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United States Department of Agriculture,

BUREAU OF CHEMISTRY.

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A PLAN FOR COOPERATING IN THE STUDY OF AVAILABLE PLANT FOOD.

At the San Francisco meeting of the American Association of Agricultural Colleges and Experiment Stations in 1899, a committee on cooperative work between stations and the Department was appointed.

The report of this committee was most favorable, especially with reference to the desire for cooperation on the part of station workers. Among the suggestions from the committee is the following:

“A fourth type of cooperation is seen in those investigations which require the combined work of many stations, for example, an attempt to obtain definite information on the changes produced in plants by environment, and the value of this factor in the study of the varying effects of soil and climate in some single crop. Such experiments call for the work of many stations at the same time. It is manifest that the uniformity and efficiency of the work would require that such cooperation be arranged and supervised by some central organization; and it is equally manifest that the Department is best prepared to undertake the task.”

In accordance with this suggestion, the Bureau of Chemistry two years ago arranged for cooperative experiments as to the effect of environment on the chemical composition of plants, and it now desires to arrange a cooperative study of the methods for determining available plant food in soils, not only between the stations and the Department of Agriculture, but between the stations themselves and farmers who will be interested in the work.

The intention is to arrange a systematic plot study, in which the seeding of the plots, sampling of the soil, harvesting of the crop, and the chemical study of the soil will be the same in every detail, and will be carried out by each of the stations taking part in the work, there being a distribution of the material and results obtained from each station among all stations taking part. From the analysis of the crop will be determined the ability of the soil to supply plant food, while upon the soil sample, taken previous to planting, efforts will be made to secure chemically, as near as possible, the result obtained by the crops.

As a basis for analytical work, a method for the estimation of available P_2O_5 and K_2O is proposed by this Bureau. Detailed instruction

as to the use of this method is given, and every station is asked to follow it minutely. In addition to this, it is asked that other methods be suggested by the stations, which will be submitted to all taking part, and, upon the general approval of any one method, it will be used by each according to the detailed instructions as arranged by the author, it being understood that no one will offer a method for trial until he has worked it out to his own satisfaction. After the work above indicated has been completed, individual study on the soils can be taken up, and where the study gives promise of success and the method has been reduced to its simplest form, it can be sent to the other stations cooperating, with the expectation that it shall be used according to explicit directions.

If all proposed methods have been carefully worked over by their authors, it will require but little time for the stations to apply the methods and send in the results, which results will be distributed. In instances where the stations are too busy on other lines to make a specialty of the work, it will be sufficient for them to do only the proposed work, leaving it for such as are especially interested in this line to formulate and experiment on methods.

In this it may be seen that there is much room for individual effort upon collective material, while those who supply the collective material will have access to all data and proposed methods, with the privilege of suggesting and amending.

It is arranged to make a study of one soil with four kinds of crops, this Bureau undertaking to supply the seed and all apparatus as far as possible, analyze the crops, receive the soil samples and prepare and distribute the subsamples for analysis, in addition to running a control-pot of each plot.

The work in detail is as follows:

Select a plot 60 feet square on even, homogeneous soil—if possible, one which has never had any fertilizer, especially P_2O_5 nor K_2O . It is immaterial whether old or new ground is used, it being hoped that all kinds will appear in the collection of results. On this plot run lines from the centers of the opposite sides, as illustrated in the diagram (fig. 1).

This makes four plots of about 1-50 acre each. In the center, at the intersection of the lines, the soil sample is to be taken. In taking this sample, use the measure which will be sent for laying off the surface. This measure consists of a tape attached to three pegs, so as to form an equilateral triangle of 3 feet on the sides. After putting one peg in the ground, draw tight one side of the tape and drive in the second peg; then draw tight the two remaining sides of the tape and drive in the third peg. With a spade dig out the enclosed area to a depth of 9 inches, having the sides of the hole vertical. Place all of this soil in the sacks which will be sent,

being careful to have the pit cleaned down to an even depth of 9 inches. The sack should be labeled: "First nine inches," with the name of the station. Then obtain the second 9 inches in a similar manner, being careful to label accordingly. These sacks should be forwarded to the Department, where each sample will be weighed, thoroughly mixed, put through a 2-mm. sieve, subsampled, and distributed, about forty pounds being retained for the control-pot experiment. The moisture and basicity of all the samples will be determined and the figures supplied, so that the results will be on the same moisture-free basis, with the same correction for basic matter. These figures will be sent out with the samples.

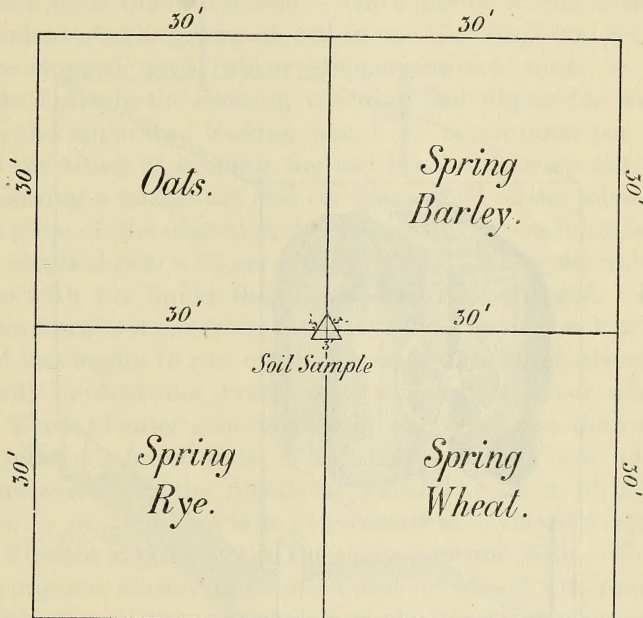


FIG. 1.—Experiment plot, 60 feet square, 1.50 acre in each small square.

In the seeding of the plots the soil is to be prepared by ordinary plowing and smooth harrowing. Packages containing the four kinds of seed will be supplied, and the entire contents of each package are to be sown broadcast as uniformly as possible and covered with a harrow. This will give a uniform rate of seeding with a uniform variety of seed. The plots will require no further attention until harvest time. During the interval, work on the soil samples can be progressing. When the crops are ready for harvesting use the triangular cord measure, which will be supplied, and lay off a definite area in the center of each of the four plots. This measure will be a cord with three pegs attached, so that when all sides are drawn tight it forms an equilateral triangle of 6.3 feet on each side,

equaling nearly 1-2500 of an acre. The method of procedure is the same as that prescribed for the soil sample, namely: Drive in one peg; stretch one side of the tape and drive in the second peg; then stretch the two remaining sides and drive in the third; cut the inclosed crop even with the ground, taking care to lose no grain by shattering. Place the total crop in the bag which is supplied, label carefully and forward to the Department, where it will be dried, weighed, ashed, and analyzed, and the material and analyses distributed, the whole being calculated to pounds per acre of the different parts of the crop removed.

One method for the determination of available P_2O_5 and K_2O ,

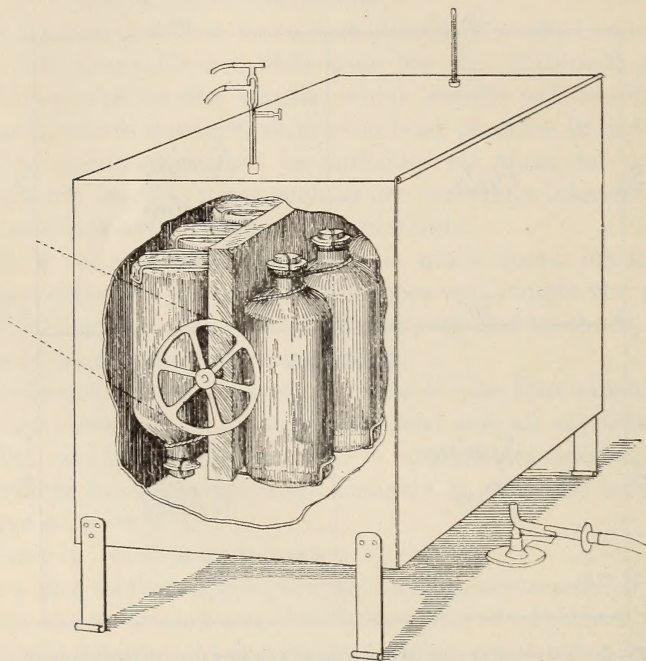


FIG. 2.—Shaking machine for soil extraction at constant temperature.

which this Bureau proposes, has been worked out in connection with a very elaborate research. It has been printed as a contribution from this Bureau in the *Journal of the American Chemical Society*, Vol. 24, page 79, and a reprint has been prepared for distribution.

A very simple and inexpensive form of shaking machine (fig. 2) has been designed for this work, which, with the accompanying flasks, will be supplied. By the use of this apparatus it is possible to have a uniform agitation and temperature throughout the work. The machine may be run by any motive power at hand at the speed of about 40 revolutions per minute. The temperature is kept at $40^{\circ}C$. by means of a flame under the air chamber, and owing to the

stirring of the air inside, and especially by use of a thermostat, the temperature can be kept within one-half degree of that specified.

The method of digestion in detail is as follows:

Weigh and place in one of the digestion flasks the quantity which will be specified in a supplementary table, this weight being based on the moisture found to be in the sample and equivalent of 186 grams of dry soil. Measure out the number of cubic centimeters of N-10 HCl, which will be given in the supplementary table, and dilute to 1 liter. This table is based upon the basicity of each soil sample. Half fill the flask containing the soil; shake with the diluted HCl thoroughly, so as to displace the air held in the soil, and then fill up to the neck with the dilute acid. The contents of the flask will be the equivalent of 200 grams of soil in one litre of N-200 HCl. The flasks are stopped with rubber stoppers, which must be tied in. Fasten the flasks in the shaking machine and digest for five hours at 40° C., the apparatus making about 40 revolutions per minute. This has the effect of keeping the soil constantly suspended in the liquid, insuring a maximum and constant effect of the solvent.

At the close of the digestion the flask with its contents is shaken well and emptied into a 32 cm. fluted filter. Unless the sediment is poured on with the liquid the filter will become clogged. The first part of the filtrate is generally cloudy and is poured back as soon as the liquid has begun to run clear. A small filter first placed in the funnel will prevent the breaking of the larger paper when it is loaded. These filtrates generally drain through in less than an hour, and the volume is found to be so near 800 c. c. that it is not necessary to measure. To the filtrate is added 2 or 3 c. c. of HNO₃ and about 10 c. c. of HCl, and it is evaporated to dryness in a porcelain dish 6 or 8 inches in diameter on the steam or water bath. The HNO₃ destroys organic matter in solution and drives off any ammonium chloride which may have resulted from the liberation of ammonia in the process of digestion, and which, if present, would subsequently interfere with the determination of potash. To the residue in the dish is now added strong HCl, at first slowly, to prevent loss by spurting. It is then evaporated until the nitrates have become completely converted into chlorides, which can be told by the absence of a greenish tinge and fumes upon the addition of HCl to the dry residue.

It sometimes requires from 50 to 100 c. c. of HCl before the conversion is completed. When complete, the solution is evaporated to dryness and cooled; then a few drops of HCl are added and about 20 or 30 c. c. of water, and the whole is warmed on the bath until solution is secured.

Filter to remove silica and wash until clean. To the filtrate, which corresponds to 160 grams of soil, in a 3 or 4-inch porcelain dish, add

about 1 c. c. of the ordinary platinic chloride solution and evaporate on the steam or water bath according to the usual method for determination of K_2O , being careful to trail the liquid around the side of the dish as it becomes concentrated and to remove just before dryness, so as to have the evaporation completed during the cooling of the dish. This procedure insures a very perfect formation of crystals of the double salt. To the dish is now added about 20 or 30 c. c. of acidulated alcohol. The preparation of this reagent will be described later. The dish is allowed to stand 3 or 4 minutes with an occasional stirring, then decanted into a 7 cm. filter. The salt is now washed into the filter with 90 per cent alcohol, as usual, until free of platinic chloride. Now wash with the ordinary Lindo solution of ammonium chloride (200 grams per liter of water), using about 20 c. c., and allow these washings to mix with the previous filtrate. The filtrate is stirred and set aside overnight to allow the ammonium platinum chloride to settle. The salt in the filter is then washed with 90 per cent alcohol until free of ammonium chloride. The filter is then dried and the contents washed through with boiling water into a previously weighed small platinum dish, which, after evaporation and drying in a bath for thirty minutes at $100^\circ C.$, is weighed. The calculated weight of the K_2O is to be divided by 160 grams. This is to be expressed in parts per million, a part per million being the equivalent of the fourth decimal place in percentage.

The acidulated alcohol is prepared by conducting HCl gas into cool 90 per cent alcohol until 1 c. c. of the alcohol neutralizes about $2\frac{1}{2}$ c. c. of normal alkali, using phenolphthalein as an indicator.

The method of acidifying is as follows:

Put 200 or 300 c. c. of strong HCl into a rather large flask, connect by means of a glass tube with a flask holding about one-half liter of 90 per cent alcohol, this flask being kept cool by surrounding with ice or running cold water. The tube containing the gas extends to the bottom of the alcohol. A trap is interposed between the flasks to prevent the alcohol running back into the acid in case the pressure is varied. The flask containing the acid is heated moderately until water begins to pass over, when the process is discontinued, to be renewed with a fresh portion of acid if the alcohol is found to be not sufficiently acidulated. The apparatus as used in this laboratory is illustrated in fig. 3.

After the filtrate from the potash determination above referred to has stood over night, nearly all of the liquid can be decanted into a porcelain dish of 5 or 6 inches diameter. Then with ordinary alcohol wash directly into a filter, washing two or three times, and adding the washings to the decanted liquid in the dish. Any sediment that may have gone over in the decantation can do no harm. The evaporation should be carried on very slowly on a steam or

water bath, else the alcohol will boil over. When the alcohol has evaporated there will be left a large amount of ammonium chloride in the residue. Care must be taken that all the alcohol is volatilized, after which the residue is dissolved in about 30 or 40 c. c. of water. To the solution add 2 or 3 c. c. of HNO_3 ; cover and heat on the bath very gradually (else there will be a violent ebullition due to the liberation of nitrous oxide). When ebullition has subsided, remove the cover and evaporate to dryness; then to the dish when cooled add two or three drops of HNO_3 , 10 or 15 c. c. of water, and warm until solution is effected. Wash from the dish with a minimum quantity of water into a beaker of suitable size for the determination of P_2O_5 , volumetrically.

To this solution add about a teaspoonful of dry ammonium nitrate; let stand in a water bath at a temperature of about 50°C .; add 10 or

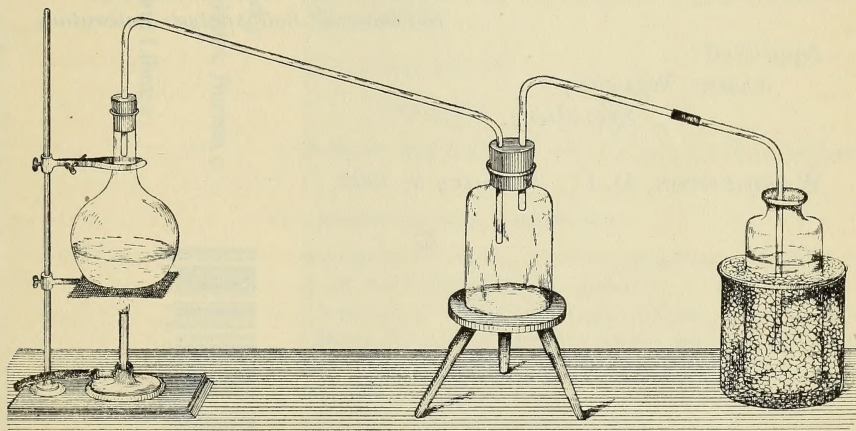


FIG. 3.—Apparatus for preparing acidulated alcohol.

15 c. c. of the ordinary molybdate solution, and let stand about thirty minutes, with an occasional shaking. Decant through a 9 cm. filter; wash the precipitate into the filter, and continue the washing until free of acid, by test with phenolphthalein or litmus. It generally requires about 200 c. c. of water for this washing. The filter and its contents are returned to the beaker, and by the use of a stirring rod and a little water the paper is beaten into a pulp. The titration is made in the usual way, with KOH and HCl , using phenolphthalein as an indicator. In this laboratory the alkali used is one-half the official strength; that is, 100 c. c. neutralizes 16.19 c. c. of normal acid. This makes 1 c. c. = .0005 gram P_2O_5 . The weight of P_2O_5 here found is to be divided by 160 grams, and the result expressed in parts per million, as was that for K_2O .

As will be seen, in this method the total weight of soil is used in each determination instead of dividing the solution, as is generally

done. The large amount used adds greatly to the accuracy of the results. Having found the parts per million of these ingredients in the soil, and having the dry weight of the original sample, which was taken over a definite area, it is a very simple matter to calculate to pounds per acre for the depth to which the sample was taken.

It is believed that this method will give the amount of plant food which is in condition to be taken up by the oat plant, provided there is a suitable balance in such plant food. It is the result of experience in a large number of analyses. The calculation in pounds per acre in this work is on the assumption that plant food does not exist to a greater depth than 18 inches.

It is desired that those who wish to cooperate in the work will notify this Bureau as soon as possible, as arrangements should be made without delay.

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Approved:

JAMES WILSON,

Secretary.

WASHINGTON, D. C., *January 3, 1902.*

