true with these results. In most cases different possibilities were weighted and the most logical values taken. These are believed to be approximately correct.

CONCLUSION

In general the plastometer has given very interesting data. It has many times enabled the laboratory to advise exactly what product will satisfy a particular requirement. Also,

it has been used in conjunction with the chemical analysis to tell what product would exactly match a submitted product. Some of the data obtained indicated that the empirical formulas of the first degree as used at present will have to be modified. Investigation will also have to be carried further as to the effect of final temperature to which the pastes are heated as well as the effect of different ingredients on the starches.

The Tyndallmeter Reading of Soil Dispersoids¹

PRELIMINARY REPORT

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The work herein reported was performed to obtain supplementary data for a bacteriological investigation so that we might gain a better knowledge of the conditions under which the bacterial flora of the soils was maintained. A complete report will be published in the near future.

Investigators have in the past made determinations of the dispersoid content of soils by the water-vapor adsorption, dye or gas adsorption, and suspension methods. The first two usually require a modification of procedure for each soil type and on this account are not well adapted for the study of a variety of soils. The suspension method gives the most promise and, if a satisfactory procedure can be developed, ought to yield fairly close results.

In this investigation a suspension method was employed in order to obtain a measure of the concentration of the disperse phase in a soil by means of a Tyndallmeter. The suspensions from the naturally moist soils were examined for dispersoid content after they had stood for 24 hrs. No attempt was made to determine the size of the organic or inorganic particles in the disperse system but, if it is assumed that the size of the particles left in suspension after 24 hrs. settling was approximately the same for the different soils, the strength of the Tyndall beam may be taken as roughly proportional to the concentration of dispersed material.²

In any case, since all the suspensions were prepared by a uniform procedure, the factors obtained bear sufficient relation to each other to furnish a good index of the comparative concentrations of the small particles which may be important factors in the maintenance of soil fertility.

APPARATUS

In 1919 Tolman and Vliet³ published a description of an apparatus which they had perfected for measuring the strength of the Tyndall beam produced by suspensions, colloidal solutions, smokes, and mists. The specifications for the Tyndallmeter may be found in the publication referred to. The apparatus with the equipment necessary for its operation is shown in the figure.

The Tyndallmeter is designated by the letter A on its triangular support. Readings are made with the apparatus by inserting the suspension in its container in the chamber B, and examining it through the eyepiece of the Macbeth illuminometer just above. The illuminometer is connected by wires with the millimeter C, which in turn is connected with the three dry cells. The lamp which furnishes the light for the Tyndall beam is at the lower end, D, of the Tyndallmeter and is connected by a wire with the positive pole

of the storage battery E. The negative pole of this battery is connected with the carbon compression rheostat F, which in turn is joined to the ammeter G, from which the current passes to the lamp of the Tyndallmeter through the switch H. The ammeter G is kept set at three amperes when readings are made.

The wooden horse which has been employed for the support of the Tyndallmeter is simple in construction but very convenient for adjusting the apparatus to any angle that suits the observer.

Although all readings of soil dispersoids in the Tyndallmeter are made in a dark room, it is found very trying to the

FIG. 1

eyes to determine the end-point, owing to the difficulty of matching the intensity of the lights when the colors are different. A screen placed in the eyepiece makes the readings much easier to obtain. It is prepared essentially as described by Crabtree⁴ by clearing an unexposed Hammer photographic dry plate in a regular hypo solution and then thoroughly washing and drying it. The plate, gelatin side up, is put in a developing tray containing a filtered solution of 0.1 per cent of Aniline Brown and allowed to adsorb the color for 10 min. The tray should be tilted back and forth for a short time when the plate is first immersed and then at intervals, as this procedure tends to make a more uniformly colored plate. At the end of this time the plate is rinsed for 1 min. in ice cold water and dried where it will be free from dust. The plate is cut in small squares, and three thicknesses are placed in the eyepiece. A test with and without the screen showed that more uniform readings were obtained with it.

STANDARDIZATION

It is necessary to standardize the light of the illuminometer and that for the Tyndall beam.

⁴ "How to Make, Fit and Use Light Filters," *Amateur Photographer and Photographic News*, 49 (1909), 173.

¹ Received August 3, 1921.

² R. C. Tolman, L. H. Reyerson, E. B. Vliet, R. H. Gerke, and A. P. Brooks, "The Relation between Intensity of Tyndall Beam and Concentration of Suspensions and Smokes," *J. Am. Chem. Soc.*, 41 (1919), 300.

³ "A Tyndallmeter for the Examination of Disperse Systems," *J. Am. Chem. Soc.*, 41 (1919), 297.

The illuminometer light is standardized with the apparatus made for that purpose by Leeds and Northrup Co. Since the standardization has been described by Tolman and Vliet it will not be repeated here. In our work the lamp is adjusted so that the actual number of apparent foot candles is only one-half as many as indicated by the illuminometer scale. The number of mil-amperes required to keep the lamp at this adjustment is maintained in all tests.

The light for the Tyndall beam is standardized by means of a silica suspension prepared in the following manner: One-half gram of silica, ground to pass a 200-mesh sieve, is suspended in one liter of distilled water in a 2-liter flask, and after 2 min. 300 cc. of the suspension are withdrawn by a pipet. The tip of the pipet is kept 2 cm. below the surface. Two hundred cc. of distilled water are added to the 300 cc. and the resulting dilution is employed as the standard.

The special glass tubes in which the dispersoid is examined are 12 cm. long by 4.3 cm. in outside diameter and the glass is approximately 1 mm. thick. The tubes are first filled with distilled water and closed with a 1-hole rubber stopper to prevent the inclusion of air bubbles, and are examined in the apparatus to determine whether any correction is necessary for the reflection of light from the glass. The correction is so small that it has been ignored. The silica standard is then placed in the tube and ten consecutive readings made by each of the two observers. Five readings should be made by approaching the end-point from the zero and five from the maximum end of the scale as described by Tolman and Vliet. This standard solution is kept in a well-sealed flask and employed for the frequent standardization of the apparatus.

METHODS

The soil samples are passed through a 7-mesh sieve to remove plant tissue and small stones and to produce a uniform texture, and they are then thoroughly mixed. The sifting and mixing are done as rapidly as possible to prevent loss of moisture by evaporation. The moisture content of the soil is then determined by air-drying two 25-g. quantities from each sample.

When the percentage of moisture is known, four portions are weighed out from a sample so that each one is equivalent to 5 g. of air-dry soil. These portions are placed in 1-liter Erlenmeyer flasks, and 500 cc. of distilled water are added. The flasks are stoppered and shaken vigorously for 5 min. and allowed to stand for 1 min., when 50 cc. of the suspension are withdrawn from each with a pipet by inserting the tip 2 cm. below the surface. The four 50-cc. aliquot portions are run into a 2-liter Erlenmeyer flask and diluted, if necessary, with a measured quantity of distilled water so as to give a reading between 1 and 10 on the Tyndallmeter scale. The investigator will soon find that he can readily estimate the concentration between these points. If the suspension is more disperse than desired a second 50 cc. from each flask may be added to it. The composite suspension is shaken vigorously for 1 min. and allowed to stand 24 hrs. At the end of this time all but 200 cc. of the supernatant suspension is siphoned into a flask and well shaken for 1 min. to insure uniformity. Two of the special tubes are filled with the suspension and read in the Tyndallmeter. A similarly prepared composite suspension is also read as a check on the first one.

The factor for the concentration of the disperse system is obtained by converting the actual apparent foot candle reading to the theoretical value for 1 g. of air-dry soil. The formula employed for this purpose is

$$X = \frac{vdr}{2w}$$

where X is the factor for the concentration of dispersoid per gram of air-dry soil; v is the total volume of the final sus-

pension; d is the quotient of the initial total volume, generally 2000, by the aliquot taken for the final suspension, usually 200; r is the Tyndallmeter reading; w is the weight in grams of air-dry soil employed to make the initial total volume of suspension.

TABLE I—READINGS OF SUSPENSIONS FROM SOIL, 32

Suspension A ₁		Suspension B ₁	
3.00	2.60	3.02	2.91
2.98	2.55	3.07	2.79
2.90	2.65	3.10	3.03
2.90	2.80	2.90	3.13
2.95	2.65	3.05	2.93
2.90	2.85	2.88	2.75
2.80	2.69	3.05	3.00
2.85	2.90	3.00	2.80
2.91	2.70	3.00	2.91
2.85	2.79	2.97	3.04
Av.	2.904 2.718	3.004	2.929
Suspension A ₂		Suspension B ₂	
3.02	2.87	3.15	3.10
3.10	2.79	3.04	2.85
2.85	2.84	2.94	2.85
2.85	2.91	2.95	3.05
3.10	2.71	2.96	3.07
2.90	3.09	3.00	2.91
2.98	3.02	3.10	3.20
2.88	2.78	3.11	3.02
2.85	2.85	3.00	2.77
2.85	2.90	2.95	2.97
Av.	2.938 2.876	3.020	2.979

DATA

The factor for the concentration of disperse phase obtained by the method outlined has been determined in thirty-one soils of different agricultural values. The first eight (6 to 13, inclusive) were kindly sent in by Dr. A. W. Sampson of the Great Basin Experiment Station, Ephraim, Utah. They are soils that have been overgrazed and are now supporting weed growth in different stages of succession. The soils numbered 31 to 34, inclusive, were collected by the junior author as representatives of types used in Massachusetts cranberry bogs. For all the remaining samples from different parts of the country our thanks are due to Dr. C. F. Marbut, in charge of Soil Survey Investigations, Bureau of Soils, U. S. Department of Agriculture.

Most of the soils gave duplicate suspensions that checked very closely, but a few, on account of their nature, gave suspensions showing greater differences. In Table I is given an example of the results that may be obtained by carefully following the procedure outlined.

TABLE II

SAMPLE No.	Soil Group	Soil Type	Source	Mean Reading	Dispersoid Concentration Factor
10	Clay and clay loam	Clay loam	Utah	1.016	191
7		Clay loam	Utah	1.685	211
12		Clay loam	Utah	1.117	356
9		Clay loam	Utah	1.031	387
11		Clay loam	Utah	1.656	621
25		Clay	Texas	2.850	712
8		Clay loam	Utah	3.063	1532
13		Clay loam	Utah	5.538	2077
30		Clay	Alabama	7.380	3690
6		Clay loam	Utah	8.230	4115
26	Silt and silt loam	Clay loam	Utah	14.390	7195
24		Clay	Texas	22.840	22840
18		Loam	Indiana	1.634	82
28		Silt loam	Idaho	1.118	223
20		Silt loam	N. Dakota	2.631	263
21		Silt loam	N. Dakota	5.558	278
37		Silt loam	Idaho	3.577	711
14		Silt loam	Kentucky	2.178	1089
17		Silt loam	Nebraska	7.519	2820
15		Silt loam	Kentucky	8.198	4057
22	Sand and sandy loam	Silt loam	New York	11.150	5575
27		Silt loam	N. Carolina	12.600	6300
16		Silt loam	Nebraska	14.620	7500
33		Sand	Massachusetts	Trace	Trace
19		Sandy loam	Indiana	1.085	51
29		Sand	Alabama	1.470	74
36		Sand	Michigan	4.730	237
35		Sandy loam	Michigan	1.316	329
31		Sandy muck	Massachusetts	Trace	Trace
34		Bogland	Massachusetts	1.510	76
32		Woodland muck	Massachusetts	2.920	439

The average reading of Suspensions A₁ and A₂ is 2.859; of Suspensions B₁ and B₂, 2.984. The mean of these readings

is 2.922, and the variation of the averages from this is 2.2 per cent.

In Table II are the results obtained from the thirty-one soils. It will be noticed that the mean readings do not increase in the same order as the dispersoid concentration factors. This difference is due to the fact that the mean readings were made with dilutions of dispersoid that varied for the different soils.

An examination of the readings obtained with these soils shows that the average error from the mean is 2.54 per cent. In obtaining this figure the results of five soils are omitted

on account of abnormally high percentage errors. No effort was made to get closer checks on these samples, because four of the factors were so low and one so high that an exact check would not in any case have altered the relative values of the soils.

The relation of these dispersoid concentration factors to the other data that have been obtained will be discussed in a future report.

SUMMARY

A procedure is given for obtaining by means of a Tyndall-meter a factor for the concentration of dispersoid in a soil.

The Determination of Glycerol in the Presence of Sugars¹

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The accurate estimation of glycerol in transparent soaps is attended with difficulty on account of the cane sugar which is almost invariably present.

AVAILABLE METHODS FOR GLYCEROL ANALYSIS

It is obvious that the sugar will oxidize, in part at least, and give rise to uncertain results if an attempt is made to determine glycerol by the Hehner method,² in which the sample is oxidized with potassium dichromate in acid solution. The acetin method of Benedikt and Cantor³ is open to the objection that sugars, having numerous hydroxyl groups, are acetylated by acetic anhydride in much the same manner as glycerol.⁴

The alkaline permanganate method of Benedikt and Zsigmondy⁵ should theoretically work on a mixture of glycerol and sugar, since it depends on the supposition that glycerol only is oxidized to oxalic acid by potassium permanganate in alkaline solution. As a matter of fact, however, invert sugar was subsequently found to be quantitatively oxidized to oxalic acid by this method. A test made in our laboratory showed that an amount of oxalic acid equivalent to practically 100 per cent of the theoretical quantity was produced when pure invert sugar was subjected to oxidation with alkaline permanganate in the usual way. This method as applied to straight glycerol analyses has been adversely criticized by Lewkowitsch⁶ and by Allen.⁷ Experiments in our laboratory have indicated that the method gives low results on crude and C. P. glycerols. The method is tedious and cumbersome, and a more convenient method capable of giving better results on glycerol was therefore sought. The isopropyl iodide method of Zeisel and Fanto⁸ was not considered because it has been sharply criticized by Lewkowitsch,⁹ and is moreover complicated, tedious, and of doubtful accuracy in presence of sugar.

The most usual method of determining glycerol in the presence of sugar is of course that of Donath and Mayrhofer,¹⁰ or one of its various modifications, in which the sample is

treated with lime or baryta to render the sugars insoluble, and the glycerol is extracted from the nearly dry mixture with various solvents. Any chemist who has ever tried this method cannot but have been impressed with its tediousness, many chances of losses, and resultant inaccuracy.

The method of Cook,¹¹ which is really an adaption of the Shukoff and Schestakoff¹² method for the determination of glycerol in meat and meat extracts, would not appear to be applicable in the presence of sugar since it is, in effect, merely an acetone extraction of the sample which has been dehydrated by mixing with sand and anhydrous sodium sulfate, and does not render sugar insoluble if present. Methods such as precipitation of glycerol with copper sulfate,¹³ distillation of glycerol *in vacuo* from a solution containing sugar or salts,¹⁴ distillation *in vacuo* with the aid of sandalwood oil¹⁵ or precipitation as sodium glyceride¹⁶ were examined and found inapplicable to the present problem.

EXPERIMENTS WITH THE DONATH AND MAYRHOFFER METHOD

An attempt was made to apply this method to the present problem, using known weights of pure sugars and glycerol of known purity. Glycerol is normally determined in a sample of soap by cracking with dilute sulfuric acid and determining glycerol in the acid filtrate by the dichromate or acetin methods. In the case of a transparent soap containing cane sugar, the acid filtrate would contain the sugar in the form of invert sugar. Hence in the experiments known equal weights of pure dextrose and pure levulose, *i. e.*, invert sugar, were used.

These results showed that continued extraction with acetone gradually dissolved the sugars. The difficulty of determining when extraction was complete and of removing all of the solvent without losing glycerol was so great and the results were so unreliable that no further work was done with the method.

QUANTITATIVE OXIDATION OF SUGAR AND GLYCEROL BY POTASSIUM DICHROMATE IN ACID SOLUTION

It seemed to the authors that it should be possible to oxidize a mixture of sugar and glycerol quantitatively with dichromate. Since the sugar in such a mixture could be readily determined by a method like that of Munson and Walker¹⁷ it would be possible to calculate the glycerol.

¹ Received January 26, 1921.

² *J. Soc. Chem. Ind.*, **17** (1898), 330.

³ *Ibid.*, **7** (1888), 696.

⁴ The acetin method was applied in our research laboratory to 0.5 g. of invert sugar. As dextrose and levulose both contain five hydroxyl groups a pentacetate might be expected to form under suitable conditions. It was found that invert sugar apparently took on 4(C₂H₅O₅) groups, or, as is more likely, acetylated to a mixture of acetates, whose net average was a tetracetate, since the above quantity of invert sugar gave a recovery of 95.3 per cent on the assumption that tetracetates were formed.

⁵ *J. Soc. Chem. Ind.*, **5** (1885), 610.

⁶ "Oils, Fats and Waxes," 5th Ed., I, 447.

⁷ "Commercial Organic Analysis," 4th Ed., XI, 457.

⁸ *Z. landw. Versuchs.*, **5** (1902), 729.

⁹ *Loc. cit.*, I, 450.

¹⁰ *Z. anal. Chem.*, **20** (1881), 383.

¹¹ *J. Assoc. Official Agr. Chem.*, **1** (1915), 279.

¹² *Z. angew. Chem.*, **18** (1905), 294.

¹³ *C. A.*, **6** (1912), 1072.

¹⁴ *Ibid.*, **8** (1914), 772.

¹⁵ *J. Am. Pharm. Assoc.*, **4** (1915), 75.

¹⁶ *C. A.*, **11** (1917), 216.

¹⁷ *J. Am. Chem. Soc.*, **28** (1906), 663; **29** (1907), 541; **34** (1912), 202.