

that they are likely to be of considerable service in a sanitary examination of water.

It is hoped that those who are interested in the subject will put these methods to a practical test in order that their true value may be ascertained, through the experience resulting from their extensive employment.

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[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE U. S. DEPARTMENT OF AGRICULTURE. NO. 29.]

### ON THE INFLUENCE OF VEGETABLE MOLD ON THE NITROGENOUS CONTENT OF OATS.

BY H. W. WILEY.

Received June 7, 1897.

IN growing oats in pots containing vegetable soils from Florida, I noticed that the content of nitrogen in the product was much greater than in oats grown in common soils under the same cultural and climatic conditions. Previous to the beginning of the experiments described below I had noticed a peculiar condition of sugar-cane grown in Florida, on what is there known as muck soils. These soils are composed of vegetable mold, produced under the water in shallow lakes and along the banks of streams. A full description of these soils may be found in my paper on the "Muck Soils of Florida," published in *Agricultural Science*, 7, 106.

The condition referred to in the sugar-cane was manifested by a brownish color in the juice, which was extremely persistent, affecting even the color of the nearly pure sugar made therefrom. From the character of this coloration there is no doubt of its being due to an actual absorption by the growing sugar-cane of some of the components of the vegetable soil or humus. The fact that plants, under certain conditions, have the faculty of absorbing humus has been subsequently confirmed by the experiments of Snyder.<sup>1</sup> The vegetable soils in which the oats were grown contain in the air-dried state over eighty per cent. of organic matter and less than ten per cent. of sand and other mineral ingredients. The composition of four samples is shown in the following table:

<sup>1</sup> Bull. No. 41, Agricultural Experiment Station of Minnesota.

	Laboratory number.			
	13747 Per cent.	13748 Per cent.	13749 Per cent.	13750 Per cent.
Moisture .....	10.30	11.52	8.84	9.35
Organic matter and combined water .	83.30	82.21	82.77	83.72
Total nitrogen .....	2.54	2.59	2.76	2.68
Albuminoid nitrogen .....	2.44	2.37	2.73	2.52
Amid nitrogen .....	0.10	0.22	0.03	0.16
Nitrogen as nitrates and ammonia salts	0.03752	0.07952	0.05600	0.08120
Nitrogen as ammonia salts .....	0.03698	0.05600	0.03510	0.03360
Nitrogen as nitrates.....	0.00054	0.02352	0.02090	0.04760

Sample No. 13,747 is a virgin soil from the best muck land on the Department Experiment Station near Narcoossee, Florida.

No. 13,748 is from the poorest virgin soil, sample No. 13,749 from a cultivated plot producing good sugar-cane, and No. 13,750 from a cultivated plot where cane failed to grow.

In the mineral constituents of the soil the following quantities of potash and phosphoric acid were found.

	13747	13748	13749	13750
Per cent. of moisture in air-dried material	10.30	11.52	8.84	9.35
Loss on ignition.....	92.87	92.91	90.80	92.36
Mineral matter <sup>1</sup> .....	7.13	7.09	9.20	7.64
Potash <sup>1</sup> .....	0.056	0.057	0.055	0.055
Phosphorus pentoxide <sup>1</sup> .....	0.066	0.057	0.088	0.173

It is seen from the above data that the soils are deficient both in potash and phosphoric acid, containing generally only about 0.05 per cent of these ingredients except in one instance, where the percentage of phosphoric acid reached almost 0.2. While the data show the almost entire absence of potash, yet the cultures in pot experiments have demonstrated the fact that these soils respond more promptly to treatment with a phosphatic fertilizer than any other, and that they are capable of acting upon the finely ground mineral phosphates of Florida in a manner which makes them easily available to the growing crop.

The soil from the best muck land was found to be entirely free of nitrifying ferments, a culture seeded with it showing no nitrification after forty days. The practical absence of nitric acid in the air-dried sample is therefore not surprising. On the other hand, the molds which produce ammonia in an acid soil appeared to be fairly active. On these soils the oats obtained in 1895 and 1896 were grown. It seems probable from the data

<sup>1</sup> Per cent. on dry material.

obtained that the two samples from the best and poorest soils were interchanged before reaching the laboratory. The crop secured in 1894 was grown on other soils, obtained from the same fields and entirely like those described above in every particular.

The experiments made in 1894 were of a preliminary nature and had for their object a study of the conditions of growth of oats and other crops, with a view to more systematic experiments subsequently. The analytical data relating to the nitrogen in those crops, however, are of so much interest as to demand a rather detailed discussion.

The pots used in the 1894 experiments were made of clay, with perforated bottoms. They were painted with black asphalt paint within and with a white lead paint without. They were thirty cm. in diameter and thirty-seven cm. deep. The bottom of each pot was covered with broken stone and the well mixed vegetable soil placed over this until the pot was full.

STATEMENT SHOWING THE AMOUNT AND KIND OF FERTILIZER ADDED TO EACH POT.

No. of pot.	Fertilizer used.
1	None.
2	36 grams lime mixed with soil.
3	3.6 " $K_2SO_4$ mixed with upper 6 inches of soil.
4	5.4 " Florida phosphate mixed with upper 6" soil.
5	5.4 " " " " " 6" "
6	5.4 " slag " " " " 6" "
7	5.4 " superphosphate " " " " 6" "
8	3.6 " $(NH_4)_2SO_4$ .
9	3.6 " $K_2SO_4$ , 3.6 grams $(NH_4)_2SO_4$ ; 5.4 grams Florida phosphate.
10	36 " lime; 5.4 grams Florida phosphate.
11	36 " " 3.6 grams $K_2SO_4$ .
12	5.4 " Florida phosphate; 3.6 grams $K_2SO_4$ .
13	5.4 " superphosphate; 3.6 grams $K_2SO_4$ .
14	None.
15	36 " lime
16	3.6 " $K_2SO_4$ .
17	5.4 " Florida phosphate.
18	5.4 " slag phosphate.
19	5.4 " superphosphate.
20	3.6 " $(NH_4)_2SO_4$ .
21	5.4 " slag phosphate; 3.6 grams $K_2SO_4$ .
22	36 " lime; 5.4 grams $NaNO_3$ .
23	None.
24	36 " lime; 5.4 grams Florida phosphate; 3.6 grams $K_2SO_4$ ; 3.6 grams $(NH_4)_2SO_4$ .
25	5.4 " $NaNO_3$ .
26	36 " lime; 3.6 grams $H_2SO_4$ ; 5.4 grams superphosphate.

*Nitrogen in the Crop.*—The weight of the crop produced and the percentage of nitrogen in the roots, straw, and grain, respectively, are given below. In this connection attention should be called to the fact that, in some instances, a few of the grains were carried off by the sparrows in spite of the watchfulness of the attendant.

TABLE SHOWING WEIGHT OF CROP FROM EACH POT AND QUANTITY OF NITROGEN IN ROOTS, STRAW, AND GRAIN.

Pot No.	Weight of roots. Grams.	Nitrogen in roots. Per cent.	Weight of straw. Grams.	Nitrogen in straw. Per cent.	Weight of Nitrogen in grains and chaff.		Average per cent. nitrogen in grains and straw. Crop 1894.
					Grams.	Per cent.	
1	4.920	1.82	15.512	2.05	1.530	2.69	2.10
2	2.970	1.62	15.070	2.02	3.120	2.75	2.15
3	4.450	1.79	13.250	2.13	1.510	2.75	2.21
4	8.860	1.34	36.010	1.29	2.160	2.58	1.36
5	6.560	1.29	36.320	1.34	4.020	2.69	1.47
6	6.820	1.29	35.100	1.12	5.920	2.52	1.32
7	10.350	1.23	32.470	1.26	3.850	2.55	1.40
8	5.670	1.74	14.760	2.26	0.770	2.24	2.25
9	8.850	1.68	43.530	1.85	9.430	2.80	1.85
10	6.680	1.79	17.920	2.29	1.620	2.80	2.83
11	5.280	1.82	16.530	2.18	1.460	2.69	2.22
12	7.370	1.23	38.150	1.15	3.870	2.46	1.27
13	7.150	1.06	44.150	1.11	6.560	2.41	1.27
14	1.520	1.57	4.330	2.24	0.760	2.58	2.29
15	1.400	1.79	6.080	2.69	0.380	2.58	2.68
16	0.750	1.68	2.070	2.21	0.300	2.52	2.25
17	6.100	1.18	34.830	1.40	2.530	2.69	1.49
18	7.150	1.46	32.650	1.47	2.170	2.58	1.54
19	6.030	1.34	33.230	1.75	2.370	2.75	1.82
20	1.260	1.90	3.520	2.63	0.320	2.80	2.64
21	5.710	1.12	32.980	1.46	1.720	2.46	1.53
22	1.860	1.79	5.830	2.63	1.370	2.58	2.62
23	1.430	1.68	3.880	2.52	0.420	2.46	2.51
24	3.230	1.85	6.320	2.69	1.530	2.46	2.65
25	1.780	1.85	4.530	2.69	0.920	2.80	2.71
26	4.870	1.74	13.270	2.69	3.080	3.14	2.77
Averages,		1.56		1.97		2.63	2.02

The surprisingly high content of nitrogen in all parts of the crop is forcibly shown by the above data. In the unfertilized soils, where the weight of the crop was a minimum, the content of nitrogen is almost uniformly higher than in the fertilized samples, though of course the total weight of nitrogen entering the

crop is much less. The remarkable effect of phosphatic fertilizers in diminishing the percentage of nitrogen is strikingly shown in Nos. 4, 5, 6, 7, 12, 13, 17, 18, and 21. When the phosphate was applied in connection with nitrogenous fertilizers the reduction in the percentage of nitrogen was not so marked, as is shown by Nos. 9, 24, and 26. The data show that in these soils a phosphate is the only required fertilizer, and that it works equally well in each of the three forms in which it was used. In other words, these vegetable soils permit of the easy absorption of the finely ground phosphates without previous treatment with sulphuric acid.

The mean percentages of nitrogen in the three parts of the crop are very high, and this is especially the case in the straw and grain. In this connection it should be mentioned that the grains were ground and prepared for analysis without removing the chaff.

These results were so interesting that it was deemed advisable to repeat the experiments with fresh samples of the vegetable soil. Accordingly, in 1895, twelve pots were filled with vegetable soil, representing four different characters of soil, resembling, in their chemical analyses, the soils already mentioned. Since the roots of the oats crop are never harvested, it was deemed sufficient to confine the examination for nitrogen to the straw and grains. To facilitate the work the straw and grains were ground together so that one analysis was made to do for both. The fertilization of the pots is shown by the following tabular statement :

No. of pot.	
15	Blank.
17	Phosphatic slag.
24	Florida phosphate rock.
26	Blank.
29	Acid phosphate.
18	“ “
23	Blank.
7	Phosphatic slag.
28	“ “
25	Florida phosphate.
6	“ “
16	“ “
5	Blank.

The weights harvested from each pot, and the percentages of nitrogen in the straw and grains combined are shown in the following table :

OATS.			
Florida soil, 1895.			
Pot No.	No.	Weight of crop. Grams.	Nitrogen. Per cent.
15	14,439	14.70	1.82
17	14,440	56.70	2.20
24	14,441	69.50	1.53
24	14,442	37.20	0.90
26	14,443	55.80	1.01
27	14,444	47.20	0.98
29	14,445	53.50	0.95
18	14,449	52.03	1.92
23	14,450	29.30	1.03
7	14,451	51.41	1.05
28	14,452	54.55	0.72
25	14,461	40.71	0.72
6	14,502	37.89	0.92
16	14,503	52.99	2.12
5	14,504	5.59	1.37

While the percentage of nitrogen is not nearly so high as in the results previously given, yet in many instances it is quite excessive. This is particularly so with Nos. 16 and 17, both of which had received phosphatic manures, the nitrogen in these samples being greater than in the unfertilized samples, in which the crop was almost a total failure. The effects of the phosphatic fertilizers, noticed in the first year's experiments, of diminishing the percentage of nitrogen, are not verified by the experiments made in 1895.

*Experiments made in 1896.*—Experiments made with the Florida vegetable soils were repeated in 1896 with a larger pot, made of metal, containing nearly twice as much soil as in the pots used in the previous years, but with no increase in superficial area. The soils in 1896 were not fertilized, but the character of the fertilizer carried over from the previous year is given in the subjoined table. The pots for 1896 being larger, were filled from two or more pots of the year previous, but the soil received no further fertilization. Any effect of fertilizer, therefore, which will be noticed in the crop is due to the continuous effect of the fertilizer from the previous year.

The character of the soil in each pot and the nature of the fertilizer residual from the amount added the previous year are shown in the following tabulation :

Pots 135, 136, 137, and 138 were filled with the Florida soil, No. 13,747.

Pots 139, 140, 141, and 142 contain soil No. 13,748.

Pot 143 contains soil No. 13,749.

Pots 144, 145, and 146 contain soil No. 13,750.

The residual fertilization of each of the pots is as follows :

No. of pot.	Character of fertilizer.
135	No fertilizer.
136	Florida phosphate.
137	Phosphatic slag.
138	Acid phosphate.
139	No fertilizer.
140	Florida phosphate.
141	Phosphatic slag.
142	Acid phosphate.
143	Florida phosphate.
144	No fertilizer.
145	Florida phosphate.
146	Phosphatic slag and acid phosphate.

The weight of the crop harvested from each pot and the percentage of nitrogen in the straw and grain combined are given in the following table :

OATS.			
Florida soil, 1896.			
Pot No.	No.	Weight of crop. Grams.	Nitrogen. Per cent.
135	15,515	27.4	1.71
136	15,516	46.6	1.53
137	95,517	33.8	1.09
138	15,518	35.6	1.23
139	15,519	19.9	1.98
140	15,520	46.1	1.81
141	15,521	51.6	1.75
142	15,522	52.2	1.79
143	15,523	39.5	1.11
144	15,524	38.7	1.01
145	15,525	37.1	0.97
146	15,526	33.4	1.06

The above data show that the unfertilized soils had been

improved by two years of cultivation, so that they produced nearly as large a crop, at least in one instance, as those which had received fertilizers in the previous year. In two instances, *viz.*, pots 135 and 139, the unfertilized crop being small, showed the highest percentage of nitrogen, while in a third instance, *viz.*, 144, the percentage of nitrogen was quite low, but in this case the crop was quite large. In general, therefore, the data show that the addition of phosphatic fertilizers, as in the first year's experiments, tended to diminish the actual percentage of nitrogen in the crop harvested.

For the purpose of comparison, the nitrogen was determined in the straw and grains of oats grown in ordinary agricultural soils from six different states in 1895 and 1896. These samples were selected without reference to the content of nitrogen which they contained, but indiscriminately, for the purpose of securing samples of representative or typical soils. The average content of nitrogen in fourteen samples of straw and oats grown in 1895 in common agricultural soils from Missouri, Michigan, Illinois, Wisconsin, Maryland, and the District of Columbia was 1.13 per cent. These crops were grown at the experimental vegetation house of the Division of Chemistry under exactly the same conditions as attended the growing of the crops on the Florida soils, fourteen samples of which the same year showed an average content of 1.30 per cent. of nitrogen in the straw and grains. In 1896 the average content of nitrogen in twelve samples of oats grown on the representative agricultural soils from Missouri, Michigan, Illinois, Wisconsin, Maryland, and the District of Columbia was 1.04 per cent., while in twelve samples grown under identical conditions on the vegetable Florida soils the average content of nitrogen was 1.42 per cent. Thus, leaving out of the comparison altogether the data obtained from the analysis of the 1894 crop, it is seen that in the case of oats the content of nitrogen in the grain and straw is very much larger when the crop is grown on the vegetable mold than when it is grown on the ordinary agricultural soils from different parts of the country. When it is remembered that these vegetable soils are extremely rich in nitrogen, as was shown in the analyses given in the first instance, and when it is further considered that they are quite deficient in nitrifying ferments, it is fair to con-



clude that at least a portion of this excess of nitrogen which they contain is assimilated directly from the vegetable mold without previous oxidation to nitric acid.

*Relative Proportions of Proteid and Non-Proteid Nitrogen.*—The relative distribution of the nitrogen in the various samples between the proteid and non-proteid forms is also a subject of interest. In the crop of 1894 the average percentage of nitrogen in the whole crop was 2.02. The quantity of material at hand did not permit of the separation of the nitrogen in all the samples. This separation, however, was accomplished in thirty-one instances, *viz.*, in fourteen samples of the roots, eleven samples of the straw, and six samples of the oats and chaff. The average percentage of total and amid nitrogen in the classes named was as follows :

	Roots. Per cent.	Straw. Per cent.	Oats and chaff. Per cent.
Total nitrogen (average).....	1.45	1.99	2.63
Amid " " .....	0.32	0.70	0.35

In the 1895 crop the separation was accomplished in fifteen samples of straw and grain together. The average percentage of total nitrogen in the samples examined was 1.27, and of amid nitrogen 0.44.

In the crop for 1895 the separation was accomplished in eleven instances, and the average percentage of total nitrogen found was 1.45, and the percentage of amid nitrogen was 0.29.

Comparing these relative percentages of amid and proteid nitrogen with crops grown in ordinary soils, it is found that in twelve samples grown in miscellaneous soils from different parts of the country in 1895 the percentage of total nitrogen was 1.15, and of amid nitrogen was 0.24, and in 1896, in fifteen instances of crops grown on miscellaneous agricultural soils from different parts of the country, the percentage of total nitrogen was 1.18, and of amid nitrogen 0.20.

It will be seen from the above data that the content of amid nitrogen in the crops grown in the vegetable soils was abnormally high, and that the content of proteid nitrogen in the crops grown on the vegetable soils was fairly comparable with the content of proteid nitrogen in the crops grown on miscellaneous soils. This is another important observation to be considered

in connection with the nitrogen content of the crop, and it is fair to infer from the data collected during the three seasons that the tendency of the vegetable soil rich in nitrogen is to increase the total nitrogen content of oats grown therein, but that this increase is chiefly due to the content of non-proteid nitrogen.

#### CONCLUSIONS.

(1.) Oats grown upon humus soils contain about twenty-five per cent. more nitrogen than those which are grown upon ordinary agricultural soils.

(2.) The increase in the amount of nitrogen noted above is found chiefly in the amid, and not in the proteid nitrogen.

(3.) Fertilization of humus soils, such as were used in these experiments with potash and nitrogenous fertilizers, did not have any appreciable effect upon the quantity of the crop.

(4.) The use of phosphatic fertilizers in connection with these humus soils greatly increases the quantity of the crop and diminishes the percentage of nitrogen contained therein. This diminution in the percentage of nitrogen appears to have resulted chiefly from the increase in the crop, and not to any deleterious influence of the phosphatic fertilizer.

(5.) The three forms of phosphatic fertilizer employed, *viz.*, finely ground soft Florida phosphate, phosphatic slag, and acid phosphate exert an almost identical influence in increasing the quantity of the crop.

(6.) Oats grown upon humus soils absorb directly therefrom a portion of the nitrogen contained therein, chiefly in the form of non-proteid nitrogen.

My thanks are due Mr. T. C. Trescot for his valuable assistance in the nitrogen determinations.

#### NOTES ON THE DETERMINATION OF INSOLUBLE PHOSPHORUS IN IRON ORES.<sup>1</sup>

BY CHARLES T. MIXER AND HOWARD W. DUBOIS.

Received June 11, 1897.

ONLY within the past few years have chemists recognized the importance of the fact, that comparatively large amounts of phosphorus may occur in the siliceous residue left

<sup>1</sup> Read at the Chicago Meeting, 1897, of the A. I. M. E.