	0.52	21.51	22.70	27.99	65°	no color	no color	"	113.9
45188a	Shells	12.32	9.25	8.65	0.31	6.37	0.75	5.62	23.34	18.70	off the scale	20	43	"	130.2
45188b	Seeds	5.82	3.84	1.83	0.2	20.97	0.6	20.37	24.70	27.39	64°		no color	"	110.1
45189a	Shells	.....	10.00	9.50	0.1	6.80	0.25	6.55	18.05	24.10	......	20	23.5	"	114.8
45190a	Shells	15.90	9.50	8.78	0.1	5.82	0.7	5.12	24.60	20.05	......	.....	.....	Petroleum spirit	134.6
45190b	Seeds	6.63	3.40	....	0.04	20.36	0.27	20.09	21.30	29.45	64°	no color		.....	.....
45190c	Stems	3.66	.....	....	....	3.24	0.3	2.94	.....	31.26	......	.....	.....	.....	.....

TABLE II.—INFLUENCE OF ADDING KNOWN AMOUNTS OF OLIVE OIL.

Lab. No.	Description.	Loss at 105° C after deducting volatile. Per cent.	Total ash. Per cent.	Ash soluble in water. Per cent.	Ash insoluble in 10 per cent. HCl. Per cent.	Total ether extract. Per cent.	Volatile ether extract. Per cent.	Non-vol. ether extract. Per cent.	Alcoholic extract. cent.	Crude fiber. Per cent.	Butyrorefract. reading 31° C.	Tintometer Yel.	Red.	Method of extraction.	No. I.
45191a	Commercial ground Pimiento	17.95	7.35	....	0.18	13.18	0.75	12.47	26.98	18.60	72°	20	16.5	Petroleum spirit	119.4
45191b	5% olive oil added	.....	..	....	....	.....	....	16.80	20.00	17.87 (calc.)	65°	20	12.5	"	116.9
45191c	10% oilve oil added	.....	.....	....	....	.....	....	21.18	18.75	16.74 (calc.)	64°	20	11.2	"	114.6
45191d	Olive oil used	.....	....	....	....	.....	....	......	.....	.....	59°	....	.....	.....	83.5

TABLE III.—ANALYSES OF SOME COMMERCIAL GROUND PIMIENTO.

Lab. No.	Description.	Loss at 105° C after deducting volatile. Per cent.	Total ash. Per cent.	Ash soluble in water. Per cent.	Ash insoluble in 10 per cent. HCl. Per cent.	Total ether extract. Per cent.	Volatile ether extract. Per cent.	Non-vol. ether extract. Per cent.	Alcoholic extract. cent.	Crude fiber. Per cent.	Butyrorefract. reading 31° C.	Tintometer Yel.	Red.	Method of extraction.	No. I.
45192	Non-vol. extract dried to constant wt. in air oven	8.40	7.0	5.65	0.19	19.31	1.35	17.96	27.04	17.0	67.5°	.....	.....	Petroleum spirit	111.7
45193		14.10	7.32	6.59	0.25	13.76	1.00	12.76	20.14	18.74	69°			"	113.3
45194		15.87	7.46	....	....	12.87	0.55	12.32	28.35	19.02	......	20	20	....	.....
45195	" "	10.72	7.50	....	0.4	16.35	0.4	16.45	26.10	.....		20	17	Doolittle-Ogden modified	119.5

of seeds increases the ether extract, and lowers the ash, iodine number, refractive index and color.

The presence of stems increases the ash and fiber and reduces the ether extract, and lowers the iodine number and color.

Olive oil increases the ether extract, lowers the iodine number, ash, refractive index, fiber and color.

Iodine Number of Non-Volatile Ether Extract— Methods.—In our opinion the point of prime importance in this connection is the proper determination of the non-volatile ether extract. We have shown in another paper[1] the influence of the method of heating on the composition of the non-volatile ether extract of spices. It is sufficient to say at this point that "heating to constant weight at 110° C." as stated in the official method of the A. O. A. C., gives non-volatile ether extracts of varying composition and consequently of varying iodine absorption capacity. We think that this point more than any other will account for the widely divergent results obtained by the different analysts, in the coöperative work of the A. O. A. C. reported by Winton. We also observe that this point has given rise to the opinion that satisfactory duplicate iodine numbers cannot be obtained on two portions of a sample of paprika, extracted by the A. O. A. C. method. It is our experience that satisfactory duplicates can be obtained if the extraction is carefully conducted and the extract brought to constant weight in a vacuum oven. In the case of paprika, heating to constant weight in an ordinary air oven at 110° C. not only frequently completely decolorizes the extract but oxidizes it so much that a considerable portion of it will no longer dissolve in chloroform when the iodine number is determined.

In our early work on paprika, we extracted a ten-gram portion of the ground sample with redistilled petroleum spirit (88°–90° Bé.) repeatedly in a cylinder and then transferred to a filter and continued washing until the extract was colorless. Then the petroleum spirit was distilled off, volatile extractives removed, and a sufficient quantity of the non-volatile extract was thus obtained to make duplicate iodine determinations and a refractive index reading. Later, after the publication of the paper by Doolittle and Ogden, and the modification of their method of extraction by W. Denis, we adopted this modified method as more convenient. It might be noted that the petroleum ether extraction method gives results which check within 1 per cent. with this latter method. Later we found that we could obtain very satisfactory results by using the non-volatile ether extract—obtained by the regular A. O. A. C. method (using a 5-gram sample)—when the precautions above mentioned were employed. By dissolving this extract in chloroform and making to a definite

[1] THIS JOURNAL, **2**, 47.

volume, satisfactory duplicate iodine values can be determined on aliquots. This latter method we find most convenient as it saves a separate extraction. The Hanus method was employed in making all iodine numbers.

In connection with the iodine value it might be interesting to call attention to the iodine number of the shells of No. 45189*a*, which was an old sample of very inferior quality at the time the determination was made, although it was pure Spanish pimiento. It will be noted that it has the characteristic high ash—ether extract and fiber are normal—but the extract has oxidized with consequent lowering of the iodine number. Its inferior quality will be revealed from a comparison of its color reading with that from fresh, high-grade shells, No. 45187*a*.

Refractive Index.—It is interesting to note that while we have been able to read the degree of refraction of the non-volatile ether extract of all ground commercial paprika on the butyro-refractometer, that when we came to make the reading on the shells only, we found that the reading was too high for this scale and had to be read on the Abbé instrument. Therefore, while the refractive index, or better the butyro-refractometer reading in itself is of but little value in detecting adulteration with oil, it will be seen from the appended table that it is of value in conjunction with the other tests.

Tintometer Reading.—We have found either the Lovibond or Dubosc instrument convenient for measuring the color of the extract of paprika. We employ the alcoholic extract as obtained by the official A. O. A. C. method, taking 10 cc. of this extract and diluting to 100 cc. with 95 per cent. alcohol.

TABLE IV.—COMPARISON OF THE INFLUENCE ON THE IODINE NUMBER OF DIFFERENT METHODS OF EXTRACTION, TIME OF EXTRACTION, AND METHOD OF HEATING NON-VOLATILE ETHER EXTRACT.

Lab. No.	Time of extraction, hours.	Method of extraction.	Method of heating.	Iodine No.
45187*a*	....	Petroleum ether	Vacuum oven 10 cm. Hg.	*a* { 137.2 137.2
45187*a*	....	Doolittle-Ogden modified	" "	*a* 138.0 *b* 138.3
45187*a*	24	A. O. A. C.	" "	*a* 138.0 *b* 137.7
45187*a*	100	Continuous extraction with anhydrous alcohol-free ether	" "	123.6
45187*a*	100	Continuous extraction with petroleum spirit 88-90° Bé.	" "	120.9
45188*a*	24	A. O. A. C.	" "	130.2
45188*a*	24	A. O. A. C.	Official A. O. A. C. to constant wgt. in air oven at 110° C.	123.0

The color readings given in the table were made on this basis. It will be noted that olive oil and seeds lower the color reading very markedly, and we find this determination of great help in selecting paprika,

and in judging whether goods received are equal in quality to samples originally submitted.

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THE PREPARATION OF CURD FROM BUTTERMILK.[1]

By J. L. SAMMIS.

Received January 24, 1910.

The filtration of colloidal precipitates is often a difficult operation. The difficulty of collecting curd from heated buttermilk by filtration has heretofore prevented the practical creamery man from preparing from buttermilk anything resembling the well-known cottage cheese made from skim milk. It has long been known[2] that curds from skim milk or from mixtures of skim milk with not too much buttermilk can be easily collected by filtration on cheese-cloth, but no reliable method has been accurately described[3] for handling buttermilk alone, desirable as such a method is.[4]

Previous attempts at this University to obtain a filterable curd from pure buttermilk and to produce' from it a product having any desired moisture content and consistency from creamy to dry and pasty had progressed only so far as to show that a creamy product "Buttermilk Cream,"[5] could sometimes be obtained, but not far enough to develop a reliable method for its manufacture or for controlling its moisture content. Neither the yield nor the moisture content of the product is stated.

The present study was begun in September, 1909, at the suggestion of Professor Farrington, of the Dairy Department. The following described method[6] was devised by the writer and it has been used daily for about four months at the University Creamery with uniform success.

It includes special heat treatment of the buttermilk for two purposes: first, to obtain the curd in a form which can be readily filtered on cheese-cloth, and, second, to regulate the moisture content of the curd so as to give it any desired consistency. Buttermilk of 0.5 to 0.6 per cent. acidity, made by churning either raw or pasteurized cream, is placed in any suitable container provided with means for heating. The buttermilk is stirred and heated to 25°–26° C., is then covered and left undisturbed at this temperature for $1^1/_2$ to 2 hours.

The purpose of this operation is to bring the butter milk quickly to a temperature at which it will slowly coagulate and to let it become quiet before coagulation occurs. As the granules of curd separate from the quiet liquid, they cohere forming a fragile floating mass, which shrinks and becomes tougher, as the whey is expelled, during the next two hours. If stirred too long or heated too high at first, the buttermilk will curdle while in motion, and the grains of curd will not cohere or float but will settle in the form of a fine flocculent precipitate which can not be filtered on cheese-cloth.

Then shrunken cohering mass is stirred gently at the end of two hours, while heating to 38, 55, or 60° C., and then left quiet to settle. This leaves the curd in the form of coarse flocks which will easily filter on cheese-cloth at any time thereafter. The higher the temperature of this second heating, the drier the final product will be after draining. The second heating causes more complete dehydration of the curd, and the whey thus formed is removed in the final draining process.

If the second heating be for 15 minutes at 38° C. the final product is a thick creamy fluid with a glazed surface containing about 82 per cent. of moisture, shown at the left of the figure. It has been called "buttermilk cream," as stated above.
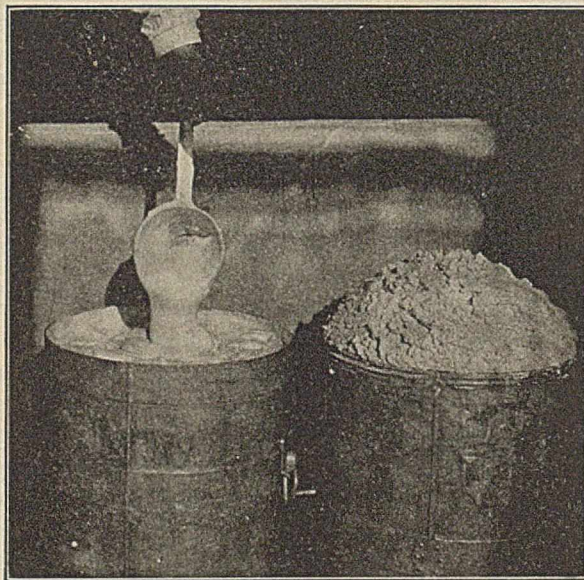
"Buttermilk cream" and "Buttermilk cheese."

If the second heating be continued for 1 hour or more at 55° C. before draining on cheese-cloth, the final product resembles a good quality of cottage cheese in appearance, as shown at the right of the figure, and the moisture content is about 77 per cent. If the second heating be continued at 60° C. for 3 hours, the appearance of the product after draining is practically the same as when 55° is used, but the moisture content is reduced to about 72 per cent. and the yield is somewhat reduced. In any case, after the second heating most of the clear whey can

[1] Published by permission of the Director of the Wisconsin Agricultural Experiment Station.

[2] See Knoch, *Die Magermilch Verwertung*, Liepsic, 1903, p. 90–91.

[3] Anonymous, *Hoard's Dairyman*, **28**, 86 (1897).

[4] See R. K. Duncan, THIS JOURNAL, **1**, No. 8, 602.

[5] "Buttermilk Cream, a New Product," *Hoard's Dairyman*, **40**, 20, March 19, 1909.

[6] From the advance sheets of this paper, E. H. Farrington has published part of the method in *Hoard's Dairyman* for Dec. 31, 1909, p. 1424, from which it was copied in other dairy papers.

be decanted from the curd, which nearly all sinks after being stirred and heated the second time. The remaining curd and whey is poured at once into a cheese-cloth bag, returning the first portions which run through, till the drippings are water-clear. The curd in the bag is stirred occasionally since the layer next the cloth drains fastest, and delays the draining of the interior of the mass. It is left over night in the bag. In the morning it is seasoned with 1 per cent. of salt and, if desired, with 0.1 per cent. of white pepper or in other ways. The dry product, which may be called *buttermilk cheese,* is eaten like cottage cheese. It can also be used as a diluent for other materials, as an article of food; or with preservatives, it can probably be used as a basis for various preparations.

The curd from buttermilk differs in one respect from skim milk curd or cottage cheese. Buttermilk curd is always exceedingly fine-grained in its ultimate texture, so that it can be quickly rubbed to a paste with water between the fingers.

Further study is in progress. It is hoped that the method described for precipitating and filtering buttermilk curd may be found useful by those who have other colloidal material to filter.

UNIVERSITY OF WISCONSIN, AGRIC. EXPT. STATION,
January, 1910.

THE QUANTITATIVE DETERMINATION OF CANE SUGAR BY THE USE OF INVERTASE.

By C. S. HUDSON.

Introduction.—The general method now in use for estimating cane sugar is the procedure of Clerget, in which the sugar is hydrolyzed by acid and the resulting invert sugar estimated either by its reduction of Fehling's solution or by the change in optical rotation which results from the hydrolysis. It has long been known that the presence of other hydrolyzable substances with the cane sugar interferes in the determination because the hydrolysis by acid is then not confined to the cane sugar. This method would be greatly improved if some substitute for the acid were known which would hydrolyze the cane sugar without attacking other acid-hydrolyzable substances; it is the purpose of this article to show that the enzyme invertase in most cases fulfils this requirement and that its use to supplement or even replace acids in the quantitative estimation of cane sugar is of great value in analytical work. The use of the enzyme in studying the sugars which occur so widely distributed among plants is particularly recommended.

The Preparation of Invertase.—To prepare a stock solution of invertase, break up five pounds of pressed yeast, which may be either baker's or brewer's yeast,

add 30 cc. of chloroform to it in a closed flask and allow it to stand at room temperature (20°) over night. By the morning the solid mass will have become fluid and it should then be filtered through filter paper, allowing several hours for draining. To the filtrate add neutral lead acetate until no further precipitate forms and again filter. Precipitate the excess of lead from the filtrate with potassium oxalate and filter. To this filtrate add 25 cc. of toluene and dialyze the mixture in a pig's bladder for two or three days against running tap water. The dialyzed solution is colorless, perfectly clear after filtration, neutral to litmus, has a solid content about one-half of one per cent., an ash content of a few hundredths of one per cent., will keep indefinitely in an ice box if a little toluene is kept on its surface to prevent the growth of microorganisms, and is exceedingly active in inverting cane sugar. The invertase solution does not reduce Fehling's solution.

The Keeping Qualities of the Invertase Solution.— The stock solution of invertase prepared as described keeps perfectly. A quantity of it has been tested during a period of fifteen months and no change in its general appearance or inverting activity noted. Three other preparations of less age have also shown no appreciable change. All of these solutions have been kept in an ice box and toluene was added in excess of saturation in each case. Another preparation has been observed by H. S. Paine, of this bureau, who found that the preparation did not change in inverting activity during a period of three months. This sample was kept at room temperature (20° to 30°) during the day and in an ice box at night, thus duplicating the conditions which occur where many analyses are carried out each day with the use of the invertase solution.

The Rotatory Power of the Invertase.—The stock solutions of invertase have a slight rotatory power and in a 400 mm. tube they give a dextro-rotation of 1.0° V. In the method to be described further on, the inversion of 95 cc. of sugar solution is accomplished by 5 cc. of the invertase solution, and therefore the correction to be applied for the rotation of the invertase solution is 0.05° when the reading is made in a 400 mm. tube or 0.025° when a 200 mm. tube is used. For all ordinary work these corrections are negligible and they can always be accurately determined when the accuracy requires them.

The Numerical Formula for the Determination of Cane Sugar by Invertase.—If S denote the polarization of pure cane sugar before inversion and I that after inversion, the numerical factor of inversion, which can be found by experiment, is I/S. This factor varies greatly with the temperature, due to the influence of the latter on the rotation of fructose. H. S. Paine and the author have measured this factor at 20° when invertase is used as the inverting agent

on sugar solutions of 7 per cent. strength and have found its value to be 0.317; similar experiments have given the factor 0.324 for the inversion by hydrochloric acid when the solution is kept at room temperature over night and read in the acid condition. For the same acid solution after neutralization the factor 0.317 was found, showing that it is the acidity which causes the difference between the factors for invertase and unneutralized acid solution. To check this point, the same quantity of hydrochloric acid was added to the solution which had been inverted by invertase and had given the factor 0.317; it was found that its rotation changed immediately to a greater negative value which corresponded to a factor 0.324, thus verifying the above conclusion in a different way. The inversions by invertase which gave the factor 0.317 were made in a solution which had been acidified with acetic acid, as invertase acts only in an acid solution; the two acid strengths of 0.01 and 1.0 normal were used and the factor was found to be the same for both, 0.317. Other experiments have been made in which the solution was weakly acidified with other acids to produce an acid medium in which the invertase would act and for the inversion by invertase in these weak acid solutions (five ten-thousandths normal) the following values of the factor I/S at 20° were found: Nitric 0.315, sulphuric 0.315, oxalic 0.314, citric 0.314, tartaric 0.320, phosphoric 0.315, arsenic 0.315, hydrochloric 0.315, hydriodic 0.315. The average of these values is 0.315, which is quite close to that given for acetic acid and for the neutralized hydrochloric inversions, 0.317; a difference of four-tenths of a degree in the temperature at which the readings of the inverted solutions were made would account for this variation. Chas. A. Browne[1] has found the value 0.325 for the inversion by hydrochloric acid when the acid solution is read, and the value 0.317 after neutralization, numbers which are practically identical with those given here. Weber and McPherson[2] find at 20° the factor 0.317 for inversions by hydrochloric acid, correction being made for the influence of the acid. L. M. Tolman[3] finds the factor 0.318 for acid inversions, the acid being corrected for, and states that "if an inverting agent could be obtained which had no effect on the invert sugar the question of concentration [influencing the factor] would practically be eliminated. But the elimination of this error would necessitate the determination of a new factor which would in all probability be 141.79 (*i. e.*, 0.3179) or perhaps a little less." Invertase fulfils this requirement and its factor is 141.7. It is thus certain that the factor for the inversion of cane sugar by invertase is considerably less than the factor for the usual hydrochloric acid inversion, that its value at 20° approxi-

[1] U. S. Dept. Agric., Bureau of Chem., Bulletin **110**, p. 44.
[2] *J. Amer. Chem. Soc.*, **17**, 324 (1895).
[3] *Ibid.*, **24**, 523 (1902).

mates 0.317 closely and that the difference between the factors for invertase and hydrochloric acid is due to the effect of the acid on the rotatory power of invert sugar. These experiments also prove that the hydrolysis of cane sugar by invertase is a complete one, for just as much invert sugar was formed by the invertase inversion as by that due to the strong hydrochloric acid. The usual formula for the per cent. of cane sugar may therefore be used with the new factor 141.7 when the inversion is carried out by the use of invertase.

$$\text{Per cent.} = \frac{S - I}{141.7 - \dfrac{T}{2}} \times 100.$$

The Influence of Acids and Alkalis on the Activity of Invertase.—Invertase acts only in an acid medium, and the slightest alkaline reaction stops its action instantly. Weak alkalinity does not destroy it, however, for if the acidity be restored the invertase resumes its original activity. But it is quite important that the medium be only weakly acid, for invertase is rendered inactive and even destroyed by acidities above hundredth-normal hydrochloric. As this acidity is itself rather weak, it seems best to avoid the use of strong mineral acids entirely in preparing the sugar solution for the action of invertase, and to use in their place a weak organic acid such as acetic, for invertase is quite active in acetic acid of 5 per cent. strength.

In the accompanying figure there is given the measured activity of invertase in solutions of hydrochloric acid and of acetic acid. These activities were found by mixing at 30°, 100 cc. of 0.2 normal (approx. 7 per cent.) cane sugar solution with 5 cc. of the appropriate strength of acid and 5 cc. of a stock invertase solution, and measuring the velocity-coefficient (k) of the resulting inversion by the usual formula $k = \dfrac{1}{t} \log \dfrac{R_0 - R_\infty}{R - R_\infty}$ where R_0 is the initial rotation of the solution, R_∞ the value after complete inversion, R the rotation at the time t (expressed in hours), and log the common logarithm. Care was taken to make the samples alkaline before each reading of R in order to stop the action of the invertase
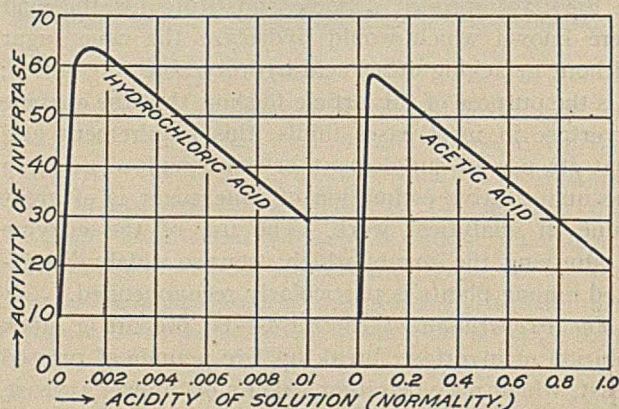

Fig. 1.—Influence of strong and weak acids on the activity of invertase.

and complete the mutarotation of the invert sugar. The figure shows that invertase is active in only a very small range of acidity when the latter is due to a strong acid like hydrochloric, which is almost completely dissociated in solution, but that if a weak acid like acetic is used, its dissociation is so slight that quite strong solutions can be used without bringing the actual acidity (*i. e.*, hydrogen—ion concentration) up to a strength which is inimical to the action of invertase. As it would be difficult in practical work to adjust the acidity of the solutions very accurately, it is recommended that the solutions be all made acid to litmus with acetic acid, as even a considerable excess of acetic acid will do no harm.

Directions for Making the Analysis.—Dissolve 26 grams of the substance to be analyzed for cane sugar in water, clarify with the usual substances (neutral or basic lead acetate or alumina cream or kaolin) and make up to 100 cc. volume at 20°. Filter and read the polarization of the filtrate, S, for a 200 mm. tube. Remove the excess of lead from the filtrate, if lead has been used as clarifying agent, with sodium carbonate or potassium oxalate and filter. To 50 cc. of the filtrate add acetic acid by drops until the reaction is acid to litmus, add 5 cc. of the stock invertase solution and make up the volume to 100 cc. Add a few drops of toluene to the solution to prevent the growth of microorganisms, shaking so as to saturate and allow to stand at any temperature between 20° and 40° over night. Under usual conditions about six hours time is required to accomplish complete hydrolysis. In the morning bring the temperature to 20° and read the rotation of the solution, I, for a 400 mm. tube. The percentage of cane sugar present is then calculated by the formula given on page 144. The percentage can also be found by the cuprous oxide reduction before and after inversion by the invertase.

The Influence of Mutarotation on the Invert Polarizations.—The temperature does not influence the rotation of glucose but changes that of fructose greatly and the change of rotation lags behind the change of temperature. This lag is due to the mutarotation of fructose and practically disappears in inversions by hydrochloric acid for the reason that the acid is a strong catalyst of the mutarotation. In solutions inverted by invertase the lag for fructose which follows a change in the temperature of the solution lasts about ten minutes at 20°, and care should therefore be used in reading the invertase inversions to see that the rotation remains constant for a few minutes. It is the writer's practice, after the invert reading is obtained, to add a pinch of cane sugar to the solution and observe its rotation for a few minutes; if it gradually changes, showing that the cane sugar added is being inverted, one is certain that the invertase is active and has accomplished a complete inversion over night.

Tests of the Invertase Method. 1. Test on Pure

Cane Sugar.—Direct polarization (S) 100°, invert polarization, with hydrochloric acid, —33.2, consequently the per cent. sugar by Herzfeld's formula,

$$\frac{S - I}{142.7 - \dfrac{T}{2}} \times 100$$

is 100.4. The inversion by invertase gave I = —31.9, and the percentage by the above formula with the constant 141.7 for invertase, as described before, is 100.1. The polarizations in all the tests were made at 20°.

2. Test on Louisiana Cane Syrup.—This syrup had been treated with SO_2 in the process of manufacture. Direct polarization was 55.6, invert polarization by hydrochloric acid —14.9, by invertase —13.8, giving the percentage of cane sugar by hydrochloric inversion as 53.1, and by invertase inversion 52.7 per cent.

3. Test on Bagasse.—S. F. Sherwood of this bureau recently obtained the following percentages of cane sugar in samples of Cuban bagasse, using the hydrochloric acid and also the invertase methods of inversion and determining the invert sugar for both methods by the change of polarization and also by the increased reduction of Fehling's solution.

ANALYSES OF BAGASSE.

Per cent. cane sugar.

Sample No.	Hydrochloric acid inversion.		Invertase inversion.	
	Polarization.	Reduction.	Polarization.	Reduction.
1	23.6	23.9	23.6	23.3
2	16.4	16.1	15.8	16.0
3	12.4	13.4	13.4	13.3
4	7.2	8.0	8.7	7.7

These three substances, pure cane sugar, cane syrup and bagasse, show practically the same cane sugar percentage by the hydrochloric acid and the invertase methods. There will now be given an example of a very different type of analysis.

4. Test on the Sotol Plant (Dasylirion Texanum).[1]— The soluble matter from 26 grams of the whole plant was dissolved to 100 cc. and the polarization S found to be —9.2. Inversion by hydrochloric acid gave I = —16.5, by invertase I = —9.7. The percentage of cane sugar determined by acid inversion was 5.5, by invertase inversion, 0.4. The analysis shows such a striking disagreement between the two methods that a further determination of the reducing sugar before and after the hydrolyses was made with Fehling's solution. The true carbohydrate composition of this plant was found by this method to be:

Per cent.

Reducing sugars..................................... 2.8
Cane sugar (by invertase inversion)................. 0.7
Unknown carbohydrate (by acid inversion).......... 13.3

Here then is a substance which contains only a trace of cane sugar but a large quantity of a very easily hydrolyzed carbohydrate; further work on the nature of this unknown carbohydrate is in progress.

[1] This interesting plant occurs in the waste dry regions of New Mexico and Texas; a description of it is given in Prof. W. L. Bray's "The Vegetation of the Sotol Country in Texas," Univ. of Texas Bulletin No. 60.

5. *Test on Commercial Glucose.*—A 26 per cent. solution of "mixing glucose," which is prepared by the acid hydrolysis of starch, gave a rotation of 86.0° V. Inversion by hot hydrochloric acid changed this to 85.2°, by cold acid 85.3°. Five drops of invertase solution were added to a third portion and after standing over night its rotation was still 86.0°. The method of manufacture of this mixing glucose by hot acid hydrolysis makes it certain that no cane sugar can be present in it, and the method of analysis in which invertase is employed shows no cane sugar, but the usual acid methods give a slight decrease of rotation. Weber and McPherson[1] have particularly investigated this error in the acid methods of hydrolysis and devised a correction for it, but the invertase method is certainly preferable.

6. *Commercial Glucose with Added Cane Sugar.*— Commercial glucose is frequently flavored with a small quantity of cane syrup to make it more palatable and is then sold for table use. An analysis for cane sugar of such "cane-flavored" glucose gave the following: Rotation of a 13 per cent. solution of the sample in a 200 mm. tube 75.0 °V., rotation after inversion with hot hydrochloric acid 72.7°, with cold hydrochloric acid 73.8°, inversion by invertase 74.0°. These results show that the acid methods indicate more cane sugar than does the invertase hydrolysis and there is no question but that the invertase determination is more nearly correct.

The Hydrolysis of Raffinose by Invertase.—The peculiar trisaccharide raffinose, which occurs in cotton seed, in wheat, and in the sugar beet, is hydrolyzed by invertase, and the method for estimating cane sugar by inversion with invertase is accordingly not applicable in case raffinose is present. Raffinose is also hydrolyzed by hydrochloric acid, and therefore the acid hydrolysis has here no advantage over that by invertase. An experiment was performed in which a 10 per cent. solution of very pure recrystallized raffinose, of specific rotation 123° (circular), was hydrolyzed with the stock invertase solution, and the rotation was found to change to 67.6° at 20°, where it remained constant.

The Action of Invertase on Other Acid-hydrolyzable Substances.—The stock solution of invertase has been found to be entirely incapable of hydrolyzing lactose, maltose, starch, dextrin, cellulose, pentosans, amygdalin or salicin. So far no action on any substances except cane sugar and raffinose has been detected, but it appears probable that the tetrasaccharide stachyose, which occurs in some plants, would be split into fructose and a trisaccharide.

Summary.—The enzyme invertase accomplishes a complete inversion of cane sugar and is without action on starch, dextrin, maltose, lactose, pentosans and natural glucosides. A procedure for preparing

[1] *J. Amer. Chem. Soc.*, **17**, 312–20 (1895).

stock solutions of invertase from pressed yeast has been worked out and the solutions found to be permanent in inverting power, the measurements having been made now for a period of fifteen months. The invertase inverts only in an acid medium and the best acid to use is found to be acetic. Full directions for the method of analysis are given and the possible errors due to the slight rotatory power of the invertase solution and the mutarotation of the invert sugar are investigated and means taken to avoid them. The formula for the percentage of cane sugar is found to be $\dfrac{(S-I)}{141.7 - \dfrac{T}{2}} \times 100$, where S and I are

the direct and invert readings of the solution, T the temperature and 141.7 the inversion constant, which differs from that for the inversion by hydrochloric acid, *i. e.*, 142.7. This difference is shown to be due to the influence of the acidity on the rotation of invert sugar, as the hydrochloric acid solutions give after neutralization the invertase constant 141.7. It is found that raffinose is hydrolyzed by invertase and that its specific rotation is thereby reduced from 123° to 67.6°; raffinose accordingly interferes with the estimation of cane sugar by the use of invertase, just as it also interferes when the inversion is carried out with hydrochloric acid. Analyses of cane sugar in pure cane sugar, Louisiana molasses, and bagasse by the invertase method of hydrolysis give percentages which agree with those obtained by the hydrochloric acid hydrolysis. The Sotol plant (*Dasylirion texanum*) is shown to contain only a trace of cane sugar, but 13 per cent. of an unknown carbohydrate which is being further investigated.

BACTERIOLOGICAL METHODS FOR DETERMINING THE AVAILABLE NITROGEN IN FERTILIZERS.

By Jacob G. Lipman.

Attempts to determine the relative availability of nitrogenous fertilizers by measuring the amounts of nitrate formed from them under definite experimental conditions have been made by different investigators. In consequence of these investigations it is now well known that nitrate formation from organic substances bears a direct relation to the proportion of nitrogen contained in them, and in general, to the ease with which they will undergo decomposition. A much larger amount of work on the same subject has been done from the crop standpoint; that is, the relative availability of nitrogenous substances has been determined not by measuring the quantity of nitrate formed from them under definite experimental conditions, but by measuring the amounts of nitrogen furnished by each to the crop, as shown by analyses of the latter.

In view of the fact that nitrate formation from organic substances is preceded by ammonia formation, it is proper to assume that nitrification is more or less directly affected by ammonification. Substances that will ammonify readily should also nitrify readily, and *vice versa*. Nevertheless, it is theoretically possible that the relative ammonification of a group of nitrogenous substances need not correspond exactly to their relative nitrification. It, therefore, appeared advisable to make a series of tests on the relative ammonification and nitrification of a number of such substances. It was hoped, also, that among other things these tests would give some indication as to the practicability of measuring the relative availability of nitrogenous materials by their ammonification, as well as their nitrification. It is to be added here, that the experiments recorded below are hardly more than preliminary, and will be continued in the future.

The nitrogenous materials in these tests were mixed in each case with 100 grams of soil and 1 gram of calcium carbonate. The soils themselves were kept at their optimum moisture content, *viz.*, 18 per cent. In the ammonification tests additional amounts of water were supplied so as to provide in each case organic matter containing 75 per cent. of water. For instance, when 5 grams of dried blood were employed there were added 15 cc. of water aside from that added to the soil itself; when approximately 30 grams of dried manure were used the amount of water added was about 90 cc., etc. Thanks to this method it was possible to maintain uniform moisture conditions in the several soil portions. Both in the nitrification and ammonification tests the materials were added so as to supply equal amounts of nitrogen. The nitrogen content of the different substances was as follows:

	Per cent.
Ammonium sulphate	20.30
Calcium cyanamid	17.64
Dried blood	10.76
Concentrated tankage	12.63
Ground fish	7.90
Cottonseed meal	6.40
Bone meal	1.35
Solid and liquid manure, fresh	3.07
Solid manure, fresh	2.20
Solid and liquid manure, leached	1.72
Horse manure	1.31

Ammonia determinations were made at the end of six days, and nitrate determinations at the end of four weeks in the corresponding series. The amounts of material used and those of ammonia and nitrate nitrogen found were as follows:

As is shown in the table below considerable quantities of nitrogen were split off either in the form of ammonia or of nitrate. Small quantities of nitrites were also found in the nitrification series, yet they were quite uniform, and so insignificant in amount as to make it unnecessary to record them here. The proportion of the amounts of nitrogen found as am-

Soil portion No.	Additions.	Ammonia N found. Mgs.	Aver. Mgs.
1	5 grams dried blood	91.51	......
2	5 grams dried blood	88.67	90.09
3	4.26 grams concentrated tankage	312.95	......
4	4.26 grams concentrated tankage	297.99	305.47
5	6.81 grams ground fish	247.43	......
6	6.81 grams ground fish	260.03	253.73
7	8.41 grams cotton-seed meal	32.13	......
8	8.41 grams cotton-seed meal	21.42	26.77
9	39.85 grams bone-meal	78.43	......
10	39.85 grams bone-meal	102.06	90.24
11	17.52 grams solid and liquid manure, fresh	180.49	......
12	17.52 grams solid and liquid manure, fresh	170.41	175.45
13	24.45 grams solid manure, fresh	28.19	......
14	24.45 grams solid manure, fresh	29.77	28.98
15	31.28 grams solid and liquid manure, leached	62.37	......
16	31.28 grams solid and liquid manure, leached	62.37	62.37
17	29.72 grams horse manure	40.00	......
18	29.72 grams horse manure	36.85	38.42

Soil portion No.	Additions.	Nitrate N found. Mgs.	Aver. Mgs.	Increase over the untreated portions. Mgs.
1	0	5.332	.....	......
2	0	5.332	5.33	......
3	100 mgs. ammon. sulphate	21.332	.....	......
4	100 mgs. ammon. sulphate	20.720	21.26	15.928
5	115 mgs. calcium cyanamid	5.481	.....	......
6	115 mgs. calcium cyanamid	16.00	16.00	10.668
7	189 mgs. dried blood	10.56	.....	......
8	189 mgs. dried blood	10.71	10.63	4.298
9	161 mgs. concentrated tankage	16.21	.....	......
10	161 mgs. concentrated tankage	16.19	16.20	10.868
11	257 mgs. ground fish	10.75	.....	......
12	257 mgs. ground fish	10.53	10.64	5.318
13	317 mgs. cotton seed meal	10.46	.....	......
14	317 mgs. cotton seed meal	12.65	11.55	6.218
15	1504 mgs. bone-meal	7.74	.....	......
16	1504 mgs. bone-meal	7.92	7.83	2.498
17	661 mgs. solid and liquid manure, fresh	7.80	.....	......
18	661 mgs. solid and liquid manure, fresh	8.00	7.90	2.568
19	923 mgs. solid manure, fresh	7.22	.....	......
20	923 mgs. solid manure, fresh	7.29	7.25	1.118
21	1180 mgs. solid and liquid, leached	6.16	.....	......
22	1180 mgs. solid and liquid, leached	6.32	6.24	0.908
23	1122 mgs. horse maure	7.08	.....	......
24	1122 mgs. horse manure	7.14	7.11	1.778

monia or nitrate, to the amounts applied may serve as an indication of the relative availability of the different materials. These proportions, designated as nitrogen recovered, are shown in the following table:

PER CENT. OF NITROGEN RECOVERED.

	Ammonification series.	Nitrification series.
Ammonium sulphate	...	78.47
Calcium cyanamid	...	52.56
Dried blood	16.74	26.13
Concentrated tankage	56.66	53.54
Ground fish	47.16	26.15*
Cottonseed meal	4.95	30.64
Bone meal	16.65	12.31
Cow manure, solid and liquid fresh	32.60	12.67
Cow manure, solid fresh	5.39	9.46
Cow manure, solid and liquid leached	11.59	4.48
Horse manure	7.14	8.76

In comparing the figures before us we should remember that ammonia was determined at the end of six days and nitrate at the end of four weeks. There is no doubt that two or three days more would have not only increased the amounts of ammonia produced in the ammonification series, but would have modified to some extent the relative recovery. It is also true

[1] Not included in the average.

that another week or two in the nitrification series would have resulted in slight modifications in the relative availability of the different materials as measured by nitrate production. For all that the results are of considerable interest even as they are. We note, for instance, that concentrated tankage, ground fish, solid and liquid manure fresh, dried blood and bone meal in the order given yielded the largest amounts of ammonia; and concentrated tankage, cotton-seed meal, ground fish, dried blood and solid and liquid manure, fresh, in the order given yielded the largest amounts of nitrate. Concentrated tankage heads the list, in each case, while three others are also found in each group. The surmise, therefore, that easily ammonifiable substances are also easily nitrifiable is borne out by the present data. Nevertheless there are some discrepancies that are of considerable moment. It will be observed that next to concentrated tankage, cotton-seed meal proved the most easily nitrifiable of the organic materials. In the ammonification series, on the other hand, cotton-seed meal was found to possess a low rate of availability. In view of other experiments carried out by us, showing the depressing effect of soluble carbohydrates on ammonification, the low rate of availability of cotton-seed meal in the ammonification series is not without interest.

In comparing further the relative availabilities of the same materials in the ammonification and nitrification series it should be remembered that small quantities of material are transformed more thoroughly and more efficiently in the soil than larger quantities. Hence, the relations in the two series are somewhat different in this respect. It is also likely that in different soils the relations found in the two series would be modified. With that much admitted, however, it is instructive to note how rapidly some substances may be broken down by bacteria. We find, for instance, that in the ammonification series 56.65 per cent. of the nitrogen contained in the concentrated tankage was changed to ammonia in six days. Similarly, 47.16 per cent. and 32.60 per cent. in the ground fish and the solid and liquid manure, fresh, respectively were transformed into ammonia. On the contrary, the three other samples of manure proved to have a low rate of availability, both in the ammonification and nitrification series. It should be noted, here however, that the samples of manure employed in the present experiments had been previously dried on the water bath and kept in a dry state for some time. Naturally, the drying reduces the availability of nitrogenous materials, hence the returns, as noted above, are smaller than they would have been had the manure in question been used in a fresh condition. It may be of interest to cite here the results secured with similar samples of manure in our cylinder experiments extending over a period of ten years. It was found in these experiments[1] that solid manure, fresh; solid and liquid, fresh; and solid and liquid, leached gave recoveries of 22.31 per cent., 32.91 per cent. and 26.75 per cent., respectively, in a ten-year period, in which two rotations of corn, oats, wheat and grass had been completed. The manure was applied annually and the figures given above represent, therefore, the cumulative effect of the manure residue. In single seasons much lower returns have been secured in a number of instances. Thus the corresponding recoveries in the corn crop of 1908 were[2] 11.20 per cent., 17.55 per cent. and 11.50 per cent., respectively. On the whole, it is not unlikely that the methods outlined above will prove very convenient not only for measuring the relative availability of nitrogenous fertilizers, but also for determining the availability of the nitrogen in humus derived from different soils.

ACCURACY IN TAKING AND PREPARING MIXED FERTILIZER SAMPLES.

By F. B. PORTER.

The importance of accuracy in taking and preparing samples of fertilizer material is often well understood by those having charge of such work. It does not, however, always follow that the sample analyzed in the laboratory is a correct one.

The following tables, I and II, give an idea of the lack of agreement in analyses of mixed fertilizers.

Table I gives differences in analyses on samples put up in pint jars by this laboratory and sent to the laboratory "A" after taking out a four-ounce bottle sample without previous grinding.

Table II gives differences in analyses on the same shipment of goods, sampled and analyzed before shipment at this plant and at destination by the State to which it was sent. All of the above materials passed a 3-mesh screen before sampling. Samples in Table II were taken at this plant with a three-foot trier from at least 10 per cent. of the bags giving a sample weighing one pound, or over. This was quartered down to about 85 grams and then ground to pass a 20-mesh screen. Most State inspectors take their samples in the same general way and it is doubtful if any reduce the size of the particles in the one-pound samples before quartering as the work here reported indicates should be done. The above tables show greater discrepancy than can be accounted for by laboratory errors and it will be noted by comparing the two, that the differences increase where the sampling is by different parties, as in the case of Table II.

A glance at the various official methods for sampling will help very little in reducing these differences, in fact, none of them give complete and definite directions for sample preparation. The official directions of the

[1] Bull. **221**, N. J. Exper. Station, p. 23.
[2] Voorhees and Lipman, "Experiments on the Utilization of Nitrogen in Fertilizer Materials," THIS JOURNAL, **1**, 397 (1909).

A. O. A. C. simply require the fertilizer to pass a sieve with 1 mm. diameter perforation.[1] The official directions of the International Congress of Applied Chemistry only specify the number of packages or buckets to be sampled, require crushing to size of a hazel-nut and require the sample sent to laboratory to weigh 300 grams.[2] The method of the French Experiment Station merely requires the reduction to a coarse powder before

general principles upon which to base such judgment. An occasional double sampling of the same lot of goods has been the only foundation for some of the sampling systems in use. The prevalent general idea has been that the larger the sample and the smaller the individual pieces in it, the more accurate it would be. Mr. Bailey has put this into a more definite form originating the new term "Size-Weight-Per Cent.," and

TABLE I.—ANALYSES MADE ON SAME PINT JAR SAMPLE BY DIFFERENT LABORATORIES—SEASON 1908–1909.

Brands. Laboratory.	Total phos. acid.			Avail. phos. acid.			Ammonia.			Potash.		
	A	B	Diff.	A	B	Diff.	A	B	Diff.	A	B	Diff.
9–3–2	11.72	11.80	0.08	9.21	9.82	0.61	3.02	3.07	0.05	2.46	2.58	0.12
9–2–1	11.69	11.43	0.26	9.85	9.83	0.02	2.10	2.18	0.08	1.48	1.48	0.00
10–2–2	12.79	12.30	0.49	10.79	10.45	0.34	2.10	2.23	0.13	2.48	2.58	0.10
10–0–4	12.14	12.10	0.04	10.59	10.95	0.36	....	....	....	4.43	4.64	0.21
10–0–2	12.14	12.45	0.31	10.26	11.12	0.86	....	....	....	2.39	2.56	0.17
8–2–2	10.40	10.25	0.15	8.57	8.55	0.02	2.21	2.18	0.03	2.13	2.42	0.29
8–4–4	11.49	11.18	0.31	8.83	8.13	0.70	4.02	4.20	0.18	5.51	4.50	1.01
8–2–2	10.34	10.25	0.09	8.43	8.55	0.12	2.21	2.18	0.03	2.05	2.42	0.37
Average difference,		0.22			0.38			0.08			0.28	
SEASON 1907–1908.												
9–3–2	11.82	11.65	0.17	9.21	9.05	0.16	3.35	3.50	0.15	2.15	2.32	0.17
9–2–1	11.62	11.65	0.03	9.46	9.62	0.16	2.46	2.28	0.18	1.25	1.06	0.19
10–2–2	12.59	12.30	0.29	10.61	10.05	0.56	2.56	2.43	0.13	2.12	2.40	0.28
10–0–4	11.73	11.78	0.05	10.60	10.58	0.02	....	....	....	3.92	4.10	0.18
10–0–2	11.85	12.05	0.20	10.48	10.90	0.42	....	....	....	1.89	2.38	0.49
8–2–2	10.51	10.00	0.51	8.62	8.15	0.47	2.30	2.01	0.29	2.16	2.72	0.56
8–4–4	11.28	11.10	0.18	8.02	8.15	0.13	4.04	4.26	0.22	4.65	4.44	0.21
8–2–2	10.53	10.00	0.53	8.71	8.15	0.56	2.26	2.01	0.25	2.20	2.72	0.52
Average difference,		0.24			0.31			0.20			0.32	

mixing and taking the final sample.[3] The International Commission merely requires mixing and attention to moisture changes.[4] The German method requires the sample to weigh 2 kg. and to be sifted if possible, and mixed.[5] There is very little in any of the above that is of value in judging the accuracy of sampling

for coal given figures which will enable any one to take and prepare a sample on which the error in ash determination will not be more than 1 per cent.

The attempt in this paper will be to give similar figures for use in sampling commercial fertilizer. In the case of fertilizer there are as high as three constitu-

TABLE II.—ANALYSES ON SAME SHIPMENT-SAMPLING AND ANALYSIS BY DIFFERENT PARTIES.

Lab. No.	Brand.	Laboratory.	Total phos. acid.			Avail. phos. acid.			Ammonia.			Potash.		
			A C D or E.	B.	Diff.	A C D or E.	B.	Diff.	A C D or E.	B.	Diff.	A C D or E.	B.	Diff.
1258	8–2–2	C	9.60	10.18	0.58	9.22	9.75	0.53	2.36	2.24	0.12	2.75	2.08	0.63
1259	8–2–2	C	8.70	9.45	0.75	8.25	9.07	0.82	2.71	2.50	0.21	2.26	1.91	0.35
1260	8–2–2	C	9.35	8.95	0.40	8.66	8.55	0.11	2.18	2.50	0.32	1.50	1.17	0.37
1261	9–2–1	C	9.00	9.90	0.90	8.57	9.32	0.75	2.45	2.34	0.11	1.60	1.03	0.57
418	9–2–1	C	9.65	10.75	1.10	9.11	10.02	0.91	1.75	1.92	0.17	2.11	1.42	0.69
728	10–0–4	D	.....	.....	....	9.32	9.90	0.58	....	....	....	3.02	2.22	0.80
584	8.75–2–2	D	10.28	9.58	0.70	9.81	8.95	0.86	2.30	2.43	0.13	1.40	1.67	0.27
585	10–0–2	D	11.02	10.93	0.09	10.66	10.35	0.31	....	....	....	1.62	2.01	0.39
1422	8–4–4	A	10.94	10.73	0.21	9.17	9.38	0.21	3.13	3.62	0.49	2.86	4.14	1.28
1423	8–2–2	A	10.13	9.05	1.08	9.75	8.52	1.23	1.42	1.86	0.44	1.50	1.93	0.43
8.87	13–0–4	E	.....	.....	....	11.75	13.20	1.45	....	....	....	4.03	3.51	0.52
1068	10–2–2	E	.....	.....	....	10.80	10.30	0.50	1.86	1.74	0.12	1.52	1.39	0.13
1256	9–3–2	E	.....	.....	....	9.50	8.43	1.07	2.83	2.99	0.16	1.34	1.46	0.12
Average difference,				0.65			0.78			0.25			0.54	

and sample preparation. As Dr. Wiley says, the matter of correct sampling is left principally to the judgment of the one in charge.[6]

Mr. Bailey's article on "Accuracy in Sampling Coal"[7] was practically the first attempt to set forth

ents on each of which the money consideration on 1 per cent. in a ton is over 10 times as great as in the case of ash in coal. The fact that goods are pulverized to pass a 2- to 5-mesh screen before shipping makes the sampling less difficult but does not make it as easy as has been supposed. The laboratory limit of error is necessarily quite high, making it difficult to determine where its effect stops and where the sampling error begins.

To determine whether the size-weight-per cent. is the controlling factor in fertilizer sampling and to

[1] Bureau of Chem., U. S. Dept. of Agr., Bulletin 107, p. 1.
[2] Wiley's "Principles and Practice of Agricultural Analysis," Vol. 2, p. 16, 2nd Edition.
[3] *Ibid.*, p. 17.
[4] *Ibid.*, p. 20.
[5] *Ibid.*, p. 21.
[6] *Ibid.*, p. 17.
[7] THIS JOURNAL, 1, 163.

obtain figures that will enable us to sample any lot of fertilizer with a known degree of accuracy, the following experimental work was undertaken: A mixture was made of

5 lbs.	51% muriate potash
25 "	16% acid phosphate
20 "	10.5% tankage

All of these materials had passed a 3-mesh screen and had been rejected on a $4\frac{1}{2}$-mesh screen. This

remaining part of the 25-pound sample was quartered to 150 grams 10 times and these 10 samples combined and ground to pass a 20-mesh sieve. It was found that the lighter materials in this sample separated out, as sometimes happens in fertilizers, so one-half of it was ground to pass an 80-mesh sieve and this latter sample was used to determine the correct analysis of the original 50 pounds. The analyses of the 20-

TABLE III.—RESULTS OF AMMONIA DETERMINATIONS.

	Composite	Composite	74.4	36	84.2	17.8	35.2	18.2	105.9	50.6	24.7
Average weight of sample gms.											
Wt. largest particle in grams	0.0000047	0.00034	0.0067	0.0067	0.250	0.0067	0.250	0.250	0.2764	0.2764	0.2764
Sieve through which passed	.....	.....	8m.	8m.	$4\frac{1}{2}$m.	8m.	$4\frac{1}{2}$m.	$4\frac{1}{2}$m.	3m.	3m.	3m.
"Size-weight-per cent."	0.00012	0.0085	0.0045	0.0093	0.0143	0.0188	0.355	0.068	0.131	0.273	0.56
Ground to pass	80m.	20m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.
	4.42	4.48	4.52	4.61	4.65	4.35	4.84	4.80	4.46	4.65	4.29
	4.46	4.27	4.44	4.50	4.26	4.58	4.63	4.63	4.76	4.26	4.84
	4.40	4.27	4.41	4.56	4.39	4.61	4.37	4.48	4.32	4.18	4.86
	4.37	4.31	4.41	4.50	4.44	4.84	4.48	4.44	4.00	4.73	4.76
	4.44	4.27	4.35	4.46	4.52	4.54	4.63	4.22	4.13	4.18	4.56
	4.42	4.54	.....	.....	.....	.....	.....	.....	4.35	4.12	4.50
	4.46	3.90	.....	.....	.....	.....	.....	.....	4.15	4.12	4.23
	4.44	4.16	.....	.....	.....	.....	.....	.....	4.15	4.24	3.97
	4.42	4.35	.....	.....	.....	.....	.....	.....	4.53	4.78	4.29
	4.48	4.31	.....	.....	.....	.....	.....	.....	4.24	4.65	4.18
Average	4.34	4.29	4.43	4.52	4.46	4.58	4.59	4.51	4.31	4.39	4.45
Maximum error from 4.43	0.06	0.39	0.09	0.09	0.20	0.41	0.41	0.37	0.43	0.35	0.46
Error of average from 4.43	0.00	0.14	0.00	0.18	0.03	0.15	0.16	0.08	0.12	0.04	0.02
Probable error calc.	0.02	0.155	0.042	0.083	0.099	0.166	0.173	0.159	0.175	0.187	0.228
Possible error calc.	0.123	0.886	0.238	0.470	0.565	0.943	0.966	0.910	0.996	1.065	1.17

makes an extreme case on all three ingredients, which would seldom if ever occur in practice, but which does often obtain to a large extent on one or more ingredients. The above mixture was quartered once and one-half set aside as a reserve portion. The other half was quartered down to about 100 grams and this placed in a bottle. The second half from the last quartering was quartered and a 50-gram sample saved; likewise a 25-gram sample was obtained and the remaining 25 grams returned to the original.

mesh portions have been included in the tables as an illustration of other factors which may influence the results.

In the following tables the probable and possible errors are calculated from the following formulas:

$$\text{Probable error} = 0.6745\sqrt{\frac{\Sigma v^2}{N-1}}$$

$$\text{Possible error} \atop \text{once in } 10,000 = 3.84\sqrt{\frac{\Sigma v^2}{N-1}}$$

TABLE IV.—RESULTS OF TOTAL PHOSPHORIC ACID DETERMINATIONS.

	Composite	Composite	74.4	36	84.2	17.8	35.2	18.2	105.9	50.6	24.7
Average wt. sample grams											
Wt. largest particle in grams	0.00001	0.008	0.028	0.0128	0.040	0.0128	0.40	0.040	0.4607	0.4607	0.4607
Sieve through which passed	.....	.....	8m.	8m.	$4\frac{1}{2}$m.	8m.	$4\frac{1}{2}$m.	$4\frac{1}{2}$m.	3m.	3m.	3m.
"Size-weight-per cent."	0.00025	0.020	0.0086	0.1780	0.0237	0.035	0.055	0.11	0.217	0.455	0.935
Ground to pass	80m.	20m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.
	12.80	12.68	12.45	12.58	12.50	12.78	12.30	12.20	12.70	12.63	12.45
	12.65	12.78	12.65	12.58	12.60	12.83	12.38	12.30	12.55	12.85	12.35
	12.63	12.80	12.50	12.65	12.40	12.68	12.40	12.28	12.08	12.30	12.35
	12.78	12.70	12.43	12.68	12.53	12.65	12.58	12.60	11.98	11.75	11.98
	12.60	12.73	12.68	12.70	12.53	12.80	12.85	12.75	12.45	12.40	11.93
	12.55	12.64	12.85	.....	12.85	12.72	......	......	12.50	13.05	12.75
	12.65	12.63	12.83	.....	12.65	13.06	......	......	12.15	12.75	12.68
	12.70	12.75	12.75	.....	12.68	12.78	......	......	12.33	12.45	12.45
	12.63	12.68	12.75	.....	12.75	12.76	......	......	12.00	12.73	11.93
	12.63	12.78	12.80	.....	12.90	13.04	......	......	12.50	11.85	12.13
Average	12.66	12.71	12.67	12.63	12.64	12.81	12.50	12.43	12.32	12.48	12.30
Maximum error from 12.66	0.14	0.14	0.23	0.08	0.26	0.40	0.36	0.46	0.66	0.91	1.73
Error of average from 12.66	0.00	0.05	0.01	0.03	0.02	0.15	0.16	0.23	0.34	0.18	0.36
Probable error calc.	0.0523	0.0573	0.1063	0.041	0.1083	0.149	0.190	0.2380	0.294	0.312	0.326
Possible error calc.	0.298	0.326	0.408	0.234	0.417	0.850	1.082	1.355	1.676	1.780	1.858

This process was repeated 10 times, giving 30 samples. The remainder of the original 25 pounds was then crushed to pass a $4\frac{1}{2}$-mesh screen and the same process carried out five times, giving 15 samples. The large sample was again crushed and passed through an 8-mesh screen and quartered down five times as before, making a total of 60 samples. The

in which Σv^2 equals the sum of the squares of the individual errors and N is the number of tests from which the errors are taken.

To determine the sizes of the particles used in these tables, curves were located by plotting the mean of the linear dimensions of the opening in two screens and the weights of the average particle rejected by

one screen and passed by the other of these same screens. Then from these curves the weight of the largest particle passing any screen was read off, using the side of the opening in the given screen. The size-weight-per cent. was calculated as follows: Size-weight-per cent. =

$$\text{Size-weight-per cent.} = \frac{\text{weight of largest particle} \times 100}{2 \times \text{weight of sample}}.$$

Omitting the 20-mesh composite sample, the in-

enough toward the origin of the curve to give us, with reasonable accuracy, the point S on curve No. 2, sheet 1, which will be used later. It is likely that a larger number of samples would have brought all the points much nearer the curve.

In the case of potash, the irregularities were of more serious character, the average being below in several cases. Two possible reasons for this were

TABLE V.—RESULTS OF POTASH DETERMINATIONS.

	Composite.	Composite.	74.4	36	84.2	17.8	35.2	18.2	105.9	50.6	24.7
Average wt. sample grams											
Wt. largest particle in grams	0.00001	0.0008	0.0121	0.0121	0.029	0.0121	0.029	0.029	0.2937	0.2937	0.2937
Sieve through which passed	.....	....	8m.	8m.	4½m.	8m.	4½m.	4½m.	3m.	3m.	3m.
"Size-weight-per cent."	0.00005	0.004	0.0082	0.0168	0.172	0.034	0.0412	0.079	0.138	0.290	0.595
Ground to pass	80m.	20m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.	80m.
	4.42	4.23	4.59	4.56	4.11	4.40	4.56	3.96	4.27	4.24	5.88
	4.48	4.35	4.49	4.65	3.91	4.21	4.62	4.29	4.18	3.74	3.65
	4.53	4.38	4.49	4.35	4.42	4.57	4.87	4.63	4.46	4.22	3.87
	4.66	4.37	4.68	4.51	4.31	5.15	4.32	4.09	5.28	4.76	5.03
	4.56	4.25	4.41	4.41	4.27	4.19	4.08	4.43	4.70	4.71	5.40
	4.30	4.21	....	....	....	.....	....	....	4.33	4.19	3.14
	4.59	4.30	....	....	....	.....	....	....	4.87	5.11	5.07
	4.37	4.34	....	....	....	.....	....	....	4.92	4.58	4.97
	4.47	4.25	....	....	....	.....	....	....	4.67	4.80	4.96
	4.32	4.41	....	....	....	.....	....	....	4.05	4.54	4.74
Average	4.47	4.32	4.59	4.49	4.20	4.50	4.49	4.28	4.57	4.49	4.67
Maximum error from 4.47	0.19	0.26	0.21	0.18	0.56	0.68	0.40	0.51	0.81	0.73	1.41
Error of aver. from 4.47	0.00	0.15	0.12	0.02	0.27	0.03	0.02	0.19	0.10	0.02	0.20
Probable error calc.	0.08	0.124	0.085	0.08	....	0.266	0.20	0.22	0.27	0.27	0.59
Possible error calc.	0.45	0.706	0.482	0.47	1.59	1.51	1.16	1.25	1.53	1.52	3.36

gredients of which separated in the bottle as noted above, there are but two of the possible error figures which do not fall on the curve No. 1, sheet 1. These figures would probably fall on the curve had they been calculated from ten or twenty determinations instead of five.

In addition to figures in Table IV on total phosphoric acid, the possible error was also calculated for avail-

suggested. Either that the moisture from the acid phosphate and air dissolved potash from the crystals and this solution was lost on the sampling cloth, or that in some sets of samples the potash was "retained"[1] more than in others.

A second set of samples was therefore made up using dry, fine sand in place of the tankage and acid phosphate used above, care being taken not to handle these

TABLE VI.—RESULTS ON POTASH IN SAND MIXTURE.

		86.7	41.5	84.7	21.2	40.2	20.5	98.1	48	•23.4
Average wt. sample grams.										
Wt. largest particle in grams	0.00001	0.0121	0.0121	0.029	0.0121	0.029	0.029	0.2937	0.2937	0.2937
Sieve through which passed		8m.	8m.	4½m.	3m.	4½m.	4½m.	3m.	3m.	3m.
"Size-weight-per-cent." ...		0.0070	0.146	0.171	0.0286	0.0365	0.0707	0.149	0.306	0.626
Ground to pass	60m.	60m.	60m.	60m.	60m.	60m.	60m.	60m.	60m.	60m.
1	5.18	4.92	5.09	4.39	4.74	6.53	4.66	3.83	4.35	7.75
2	5.31	5.31	4.77	4.16	4.73	4.06	4.72	3.14	3.92	5.26
3	5.21	5.65	6.04	4.46	7.09	5.18	4.61	4.13	5.66	5.15
4	5.35	5.16	4.93	4.69	4.84	5.47	4.61	4.84	4.86	3.58
5	5.39	5.59	5.98	4.59	6.85	4.94	4.87	4.26	4.55	7.57
6	5.33	5.12	5.92	5.25	4.67	4.49	5.57	4.78	5.07	4.82
7	5.35	5.18	5.47	5.13	5.44	5.28	5.30	4.88	6.00	5.67
8	5.33	5.55	6.29	4.89	5.42	5.88	3.56	3.69	4.99	6.32
9	5.38	5.31	6.10	5.24	5.71	5.96	4.57	4.64	4.56	3.49
10	5.37	4.83	5.14	4.97	5.16	5.31	6.14	3.69	4.87	5.56
Average	5.32	5.27	5.57	4.78	5.22	5.31	4.86	4.19	4.88	5.52
Maximum error from.... 5.32	0.00	0.49	0.97	1.16	1.77	1.26	1.76	2.18	1.40	2.43
Error of average from... 5.32	0.14	0.05	0.25	0.54	0.10	0.01	0.46	Spoiled by	0.44	0.20
Probable error	0.047	0.191	0.416	0.462	0.594	0.484	0.559	mechanical	0.507	0.973
Possible error	0.23	0.945	2.06	2.28	2.94	2.40	2.77	losses.	2.53	5.54

able, but owing to the introduction of the second laboratory error in the insoluble, the results were not uniform.

Considerable irregularity of results was found on total phosphoric acid at first, but on making up three second sets of samples from the reserve 25 pounds, the possible error figures became uniform down far

samples when the relative humidity in the room was above 75%. Following are the results on this test.

It was found that, while this avoided the difficulties above mentioned, it was very difficult to grind and sift these samples without loss into the air, of very fine particles of potash. Because of inattention to

[1] THIS JOURNAL, May, 1909.

this detail, the results on the 3-mesh 100-gram samples were made valueless and have been omitted from the curve. The possible errors in the last set of figures, Table VI, are much higher than on the fertilizer, Table V. If, however, by simple proportion, a calculation is made as to what error to expect from the concentration of the materials used, using the ammonia results as the basis, the results from the sand sample will be found to agree closely with the calculated figures. The following table illustrates this point, all the figures being on the 3-mesh 25-gram samples.

TABLE VII.

Material used.	Concentration of ingredients tested for	Possible error found.	Possible error calculated from the ammonia figure.
Tankage.	10.5 per cent. ammonia	1.17	....
Acid phosphate.	17.0 per cent. tot. phos. acid	1.86	1.89
Mur. pot. in fert.	51.0 per cent. potash	3.36 }	5.69
Mur. pot. in sand.	51.0 per cent. potash	5.54 }	

The writer believes that the low possible errors in potash on fertilizers are due to the solution of potash in the acid phosphate and tankage moisture, making it not a case of sampling a mixture of 51 per cent. crystals, acid phosphate and tankage, but rather mixture of crystals reduced in size by solution and acid phosphate and tankage carrying potash dissolved in their moisture contents.

Such action as this would not be likely to go on to the same extent in each of the 60 samples above and would possibly account for some of the irregularities in the results obtained on potash in the fertilizer. It has been evident throughout the work, however, that a large part of the irregularities disappear as the number of samples is increased.

The above results can be put into a more convenient and practical form by deciding what possible and probable error can rightly be allowed to the process of sampling and then determine the weight of sample necessary from any sieve to give that result. It is desirable to restrict the possible error due to sampling to 0.50 per cent. with the corresponding probable error of 0.088 per cent. This added to the possible error found on the 80 m. composite sample which we have taken as the laboratory limit gives the following figures. An average of the two laboratory limit figures has been taken on the potash work.

TABLE VIII.

	Allowable possible error. Per cent.	Corresponding size-weight-per cent.
Tankage (ammonia)	0.633	0.02
Acid phosphate (phos. acid)	0.80	0.03
Muriate of pot. in fert. (potash)	0.80	0.03
Muriate of pot. in sand (potash)	0.80	0.005

These possible error figures give the points on the curves, sheet 1, marked S and from them the corresponding size-weight-per cents. given above were read off. Calculating the weight of sample for the different
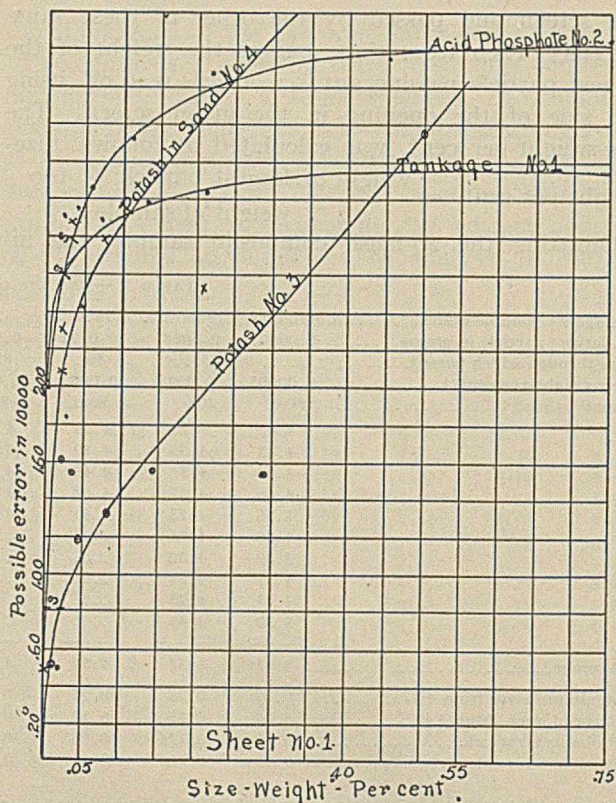
mesh screens, using these size-weight-per cents. and the weight of the largest particles for the screens, we have:

TABLE IX.

Grams of fertilizer sample necessary to give 0.50 per cent. sampling possible error.

Size of opening in screen.	Mesh of screen.	On ammonia.	On potash and phos. acid in fert.	On potash in sand.
0.253	3	1382	1535	5874
0.142	4.5	125	133	580
0.107	8	34	43	242
0.033	20	1.13	2.56	13
0.170	4	290	310	800

Plotting these grams of samples and dimensions of openings in screens gives the curves on sheet No. 2 from which the weight of sample for any screen can be determined if the dimensions of openings in the screen are known. In this way, the above figures for 4-mesh screen were obtained.

The taking and preparing larger samples of course means more work. In view of this fact, it does not seem advisable to take samples as large as indicated by the potash and sand results, Table VI, except perhaps in special cases. This is especially true until such a time as such figures as are given in Table VI have been obtained on commercial fertilizer mixtures.

In conclusion, the following points are noted:

1. Discrepancies in the analyses of the same shipment are much greater than they should be.

2. Discrepancies are largely due to sampling as shown by a comparison of averages on Tables I and II and as shown by the errors from the averages on Tables III, IV, and V.
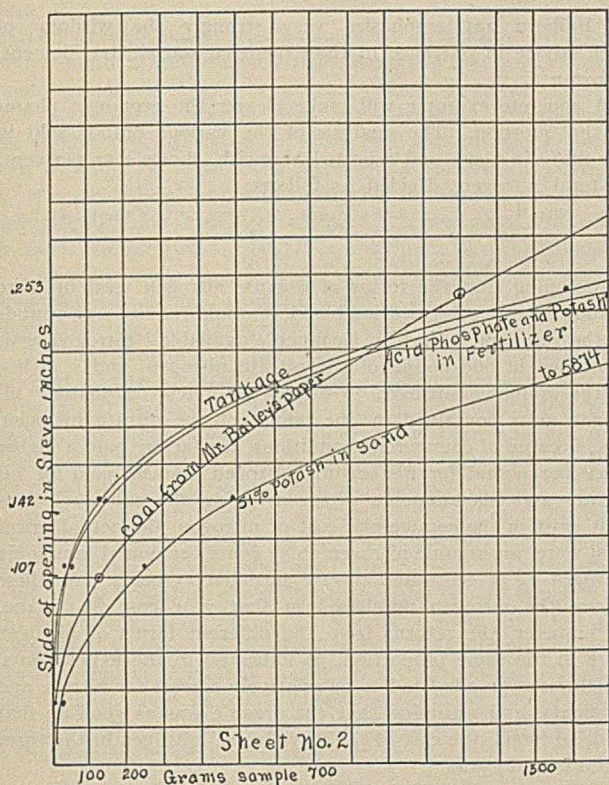
3. Possible error increases with the size-weight-per cent., giving a fairly uniform curve. Irregularities are the greatest in case of potash.

4. The possible error seems to increase in simple proportion with the percentage of the ingredient analyzed for in the material used to furnish that ingredient, provided there is not some factor which modifies this.

5. For a chosen degree of sampling accuracy the weight of sample required on goods from any known mesh or screen can be determined.

6. For a possible sampling error of 0.50 per cent. on fertilizer from a 3-mesh screen, a sample weighing 1535 grams (3½ pounds) should be taken and passed through a finer mesh sieve before quartering. Subsequent handling should be kept within the bounds indicated by sheet 2 and Table IX. These are the points at which most present fertilizer sampling systems are weak.

7. Strict adherence to the above suggestions will not obviate the difficulties due to imperfectly mixed goods, but given well-mixed goods and inspectors who will correctly take and prepare samples as indicated by the size-weight-per cent. figures given, the writer is confident that the wide variations so common to-day will disappear.

This work has been done under certain limitations, especially as to time, and it is hoped that some one will take up, confirm, and complete it. The behavior of potash seems to especially invite further attention.

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ADDRESSES.

THE COST OF AVAILABLE NITROGEN.[1]
BY DR. EDWARD B. VOORHEES.

The use of commercial fertilizers has been one of the most important factors in the development of the farming interests in this country. The present annual consumption is, as near as can be estimated, 5,000,000 tons which, at an average cost of $29.00 per ton, makes a total expenditure of $145,000,000. This great quantity of fertilizer is being used for increasing the crops of grain, hay, potatoes, fruits and market garden crops. The money is expended for nitrogen, phosphoric acid and potash, and notwithstanding the claims made for superior brands and special formulas, the returns are due to the actual amounts of nitrogen, phosphoric acid and potash that these crops are able to obtain from the total in the fertilizers used.

The value of the increased crops made from the use of any one or more of these constituents is, however, measured both by the amount that the crop obtained, and the character of the crop obtaining it. A pound of nitrogen, phosphoric acid or potash, when used in making a crop of celery, or of asparagus, or of fruit, would be worth more than if used in making a crop of wheat, rye, or hay; furthermore, the value of the user of the nitrogen or other constituents bought in a fertilizer, is measured both by the amount that the immediate crop is able to obtain, and the proportionate amount of the total that would eventually be gathered.

Of the sum annually paid for the three constituents, nitrogen, phosphoric acid and potash, about 40 per cent. is paid for nitrogen, which is the only one of the three essential elements that is likely to suffer any considerable loss in use, as the experiments conducted along this line show, on the average, not more than 70 per cent. of the quantities applied, even in the best forms, is recovered in the crops.

The case is different with phosphoric acid and potash; these are not subject to serious losses, under good methods of soil management. Practically, these constituents remain in the soil until they are taken out by plants.

From the standpoint of crop, it is evident that the utilization of nitrogen is a much more important matter than the use of phosphoric acid and potash, although the further fact that a pound of nitrogen, capable of being used in a commercial fertilizer, and without regard to form, costs from four to five times as much as a pound of "available" phosphoric acid or of potash, is an additional argument in favor of greater care in its purchase and use.

Nitrogen as nitrate is the only commercial form soluble in water, ready for immediate use by most plants; nitrogen as ammonia is also a form soluble in water, but it is less available than the nitrate. A pound of nitrate and a pound of ammonia, being definite chemical compounds, are quite as good from one source as another.

Organic forms of nitrogen have to decay first, changing to ammonia and then to nitrate, and are therefore less quickly available; besides, they vary in their rate of availability according to the source of supply and their physical character. Materials which are likely to decay quickly, as dried blood, dried meat, dried fish and cottonseed meal, do show a high rate of availability, while forms like ground leather and ground peat show a very low rate of availability. A pound of organic nitrogen varies in availability, therefore, according to its source, whether derived from dried blood or peat, or from intermediate products.

Since nitrogenous materials are variable in their rate of availability, that is, the rate at which the nitrogen in them may be absorbed by the plant, the farmer desires to know the dependence that can be placed on the different materials; he

wants available nitrogen. Hence, the chemical and physical characteristics of the various forms of nitrogen have been made the subject of every considerable study and investigation, in order that at least approximate values in respect to availability may be attached to each form. Sufficient work has been done thus far to establish a pretty safe relationship between the nitrate, ammonia and organic nitrogen, in the form of dried blood. It has not been possible, yet, to investigate fully all of the various forms of organic nitrogen, so as to assign an exact value for the different materials.

The very extensive investigations conducted by Dr. Paul Wagner, at Darmstadt, Germany, show that for the crops tested by himself and others, namely, barley, oats, rye, wheat, mangels, sugar, beets and potatoes, there was returned in the harvest 62 parts of nitrate nitrogen for every hundred parts applied, 44 parts of ammonia nitrogen for every 100 parts applied, and 40 parts of organic nitrogen for every hundred parts applied as dried blood. In no case is the recovery equal to two-thirds of the nitrogen applied; besides, there are wide variations in the amount recovered in the different forms.

In 1898, plant nutrition experiments were begun at the New Jersey Station, one object of which was to study the "relative availability" of these three forms of nitrogen, using a rotation of corn, oats, wheat and timothy; crops which, because of their long periods of growth, would be likely to absorb relatively large proportions of organic nitrogen. The results of these experiments for two rotations (10 years) are reported in detail in Bulletin 221, and show that the recovery for nitrogen as nitrate was 62.09 parts per hundred, for the nitrogen as ammonia 43.26 parts per hundred, and for organic (dried blood nitrogen) 40 parts per hundred. These results agree almost exactly with those obtained by Dr. Wagner and his associates. With the returns from nitrate, the highest recovery regarded as 100, the relative availability of the nitrogen as ammonia would be 69.7 and of nitrogen as dried blood 64.4.

These figures possess a very great practical significance as they have a direct bearing upon the economical purchase and use of the nitrogen contained in the fertilizers now offered upon the market.

Commercial conditions fix the price of the various nitrogenous materials, and the cost to the farmer of any one form is not measured by its usefulness to him, but by the cost in the market; that is, there is no strict relationship between commercial and agricultural values.

It happens that at the present time, a pound of nitrogen in the form of nitrate or of ammonia costs the farmer less than a pound of organic nitrogen; that is, the nitrogen possessing the highest rate of availability as nitrate is less expensive to him than dried blood nitrogen, or even that derived from low-grade nitrogenous materials, which do not possess any definite rate, and which must, on the average, show a much lower rate of availability than dried blood, because the mixtures contain nitrogen derived from many sources, not uniform in their content of nitrogen or in their physical character or constitution.

Garbage tankage and tanned leather scraps, for example, are used in large quantities, some of the larger eastern fertilizer factories using several thousand tons per year. The nitrogen in these products is admittedly much less available than is that in dried blood, and its cost to the manufacturers is, according to present quotations, but little more than one-half as high.

The Experiment Station has since its establishment consistently urged the farmers, in their purchase of fertilizers, to be guided not only by the quantities of the constituents present in the mixture offered, but also by the kind that is used in them, pointing out the importance of selecting brands which contain high percentages of available plant-food, more especially of nitrogen, because of its relatively greater importance, or its higher cost. The results obtained in the investigations reported

in Bulletin 221 emphasize very strongly the wisdom of such advice in reference to the expensive and elusive element, nitrogen.

A concrete example will make clearer the economic phases of the question. The analysis of the various brands sold in the State in 1909, and reported herewith, shows 2.57 per cent. of total nitrogen, divided as follows:

Nitrate......... 0.48 per cent., or 19 per cent. of the total.
Ammonia....... 0.77   "   " 30   "   "   "
Organic .... 1.32   "   " 51   "   "   "

Assuming that the forms of organic nitrogen used in these brands were as good as dried blood, it would require 1.55 pounds of the nitrogen to furnish as much "available" nitrogen as is contained in one pound of the nitrate nitrogen, and 1.43 lbs. of the ammonia nitrogen to furnish as much "available" nitrogen as is contained in one pound of the nitrate nitrogen. Yet, because of commercial conditions, the farmer paid a higher price per pound for his organic nitrogen than he paid for his nitrate and his ammonia nitrogen; using the same relations that exist in the commercial cost of nitrogen, the actual prices paid were, for organic nitrogen 26.52 cents per pound, ammonia nitrogen 23.73 cents and nitrate nitrogen 23 cents. At these prices, the nitrogen purchased in that year cost $1,157,400. If, however, the returns from the different forms of nitrogen were in the same proportion, as indicated in the experiments, which must be admitted to be relatively correct for nitrate and ammonia, and assuming that the organic was as good as that in dried blood, the cost of the "available" nitrogen in the three forms actually was

For organic........................... 41.12 cts. per lb.
For ammonia.......................... 34.04 "   "
For nitrate............................ 23.00 "   "

while the farmer should have paid, on the basis of availability,

For organic........................... 14.81 cts. per lb.
For ammonia.......................... 16.03 "   "
For nitrate............................ 23.00 "   "

and a saving of $383,940 would have been effected. If, therefore, instead of buying organic and ammonia nitrogen, nitrate only had been purchased, the same gain in crop from the use of the nitrogen could have been purchased for $773,460 instead of $1,157,400.

Assuming that practically the same relations existed for all the fertilizers made and sold in the whole country, this year, the actual cost of the nitrogen was over $60,000,000 while on the basis of available it should have cost but $43,000,000. It may be argued that the availability of the organic nitrogen is greater in the warmer climates of the South, where the bulk of the fertilizer is used; this may be true, but is probably counterbalanced by the fact that a much larger proportion of the nitrogen used there is in organic forms. It is a fact, too, that the present high cost of cottonseed meal has encouraged a larger use of the tankages and other lower grade nitrogenous products.

The point of importance, therefore, is the price that is paid for the organic forms. In the above discussion, it has been assumed that the organic nitrogen contained in the fertilizers has been derived from dried blood, or from other materials quite as good. As a matter of fact, however, dried blood does not constitute even a large proportion of the organic nitrogenous materials used, the bulk of the nitrogen being derived from products of a lower grade. Various kinds of meat and bone, tankage, dried fish, fish scrap, cottonseed meal, garbage tankage, leather meal and even peat are used to supplement products of the higher grade. These, while genuine nitrogen carriers, have been shown to have a wide range in availability, the leather and peat rating in availability as low as 4 in comparison with nitrate as 100.

It may be urged, first, that these products possess a value as sources of nitrogen; and second, they are valuable as absorbents and in improving the texture of mixtures containing

nitrates, acid phosphate and potash salts, as mixtures of chemicals only cannot be applied by machinery; and third, that proper conservation of natural resources demands that waste nitrogenous materials should be utilized. The points are *conceded.* The Experiment Station does not discourage, but strongly encourages the utilization of waste products containing nitrogen; it would be false to its duty to the farmers, however, if it did not clearly point out to them what is known of the relative agricultural value of such products. It is not solely a question of use, it is a question of cost. The cost to the farmer of a pound of nitrogen in these materials, of a value lower and more variable than the nitrate and ammonia, should be lower rather than higher than for nitrate or ammonia.

It is not economy to save refuse nitrogenous materials, if the cost of the nitrogen to the farmer is greater and his returns less than may be obtained by the use of nitrogen from materials of known value. Farmers have been and are now spending thousands of dollars for nitrogen for which they do not receive a proportionate return.

To the farmer, it is purely a business proposition. He buys nitrogen, in order that he may get a return in crop; if in one case 100 pounds of nitrogen contributes 60 pounds to the crops upon which it is applied and in another 100 pounds contributes but 40 pounds to the crops, the purchaser should not pay the same for the second as for the first, for if he did so he would pay 50 per cent. more per pound for his "available" nitrogen; that is, if the cost of the first hundred pounds were $15, the second hundred should cost but $10, when the basis of value s the amount available in each.

THE RELATION OF THE CHEMIST TO PROPRIETARY MEDICINES.[1]

W. A. PUCKNER, Secretary, Council on Pharmacy and Chemistry of the American Medical Association.

That the composition of proprietary medicines differs widely from that which is claimed for them by their manufacturers or owners has been given wide publicity through the enforcement of the Pure Food and Drugs Act and the exposures which which have been published in the *Journal of the American Medical Association's* Propaganda for reform in Proprietary Medicines. As a result of this publicity the public, and more particularly the medical profession, has become suspicious of statements made and is inclined to ask for evidence which will substantiate the promoter's claims. This demand the manufacturers (promoters, owners) have attempted to meet in two ways: the large firms have employed a staff of chemists and have advertised that their products are duly examined and tested before being offered for sale. The degree of credence which is given to the firm's protestations of honesty and reliability has to no small extent depended on the professional standing of their chemists. While it is to be expected that the employees of a firm will incline to a high opinion of that firm's products, the reports of analysis or examination vouched for by such chemists of reputation as A. R. L. Dohme, J. M. Francis, A. B. Lyons, C. E. Vanderkleed, and other chemists closely identified with progress in pharmaceutical chemistry are generally accepted without question. This condition of affairs is satisfactory to all concerned but particularly so to chemists. As secretary of the Council on Pharmacy and Chemistry of the American Medical Association, I have had ample opportunity to see the increased reliance which is placed on the services of chemists. Formerly it was often quite evident that questions dealing with the chemistry of medicinal products had been decided by the office force without consulting with those who were competent to decide the points involved. To illustrate the lack of coöperation between the scientific departments of

[1] Delivered before the Division of Pharmaceutical Chemists, Boston Meeting American Chemical Society.

pharmaceutical manufacturing houses and the commercial end (evidently because the value of the scientific staff was not appreciated), it may be mentioned that a firm which had in its employ one of the chemists just referred to, offered to supply to the Council the structural formula of an iron protein compound of rather indefinite composition. To-day such questions are placed in the hands of those who are competent to decide them, the chemists. In this and in many other ways it is plain that the manufacturer relies to a very large extent on the chemist to demonstrate the reliability of his wares.

While the large manufacturing houses thus rely on the chemists in their employ, many smaller concerns, often aptly called "pseudo-chemical companies" do not regularly employ chemists. Although there is no sharp line of demarcation, these establishments may be divided into two classes: those who belong to one class, though often not overly conversant with the products which they sell, are entirely honest and sincere, and submit their products to commercial chemists or "professors of chemistry" for analysis in order to obtain certificates which will vouch for the quality of their goods.

Although there is a tendency to use a report of an analysis of a certain specimen of a product as an indication that all other lots of this product are of the same composition, and in this way to create a false impression, the employment of chemists to check the quality of medicinal products is to the best interest of all concerned. It helps the dealer to sell his goods, it ensures to the buyer their quality and it has helped to bring about a more general appreciation of the value of the science of chemistry.

While the manufacturers of, or dealers in, proprietary products just referred to, make use of the chemist in a perfectly legitimate way, this can not be said of all who are so engaged. A very large proportion of those engaged in the sale of proprietary medicines apparently believe that "all is fair in love and war" in the exploitation of medicines, and who have regard for the truth only so long as the telling of it will help the sale of their goods. When these concerns seek the services of a chemist it is less with a view of learning the truth regarding their products than to obtain good advertising copy. These concerns are willing to pay liberally for the right kind of a chemical analysis, that is, for a report which in its entirety or in part will look well in print. The way in which these concerns regard the chemist and his work is well brought out in an editorial which appeared in the *The Druggists' Circular* (October, 1908). Here it is related how a chemist was approached with the request to sign a report of an analysis and to pocket the fee without going through (what appeared to the one who wanted the analysis) the useless formality of making any examination of the product referred to in the report submitted to the chemist for signature. While few chemists will be tempted by such crude efforts at bribery, not all are proof to a more refined invitation to certify to that which is not the truth. By this I refer to the temptation to make out a report which emphasizes all the good points of a product, to minimize or to omit all that is derogatory to it and in other ways to make the report satisfactory to him who wants to use it in the exploitation of the remedy. When it is considered that the patronage of these concerns can be retained only by writing reports that will look well in print, it is not surprising that so many certificates of analysis give no material information in regard to the medicine which they are designed to advertise but can be used to create or perpetuate a wrong conception regarding the nature of the product.

If the report as a whole is not satisfactory, the exploiter of the proprietary medicine will of course not hesitate to use any part thereof which will make good advertising material even at the risk of utterly misrepresenting the actual findings of the chemist, as shown by the report.

Since not only the reputation of the individual chemist, but also the reputation of chemists as a whole, will suffer through association with the exploiter of proprietary medicines as just described, I deem it of interest to offer the following illustrations of the way in which chemists are used as a means of exploiting a medicine in a way which discredits our profession.

In the *Journal of the American Medical Association* (October 13, 1906) the composition of a preparation known as Waterbury's Metabolized Cod-liver Oil compound was discussed. It was there stated that the product did not show any fat globules under the microscope and that it was therefore not an emulsion, that it was acid in reaction and did not become turbid on the addition of strong acids and hence was not soap or a saponification of fat, that it did not become turbid on dilution with water and hence could not contain more than traces of a fatty acid. In view of this evidence and because of confirmation by chemical examination it was stated that the preparation, although claimed to contain the product "obtained by the action of fat-splitting ferments on cod-liver oil," represented no (or at most a mere trace of) cod-liver oil. In an attempt to offset this damaging evidence the Waterbury Chemical Company published in the form of advertisements, a number of chemists "reports of analysis" which, more or less directly, denied the truth of the damaging report of the *Journal of the American Medical Association*. One advertisement purporting to be a report signed by a professor of chemistry in a college of pharmacy is rather indefinite and vague, but it contains the phrase "of course we know that the finished product contains metabolin" which, while meaningless, no doubt impressed many as some sort of a confirmation of the truthfulness of the claimed presence of "metabolized cod-liver oil." Another advertisement bearing the fac-simile signature of a professor in a western university certified that the product contained the following imposing list of constituents without giving any quantities:

Maltose and Reducible Sugars; Oils and Fatty Acids; Glycerine-Alcohol-Acetone; Acids including Hypophosphoric, Phosphoric, Tartaric, Acetic, Sulphuric and Hydrochloric; Proteids (soluble and of variable composition); Benzaldehyde very small quantity present; Bitter Principle, not Alkaloidal, vegetable character; Ferments—Enzymes, etc., including various Organized and Unorganized Ferments; Inorganic Salts, Iron, Sodium, Calcium; Further the report states that "In the fats extracted there is present free fatty acids, in which Palmitic, Stearic and Oleic Acids are identified."

While chemists will have no difficulty in assigning its real value to any report of an analysis which does not give quantities, physicians no doubt were duly impressed by the statement regarding the presence of a large number of constituents including "oils and fatty acids."

In a third advertisement the statement made in the *Journal of the American Medical Association* that the product when acidulated remained clear was definitely challenged. The advertisement appears to be a reproduction of an analytical report and shows the signer to be a doctor of medicine as well as a doctor of philosophy.

Recently (October 9, 1909) the chemical laboratory of the American Medical Association published a report of an analysis which confirmed the statements made in the *Journal of the American Medical Association* (October 13, 1906). The report shows that a specimen of the preparation purchased in the open market contained probably not more than 0.1 per cent. and certainly not more than 0.3 per cent. of fatty acids and that, for all practical purposes, it should be considered devoid of cod-liver oil. Since then the federal authorities have brought suit to confiscate a shipment of the Waterbury product, the charge being in the main that it contains no cod-liver oil and is therefore misbranded.

It is thus seen that the certificates of chemists have been used to aid in the sale of a fraudulent product. That this is contrary to the best interests of the public, our profession and of the signers of the reports goes without saying. It also is quite evident that the manufacturer of the product deliberately made use of the reports to misrepresent the facts. But it seems to me that the signers of the reports are not without blame since the misuse of chemists' certificates tend to discredit the entire profession; and since the abuse is quite common I consider it my duty to call attention to the matter by pointing out how far the signers of the reports were at fault or at least were injudicious.

Granting that the particular specimen of the product which the signer of the first report examined did contain any appreciable amount of the products produced by the action of digestive ferments on cod-liver oil, the application of the term "metabolin" to such a mixture seems inexcusable. Whether the term was used to impress the manufacturer with the scientific attainments of the writer of the report or with a view of furnishing suitable advertising material to the manufacturer, the chemist must have known that its use was unwarranted. From a letter received in reply to my request for information as to the method used to determine the amount of "metabolin" and the amount actually found (information which it is needless to say was not forthcoming), I am inclined to believe that the chemist was much surprised and chagrined to find that a portion of a letter written to the manufacturer was used as advertising copy.

According to the chemist, whose signature appeared in connection with the second advertisement mentioned, publication of his report was authorized only on condition that the complete quantitative analysis be furnished to physicians who asked for it. This being the case and granting that all of the constituents mentioned in the imposing list (including oleic acid, the demonstration of which has baffled chemists for fifty years or more) were actually identified in the specimen examined, also granting that it was not made out with a view of impressing the manufacturer with his ability, no specific fault can be found with the chemist's report but the chemist should have known that it would be used to mislead others in regard to the composition of the preparation.

While at first it would appear as if the statements of the chemist's report in the third advertisement were refuted by the published analysis of the chemical laboratory of the American Medical Association, and by the action of the federal authorities, this is not the case. Instead it is probably but another illustration of the often noted variability of proprietary medicines. The specimen reported on by the laboratory of the American Medical Association, and which was found to be practically free from fatty acids, was purchased on the open market. On the other hand, the specimen reported on in the third advertisement, according to the statement of the chemist to me, contained about three per cent. of fatty acids. From this it is evident that the chemist did nothing more than report his findings. But in view of his training as a chemist and as a physician he must have recognized that the preparation did not contain enough cod-liver oil to give it any value as a cod-liver oil preparation. Further, he must have known that his report would be used to mislead physicians in regard to the actual composition of the preparation.

To show the tendency of analysts to write reports which will make good advertising copy and to show how such enthusiastic reports may later place the chemist in an embarrassing position, the following is given: The Council on Pharmacy and Chemistry of the American Medical Association some time ago (October 20, 1906) published a report which stated that a certain product, Tyree's Antiseptic Powder, did not possess the composition which its manufacturers had claimed it to have. As in the case of the Waterbury product so in this instance the manufacturer

attempted to offset this by publication of a report of a chemist. In this analytical report which deals with the composition of the product and the purity of the materials used in its preparation it is stated that the product contains, among other things, zinc sulphate and phenol; that these two substances "of course react to form zinc phenol-sulphonate." While it is a rather common misconception that phenol and sulphates when dissolved in water react to form phenol-sulphonates, the statement in regard to its formation was so foreign to the subject of the analytical report that I wondered whether the analyst had really made this statement. Correspondence with the analyst brought out the fact that the statement was contained in the report made to the manufacturer. The analyst also informed me that he had advised the manufacturer that this statement was incorrect and should be omitted from his report. It should be added that the manufacturer, now (or at least quite recently) three years later, is still circulating the statement.

What has been said illustrates a general tendency to frame reports of chemical analyses in such a way as to create a favorable but incorrect opinion in regard to the product reported on. While the writers of such reports can rarely be accused of making any incorrect statements they often disguise or ignore the facts in such a way that their effect is the same as if a direct misrepresentation had been made. There is grave danger that the term "commercial chemist" may come to be generally interpreted as meaning a chemist who puts a commercial value on his reports; that is, a chemist whose favorable reports may be purchased. While many analysts are above reproach in this respect, the tendency is so general that individual chemists should be blamed for this condition. It seems to me that the time is ripe when chemists individually and through their societies should express their disapproval of a practice which is a discredit to the entire profession.

NOTES AND CORRESPONDENCE.

THE LENGTH OF A TECHNICAL PAPER.

The appeal of Mr. Albert E. Leach, on the editorial page of THIS JOURNAL, January, 1910, for greater brevity in the method of presentation of technical papers must be seconded unanimously by those who attend scientific meetings and, for the most part, likewise by readers of scientific journals. However, the abbreviations of strictly technical papers intended for publication, it must be conceded, if too much emphasized is quite apt to be overdone.

The articles appearing in THIS JOURNAL and in the *Journal of the American Chemical Society* are supposedly exact accounts of observations of chemical operations, with results, descriptions of chemical processes and presentations of chemical theory. The science of chemistry has been expanded to such enormous proportions that it has long since become impossible for any one mind to have more than an elementary knowledge of all the different branches. It is therefore manifestly impossible for any one, however broad one's interests and however great one's desire for information, to read with any great degree of understanding all of the more technical articles appearing even in these journals. For the author to so popularize his articles that such a reader can not only understand but can enjoy the perusal thereof would necessitate his pruning out the more technical and, therefore, the most important, portions of his contributions.

The very excellent method of printing adopted by the *Journal of the American Chemical Society*, involving the use of different sized type, emphasizes the more important matter and makes easy the perusal of technical papers by the casual reader.

Let technical papers be written by the specialist for the specialist. "Padding," which is easily recognizable, should

be condemned, but let scientific observations be recorded with special attention to detail so that, should occasion arise, work described can be repeated and processes portrayed can be reproduced with accuracy.

To record *results* is not sufficient, though to the non-specialist they are of prime interest. The specialist wants to know *how* the results were got and he demands that the details be presented with such a degree of elaboration that he himself can duplicate the results. Otherwise, he can arrive at like results only after experimentation and investigation to discover the methods.

Who is interested in an analytical method except the man who expects to use that method? Or who becomes enthusiastic over the derivation of some complex chemico-mathematical formula except the man who is himself employing such a formula, either as an end in itself, or as a means to an end?

Let him who craves popularized science read those journals devoted thereto, and let him who wants the bare results of chemical research read the *Abstract Journal of the American Chemical Society*. But let us have journals devoted to the sort of publications which will enable the specialist and the investigator to find expert knowledge first hand and in detail.

J. W. TURRENTINE.

THE DETERMINATION OF PHOSPHORIC ACID BY THE OFFICIAL VOLUMETRIC METHOD.

The Official Volumetric Method for the determination of phosphoric acid is very popular among chemists who have a large amount of work to do on account of its rapidity and ease of manipulation. Some, however, admit that they cannot use it with any satisfaction, and cling to the old "magnesia process." Others use it for low percentages and rough factory tests, and check frequently with the gravimetric method.

The writer has had about fifteen years' experience with the method, and has observed the work of many different chemists during the time; the result of this experience shows conclusively that the method is not entirely reliable under all conditions. In the first place, the process as outlined in the revised Bulletin of "Official Methods of Analysis" is faulty. Here the chemist has not only the option of making the solution by several different methods, but he is at liberty to precipitate with the molybdic solution either at 65 or 45° or even in the cold, and any one who has had experience knows that if other conditions are the same, these different temperatures will have a marked influence on the percentages of phosphoric acid obtained. There is considerable doubt whether the precipitate formed is always of the same composition even when great care is taken to have all the conditions just right; at any rate, it only takes some slight deviation from the accepted practice to materially affect the results. The presence of sulphuric acid tends to make the results high, and it is evident that if sulphuric acid is used as a solvent, or even the varying quantities which are present in fertilizers will have some slight influence on the accuracy of the determinations. High temperatures give high results, and low temperatures low results; the presence of ammonium nitrate in varying quantities also has an appreciable influence. Other conditions might be mentioned, but these are sufficient to illustrate the point; that is, the method is too delicate for the average analyst who turns out a large number of determinations. There is no use in attempting to follow the printed directions and expect accurate results. The only way to obtain good work is for each analyst to take some standard samples of known composition and adjust the working conditions in such a manner that correct results may be obtained. It will be necessary for every chemist to determine these conditions for himself, and carefully adhere to the same routine in his future work. If this plan is followed, a good analyst should be able to do fairly accurate work, but even then there will

occasionally be results which cannot be accounted for. In my laboratory, there is never a set of phosphoric acid determinations made by this process without carrying along a check sample; this serves as a safeguard to indicate any erratic condition which may have crept in. In addition to this, frequent gravimetric checks are made. No important analyses involving the settlement of large contracts are ever entrusted to the volumetric process.

It is not intended to imply that it is impossible to get accurate results by this method, but to point out some of its weak points, and to show how ill adapted it is for strictly accurate work under the usual working conditions of commercial or state inspection laboratories. While, therefore, the method is useful for certain classes of work, it is unfortunate that it was adopted as an "Official Method," by the A. O. A. C. to be used at the option of the chemist as the basis of settlement in the analysis of large and important purchases of material, or for the testing of fertilizers by our Agricultural Departments where slight deviations from the guarantee involve severe penalties, or otherwise affect the business of the manufacturer by publishing the results in bulletins which are distributed broadcast to the consumers.   F. B. CARPENTER.

UNIFORM ANALYTICAL METHODS FOR PHOSPHATE ROCK.[1]

As the discrepancies between the results of chemists in the analysis of phosphate rock have often been the cause of disputes between buyer and seller, sometimes resulting in costly litigation and usually ending in a feeling of diminished confidence by the manufacturer for his chemist, the National Fertilizer Association, at their annual meeting at Atlantic City in 1908, appointed a committee to examine the various analytical methods for phosphate rock then in use, with a view to the adoption of methods which would yield uniform results.

That year four sets of phosphate rock samples were prepared, consisting of Tennessee brown, Tennessee blue, Florida and South Carolina rocks. These were distributed among the chemists of the National Fertilizer Association and the commercial chemists who make a specialty of fertilizer analyses. Each chemist was instructed to determine moisture, phosphoric acid and iron and aluminum oxides, using the routine methods of his laboratory, and to return a complete report of his results, together with a copy of his methods. Samples were sent to approximately fifty, and reports were received from thirty-one.

The results of this work were published in detail in pamphlet form by the Association.[2] A study of the tables of results confirmed the existence of serious differences in the results of various laboratories, which could not be laid to differences in samples. The samples had been prepared with extreme care, and were as nearly uniform as it was possible to make them.

In the report of the Committee to the National Fertilizer Association, made in October that year, it was tentatively recommended that for referee work the Official gravimetric method be used for phosphoric acid and that in the methods for iron and alumina then in use, the solvent be hydrochloric acid (1–1) until the question of the effect of pyritic iron in acidulating could be definitely solved.

In April, 1909, another sample of phosphate rock was distributed together with a set of methods, which were based on a study of the results of the previous year and on comments and suggestions of the coöperating chemists. The sample was a mixture of Tennessee brown and blue rocks, and of

Florida pebble. This sample was prepared with every possible precaution to ensure uniformity, and as in the previous samples, was to be used without further grinding or handling, having been ground to the requisite fineness during the preparation of the sample.

Samples and instructions were sent to approximately fifty chemists, but reports were so slow in coming in that at the time of the meeting of the National Fertilizer Association at Atlantic City, July 6th and 7th, the committee was able to make only a preliminary report on the work done this year. This report was published in the August number of THIS JOURNAL, in 1909. A few additional results have been received since, but they caused no improvement in the general character of this work. In all, reports were received from thirty-one chemists.

It is to be regretted that the results obtained this year do not show a more decided improvement over those of last year than is the case. The committee does not believe that the point has been reached where methods to be used as official in referee work can be definitely recommended.

An interesting point shown in the results received, is that those analysts who are chiefly and solely engaged in referee work agree very closely and that the greatest variations occur in the results of some of the chemists employed in the laboratories of the various manufacturers or retained by them. It is difficult to conceive how such differences can exist as shown, for instance, in the determination of moisture, considering that it is apparently one of the simplest determinations a chemist has to make. Yet some of the worst discrepancies are to be found in the results reported on this determination.

The committee could undoubtedly have chosen chemists for this work whose results would have been quite uniform, but the idea of this work is to arrive at methods which shall be so clear and so definite that any analyst should obtain results agreeing very closely with those obtained by any other analyst using the same methods.

A final report to the National Fertilizer Association has not been made as yet. Regarding the methods to be considered, there seems to be no reason for changing the proposed method for moisture as outlined in the instructions sent out this year. Regarding the phosphoric acid, nothing has developed to change the tentative recommendation of the Committee in favor of the Official gravimetric method including a proper method of solution of phosphate rock. While the results by the volumetric method show a marked improvement over those of last year, it does not seem advisable to recommend it in preference to the gravimetric method or even as an alternative method for referee work. The Wagner citrate method and others proposed do not seem to offer any advantages over the Official gravimetric method. Regarding the determination of iron and aluminum oxides, the committee tentatively leans toward the caustic potash method. The great irregularity of the results received by the acetate method seems to offset the good agreement by those accustomed to using it. Objection has been made to the apparently complicated character of the caustic potash method, but this is not founded on fact. The determination of iron by the permanganate method is certainly not a difficult nor complicated one, and the number of solutions required need not confuse any competent analyst. The most serious objection to the caustic potash method seems to be that a correction is required for the alumina, silica, etc., but the objection to blank determinations holds good in many other methods, as for instance in the determination of nitrogen.

The committee believes that this work should be taken over by the Division of Fertilizer Chemists of the American Chemical Society and wishes to urge upon the members of the Division

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[1] Read before the Division of Fertilizer Chemists, Boston meeting, American Chemical Society.

[2] THIS JOURNAL, **1**, 41.

the great need of immediate and effective coöperative work on this subject. A large stock of the samples which were distributed this year is still on hand and should prove of the greatest value to the coöperators in this most important work. As stated in the preliminary report on the work of this year, the committee believes that standard methods alone are not sufficient to ensure uniformity. It is evident that considerable familiarity and experience with any method is required before dependable results can be obtained by it. For this reason it seems that standard methods should be accompanied by standard samples so that every chemist interested can obtain them for the purpose of verifying his work from time to time.

C. F. HAGEDORN.

BOOK REVIEWS AND NOTICES.

Beet Sugar Making and Its Chemical Control. By Y. NIKAIDO, B.Sc., M.A. Easton, Pa.: The Chemical Publishing Co., 1909. Octavo, xii + 354 pp., 65 illustrations. $3.00 net.

The author states that this book is intended as an aid to those engaging in beet sugar manufacture, who lack systematic technical training in the matter.

This the author undertakes to supply in an unusual manner in a series of chapters on general chemistry and sugar house processes, both manufacturing and analytical, which view the entire field broadly and specifically. After a chapter on definitions of chemical terms follows one on the non-metallic elements. The novel method of presentation is instanced here, where under "Nitrogen" we find a discussion of ammonia in beet juice and the difficulties arising therefrom; under "Chlorine" directions for preparation of normal hydrochloric acid and other normal solutions, acidimetry, alkalimetry, etc.

Chapter III, in similar manner, deals with the principal metallic elements, giving various collateral information about lime and iron and an analytical method for the determination of copper. Then follow chapters on Organic Chemistry (30 pages) and Cane Sugar (17 pages) setting forth the classification of organic compounds and giving the properties of sugar and its compounds met with in beet sugar manufacture.

The well-arranged chapter on the "Polariscope and Its Accessories" is accompanied by numerous well-designed illustrations, and treats the subject in a clear, comprehensive manner. Other laboratory apparatus is discussed and directions given for determination of sucrose, total solids and purity.

Chapter VIII deals with the "Practical Operations of the Beet Sugar House" and describes in considerable detail the operations, from the harvesting of the beet to the last process in making white sugar and the recovery of sugar from molasses. This chapter, giving the limits within which good work should be conducted at the various stations, will be found of considerable value to those engaged in this work. A final chapter on "Special Analysis" (39 pages) takes up all the analytical determinations ordinarily needed, not already described, with formulae for calculation of results and many examples to make the matter plain.

An Appendix contains a few tables most commonly used in sugar work.

In viewing the book broadly it is a clever compilation of material from a dozen or more authoritative treatises on various subjects so brought together as to bear most closely on beet-sugar making, considered chemically.

The book is essentially a mixture that one could wish to see sorted out more thoroughly so that all the analytical matter would appear together and so be more directly accessible. It is a question whether the theoretical part is not largely over the heads of those who have had no previous technical training, but it is all very suggestive and will doubtless prove a grateful reminder to those already conversant with the subject.

W. D. HORNE.

INDUSTRIAL AND TRADE NOTES.

Air-Nitrates in Germany.—Consul-General A. M. Thackara, of Berlin, answers as follows the queries of an American correspondent regarding the manufacture of air-nitrates and the status of farming in Germany:

Atmospheric nitrogen is utilized in making nitrates for fertilizing purposes, in accordance with two general systems in Germany, as well as in Norway, Italy, and other European countries, and in Canada. (1) The formation of the so-called calcareous nitrogen (kalckstickstoff), which has the chemical formula Ca(CN)N, and which is formed by passing nitrogen over heated calcium carbide or through a heated mixture of lime and charcoal, and (2) the direct combination of the elements in the air—oxygen and nitrogen—by the use of the electric spark and the formation of nitrate by bringing these combinations into contact with the proper calcium or other components.

There are several processes by means of which the nitrogen, which combines with the calcium carbide in the formation of calcareous nitrogen, is separated from the oxygen of the air. One is by passing air over the copper coils by means of which the oxygen is removed. Another process is to obtain free nitrogen by the partial evaporation of liquefied air. The great expense attending this latter process renders it impracticable, and the nitrogen so obtained is still much mixed with oxygen compounds. Nitrogen is also obtained by one German firm in Hamburg and Hanover by cooling the gases of combustion and removing the impurities by passing the gases through retorts filled with copper and copper oxides and then through some substance that absorbs the carbon dioxide.

The so-called calcareous nitrogen obtained by the various processes is a grayish substance containing about 20 per cent. nitrogen. As a fertilizer it is generally supposed to be as efficient as ammonium sulphate and but little inferior to saltpeter. The exact effect of the cyanide in the compound is as yet not understood. By heating, the whole of the nitrogen in the compound is changed into ammonia, from which ammonium sulphate may be formed. The practicability of the production of calcareous nitrogen depends upon cheap power supply, and hence the plants are generally located where water power is available. The substance is produced in many different countries and the estimated total production for 1908 was about 45,000 tons.

The second system by which nitrates are obtained by the direct combination of the oxygen and nitrogen of the air is much more recent in its development than the above. After numerous attempts had been made in various countries, the Norwegian inventors, Birkeland and Eyde, finally succeeded in making the process industrially practicable (described in a monograph on "Manufacture of Air-Nitrates" recently published by the Bureau of Manufactures). According to their system, the end of each of the electric poles through which a current is passing is exposed to the action of the magnet which causes the electric spark to spread out into a disk-like flame. This flame is surrounded by some material that resists combustion,

thus leaving. a disk-shaped enclosure through which the atmosphere is sucked. As the temperature at which this reaction takes place is very high and as the atmosphere passes comparatively rapidly through the flame, only from 1 to 2 per cent. of the elements in the air is transformed. The compounds formed begin to decompose when their temperature is lowered, and therefore methods are devised to rapidly decrease their temperature to the point at which they can form more stable products. For this purpose the compounds are passed through an evaporation apparatus, and after their temperature has been reduced to about 50 degrees Réaumur they are passed into an oxidation chamber and oxidized into nitrogen dioxide, from which the nitrates are made. The farthest advance in this system has been made in Norway, principally by the firm of Birkeland & Eyde, and in factories located at Notodden, Svälgfos, and Christiansand. A similar system in Germany is used at the "Badische Anilin- und Soda-Fabrik" at Ludwigshafen am Rhein. The nitrates so formed are quite pure and have thus far been consumed principally in the industries, only a small quantity having been used as fertilizers.

COST OF PRODUCTION—WORKS OF REFERENCE.

The cost and the volume of production of each system depend upon the availability of cheap power, and the production of calcareous nitrogen also depends upon the prices at which lime and coal or charcoal are available. One estimate places the cost of the production of calcareous nitrogen containing 20 per cent. nitrogen at 270 to 315 francs ($52.11 to $60.79) per metric ton (2,204.6 pounds). This would make the nitrogen in the compound cost 26 to 30 cents a kilo (2.2 pounds), while the cost of the nitrogen in compounds formed by the direct combination of the elements in the air is generally conceded to be less. The experiments made with fertilizers of either system, in comparison with Chilean saltpeter, are generally favorable to the artificial product. In sandy soil the calcium nitrate formed by the direct combination of the elements in the air brought even better results than the Chilean saltpeter.

A description and illustrations of the mechanical devices in use in the electrical system of producing atmospheric nitrogen is published by one Witt in Das neue Technisch-Chemische Institut for 1906, which may be procured for 2 marks ($0.476) at the Weidmanns'che Buchhandlung, Zimmer-Str. 94, Berlin, and a cursory description of the various processes in use in both systems, as well as the numbers of the various patents covering the same, is given in *Stahl und Eisen* for May 19, 1909, published in Dusseldorf. Copies of the German patents may be procured at 1 to 2 marks each ($0.238 to $0.476) through an American or a German patent attorney.

Another book giving an exhaustive description of the technical and industrial application of atmospheric nitrogen is issued by Doctors Donath and Frenzel, of the Technical High School at Brunn, and can be obtained from the book-dealer A. Seydel, Königgrätzer-Str. 31, Berlin. The price of this book is 7 marks ($1.67).

Nitrates in Norway.—Supplementing previous articles in Consular and Trade Reports on the manufacture of air nitrates for fertilizer in Norway, Germany, and Niagara Falls, Canada, it is learned from British consular reports that the industry is undergoing rapid expansion in the first-named country, where nearly $15,000,000 will be invested. Though there are vast water powers in the United States running to waste, which could be utilized to produce this article, nothing has yet been done in that line, although this country is buying annually about $15,000,000 worth of Chilean nitrates. The British consul's report from Christiania reads:

"Up till now about $6,000,000 has been expended on the works at Notodden and Svaelgfos and the power stations under construction at Rjukan and Vamma. When all the works

are completed, at the end of 1910, $14,600,000 will have been spent. A great point in connection with the development of this industry is that the opportunity has now arisen of opening up several industries in connection with the manufacture of nitrates, such as nitric acid, nitrate of ammonia, nitrate of potash, also sodium nitrate, which last is already being manufactured.

The Nobel syndicate, in conjunction with the Birkeland and Eyde Company, is now concentrating the weak acids, with the assistance of the gas furnaces, to an acid of such percentage as to become an article of transport, and further opportunities have thus been opened for export trade, especially from works with water power that are situated near the seaboard.

It is of interest to note that no coal is used in the production of saltpeter or other products here referred to. It is stated authoritatively that there is no probability for many years to come that the sale of saltpeter produced by the method practiced at the Notodden and Rjukanfos works will be disturbed by competition with Chile saltpeter on the question of price.

LARGE ANNUAL PRODUCTION.

When the Rujukan Falls works are fully completed, they and the Notodden works combined will represent 240,000 horse-power, with a production of saltpeter representing an export value of $6,164,000.

At some not very far distant time it is not improbable that the waterways and loughs between Skien and Notodden may be increased to such size as to allow sea-going vessels to load up at Notodden. Plans have already been worked out and are under consideration in connection therewith. At present everything has to be lightered to and from Skien.

The value of the output of nitrates in Norway in 1908 was about $536,000, and the total expenses amounted to $402,000.

The following are the companies that are producing, or will shortly produce, saltpeter from the air:

(1) The Norsk Hydro Elektrisk Kvaelstof Aktieselskab, Christiania, who are the owners of the Notodden Saltpeter-fabrikker, the power of which (35,000 h. p.) is supplied from the Svaelgfos. The capita* of the company, which is French, is $7,890,000.

(2) The Vammafos (Vamma Falls) Company, whose saltpeter works are now under construction. This company is a separate company, but half of its shares are said to be owned by the Norsk Hydro Elektrisk Kvaelstof Aktieselskab; some of the preference shares are in the hands of Norwegians. The amount of the capital can not be stated.

(3) The Rjukanfos (Rjukan Falls) Company, whose saltpeter works are also in course of erection. The capital of the company, which is one half French and the other half Scandinavian and German, is $3,376,800. This capital has been furnished by some of the shareholders of the Norsk Hydro Elektrisk Kvaelstof Aktieselskab and of the Kraft Aktieselskab, but the company is an independent one.

(4) The Matre and Tyin waterfalls are owned by the Norsk Hydro Elektrisk Kvaelstof Aktieselskab and by the Kraft Aktieselskab, but the companies formed in connection with these falls are two separate companies with separate administrations. Electro-technical works will probably be erected at both of these falls.

(5) Christiansands Elektrokemiske Aktieselskab, which is the property of the Badische Anilin- und Soda-Fabrik, Ludwigshafen, and has a capital of $53,600.

Chilean Nitrate Situation.—Consul Alfred A. Winslow, writing from Valparaiso, under date of November 30th, reviews the conditions of the nitrate mining industry in Chile:

The nitrate industry in Chile is still in very bad shape, with no prospects of any immediate relief. The price of nitrate is very low, being only $1.60 United States gold per quintal,

against $2.13 in 1907, when the industry was flourishing. The different interests are cutting prices until many of the works have shut down because they can not produce nitrate at the present prices. There has been no concerted action since the combine was dissolved last April, and the outlook is not bright for this most important industry for Chile. This question is receiving much attention on the part of the National Government, and the President of Chile has just published a decree constituting a commission composed of the director of the division of nitrate in the Treasury Department, the president of the Sociedad Nacional de Mineria, the professor of nitrate subjects in the National University, the Santiago delegate of the Nitrate Propaganda Association, the Assistant Secretary of the Treasury, and five members with a secretary, to be named by the President of Chile, which is to make a thorough study of the perplexing problems involved in the present unsatisfactory conditions of the nitrate business.

This commission is instructed to report to the Secretary of the Treasury on what means can best be employed to reduce the cost of production, on an organization for the sale of nitrate, on a systematic propaganda in the interest of an increased consumption of nitrate, and on a definite plan for the systematic location of claims in the nitrate fields, together with other matters that may present themselves.

For the first 10 months of 1909 there was an increased exportation of 2,415,396 quintals of nitrate over the same period of 1908, but at a great loss in receipts, which has cut down the profits to very small margins in all cases where they have not been entirely wiped out.

CONSUMPTION AND PRODUCTION.

A review of the Chilean nitrate trade by a British firm contains the following statistics:

The world's consumption for the past twelve months at 1,929,00 tons, against 1,732,000 tons in 1908, is highly satisfactory, showing, as it does, an increase of 11½ per cent., or 196,000 tons. Of this quantity the European Continent accounts for 1,354,000 tons against 1,275,000, an increase of 6 per cent. An increase of 7¾ per cent. is also shown by the United Kingdom, the figures being 111,000 tons, against 103,000. Consumption in the United States has not only recovered lost ground, but has beaten the previous best record of 355,000 tons in 1906 by 12 per cent. The figures are 398,000 tons, against 309,000, an increase of 29 per cent. Other countries account for 66,000 tons, against 45,000 tons, or an increase of 47 per cent. Production for the eleven months ended 30th November, at 41,106,660 quintals, against 39,236,640, shows an increase of 1,870,000 quintals, or 84,700 tons. Shipments for the twelve months to all parts amount to 2,085,000 tons, against 2,017,000, an increase of 68,000 tons.

Trade in Antimony.—As the new tariff law of the United States raised the duties on antimony in order to encourage its home production, interest attaches to the following report from Consular Agent Ernest L. Phillips, of St. Helens, on its manufacture in England mainly from foreign ores:

Basil Valentine said: "The shortness of life makes it impossible for one man thoroughly to learn antimony, in which every day something new is discovered." The crude ore stibnite or gray antimony was formerly mined at several places in Great Britain, but latterly this country receives most of its supplies from China, Australia, Chile, and Nova Scotia.

The process of extracting the antimony from the ore is a simple matter, but there are several processes employed. The crude and commercial antimony, the bulk of which used throughout the world comes from China and Japan, is the ore separated from associated earthy gangue, and this operation is performed by a simple fusion. From this there are two or three ways of obtaining the regulus of antimony. The raw antimony,

whether obtained direct from the ore or from the purified sulphide, must be calcined, in order to separate such impurities as arsenic, sulphur, etc. In the process of getting it direct, the ore is smelted together with some alkaline slag and old scrap iron. When this combination has completely fused, it is poured into conically shaped molds, and this mixture, after cooling, consists of impure antimony and sulphide of iron.

There are several methods used for purifying the regulus. One of the simplest is to charge each of the number of crucibles with this regulus along with soda, common salt, and pure oxidized antimony ore. When sufficient heat has been applied, the foreign impurities or metals become oxidized and scorified, and the star metal is thus obtained. Antimony is a bright silver gray metal; it is brittle and can be easily pulverized; it is valuable for the alloys it yields with other metals; the britannia metals, type metal, stereotype plates, Babbitt's antifriction metal, and shot, all contain a certain proportion of this valuable commodity.

The exports of star antimony metal from St. Helens to the United States during 1908 amounted to $40,419.

The importation of antimony metal into the United States during fiscal years ended June 30th has been as follows:

Country.	1904. Pounds.	1905. Pounds.	1906. Pounds.
Austria-Hungary	....	133,243	794,663
France	111,224	220,736	521,719
United Kingdom	2,124,660	3,070,522	4,521,397
Other countries	1,694,995	1,098,780	1,253,539
Total	3,930,879	4,523,281	7,091,318
Total value	205,908	312,332	795,398

Country.	1907. Pounds.	1908. Pounds.	1909. Pounds.
Austria-Hungary	1,156,027	459,564	67,427
France	422,322	1,116,225	1,108,189
United Kingdom	5,263,717	5,615,168	6,651,934
Other countries	1,968,131	855,159	1,703,217
Total	8,810,197	8,046,116	9,530,767
Total value	1,945,362	672,934	644,734

The unit value of the imports in each year per pound was as follows: 1904, 5.2 cents; 1905, 6.9 cents; 1906, 11.2 cents; 1907, 21.7 cents; 1908, 8.4 cents; 1909, 6.8 cents.

The imports of antimony ore averaged about 2,000,000 pounds for the fiscal years 1904, 1905 and 1906, while for 1907 it amounted to 3,053,082 pounds, but dropped to 1,684,774 pounds in 1908. However, in 1909 the imports increased to 6,021,877 pounds. The average value of the ore fluctuated heavily, in keeping with the price of the regulus or metal. The unit value of the ore imports in 1908 was 5.4 cents per pound. About one-half the ore in 1908 was secured from China, and about one-third *via* England.

Until the new Payne tariff act antimony ore was admitted into the Untied States free of duty, while three-fourths of 1 cent a pound was charged on the regulus or metal. The new customs law taxes the ore at 1 cent per pound, and the metal at 1½ cents per pound.

The Geological Survey states that the production of antimony in the United States during the years 1904 to 1908 has been as follows:

Year.	Short tons.	Value.
1904	3,057	505,524
1905	3,240	705,787
1906	1,766	602,949
1907	351	77,300
1908	Nil.	Nil.

A limited mining of antimony ore has been carried on in Nevada, though with the high prices in 1906–7, when the best grade of the metal reached 28 cents a pound, operations were

begun in Washington, Idaho, Utah, California, and Oregon. Many gold and silver ores are antimony-bearing, and in smelting the antimony combines with the lead of the charge, the product going on the market as antimonial lead. The alloy is used in type metal, babbitt, coffin trimmings, linings for acid tanks, toys, clock cases, and other articles for which a fairly hard metal to be gilded or otherwise finished is desired. Other uses of antimony are described by the Geological Survey:

Antimony is also used as an adulterant in solder and is considered a very objectionable element, especially when the solder is to be used for sealing cans holding provisions. Some antimony compounds are very poisonous, especially the organic compounds. The use of antimony salts and oxides is comparatively large, as shown by the importations (585,389 pounds worth $105,234) in 1908. Antimony trioxide is used as a pigment in place of white lead, zinc oxide, etc., and seems to be gaining ground, especially in Europe. The trioxide is also used for making the glass used to coat iron in enameled ware, as a reducing agent in chemical work, and as a detector of alkaloids and phenols. Antimony arsenate, arsenite, trichloride, iodide, trioxide, trisulphide, pentasulphide, tartrate and antimony and potassium tartrate are used in medicine. The trichloride, also called antimony butter, is used in bronzing iron, especially gun barrels, as a mordant for patent leather, in coloring zinc black, as a mordant for silver, and in the manufacture of lakes particularly from dyewoods.

The double salts, antimony fluoride and ammonium sulphide, antimony and potassium oxalate, and antimony and potassium tartrate (tartar emetic) are used in dyeing.

The trisulphide is used in pyrotechnics for making "Bengal fire;" the pentasulphide, or golden sulphide, in coloring rubber goods; the antimonate in coloring glass yellow; vitreous antimony sulphide made by roasting antimony sulphide, then fusing the product with fresh antimony sulphide, is also used to tint glass yellow, and for a like purpose with porcelain. The chromate, under the name of "Naples yellow," is used as a fine artist's color. Naples yellow and some of the oxides are used as ceramic colors.

Antimony in the form of metal and oxide is used in making flint glass. About $1\frac{1}{4}$ pounds of each per ton of "batch" is used.

New Potash Discoveries.—As the United States has been purchasing one-sixth of Germany's annual export of about $35,000,000 worth of potash, the following report from Consul Talbot J. Albert, of Brunswick, will be of interest:

Hitherto it has been believed that Germany was the only country in the world where deposits of potassium salts existed, and had therefore a safe monopoly of them. Now, however, comes the startling news in the press of the discovery of potassium deposits in other countries. The *Brunswick Landes-Zeitung* says:

It is already an established fact that potassium salts occur in Galatia, Hungary, Tyrol, Russia, Holland, Florida and Persia. (The United States Geological Survey has not yet been able to locate the reported Florida deposits, concerning which several press notices have appeared.—B. of M.) The latest German discoveries are located in Alsace, close to the French boundary, and the potassium deposits there will probably extend across into France, as has been the case with the salt wells.

There still comes the surprising information that potassium salts have been found in the vast Empire of China. Although this news might at first seem incredible, it has been a well-known fact for some time, but kept secret by interested parties. Samples of these salts have been tested by a chemist connected with the Potassium Mining Syndicate, and the results have given a large percentage of chloride of potassium (Chlorkalium).

All particulars, especially the localities of the discoveries in China, are naturally withheld by the combine. Should the mining of the potassium salts in China be successful, the western part of America will first come in question as a consumer. The salts could be delivered there very cheaply, as the wages of labor in Chinese mines is one-tenth of those in German mines, and especially the cheap ocean freight to the west coast of America will be an important factor.

World's Sugar Crop.—Consul-General Henry W. Diederich, of Antwerp, has prepared, under date of January 8th, the following review of the sugar production of the world for the 1909–'10 season:

As usual, various approximate beet-sugar crop estimates have been issued from time to time during recent months, by sugar factories and others interested in the production of sugar in Europe. The unusually wet and cold summer had proved very unfavorable to the development of sugar beets in most countries of Europe, but fairer skies in autumn helped considerably to improve both the quality and quantity of beets. However, the sugar contents fall much behind that of last season, amounting in Belgium to only 15 per cent., whereas in 1908 they reached the high average of 17 per cent.

The season is completely finished in Belgium, and practically so in the other countries, so that only slight variations, one way or the other, in the present approximate estimates of the beet-sugar crop, may be looked for when the final actual results are recorded at the end of the year on August 31, 1910. The International Association for Statistics, therefore, after having issued approximate statements of the sugar crop in October and November, has just completed its third and last report, at the end of December, after having made another very careful inquiry, and now publishes the following total results for 1909–'10 of the sugar-beet fields in Europe:

Country.	Beets.	Sugar.
Germany	12,846,560	1,985,460
Austria	8,100,200	1,259,100
France	6,354,900	807,500
Russia	6,888,860	1,144,150
Belgium	1,765,000	240,900
Holland	1,291,000	175,500
Sweden	894,350	125,300
Denmark	488,000	65,000
Italy[1]	1,000,000	116,000
Spain[1]	830,000	99,300
Roumania[1]	203,000	28,000
Servia[1]	63,000	8,400
Switzerland[1]	25,000	3,100
Bulgaria[1]	28,000	3,300
Total	40,777,870	6,061,010

Mr. F. O. Licht, the veteran German sugar statistician, whose work I have closely followed for a number of years and found as unerring as human effort can attain, publishes the following crop estimates, based upon his own close observations and modified by his experience of a lifetime:

Country.	1909–'10. Tons.	1908–'09. Tons.	1907–'08. Tons.
Germany	2,005,000	2,066,779	2,116,595
Austria	1,250,000	1,370,453	1,397,260
France	810,000	783,904	711,541
Russia	1,175,000	1,274,659	1,415,115
Belgium	240,000	256,442	230,797
Holland	180,000	214,343	175,184
Sweden	125,000	133,383	109,012
Denmark	65,000	66,298	52,661
Italy	116,000	165,312	135,965
Spain	100,000	119,387	123,523
Roumania	25,000	22,600	25,832
Others	18,000	15,955	16,357
Total	6,109,000	6,488,915	6,509,842

[1] Unofficial estimates of the other beet-sugar-producing countries in Europe which do not belong to the International Association.

Country.	1906–'07. Tons.	1905–'06. Tons.
Germany	2,225,021	2,403,805
Austria	1,315,686	1,478,737
France	740,291	1,065,469
Russia	1,458,823	987,474
Belgium	280,928	326,962
Holland	181,407	207,189
Sweden	157,956	125,300
Denmark	62,275	64,369
Italy	106,383	93,916
Spain	98,085	96,206
Roumania	32,000	31,444
Others	14,240	7,150
Total	6,673,095	6,888,021

(The later statistics issued by Licht on January 21st, according to Willett & Gray's bulletin, gives the 1909–'10 crop estimate at 6,185,000 tons, being 35,000 higher for Germany, 10,000 higher for Austria, 15,000 higher for France, 10,000 higher for Belgium, 20,000 higher for Holland, and 25,000 tons higher for Russia, with a less amount for "all others."—B. of M.)

As the first decade of this century has just come to its close it is interesting to study the following table and note what has been accomplished in Europe during that period (hectare = 2.47 acres):

Season.	Area (hectares).	Beets. Tons.	Pounds per acre.	Sugar. Tons.	Pounds per acre.
1909–'10	1,832,590	40,496,720	20,003	6,109,000	3,009
1908–'09	1,808,158	41,270,021	20,704	6,488,905	3,252
1907–'08	1,872,419	42,510,724	20,303	6,509,842	3,116
1906–'07	1,821,473	45,275,316	21,440	6,673,095	3,270
1905–'06	1,871,229	48,267,128	23,003	6,888,021	3,294
1904–'05	1,610,800	31,962,027	17,695	6,667,980	2,583
1903–'04	1,755,692	40,311,209	20,479	5,860,991	2,999
1902–'03	1,786,964	38,732,557	19,326	5,530,961	2,792
1901–'02	1,962,685	49,546,329	22,511	6,722,051	3,057
1900–'01	1,842,142	42,466,828	20,550	5,970,070	2,869

CANE SUGAR RESULTS.

It is extremely difficult at present, even approximately, to give the crop estimates of cane sugar which is being produced

	1909–'10. Tons.	1908–'09. Tons.	1907–'08. Tons.	1906–'07. Tons.
Cuba	1,800,000	1,514,000	963,000	1,431,000
Porto Rico	270,000	222,000	190,000	181,000
Haiti and Dominican Republic	60,000	75,000	54,000	58,000
Jamaica	7,000	5,000	13,000	14,000
Barbados	30,000	12,000	33,000	35,000
Trinidad	45,000	41,000	39,000	40,000
Martinique	36,000	38,000	38,000	38,000
Lesser Antilles	45,000	40,000	40,000	45,000
Antigua and St. Kitts	25,000	25,000	23,000	30,000
Guadeloupe	35,000	25,000	36,000	39,000
St. Croix	13,000	12,000	12,000	10,000
Brazil	275,000	280,000	195,000	250,000
Argentina	130,000	162,000	110,000	119,000
Peru	130,000	150,000	135,000	169,000
Venezuela	3,000	3,000	3,000	3,000
Demerara	100,000	116,000	99,000	116,000
Surinam	12,000	12,000	12,000	9,000
Central America	50,000	50,000	50,000	50,000
Mexico	130,000	115,000	113,000	105,000
Egypt	45,000	40,000	42,000	65,000
Natal	54,000	40,000	41,000	36,000
Mauritius	225,000	188,000	165,000	214,000
Reunion	45,000	39,000	39,000	43,000
Fiji Island	70,000	65,000	68,000	40,000
Formosa	160,000	121,000	71,000	65,000
Java	1,150,000	1,187,000	1,244,000	1,115,000
Other East Indies	2,120,000	1,927,000	2,141,000	2,450,000
Queensland	140,000	151,000	188,000	175,000
New South Wales	15,000	15,000	29,000	23,000
Philippines	115,000	108,000	132,000	111,000
Hawaii	475,000	448,000	486,000	383,000
Louisiana	330,000	320,000	330,000	230,000
American home-grown beet	420,000	386,000	455,000	435,000
Total	8,560,000	7,932,000	7,595,000	8,127,000

in many countries all over the globe outside of Europe, and which is in various stages of growth and maturity. No one can tell to-day what quantities of sugar will be garnered from the cane fields of the East and West Indies, the two leading cane-producing countries. But for all practical purposes the above estimates may be safely made.

Finally, what has become of the world's sugar crop during the past four years; how much of it was left at the end of each year, after satisfying the demands of trade? What the results are likely to be during the present year, ending August, 1910, may be learned from the following table, showing actual returns of the world's sugar trade since 1905:

	1909–'10. Tons.	1908–'09. Tons.	1907–'08. Tons.
Initial stock September 1st	1,650,000	2,002,000	2,014,000
Production of beet sugar	6,109,000	6,494,000	6,510,000
Production of cane sugar	8,500,000	7,932,000	7,610,000
Total	16,259,000	16,428,000	16,134,000
Balance on hand August 31st	1,600,000	1,650,000	2,002,000
World's consumption	14,659,000	14,788,000	14,132,000

	1906–'07. Tons.	1905–'06. Tons.
Initial stock September 1st	1,810,000	1,767,000
Production of beet sugar	6,673,000	6,888,000
Production of cane sugar	8,127,000	7,119,000
Total	16,610,000	15,774,000
Balance on hand August 31st	2,014,000	1,809,000
World's consumption	14,596,000	13,965,0

Thus it will be seen that the production of beet sugar has steadily grown less during recent years, while that of the cane has increased to such an extent as not only to cover the shortage in beet sugar, but also to add to the market a sufficient quantity to supply the world's demand for sugar, with a limited surplus at the end of the season, scarcely enough to keep the world supplied for six weeks. It also may be interesting to learn the fact that of the average annual world's sugar crop, about two-thirds, or 10,000,000 tons are used in Europe, the United States, and Canada, while the other third is absorbed by the rest of the world.

Production of Radium.—AUSTRIA: Consul Will L. Lowrie, of Carlsbad, transmits the following translation of an article which recently appeared in the Vienna Neue Frei Presse concerning the production and sale of radium in Austria:

At the Ministry of Public Works many conferences have been held lately to discuss the place where radium may be sold and to arrange details of putting the mineral on the market. The quantity to be disposed of consists of the recent shipment from Joachimsthal which was sent for experimental purposes, and which has been examined to determine the amount of pure radium it may contain. Already it is certain that the ore sent from Joachimsthal will yield not less than the amount of pure radium expected—1 gram. As has been said, this will be sold and the price is fixed at 380 crowns ($77.14) a milligram. Persons or institutions which contemplate purchasing radium may have their addresses noted at the Ministry of Public Works, and already, before the official selling has begun, many applications have been filed, not only from the Empire but also from foreign countries. Many hospitals are among the applicants. Special means for packing the radium have been adopted owing to the danger from the rays. Since this shipment from Joachimsthal, a like amount of radium has been produced at the Imperial factory located at that place. It is hinted that if the chemical works at Joachimsthal are carried on in an adequate manner a number of other extremely valuable radioactive substances beside radium, as actinium and jonium, may be obtained, which in some regards, as, for instance, in the power of throwing out rays, may surpass radium.

ENGLAND: The following information concerning the establishment of a factory in London for the manufacture of radium according to a secret process discovered by Sir William Ramsay is taken from a London newspaper:

It is claimed that by this process the production of radium will be reduced from nine months to six or seven weeks, and the British Radium Corporation has been formed for taking over the radium rights of the Trentwith mine at St. Ives, where the discovery of pitchblende ore was made (previously referred to in Consular and Trade Reports). The services of Sir William Ramsay have been retained by the corporation.

The total quantity of radium which has thus far been recovered for scientific use throughout the world is estimated not to exceed one-fourth of a pound. The total stock in the London Hospital is but about $16^{1}/_{4}$ milligrams.

A little while ago the pitchblende containing this wonderful element was actually regarded as a nuisance by the old miners of St. Ives, so that it was cast aside on the rubbish heap, a heap which grew to mountainous proportions, and from which to-day the company is obtaining tons of material.

American Exposition at Berlin.—POSTPONED FOR ONE YEAR: Special Agent Julien L. Brodé writes from Berlin, under date of February 7th, on the progress and prospects of the forthcoming American exposition in the German Imperial capital, the date of which has been changed since this report was written to 1911:

It is announced that William C. Brumder, of Milwaukee, Wis., one of the proprietors of the Germania-Abend Post there, has been appointed by the Department of State as honorary commissioner-general to the American Exposition at Berlin. Four honorary commissioners have also been selected, namely, Herman A. Metz, until recently comptroller of the city of New York; John Ball Osborne, chief of Bureau of Trade Relations at Washington; Irwin M. Laughlin, secretary of the United States Embassy at Berlin; and Hugh Gordon Miller, an attorney of New York, who was New York Commissioner to the Jamestown Exposition at Norfolk.

Former Comptroller Metz, of New York City, chairman of the American executive committee of the Berlin Exposition, gave out the following statement on February 28th:

In view of the apparent opposition which has developed in Germany against the American Exposition to be held in Berlin during the coming summer, the American executive committee has decided upon a postponement until 1911.

The members of the executive committee, and the gentlemen who agreed to serve on the advisory and other committees, did so for the purpose of showing their good will toward Germany and German interests and to promote the commercial relations between the two countries and in anticipation of similar coöperation on the part of the commercial interests of Germany and for no other reason.

As apparently this is not entirely understood, the members of the executive committee are of the opinion that it might be advisable to make the exposition a German-American affair in which both countries should participate, and thus prove it is not intended to be an American industrial invasion. With this object in view the majority of the members of the executive committee will visit Berlin in the coming summer for the purpose of bringing about, if possible, such a German-American exposition, and the United States Government has been requested to continue the honorary commission which was appointed to represent it at the exposition this summer.

OFFICIAL REGULATIONS AND RULINGS.

Only a few of the more important rulings are reported here. For others the reader is referred to the publications of the Board of Food and Drug Inspection and to Treasury Decisions.

NOTICE OF JUDGMENT No. 147, FOOD AND DRUGS ACT.
Adulteration and Misbranding of Lemon Extract.

In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 10th day of November, 1908, in the District Court of the United States for the Eastern District of Virginia, in a prosecution by the United States against the Suffolk Drug and Extract Company, a corporation of Suffolk, Va., for violation of Section 2 of the aforesaid act in shipping and delivering for shipment from Virginia to North Carolina an adulterated and misbranded lemon extract, the said Suffolk Drug and Extract Company entered a plea of guilty and the Court imposed a fine of $25.

The facts in the case were as follows:

On August 24, 1907, an inspector of the Department of Agriculture purchased from J. J. Medford, Oxford, North Carolina, a sample (I. S. No. 2094) of a food product labeled: "Pure Brand Extract Lemon. Artificial Coloring. Manufactured by Suffolk Drug & Extract Co., Inc., Suffolk, Va." The sample was analyzed in the Bureau of Chemistry of the United States Department of Agriculture and the following results obtained and stated:

Alcohol by volume (per cent.)	44.00
Lemon oil (per cent.)	0.18
Citral	0.02
Solids	0.10
Color: artificial color declared.	

Lemon extract, or flavor, as recognized by reliable manufacturers and dealers, is the flavoring extract prepared from oil of lemon, or from lemon peel, or both, and contains not less than five per cent. by volume of oil of lemon. The analysis of the aforesaid sample disclosed that there was only 0.18 per cent. of oil of lemon in this article, hence it was adulterated within the meaning of Section 7 of the act in that an inferior extract, artificially colored in a manner whereby its inferiority was concealed, was substituted wholly for the genuine article which it purported to be, and was misbranded within the meaning of Section 8 of the act in that it was labeled "Extract Lemon," which statement was false, misleading and deceptive because it was not lemon extract, but a very poor imitation thereof.

It appearing from the aforesaid analysis that the article was adulterated and misbranded, the Secretary of Agriculture gave notice to J. J. Medford, the dealer from whom the sample was procured, and also to the Suffolk Drug and Extract Company, the manufacturer and shipper, and gave them an opportunity to be heard. The said company being the party solely responsible for the adulteration and misbranding of the article and failing to show any fault or error in the result of the aforesaid analysis, and it being determined that the article was adulterated and misbranded, on July 2, 1908, the said Secretary reported the facts and evidence (F. & D. No. 133) to the Attorney-General, by whom they were referred to the United States Attorney for the Eastern District of Virginia, who presented the facts to the grand jury, by whom an indictment was duly returned against the said Suffolk Drug and Extract Company with the result hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.

January 15, 1910.

NOTICE OF JUDGMENT No. 152, FOOD AND DRUGS ACT.
Adulteration and Misbranding of Pineapple and Lemon Extracts.

In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on June 8, 1909, in the District Court of the United States for the Southern Division of the Southern District of Alabama, in a prosecution by the United States against the Mobile Drug Company, a corporation of Mobile, Alabama, for two violations of Section 2 of the aforesaid act in shipping and delivering for shipment from Alabama to Mississippi adul-

terated and misbranded pineapple and lemon extracts, the said Mobile Drug Company entered a plea of guilty and the Court imposed upon it a fine of $25.

The facts in the case were as follows:

On February 22, 1908, and March 13, 1908, an inspector of the Department of Agriculture purchased from Mrs. Sophia Fields, Escatawpa, Mississippi, and R. W. Fagan & Company, Waynesboro, Mississippi, respectively, samples (I. S. Nos. 5051 and 2126-a) of food products labeled, respectively: "Pure Concentrated Extract of Pineapple for Flavoring Ice Cream, Custards, Jellies, Pastry, etc. Mobile Drug Co., Mobile, Ala.," and "Concentrated Extract of Lemon. Guaranteed ——— June 30, 1906. Serial No. 6901. Mobile Drug Co., Mobile, Ala." The samples were analyzed in the Bureau of Chemistry of the United States Department of Agriculture and the following results obtained and stated:

I. S. No. 5051:

Specific gravity	0.9447
Alcohol by volume (per cent.)	45.40
Esters as ethyl butyrate (grams per 100 cc.)	0.696
Coloring matter	Turmeric

I. S. No. 2126-a:

Specific gravity (15.5° C.)	0.8748
Alcohol by volume (per cent.)	73.28
Solids (grams per 100 cc.)	0.21
Lemon oil by polarization (per cent.)	3.22
Lemon oil by precipitation (per cent.)	3.30
Color	Turmeric

A flavoring extract, as recognized by reliable manufacturers and dealers, is a solution in ethyl alcohol of proper strength of the sapid and odorous principles derived from an aromatic plant, or parts of the plant, with or without its coloring matter, and conforms in name to the plant used in its preparation; and lemon extract, or flavor, as recognized by reliable manufacturers and dealers, is the flavoring extract prepared from oil of lemon, or from lemon peel, or both, and contains not less than five per cent. by volume of oil of lemon. The analysis of sample I. S. No. 5051 disclosed practically the total absence of the sapid and odorous principles of the pineapple, hence the article was adulterated within the meaning of Section 7 of the act in that an imitation extract, artificially colored in a manner whereby its inferiority was concealed, was substituted wholly for the pineapple extract which it purported to be, and was misbranded within the meaning of Section 8 of the act in that it was labeled "Pure Concentrated Extract of Pineapple," which statement was false, misleading and deceptive because it was not an extract of pineapple, but merely an imitation extract. The analysis of sample I. S. No. 2126-a disclosed the fact that it contained only 3.3 per cent. of oil of lemon and was artificially colored, hence the article was adulterated within the meaning of Section 7 of the act in that an inferior extract, artificially colored in a manner whereby its inferiority was concealed, was substituted wholly for the genuine product which it purported to be, and was misbranded within the meaning of Section 8 of the act in that it was labeled "Concentrated Extract of Lemon," which statement was false, misleading, and deceptive because it was not concentrated extract of lemon, but an inferior grade lemon extract, artificially colored in a manner whereby its inferiority was concealed.

It appearing from the aforesaid analyses that the articles were adulterated and misbranded, the Secretary of Agriculture gave notice to Mrs. Sophia Fields and R. W. Fagan & Company, the dealers from whom the samples were purchased, and also to the Mobile Drug Company, the manufacturer and shipper, and gave them an opportunity to be heard. The Mobile Drug Company being the party solely responsible for the adulteration and misbranding of the articles and failing to show any fault or error in the results of the aforesaid analyses and it being determined that the articles were adulterated

and misbranded, on January 6, 1909, and February 27, 1909, respectively, the said Secretary reported the facts and evidence (F. & D. Nos. 306 and 486) to the Attorney-General, by whom they were referred to the United States Attorney for the Southern District of Alabama, who filed an information against the Mobile Drug Company with the result hereinbefore stated.

JAMES WILSON,
January 15, 1910. *Secretary of Agriculture.*

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(Notice of Judgment No. 175, Food and Drugs Act.) *Adulteration of Water.*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 19th day of August, 1909, in the Police Court of the District of Columbia, judgment was entered in the case of the United States against Frank H. Finley, trading as F. H. Finley & Sons, a prosecution upon an information in substance charging said defendant with having sold and offered for sale in the District of Columbia 24 bottles of a liquid substance intended for use as drink by man, labeled "Diamond Distilled Water," which was adulterated in that it was injurious and deleterious to health and contained poisonous and deleterious ingredients, and consisted wholly or in part of filthy, decomposed, and putrid vegetable substances. The defendant pleaded not guilty to the above information, and the case was tried on August 4 and 5, 1909. After hearing the testimony and arguments of counsel, the court found the defendant guilty as alleged in the information and imposed upon him a fine of $10.

The facts upon which the above prosecution was based were as follows:

An inspector of the United States Department of Agriculture purchased a sample of the water labeled as above described from F. H. Finley & Sons, 208 Massachusetts Avenue, N. E., Washington, D. C. This sample was analyzed in the Bureau of Chemistry of the United States Department of Agriculture and found to contain the colon group of organisms. The analysis having disclosed an adulteration of said water, F. H. Finley & Sons were duly notified of the results obtained, and were given an opportunity to be heard and were heard in regard to said adulteration. It appearing that there had been a violation of the act, the Secretary of Agriculture, on May 19, 1909, reported the facts to the Attorney-General. The case was then referred to the United States Attorney for the District of Columbia, who filed the above information, with the result hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.
WASHINGTON, D. C., January 28, 1910.

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(Notice of Judgment No. 179, Food and Drugs Act.) *Adulteration and Misbranding of Cottonseed Feed Meal.*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 18th day of June, 1909, in the District Court of the United States for the Western District of North Carolina, judgment was rendered in the case of the United States v. 120 Sacks of Cottonseed Feed Meal, wherein a libel was under Section 10 of the aforesaid act, alleging in substance that 120 sacks of a product designated as cottonseed feed meal and labeled: "Creamo Brand Feed Meal. Manufactured by the Tennessee Fibre Co., Memphis, Tenn. Guaranteed Analysis—Protein 22%, Fat 5%, Crude Fibre 28%," which had been shipped from Memphis, Tenn., to Asheville, N. C., and there found in original unbroken packages, were misbranded in that the label on said sacks represents the product to contain protein 22 per cent., fat 5 per cent., crude fibre 28 per cent., when in truth and in fact but 18.73 per cent. of protein, 4.69 per cent. fat, and 25.04 per cent. crude fibre, and approximately 50 per cent. cottonseed

hulls were present in said product, and were adulterated in that the said contents were designated as cottonseed feed meal with intent to deceive and mislead the purchaser as to its quality.

The libel prayed process against all claimants to said feed meal, and seizure and condemnation of the same.

The Asheville Grocery Company appeared as respondent in the above proceedings, whereupon the court, after parties had agreed to make statements of their evidence, found for the libelant and rendered the following decree:

IN THE DISTRICT COURT OF THE UNITED STATES, WESTERN
    DISTRICT OF NORTH CAROLINA. AT ASHEVILLE.

UNITED STATES OF AMERICA   ⎞
                vs.   ⎟
120 SACKS OF COTTON SEED FEED MEAL. ⎠
        DECREE OF CONDEMNATION.

This cause coming on to be heard, and it appearing to the Court that upon the Libel filed herein, Warrant of Arrest was duly issued and served on the 7th day of June, 1909, and that by virtue of said Warrant, the Marshal has seized and now holds 116 sacks of Cotton Seed Feed Meal of the approximate value of $200.00 the said 116 Sacks of Cotton Seed Feed Meal, having been seized from the premises and in the possession of the Asheville Grocery Co. a partnership formed and doing business in the city of Asheville, N. C., within the said District and that the said Cotton Seed Feed Meal is now in storage and in the custody of the said Marshal; and it appearing that the Asheville Grocery Co. the respondent herein the owners of the said 116 bags of Cotton Seed Feed Meal, so seized were duly warned to appear herein and that due and legal notice and proclamation was given to all persons having or claiming to have any right, title or interest therein or in or to said property, to appear and answer said libel, and that said Asheville Grocery Co. have so appeared; the libelant and respondent each making a statement to the Court of their evidence and agreeing in open court to submit the same to the court and the court being now fully advised in the premises, finds for the libelant and finds that the said 116 sacks of Cotton Seed Feed Meal contains articles of food and that the said sacks are misbranded within the meaning of the Act of Congress of June 30, 1906, the same having been transported in interstate commerce from the City of Memphis, Tenn., consigned to the Asheville Grocery Co., at Asheville, N. C., being all of such consignment found in original unbroken packages, that is, the Court finds that the said articles of feed are misbranded in violation of the said Act of Congress in that said sacks and each of them contain protein 18.73 per cent., fat 4.69 per cent., crude fibre 25.04 per cent. and contain approximately 50 per cent. hulls, and that the said articles of feed were transported in interstate commerce and consigned and delivered to the claimant aforesaid, Wholesale dealers at Asheville, N. C.

The Court further finds that the articles of feed contained in said 116 sacks of Cotton Seed Feed Meal is not adulterated, poisonous or deleterious, but that the violation of said Act of Congress is in the misbranding of the said sacks and that the same were consigned only to a wholesale dealer and not sold to the public for consumption.

Wherefore, it is Ordered, adjudged and decreed by the court that the said 116 sacks of Cotton Seed Feed Meal with the contents as aforesaid, be and they are hereby declared to be misbranded in violation of the Act of June 30th, 1906, as is charged in said libel, and it is further ordered that the said 116 sacks of Cotton Seed Feed Meal, with the contents as aforesaid, be, and they hereby are condemned and forfeited as provided for in the said Act of June 30, 1906. It is provided, however, that upon the payment of all the cost in the proceeding herein including all Court, Clerk's and Marshal's costs and costs of hauling, storage, watchmen and all other costs incident to or contracted in this proceeding, and the execution and delivery

by the said Asheville Grocery Co. to the libelant of a good and sufficient bond in the penalty of $250.00, conditioned that the said sacks of Cotton Seed Feed Meal, with the contents as aforesaid shall not be sold, or otherwise disposed of, contrary to the provisions of the said Act of June 30, 1906, or to the laws of any state, territory, district, or insular possession, that the said Marshal shall redeliver the said 116 sacks of Cotton Seed Feed Meal with such of their contents as they now contain or may contain at the time of such redelivery to the Asheville Grocery Co. in lieu of the retention and destruction thereof; The Clerk of this court will attach the costs in accordance with this order and furnish a copy thereof to claimants.

This June 18, 1909.

                    WM. T. NEWMAN,
                        U. S. Judge presiding.

We consent to this decree:
    THOS. J. RICKMAN, *Atty. for claimant.*
    A. L. COBLE, *Asst. U. S. Atty.*

The facts preceding the filing of the above libel were as follows:
On or about June 4, 1909, an inspector of the United States Department of Agriculture found in the possession of the Asheville Grocery Company, in original unbroken packages, 120 sacks of product labeled as above described, which had been sold, invoiced, and shipped as Cotton Seed Feed Meal to the said company by the Tennessee Fibre Company, of Memphis, Tenn. A sample taken from the above consignment was analyzed in the Bureau of Chemistry of the United States Department of Agriculture and found to contain protein 18.73 per cent., fat 4.69 per cent., crude fibre 25.04 per cent., and hulls, approximately, 50 per cent. The misbranding and adulteration disclosed by this analysis was reported by the Secretary of Agriculture, on June 5, 1909, to the United States Attorney for the Western District of North Carolina, who filed the above libel, with the result hereinbefore stated.

                    JAMES WILSON,
                        Secretary of Agriculture.
WASHINGTON, D. C., January 28, 1910.

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(Notice of Judgment No. 180, Food and Drugs Act.) *Misbranding of a Drug—"Gowan's Pneumonia Cure."*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on December 16, 1908, in the District Court of the United States for the Northern District of Illinois, a judgment was rendered in the case of the United States *v.* Gowan Medical Company, a corporation organized and existing under the laws of the State of North Carolina and transacting business through a branch office at Chicago, Ill., a prosecution upon an information in substance charging said defendant corporation with having delivered to the Baltimore and Ohio Railroad Company, at Chicago, Ill., for shipment to Washington, D. C., four cases of a certain drug called "Gowan's Pneumonia Cure," which was misbranded in the following particulars, *viz:*

1. On a green circular enclosed in the carton and surrounding each of the bottles containing this drug, and thereby made a part of the labels descriptive of the said preparation, occurred this statement: "It is entirely different from any other remedy containing new principles never before applied; consequently, it cannot be substituted;" which said statement was then and there false and misleading in this, that all the ingredients in said preparation were and are well and commonly known and are constantly applied, singly or in combination, in the very manner directed by the instructions accompanying this preparation, and commonly used for the affections of the lungs, throat, and other portions of the body similarly affected.

2. On a green circular enclosed in the carton and surrounding each of the bottles containing this drug, and thereby made a part of the labels descriptive of said preparation, occurred

this statement: "Supplies an easily absorbed food for the lungs that quickly effects a permanent cure;" which statement was false and misleading in this, that there is no such thing as a food for the lungs separate and apart from a food that nourishes the whole body.

3. On a white circular also enclosed in the carton and surrounding each of the bottles containing the drug, and thereby made a part of the labels descriptive of said preparation, occurred this statement: "It was endorsed and advertisement accepted by the *American Medical Journal*, as a valuable therapeutic agent;" which statement was false and misleading in this, that the said preparation was never advertised in the *American Medical Journal* and was never endorsed by the said *American Medical Journal*.

The information charged a further misbranding in that the labels printed upon the cartons containing the bottles filled with this preparation did not bear a statement of the quantity of opium contained in said preparation in a manner that could be easily read by the purchaser; but the statement of the amount of opium contained therein was printed in inconspicuous type in such an inconspicuous place that the proper notice of the poisonous contents of said preparation was not easily conveyed to the purchaser or person to whom it might be transferred.

On the aforesaid date, the defendant pleaded guilty to the above information and was fined $200. The facts on which the prosecution was based were as follows:

On November 22, 1907, an inspector of the United States Department of Agriculture purchased from the Washington Wholesale Drug Exchange a sample of a drug preparation labeled and branded as above described, which had been shipped to said Exchange by the Gowan Medical Company from Chicago, Ill., on or about November 18, 1907. This sample was analyzed in the Bureau of Chemistry of the United States Department of Agriculture and was found to consist of stearin, lard, turpentine, camphor, phenol, quinine sulphate, opium, and a trace of quinine alkaloid. The analysis having disclosed a misbranding of the drug, the said Washington Wholesale Drug Exchange and the said Gowan Medical Company were duly notified of the charges and were given an opportunity to be heard and were heard in regard to said misbranding. It appearing that there had been a violation of the act, the Secretary of Agriculture, on May 28, 1908, reported the facts to the Attorney-General. The case was then referred to the United States Attorney for the Northern District of Illinois, who filed the above information, with the result hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.
WASHINGTON, D. C., January 28, 1910.

(Notice of Judgment No. 181, Food and Drugs Act.) *Misbranding of a Drug—"Eyelin."*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 21st day of December, 1908, in the District Court of the United States for the Northern District of Illinois, a judgment was rendered in the case of the United States against the Eyelin Company, a corporation of Chicago, Ill., a prosecution upon an information in substance charging said defendant corporation with having delivered to the United States post-office at Chicago, Ill., for shipment to Washington, D. C., a quantity of a drug preparation contained in a circular tin box, upon one side of which were printed the following words;

One Dollar Repairs and Rejuvenates
Trade Mark EYELIN Registered
The Eye and Sight
The EYELIN CO., Chicago, U. S. A.

and upon the other side of which were printed directions for the use of said drug, said box being surrounded by a circular

entitled: "How to Use Eyelin and Your Eyes," which said tin box and surrounding circular were placed in a blue pasteboard box, upon the front of which were placed the following printed words:

Reshapes and Rejuvenates,
Trade Mark EYELIN Registered,
The Eye and Sight.

which said drug was misbranded in the following particulars:

(*a*) The label printed upon one face of the tin box containing said drug contained this statement: "Repairs and Rejuvenates the Eye and Sight," which said statement was false and misleading in this, that the perfumed vaseline enclosed in the circular tin box aforesaid, and constituting said preparation, had no properties capable of repairing the eye and the sight; and

(*b*) The label upon the blue pasteboard box in which the circular tin box containing the drug and the circular were placed contained this statement: "Reshapes and Rejuvenates the Eye and Sight," which statement was false and misleading in this, that the perfumed vaseline enclosed in the said tin box, constituting the preparation, had no properties capable of reshaping and rejuvenating the eye and sight.

On December 16, 1908, the defendant pleaded guilty to the information and the court imposed upon it a fine of $10.

The facts upon which the above prosecution was based were as follows:

On or about February 13, 1908, an inspector of the United States Department of Agriculture purchased from the Eyelin Company, No. 1403 Washington Boulevard, Chicago, Ill., a box of the drug preparation heretofore described, the same being delivered to him at Washington, D. C., through the United States mail. This sample was analyzed in the Bureau of Chemistry, United States Department of Agriculture, and found to consist essentially of perfumed or flavored vaseline, wherefore it was deemed misbranded, and the said Eyelin Company was duly notified of the charge and given an opportunity to be heard, and was heard in regard to said misbranding.

It appearing that there had been a violation of the act, the facts were reported to the Attorney-General on June 25, 1908, by the Secretary of Agriculture. The case was referred to the United States Attorney for the Northern District of Illinois, who filed an information against the Eyelin Company, with the result hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.
WASHINGTON, D. C., January 28, 1910.

(Notice of Judgment No. 182, Food and Drugs Act.) *Misbranding of a Drug—"Bromo Febrin."*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 29th day of October, 1909, in the District Court of the United States for the District of Maryland, judgment was rendered in the case of the United States *v.* William H. Smaw, trading as W. H. Smaw & Company, a prosecution upon an information in substance charging said defendant with having shipped from Baltimore, Md., to Detroit, Mich., one dozen packages of a drug called "Bromo Febrin," which was misbranded in this, that the packages containing the same failed to bear a statement of the quantity or proportion of acetanilid contained therein; and which was further misbranded in that packages bore the following statement: "Each Powder contains 4 Grains of Acetanilid," which said statement was false and misleading for the reason that said powders contained more than 4 grains of acetanilid, *viz.*, nearly 6 grains; and which was further misbranded in this, that said packages bore the statement "Sure Cure for Headache and Neuralgia," which statement was then and there false and misleading because the article in said packages

was not a sure cure for headache and neuralgia; and which was further misbranded in this, that said packages bore this statement "Permanent in Results," which statement was false and misleading for the reason that the article contained therein was not permanent in results; and which was further misbranded in this, that the packages containing the same bore this statement "It is Absolutely Safe," which said statement was false and misleading because said drug was not absolutely safe.

The defendant pleaded guilty to the information on the aforesaid date, and the court imposed a fine upon him of $20.

The facts on which the above prosecution was based were as follows:

On or about February 1, 1909, an inspector of the United States Department of Agriculture purchased from the Michigan Drug Company, of Detroit, Mich., a sample of the drug heretofore described, which was contained in a consignment shipped to said dealers from Baltimore, Md., by William H. Smaw, trading as W. H. Smaw & Company. This sample was analyzed in the Bureau of Chemistry of the United States Department of Agriculture, and each powder found to contain nearly 6 grains of acetanilid. The analysis having disclosed a misbranding of the drug, the Michigan Drug Company, and the said William H. Smaw, were duly notified thereof and were given an opportunity to be heard, and were heard in regard to said misbranding.

It appearing that there had been a violation of the act, the Secretary of Agriculture, on July 31, 1909, reported the facts to the Attorney-General. The case was thereupon referred to the United States Attorney for the district of Maryland, who filed an information against the said William H. Smaw, with the result hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.
WASHINGTON, D. C., January 28, 1910.

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(Notice of Judgment No. 183, Food and Drugs Act.) *Adulteration and Misbranding of Powdered Colocynth.*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 7th day of October, 1909, in the District Court of the United States for the District of Maryland, judgment was rendered in the case of the United States *v.* Gilpin, Langdon & Company, Inc., a prosecution upon an information in substance charging said defendant corporation with having shipped from Baltimore, Md., to Cincinnati Ohio, 3 pounds of a certain drug, which was then and there adulterated, in that it was then and there sold under a name, to wit, "Powdered Colocynth," recognized in the United States Pharmacopoeia and National Formulary, and then and there differed from the standard of strength, quality, and purity as determined by the test laid down therein, in this, that the standard of strength, quality, and purity as determined by said test requires that in colocynth ready for use the seeds should be separated and rejected, whereas, the colocynth shipped as aforesaid contained a mixture of pulp and seeds; and which said drug was misbranded in this, that the packages containing the same then and there bore a statement regarding the ingredients and the substances contained therein which was false and misleading for the reason that it represented said drug as consisting of powdered colocynth, whereas, in truth and in fact, said contents consisted of powdered colocynth mixed with a quantity of seeds.

On the aforesaid date the defendant pleaded guilty to the information, and the court imposed upon it a fine of $25.

The facts on which the above prosecution was based were as follows:

On or about August 11, 1908, an inspector of the United States Department of Agriculture purchased a sample of the drug heretofore described from Theodore Rosenthal, Eighth and

Vine streets, Cincinnati, Ohio, which sample was contained in a consignment of said drug shipped to said dealer from Baltimore, Md., by Gilpin, Langdon & Company. This sample was analyzed in the Bureau of Chemistry of the United States Department of Agriculture and found to consist of the entire fruit, pulp, and seeds ground. The analysis having disclosed an adulteration and a misbranding of the drug, the said Theodore Rosenthal and the said Gilpin, Langdon & Company were duly notified of said charges, and were given an opportunity to be heard, and were heard, in regard to said adulteration and misbranding; whereupon, it having appeared that there had been a violation of the act, the Secretary of Agriculture, on July 28, 1909, reported the facts to the Attorney-General; the case was then referred to the United States Attorney for the District of Maryland, who filed an information against the said Gilpin, Langdon & Company, Inc., with the results hereinbefore stated.

JAMES WILSON,
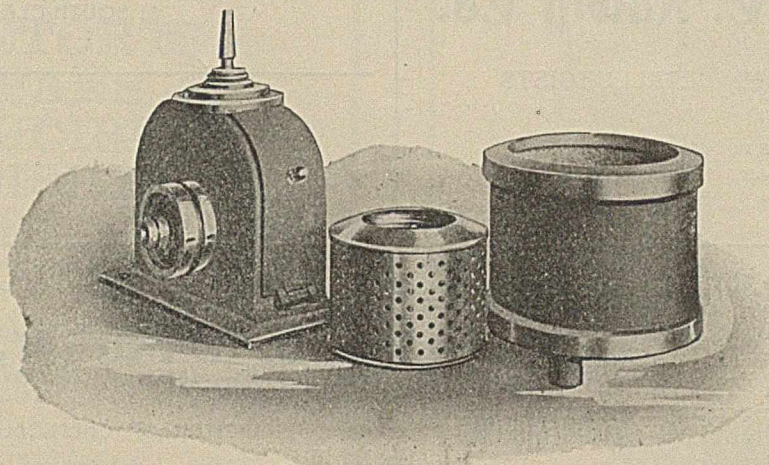*Secretary of Agriculture.*
WASHINGTON, D. C., January 28, 1910.

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(Notice of Judgment No. 184, Food and Drugs Act.) *Misbranding of a Drug—"Radol."*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on February 3, 1909, and October 1, 1909, respectively, judgments were rendered in two cases of the United States against Dennis Rupert Dupuis, both prosecutions upon informations identical in terms except as to the dates of the shipments alleged. The first information, in one count thereof, in substance charged the defendant with having shipped from St. Louis, Mo., to Washington, D. C., a certain bottle then and there labeled "This bottle contains Radol (Registered Trade Mark), a radium impregnated fluid prepared according to the formula and under the supervision of Dr. Rupert Wells, St. Louis, Missouri. This fluid is not expected to retain its radioactivity beyond forty days from the date of this label. For External use. Name, E. G. Henson. Address, Washington, D. C. Date Feb. 22, 1908. Directions: To be used as directed by letter. Signature, D. R. Wells, M.D. Keep this bottle in a dark cool place;" the contents of which said bottle were misbranded in that the statements upon the label were false and misleading for the reason that said bottle contained a liquid which was not radium impregnated and which had no radioactivity beyond that of ordinary water, and further that the name Dr. Rupert Wells was a false and fictitious name, and that said fluid was not prepared according to the formula of a Dr. Rupert Wells or under his supervision; and in the second count thereof charged said defendant with having shipped from St. Louis, Mo., to Washington, D. C., a certain bottle then and there labeled "This bottle contains Radol (Registered Trade Mark), a radium impregnated fluid prepared according to the formula and under the supervision of Dr. Rupert Wells of St. Louis, Missouri. This fluid is not expected to retain its radioactivity beyond forty days from date of this label. For internal use. Name, E. G. Henson. Address, Washington, D. C. Date, Feb. 22, 1908. Directions: Take one tablespoonful in a wine glass of water before each meal and at bed-time. Signature, D. R. Wells, M.D. Keep this bottle in a cool dark place;" the contents of which said bottle were misbranded in that the label was false and misleading in the following particulars: The said liquid was not radium impregnated and had no radioactivity, and further in that the name Dr. Rupert Wells was a false and fictitious name and that said fluid was not prepared according to the formula of a Dr. Rupert Wells, or under the supervision of a Dr. Rupert Wells, and which was further misbranded in that the said liquid contained approximately 6.99 per cent. of alcohol, and that the label upon said bottle did not state the proportion of alcohol

contained in said liquid or that the liquid contained any alcohol.

The defendant pleaded guilty to both informations and on the dates aforesaid was fined $100 and $50, respectively.

The facts upon which the prosecutions were based were as follows:

An inspector of the United States Department of Agriculture, upon two occasions, purchased samples of the drug heretofore described from Dennis Rupert Dupuis, St. Louis, Mo. The samples were analyzed in the Bureau of Chemistry of the United States Department of Agriculture and found not to be radium impregnated liquids, not to have any radioactivity beyond that of ordinary water, and to contain approximately 6.99 per cent. of alcohol. The analyses having disclosed an apparent misbranding of the liquids, the said Dennis Rupert Dupuis was duly notified thereof in each case and given an opportunity to be heard, and was heard, in regard to said misbranding. Thereupon it appeared that there had been violations of the act, and the Secretary of Agriculture, on December 8, 1908, and April 23, 1909, reported the facts to the Attorney-General. The cases were referred to the United States Attorney for the Eastern District of Missouri, who filed the informations against the said Dennis Rupert Dupuis, with the results hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., February 7, 1910.

(Notice of Judgment No. 189, Food and Drugs Act.) *Adulteration and Misbranding of Vinegar.*—In accordance with the provisions of Section 4 of the Food and Drugs Act of June 30, 1906, and of regulation 6 of the rules and regulations for the enforcement of the act, notice is given that on the 27th day of August, 1909, in the District Court of the United States for the Eastern District of Michigan, in a prosecution by the United States against the Gordon Vinegar Company, a corporation of Pontiac, Mich., for violation of section of the aforesaid act in shipping and delivering for shipment from Michigan to Ohio an adulterated and misbranded vinegar, the said Gordon Vinegar Company entered a plea of *nolo contendere* and the court sentenced it to pay the cost of the case.

The facts in the case were as follows:

On February 18, 1909, an inspector of the Department of Agriculture purchased from the W. W. Harper Company, Zanesville, Ohio, a sample of a food product labeled: "Gordon Vinegar Co. 46. Apple Cider Vinegar. Fermented. Pontiac, Mich.," which was part of a shipment made by the Gordon Vinegar Company from Pontiac, Mich., to the said W. W. Harper Company on or about December 26, 1908. The sample was analyzed in the Bureau of Chemistry of the United States Department of Agriculture and following results obtained and stated:

Solids	1.91
Reducing sugar invert	1.16
Per cent. sugar in solids	60.8
Polarization, direct, temp. °C. 26	—2.6
Polarization, invert, temp. °C. 26	—2.6
Ash	0.26
Alk. sol. ash (cc. N/10 acid per 100 cc.)	29.1
Sol. phos. acid (mgs. per 100 cc.)	1.5

Insol. phos. acid. (mgs. per 100 cc.)	11.1
Acid, as acetic (wines tartaric)	4.64
Volatile acid, as acetic	4.64
Fixed acid, as malic (wines, tartaric)	0.0
Lead precipitate	Small.
Color, degrees, brewer's scale 0.5 in	4.0
Color removed by Fuller's earth (per cent.)	65.0
Ash in solids (per cent.)	13.9
Salicylates and benzoates	Negative.

$$\text{Ratio } \frac{P_2O_5 \text{ water sol.}}{P_2O_5 \text{ total}} \text{(per cent.)} \quad \quad 11.9$$

Vinegar, cider vinegar, apple vinegar, as recognized by reliable manufacturers and dealers, is the product made by the alcoholic and subsequent acetous fermentations of the juice of apples. The analysis of the aforesaid sample disclosed that it contained dilute acetic acid, or distilled vinegar, and a foreign material high in reducing sugars. Hence the article was adulterated within the meaning of Section 7 of the act in that a mixture of dilute acetic acid, or distilled vinegar, and a foreign material high in reducing sugars had been substituted wholly or in part for the vinegar which it purported to be, and was misbranded within the meaning of Section 8 of the act in that it was labeled "Apple Cider Vinegar," which statement was false, misleading, and deceptive because it was not an apple cider vinegar, but a mixture of dilute acetic acid, or distilled vinegar, and a foreign substance high in reducing sugars.

It appearing from the aforesaid analysis that the article was adulterated and misbranded, the Secretary of Agriculture gave notice to the W. W. Harper Company, the dealer from whom the sample was procured, and also to the Gordon Vinegar Company, the manufacturer and shipper, and gave them an opportunity to be heard. The Gordon Vinegar Company being the party solely responsible for the adulteration and misbranding of the article and failing to show any fault or error in the result of the aforesaid analysis, and it being determined that the article was adulterated and misbranded, on July 26, 1909, the said Secretary reported the facts and evidence to the Attorney-General, by whom they were referred to the United States Attorney for the Eastern District of Michigan, who filed an information against the Gordon Vinegar Company, with the result hereinbefore stated.

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., February 7, 1910.

CORRECTION.

My attention has been called to the fact that in my paper in the December number of THIS JOURNAL on the "Volumetric Estimation of Potassium" some error in reference occurred. On page 796 it was stated that the average of a number of results secured by Mr. W. A. Drushel was 104.2 per cent., and the individual results were given. This it proves is an error on my part; as a matter of fact the results of his Table I were secured by the use of the Adie and Wood method, hence should be excluded from the average. This makes a much better general showing for his method, and I am glad to take this occasion to rectify my mistake in the matter.

L. T. BOWSER.

PROFESSIONAL DIRECTORY