

taking special precautions (avoidance of draughts, etc.), I have been able to avoid any variation greater than one-hundredth degree for over four hours. Temperature variations were noted with the aid of a Beckmann thermometer.

I should like to state here that, from my experience with thermostats, I have been convinced that the fineness of the regulation of the temperature demands not so much sensitiveness, *i. e.*, large linear motion for small temperature change in that part of the regulator where the cut-off is, but rather rapidity of response on the part of the expansive medium to temperature changes. In the ordinary bulb regulator, the lag of the expansion and contraction of the toluene is the most fruitful source of variation, when the sensitiveness of the regulator has been otherwise highly developed. This defect is to be best met, not by increasing the quantity of expansive liquid, but by distributing it in such a way that there will be greater surface exposed; *i. e.*, in annular or spiral bulbs. I have made no experiments in this direction as the apparatus above described was wholly sufficient for my purposes.

Certain modifications of the above apparatus will naturally suggest themselves, such as use of platinum wires instead of lamps, etc., but the apparatus as described has worked so satisfactorily that I have let well enough alone.

Certain advantages, such as affording no danger from fire, cleanliness, etc., need not be enumerated here.

A METHOD FOR THE DETERMINATION OF THE AVAILABILITY OF ORGANIC NITROGEN IN COMMERCIAL FERTILIZERS.

BY JOHN PHILLIPS STREET.

Received March 12, 1901.

OF the three essential plant-food elements, nitrogen, phosphoric acid, and potash, nitrogen is by far the most important, not only because it is the most expensive, but also because it is the chief factor from a fertilizing standpoint in the early growth of the plant. The nitrogen of sodium nitrate and that of ammonium sulphate, on account of their solubility in water, are known to be readily available to the plant, and satisfactory methods for their detection exist. The great demand for nitrogen by the farmers, however, and the desire to utilize as far as possible

all by-products have led fertilizer manufacturers to search for forms of organic nitrogen for use in the mixing of their various brands. Among those most commonly used are dried blood, dried fish, castor pomace, hoof meal, ground bone, tankage, cottonseed meal, and linseed meal. Practical experience has shown all of these forms of organic nitrogen to be quite available to the plant, and has marked them as excellent sources of plant food. The manufacturers, however, did not stop with the use of these materials, but sought still others, which were cheaper, and whose bulk was of great assistance as a filler in their fertilizers. Such materials as garbage, leather, shoddy, felt waste, and hair, came into use, and while their employment has been by no means general, it is nevertheless important to agricultural chemists that they should be able to detect these materials in mixed fertilizers, for vegetation tests have shown them to be either entirely worthless, or at any rate not available to the plant until after a comparatively long lapse of time.

The analyses made at the New Jersey Experiment Station during 1900, showed that the cost per pound of the nitrogen in different organic materials was as follows :

	Cents.
Dried blood.....	14.2
Dried fish.....	13.7
Cottonseed meal	14.5
Ground bone.....	16.4
Wool waste	7.0 to 0.5
Hair.....	5.1 to 4.6
Leather.....	5.0

The present laboratory methods do not determine the source of the organic nitrogen in fertilizers, and it is evident at once that the manufacturer using wool waste, hair, or leather, as the source of his nitrogen, is not justified in charging for that nitrogen the same price that he would be warranted in charging if the nitrogen of blood, fish, bone, or cottonseed meal had been utilized in making his fertilizers. To call this simply total nitrogen is manifestly unfair to the honest manufacturer, and at the same time misleads the purchaser.

At both the Connecticut and New Jersey Experiment Stations the complete fertilizers analyzed during one season were subjected to digestion with pepsin-hydrochloric acid, with the following results :

Connecticut.			New Jersey.		
Per cent. soluble.			Per cent. soluble.		
3	over	90	3	between 80 and 90	
22	between 80 and 90		13	"	70 " 80
62	"	70 " 80	16	"	60 " 70
18	"	60 " 70	11	"	50 " 60
12	"	50 " 60	4	"	40 " 50
6	"	40 " 50	2	under	40
2	under	40			

A fertilizer showing 60 per cent. of its organic nitrogen soluble in pepsin may be considered as above suspicion, and one falling as low as 50 per cent. must not necessarily be condemned, while one below 50 per cent. must be regarded as suspicious. From the above table it will be seen that in Connecticut eight samples, or 6 per cent., fall in the suspicious class, and in New Jersey 6, or 12 per cent. These results show that, although their number is limited, there are suspicious fertilizers on the market, and that the necessity does exist for an accurate method to determine nitrogen availability.

For many years, therefore, agricultural chemists have employed the above-mentioned pepsin-hydrochloric acid method,¹ generally used in biochemical laboratories, as a test of the availability of organic nitrogen. Vegetation tests, and chemical tests by this method on the same materials have in general shown close agreement in most classes of organic nitrogenous compounds, but experiments at the Connecticut and New Jersey State Experiment Stations have shown that for certain classes of materials, notably hoof meal, it gives but little indication of their value. The following tabulation shows some comparative results obtained by the writer on certain well-known materials :

	Vegetation tests.	Availability by Pepsin digestion.
Dried blood	68	68
Dried fish	66	51
Hoof meal	62	39
Steamed bone	60	56
Tankage	59	56
Wool waste	31	22
Raw leather	2	14

A comparison of these results shows that with the exception of raw leather, the pepsin method tends to give lower results than were secured by the vegetation tests. The pepsin results, how-

¹ Report of Conn. Expt. Station, 1893, 219.

ever, are quite satisfactory, except in the case of hoof meal, where the pepsin solubility is 23 per cent. lower than by the actual culture test.

Not only is the pepsin-hydrochloric acid method unreliable with certain materials, but it is so chronokleptic that its use is almost impossible in the average agricultural or commercial laboratory. The putrefactive method, employed in several laboratories, is open to these same objections.

While the writer was referee on nitrogen for the Association of Official Agricultural Chemists in 1895, Mr. S. H. T. Hayes, of Cornell University, submitted to him a paper in which he tested various methods for determining availability, including fractional treatment with sulphuric acid, digestion in a saturated solution of barium hydroxide, and digestion in alkaline, acid, and neutral solutions of potassium permanganate. The results obtained by Hayes¹ were not at all conclusive, but those secured by the permanganate digestion were promising, and were deemed by the writer worthy of further trial.

DIGESTION WITH POTASSIUM PERMANGANATE.

The method, as outlined by Hayes, consists in treating 1 gram of the material in a 500 cc. flask with 100 cc. of potassium permanganate solution, containing 16 grams potassium permanganate and 200 grams caustic soda per liter. Digest at a low temperature for one hour, then increase the temperature and distil for one hour, and titrate as usual. For the acid solution, the method consists in weighing 1 gram of material into a 500 cc. flask, add a little paraffin and 100 cc. of potassium permanganate solution (16 grams potassium permanganate and 100 cc. concentrated sulphuric acid to the liter). Connect with distilling apparatus, and heat at a temperature just below boiling for one hour. Then add 50 cc. of saturated caustic soda solution, distil for thirty minutes, and titrate as usual.

After making a number of tests, it was evident that the methods, as suggested, were difficult to control. It was almost impossible to carry on the digestion without a boiling of the liquid, and it was very difficult to maintain uniform conditions in two different sets of tests. Comparative results obtained by seven agricultural chemists working in different laboratories showed a wide divergence, and it was believed by the writer that this was

¹ U. S. Dept. of Agr. Dept. of Chem. Bull., 47, 112-116.

due more to this inability to control the temperature, and keep the conditions uniform, than to any inherent defect in the principle of the method itself.

It also seemed that the great variations in bulk of the nitrogenous materials examined required that the comparison should be based on the same quantity of organic nitrogen rather than on equal weights of materials. With these ideas in mind, the writer devised the following method :

Weigh an amount of the material equivalent to 0.075 gram of nitrogen into a 500 cc. Erlenmeyer flask, add 100 cc. of neutral, 1.6 per cent. potassium permanganate solution, and digest on a steam-bath for thirty minutes, shaking occasionally to moisten any particles of the material adhering to the sides of the flask. Filter and wash thoroughly, using from 125 to 150 cc. of water. Determine the total nitrogen in the undigested residue by the ordinary Kjeldahl method.

Mr. C. H. Jones,¹ of the Vermont Experiment Station, has also modified Hayes' method, by using an amount of the material equivalent to 0.045 gram of organic nitrogen with the alkaline potassium permanganate solution. The results secured by this method have on the whole been very acceptable, but with cotton-seed meal, a material of known value, it fails utterly as a test of availability.

To test the method suggested by myself, I have examined eighty-four samples of organic nitrogenous matter, representing twenty-eight different classes of fertilizing material. The results will be found in the table below, where the total organic nitrogen and the percentage digestibility in potassium permanganate are given.

AVAILABILITY OF ORGANIC NITROGEN.		
	Organic nitrogen. Per cent.	Digestible. Per cent.
Dried blood	12.41	95.6
" "	13.20	95.2
" "	14.01	95.0
" "	13.60	94.4
" "	13.73	94.3
" "	14.04	94.0
" "	11.63	91.9
" "	11.39	91.8
" "	13.10	91.0

¹ U. S. Dept. of Agr., Div. of Chem., Bull. 57, 51.

	Organic nitrogen. Per cent.	Digestible. Per cent.
Dried blood	13.41	89.8
“ “	11.06	87.4
Average.....	12.87	92.8
Ground horn.....	14.85	95.9
Concentrated tankage	11.13	93.7
Ammonite	13.00	93.6
Cracklings.....	13.22	93.5
Hoof meal	14.47	92.2
“ “	14.04	89.7
Average.....	14.26	91.0
Castor pomace.....	6.12	90.0
Peanut pulp.....	8.52	89.3
Dried fish.....	8.78	90.2
“ “	7.97	90.0
“ “	7.53	88.7
“ “	8.80	88.5
“ “	9.53	88.0
“ “	8.69	87.6
“ “	7.39	87.1
“ “	6.82	86.7
“ “	8.28	86.5
“ “	8.83	86.3
“ “	9.43	86.1
“ “	4.57	86.0
“ “	8.37	85.0
“ “	7.80	84.7
“ “	6.50	82.9
“ “	8.34	82.3
“ “	7.75	82.2
“ “	8.23	82.1
“ “	5.07	81.9
“ “	8.44	81.4
“ “	7.49	79.6
“ “	6.82	78.4
“ “	5.07	74.7
“ “	6.47	74.4
“ “	6.86	70.1
“ “	5.19	69.3
“ “	6.96	65.8
Average	7.48	83.9
Tankage	6.90	91.5
“	7.67	89.5
“	5.88	87.1
“	6.42	85.4

	Organic nitrogen. Per cent.	Digestible. Per cent.
Tankage	5.69	84.0
“	6.65	81.8
“	5.67	79.8
“	5.89	79.7
“	5.34	79.7
“	5.16	65.0
Average.....	6.13	82.4
Cottonseed meal.....	7.33	79.6
“ “	6.74	78.6
Average.....	7.04	79.1
Linseed meal.....	5.94	77.7
Horse meat.....	4.29	70.4
Steamed bone	2.78	87.0
“ “	2.25	84.8
Button bone.....	3.33	84.0
“ “	3.93	76.0
Raw bone.....	3.40	66.9
“ “	4.12	66.3
“ “	4.05	66.2
Bone sawings.....	3.85	77.6
Soft bone	2.37	71.6
Dissolved bone.....	1.95	62.7
Wool waste.....	2.58	77.6
“ “	2.52	75.1
“ “	2.51	75.0
Dissolved tankage	5.02	65.9
“ “	2.93	63.1
King crab.....	8.78	72.5
“ “	9.12	52.5
Hair manure.....	5.76	54.6
Garbage fertilizer.....	2.95	59.2
“ “	3.20	44.1
Burned garbage	1.95	51.5
Steamed leather	6.87	39.5
Treated leather.....	7.22	33.4
Raw leather.....	7.56	25.5

These results are remarkably striking, the difference in the effect of the permanganate solution in the various ammoniates being very noticeable. The percentage digestibility varies from 95.9 per cent. in horn to 25.5 per cent. in raw leather. The other materials range in availability between these limits in the approximate order vegetation cultures would lead one to expect. The ground horn sample is, perhaps, somewhat above the average of

that material, it having been carefully prepared in my laboratory, and being nearly five years old and in a pulverulent condition. Of the six classes of bone examined, steamed bone shows the highest digestibility. This is doubtless due to the fact that the process of steaming had in a large measure removed the fatty matter; raw bone, in which much fat was present, is nearly 20 per cent. lower. The availability of the dissolved bone is slightly below that of raw bone, seeming to indicate that the treatment with sulphuric acid had little favorable effect on the nitrogenous matter in the bone; the same conditions obtain in the case of dissolved tankage, which shows a digestibility 18 per cent. lower than the untreated tankage. The remaining materials occupy about the rank agricultural experience would assign them.

It must be remembered in using this method, that the figures obtained are relative, not absolute. It is not claimed that it shows the exact amount of organic nitrogen available to the plant, but it does serve as a means of distinguishing high-class from low-class ammoniates. The results would seem to indicate that in any material showing by this method a digestibility of 60 per cent., or under, the nitrogen was obtained from an inferior source. It certainly could not have been obtained from blood, hoof meal, dried fish, tankage, or cottonseed meal, but was probably furnished by hair, garbage, leather, or some very inferior bone.

The table also shows the variations which may be found in working with materials of the same class, and indicates that the name of a fertilizer is not always a safe guide for the agriculturalist. The variations are particularly striking in the case of fish and tankage, where the digestibility ranges from 90.2 to 69.3 and 91.5 to 65.0 per cent., respectively. These variations are doubtless due to the varying amounts of fatty material in these products, and show still further the value of this method in the detection of such conditions.

Experiments have shown that this method is equally applicable to complete fertilizers, the only variation being that the material is washed on a filter with cold water, prior to the digestion, to remove such soluble salts as might interfere with the subsequent action of the permanganate.

It is admitted that the only true test of the availability of any fertilizer is the plant itself, but it is manifest that this method of examination is not open to all analysts, and to most is an impossi-

bility. It is hoped, therefore, that this simple method may be of value in furnishing chemists with a means of determining within reasonable limits the relative availability of the various nitrogenous materials submitted to them for examination.

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AN APPARATUS FOR DETERMINING FAT.

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THE Knorr apparatus¹ for the extraction of fat, which was a great improvement over preceding forms, enabled one to dispense with the ground-glass and cork connections formerly in use, by the employment of a mercury seal.

The mercury is carried in a channel which encircles the neck of the flask at the base. In order to maintain the connection between the flask and the glass part immediately above it, the two are held together by rubber bands. To avoid the use of the bands the condensers may be made movable so they can be lowered sufficiently to connect with the flasks, or if the condensers are stationary the bath which supports the flasks may be made movable. In many instances, particularly in the case of batteries of considerable size, neither of these plans is convenient and at the best, unless great care is exercised, the flasks are liable to break during the adjustment. The use of rubber bands is unsatisfactory owing to their liability to slip or break.

A later modification of the Knorr flask was one which had a glass channel attached to the neck of the flask near the top. This modification enabled one to reduce the quantity of ether liable to collect outside of the neck, but the flask was necessarily fragile and expensive. The apparatus described below was designed to remove some of the difficulties connected with that devised by Knorr and to accomplish certain other desirable objects. The improvements attempted may be summarized as follows:

1. The use of a simple flask which can be readily cleaned and replaced at small expense.
2. The employment of a rubber cup to carry the requisite

¹ Bull. 28, U. S. Dept. of Agriculture, Div. of Chemistry (1890), p. 96.