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A PIPETTE METHOD OF MECHANICAL ANALYSIS OF SOILS BASED ON IMPROVED DISPERSION PROCEDURE

By L. B. OLMSTEAD, *Physicist*, LYLE T. ALEXANDER, *Junior Physicist*, and H. E. MIDDLETON, *Associate Physicist*, *Division of Soil Chemistry and Physics, Soil Investigations, Bureau of Chemistry and Soils*<sup>1</sup>

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INTRODUCTION

In all methods of mechanical soil analysis some separations are based on the well-known fact that the smaller particles in water suspensions fall more slowly than larger ones. In elutriation methods the smaller particles are borne upward by a rising water column while larger particles fall. In the early work of the Bureau of Soils the Osborne (20)<sup>2</sup> beaker method was used to make silt and clay separations. Whitney (5) apparently was the first to use the centrifuge to increase the settling velocities of soil particles. The centrifuge method was then worked out independently by Hopkins (12) and Snyder (27). In 1904 the Bureau of Soils published its centrifuge method (5), which with modifications (9) has been used until recently.

The centrifuge method was much more rapid than the beaker method and gave better dispersion than elutriation methods. In this method the silt and clay were separated from the sands by decantations from a shaking bottle, and the silt was separated from the

<sup>1</sup> The authors desire to express their appreciation to those who have assisted in this work. Thanks are due especially to G. B. Bodman of the University of California for soil samples for analysis; to W. H. Fry for microscopical examination of soil separates; to Hubert Lakin, Mrs. Dorothea M. Darnell, and William Stevens of the soil physics laboratory for assistance in the analytical work; and to C. J. Crawley and J. N. Hall of soils investigations' instrument shop for aid in the design and for the construction of apparatus.

<sup>2</sup> Reference is made by italic numbers in parentheses to Literature cited, p. 21.

clay by centrifuging. With trained men checking their silt and clay separations with a microscope micrometer, the analyses generally were reproducible. Dispersion was obtained by shaking a 5-gm. soil sample with 150 c. c. of distilled water containing 1 c. c. of concentrated ammonia. As many as 40 centrifugings were necessary in some cases to give a complete separation. In such cases rubbing the wet sample with a rubber policeman hastened the operation. Later (6) rubber balls with lead centers were put into the shaking bottles to assist in cleaning colloid coatings from sand grains and in breaking colloidal aggregates.

About 1919, when attempts were made to determine quantitatively the amount of colloid in soils, it was found that dispersion was not always so complete as had previously been believed. Davis and Middleton (8) then studied a number of methods of dispersing soils preparatory to their separation by the centrifuge method. They concluded that some chemical assistance was necessary for complete dispersion and recommended treatment with 0.1 N HCl for one hour followed by washing in the centrifuge to remove excess acid and exchanged ions. The sample was then shaken seven hours in 0.05 N NaOH.

The introduction of the pipette method developed independently by Robinson in Wales (23), Krauss in Germany (16), and Jennings, Thomas, and Gardner in the United States (13), necessitated a more stable and more complete dispersion than was required in the centrifuge method. A study of dispersion was undertaken by the first commission (soil physics) of the International Society of Soil Science, that resulted in the adoption at the First International Soil Science Congress held in Washington in 1927 of two methods for mechanical soil analysis (19). One method, called the practical method, secured dispersion by shaking with ammonia, boiling, and rubbing. The other method, designed to secure complete dispersion, prescribed (1) treatment with hydrogen peroxide to remove organic matter, (2) treatment with 0.2 N HCl followed by washing to remove alkaline earth carbonates and adsorbed bases, and (3) treatment with ammonia or sodium carbonate to secure dispersion. The latter method will be referred to hereafter as the international method. No attempt was made to fix the size classes, but an arbitrary relationship between sedimentation velocity and particle size was established.

These two methods, one intended to give partial and the other complete dispersion, represent two diverse purposes of mechanical analysis. In so-called practical methods such as the first the endeavor is made to avoid removing the coating from soil particles or breaking apart slightly cemented sand grains or aggregates of finer material which would not be resolved in tillage operations or under severe weather conditions in the field. Hilgard (11, p. 88) objects to the use of acids to disintegrate compound particles, particularly in calcareous sands. In practical methods it is difficult to determine just when a proper degree of dispersion has been reached. For example, Bouyoucos (4), in an effort to secure an analysis of the ultimate natural soil structure, does not give the sample as rigorous treatment as the international practical method proposes.

A mechanical analysis based on complete dispersion does not give so true a picture of field structure and associated physical properties as does the practical method, but it does give information more val-

uable for most purposes. It gives the amount of coarser material stripped of adhering material, the amount of colloid, and usually the approximate amount of organic matter. Since the colloid is the seat of most soil chemical reactions and governs or strongly influences most of the physical properties, the quantitative determination of the finest soil fraction is the most important single determination in a mechanical analysis. In the case of concretions of irreversible colloidal material having adsorptive properties less than that of dispersed colloid but more than that of solid mineral grains, there is some question as to how far dispersion should be carried.

This bulletin reports an investigation of methods of obtaining dispersion of soil material, with special reference to the effect of acid treatment and the use of alkaline dispersing agents. A method for the removal of organic matter from manganese soils is included. A procedure for mechanical soil analysis, based on the most satisfactory of several methods of soil dispersion investigated, is here outlined. This is the method now used in the Bureau of Chemistry and Soils. This method is designed to secure complete dispersion of soil material below 2 mm. in diameter after organic matter and water-soluble material have been removed. Sieves are used to make all separations except those at 5  $\mu$  and 2  $\mu$ , where the pipette is used.

### DISPERSION

Good dispersion is the key to accurate mechanical analysis. By complete dispersion is meant the removal of colloid coatings on sand and silt grains and the separation of aggregates into single grains or groups smaller than 2  $\mu$  equivalent diameter. The problem of dispersion is, (1) to obtain, and (2) to maintain suspensions free from aggregation. The chemical and mechanical aids to dispersion considered are treatment with (1) hydrogen peroxide, (2) hydrochloric acid, (3) water, and (4) dispersing agents<sup>3</sup>; electro dialysis; and shaking.

#### HYDROGEN PEROXIDE TREATMENT

In the analysis of soils high in organic matter G. W. Robinson (22) found that dispersion was aided by the removal of organic matter with hydrogen peroxide. He ascribes this effect to the removal of material which cements soil particles into aggregates strongly enough to resist ordinary dispersion methods. Although it is not necessary to remove organic matter in order to secure dispersion of the mineral portion of the soil (14), its presence interferes with the separation of the material into size classes. For this reason all samples for routine analysis in this laboratory are treated with 6 per cent  $H_2O_2$ , in accordance with the international method.

When manganese dioxide is present in sufficient quantity it is not possible to remove the organic matter by the usual treatment. Since manganese dioxide is the only form of manganese which catalytically decomposes hydrogen peroxide so rapidly that the organic matter is very slowly attacked, the obvious remedy is to convert it into some other manganese compound. W. O. Robinson (25) found that if a small fraction of 1 per cent of manganese dioxide was present it could be destroyed by evaporating the sample with a very small excess of

<sup>3</sup> Ammonium hydroxide, sodium hydroxide, sodium carbonate, and sodium oxalate.

oxalic acid. In this investigation, if manganese dioxide was known to be present or its presence was indicated by the violence of the reaction, acetic acid was added, followed by hydrogen peroxide, the purpose being to convert the manganese dioxide into the manganous form which does not interfere with the decomposition of organic matter by hydrogen peroxide.

The effect of acetic acid treatment was determined on duplicate samples of Wabash silt loam from Nebraska. The samples contained about 3 per cent organic matter and 0.11 per cent manganese, calculated as manganous oxide (MnO). The rate of decomposition of hydrogen peroxide indicated that the manganese is not in the form of manganese dioxide. To 10-gm. samples of this soil were added various amounts of finely precipitated manganese dioxide and acetic acid. The amount of carbon dioxide evolved upon the addition of 20 c. c. or more of 6 per cent hydrogen peroxide is shown in Table 1. The amount of carbon dioxide evolved decreased rapidly with the addition of manganese dioxide. The presence of 0.1 per cent (10 mg.) of manganese dioxide did not interfere with the decomposition of organic matter when 10 mg. of acetic acid was used. Blanks run by treating acetic acid with hydrogen peroxide in the presence of manganese dioxide showed practically no evolution of CO<sub>2</sub> due to oxidation of the acid.

TABLE 1.—*Effect of manganese dioxide and acetic acid on the liberation of carbon dioxide from soil treated with hydrogen peroxide*

Soil	Manganese dioxide added	Acetic acid added	Hydrogen peroxide added	Carbon dioxide evolved
	Mg.	Mg.	C. c.	Mg.
Wabash silt loam (10 gm.)	0	0	20	105.8
Do.	10	0	20	55.3
Do.	50	0	20	17.5
Do.	100	0	20	4.0
Do.	0	10	20	112.2
Do.	10	10	20	112.7
Do.	50	20	20	10.5
Do.	50	20	40	74.1
Do.	50	20	60	174.2

TABLE 2.—*Organic matter removed by the Robinson method from manganese soil treated with acetic acid*

Soil	Manganese dioxide added	Acetic acid added	Hydrogen peroxide (6 per cent) added	Organic matter removed
	Mg.	Mg.	C. c.	Per cent
Wabash silt loam (10 gm.)	100	0	40	0.81
Do.	100	100	40	1.38
Do.	100	0	60	1.19
Do.	100	100	60	1.73
Do.	100	0	80	1.48
Do.	100	100	80	1.85
Do.	0	0	( <sup>2</sup> )	3.10
Do.	10	10	( <sup>2</sup> )	2.95
Do.	100	100	( <sup>2</sup> )	2.85
Do.	200	200	( <sup>2</sup> )	2.93

<sup>1</sup>A slight excess over the equivalent amount of acetic acid was used.

<sup>2</sup>An excess of hydrogen peroxide.

When soil is treated with hydrogen peroxide the organic matter either is destroyed with the evolution of carbon dioxide or is brought into solution. Table 1 shows only the amount of organic carbon oxidized to  $\text{CO}_2$ . Table 2 gives the total organic matter removed by both means when the same soil, containing amounts of manganese dioxide, is treated with different amounts of hydrogen peroxide. The quantity of acetic acid added was a slight excess over the molecular equivalent of the manganese dioxide present. The addition of acetic acid is quite effective when the soil contains 1 per cent of manganese dioxide, 40 c. c. of hydrogen peroxide removing nearly as much organic matter when acid was present as was removed by 80 c. c. of peroxide without acid. In the case of the last four samples hydrogen peroxide was added until decomposition of the organic matter was complete. The sample was then washed and dried and the amount of organic matter determined by loss in weight, according to the method of W. O. Robinson (25), who removed 3.03 per cent of organic matter from this soil. The results show that organic matter can be completely removed in the presence of manganese dioxide up to 2 per cent. It is possible that soils containing both manganese dioxide and lime carbonates would not respond so readily to acetic acid and hydrogen peroxide treatment. The amount of peroxide required increases with the amount of manganese dioxide present. The removal of organic matter from the sample containing 2 per cent manganese dioxide required the use of excessive quantities of hydrogen peroxide. The removal of organic matter is not essential to the method described in this bulletin and may well be omitted in the case of soils high in manganese dioxide and low in organic matter.

In some cases hydrogen peroxide decomposes the organic matter slowly and incompletely. In one case, recently noted, hydrogen peroxide attacked the organic matter only slightly. After the addition of 1 or 2 mg. of manganese dioxide the organic matter was quickly attacked and apparently completely decomposed.

#### ACID TREATMENT

In the pipette method of mechanical analysis it is necessary to prepare soil suspensions in such manner that they may be kept in a sedimentation chamber for several days without coagulation into flocs larger than  $2\mu$  equivalent diameter. For this purpose treatment of the soil sample with hydrochloric acid has been found helpful because it dissolves alkaline earth carbonates and removes "exchange bases" which later might cause flocculation of the suspension. There are, however, some objections to the use of acid treatment.

In the international method the soil, after decomposition of organic matter, is treated with enough hydrochloric acid to decompose the carbonates and still leave 250 c. c. of solution of 0.2 N HCl. Such rigorous treatment dissolves sesquioxides of iron and aluminum and some silica, although it may not dissolve all the carbonates. An example of this is given by Bodman (3). When a sample of Montezuma clay adobe subsoil from California was boiled with 0.1 N HCl, the amount of acid consumed was equivalent to 9.08 per cent calcium carbonate; but if treated with 0.2 N HCl at room temperature in accordance with the international method, acid equivalent to only 1.27 per cent of calcium carbonate was consumed, and part of this

consumption must be ascribed to the solution of 0.81 per cent of sesquioxides and silica. The amount of carbonates in this soil, determined by the official method (1 p. 22-24), was 8.28 per cent. The mechanical analysis of this material is given later in Table 5.

Even though carbonates might be removed completely and easily, such removal is undesirable in some soils, such as glacial soils containing large amounts of limestone or dolomite, because it would be likely to change soil textural classifications. Other soils may have relatively permanent grains or crystals of precipitated carbonate whose presence should be expressed in the results of mechanical analysis. An example of this is the Goliad fine sandy loam subsoil from Texas. Ten-gram samples of this material were treated with varying amounts of acid, following the regular peroxide treatment. The washed residues were shaken with 5 c. c. of normal sodium carbonate and analyzed by the pipette method described later. The results are shown in Table 3. An examination of the material by William H. Fry, soil petrographer of this division, showed that the carbonate material, amounting to about 38 per cent, was present largely in the form of small calcite crystals.

TABLE 3.—*Effect of acid treatment on the results of mechanical analysis<sup>1</sup> of Goliad fine sandy loam (35 to 48 inches)*

Treatment	Sands 2.0 to 0.05 mm.	Silt 0.05 to 0.005 mm.	Clay <0.005 mm.	Colloid <0.002 mm.	Hydrogen peroxide and solu- tion loss
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
No acid.....	36.9	38.6	24.2	18.3	0.2
2 c. c. N HCl.....	36.6	37.9	24.1	18.1	1.4
10 c. c. N HCl.....	36.6	34.7	23.3	19.4	5.3
Carbonates removed+10 c. c. N HCl.....	31.4	9.6	19.9	18.0	39.0
No acid, dispersed with sodium oxalate....	37.0	34.3	28.4	21.0	.2

<sup>1</sup> Results in all mechanical analysis tables are on oven-dry basis (105° C.), and silt is reported by difference.

For some special purposes, such as dam construction or other engineering uses, an analysis based on vigorous acid treatment might be preferred for a soil such as the Goliad, although an analysis without acid treatment together with a determination of total carbonates would be preferable. Analyses by both methods are required to furnish a complete description of the soil.

These analyses (Table 3) are presented primarily to show that acid treatment may alter the amount of material in the different size classes enough to change markedly the textural classification. Incidentally they indicate that acid treatment is not essential to good dispersion, a matter which will now be examined more fully.

Since it appears that treatment with 0.2 N HCl is not always effective in removing the carbonates from soil and in some cases presents a distorted picture of the soil, it seemed desirable to ascertain whether complete dispersion can be obtained without its use.

Table 4 gives the results of mechanical analysis of a number of soils with and without acid treatment. All samples (10 gm.) were first treated with hydrogen peroxide. Those given acid treatment were allowed to stand overnight in HCl solution of sufficient strength to leave 150 c. c. of 0.2 N HCl after the carbonate content is removed.

The difference in dry weight of the sample before and after pretreatment is reported as solution loss. This comprises the loss of organic matter, sesquioxides ( $R_2O_3$ ), silica, lime carbonates, or other dissolved material. All samples were peptized by shaking with 150 c. c. of water containing 10 c. c. of 0.5 N  $Na_2C_2O_4$ . These samples embrace a wide range of soil types and are representative of soil samples received for analysis.

TABLE 4.—*Effect of acid pretreatment on mechanical analysis of soils*

Sample No.	Soil type and source	Depth	Treatment	CaCO <sub>3</sub>	Sand	Silt	Clay	Colloid	Solution	R <sub>2</sub> O <sub>3</sub>
				Per cent	2.0 to 0.05 mm.	0.05 to 0.005 mm.	<5 μ	<2 μ	loss	loss
425250	Ruston fine sandy loam (Mississippi)	Inches 50 to 70	Acid	0	56.2	13.9	29.7	25.6	0.1	0.2
425250	do	do	No acid	0	56.4	14.1	29.3	27.0	.1	.03
304041	Isabella loam (Michigan)	1 to 2	Acid	0	58.0	30.5	6.8	4.2	4.5	.5
304041	do	do	No acid	0	57.0	29.9	8.7	3.8	4.3	.1
2437209	Coxville clay loam (South Carolina)	5 to 15	Acid	0	17.7	26.2	55.1	45.5	1.2	.6
2437209	do	do	No acid	0	17.5	25.2	56.3	48.8	1.0	.05
376337	Paxton very fine sandy loam (Nebraska)	24 to 44	Acid	5.6	52.1	27.9	11.4	9.4	8.6	1.3
376337	do	do	No acid	5.6	55.1	24.5	20.0	16.1	.4	.01
-----	Houston black clay (Texas)	0 to 12	Acid	11.7	4.1	25.0	56.3	50.7	14.7	1.7
-----	do	do	No acid	11.7	5.9	28.5	63.8	53.8	1.9	.1
-----	Badob (Sudan-Gezira)	do	Acid	5.9	6.3	18.0	69.7	62.4	6.0	2.2
-----	do	do	No acid	5.9	8.4	14.7	75.9	64.6	1.0	.04
-----	Rendzina (Ceje)	do	Acid	5.1	31.8	26.4	30.2	25.7	11.6	2.5
-----	do	do	No acid	5.1	33.0	29.0	34.3	28.8	3.6	.6
-----	Podsol (Zdar)	do	Acid	0	25.7	35.4	37.1	30.9	1.6	1.3
-----	do	do	No acid	0	25.6	36.4	37.8	30.2	.2	.05

<sup>1</sup> Determination by Robinson and Holmes (26).

The last three sets of analyses are from samples submitted for analysis to some 20 laboratories by a committee of the first commission of the International Society of Soil Science. The unpublished results collected by this committee and furnished us by R. O. E. Davis give the amount of organic matter in the badob, rendzina, and podsol as 1 per cent, 4.6 per cent, and 0.4 per cent, respectively. The average (unpublished) result collected by this committee from the reporting laboratories for the percentage 2 μ clay (colloid), when hydrogen peroxide and hydrochloric acid treatment was used, was 60.5 for the badob, 26.7 for the rendzina, and 28.6 for the podsol. Joseph and Snow (14), using their method which consists of shaking with sodium carbonate, puddling, and decanting by the beaker method without the use of peroxide or acid, obtained 63.3 per cent, 29 per cent, and 27 per cent for the amount of 2 μ clay in the badob, rendzina, and podsol, respectively. W. O. Robinson (25) reports 3.6 per cent organic matter in the Houston black clay, determined by the hydrogen peroxide method, and 4.2 per cent determined by combustion. It is apparent from the results in Table 4 that the organic matter was not all removed by peroxide treatment from either the Houston black clay or the rendzina.

Recently Bodman (3) published analyses of five well-selected California soils, using two methods. At request of the writers Doctor Bodman furnished this laboratory with samples of these soils. The results of the analyses, together with those of Bodman, are shown in

Table 5. The Montezuma clay adobe soil has a high content of organic matter, and its subsoil has a high content of lime. The Fresno sandy loam has a high content of sodium carbonate; the Aiken clay loam subsoil has a high percentage of iron; and the last is the difficultly dispersible hardpan horizon of the San Joaquin sandy loam.

TABLE 5.—*Mechanical analyses of soils by comparative methods*

Soil type	Depth	Method <sup>1</sup>	Sand 2 to 0.05 mm.	Silt 0.05 to 0.005 mm.	Clay <0.005 mm.	Colloid <sup>2</sup>
	<i>Inches</i>		<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
Montezuma clay adobe.....	0 to 12.....	A	25.0	32.5	36.9	28.1
Do.....	do.....	B	24.0	39.3	31.1	15.0
Do.....	do.....	C	23.8	32.8	40.2	34.3
Do.....	32 to 40.....	A	30.8	34.1	32.3	26.2
Do.....	do.....	B	27.5	43.0	26.7	14.4
Do.....	do.....	C	27.3	34.6	37.9	31.7
Fresno sandy loam.....	0 to 12.....	A	61.8	29.3	7.5	3.7
Do.....	do.....	B	64.2	26.4	7.8	3.7
Do.....	do.....	C	62.3	28.8	8.2	5.5
Aiken clay loam.....	12 to 14.....	A	6.5	34.8	58.3	45.7
Do.....	do.....	B	7.0	35.2	57.3	43.4
Do.....	do.....	C	8.2	33.3	58.1	48.6
San Joaquin sandy loam.....	25 to 30.....	A	70.7	18.0	10.7	4.9
Do.....	do.....	B	58.4	25.5	15.5	6.4
Do.....	do.....	C	66.1	15.7	17.9	11.8

<sup>1</sup> Method A, hydrogen peroxide-hydrochloric acid pretreatment, shaken with ammonia. Method B, rubbed with ammonia, shaken with ammonia; separates treated with hydrogen peroxide. Method C, hydrogen-peroxide pretreatment, shaken with sodium oxalate.

<sup>2</sup> Methods A and B, colloid pipetted at 1  $\mu$ . Method C, colloid pipetted at 2  $\mu$ .

The results given under methods A and B are those published by Bodman, and under method C those obtained in this laboratory. Method A is the international method designed to give complete dispersion. In methods B and C hydrochloric-acid treatment is omitted, hydrogen peroxide being used as the last step in the analysis of the former and the first step in that of the latter. In method B the sample, wet with a dilute ammoniacal solution, is rubbed with a rubber pestle for 10 minutes before being shaken. In methods A and B 5-gm. samples dispersed with ammonia and in method C 10-gm. samples dispersed with sodium oxalate are used.

That the larger amount of colloid obtained by method C is not entirely due to the separation being made at 2  $\mu$  instead of 1  $\mu$  is shown by the fact that the amount of clay is also greater by this method.

The results in Tables 4 and 5, typical of hundreds of analyses of soils developed from a wide range of materials and under a wide range of climatic conditions, are presented to show that in no case is there any indication that acid treatment is necessary to dispersion. Joseph and Snow (14) reach the same conclusion in the case of the heavy alkaline soils of Sudan. In their beaker (or centrifuge) method in which the repeated puddling and decantation of suspensions produces a washing effect, flocculation merely increases the number of decantations required for complete separation into size classes. In the pipette method, however, it is necessary for the material to remain in the sedimentation cylinder without coagulation for at least two or three days. Duplicate pipettings of the same suspension taken a week apart show no evidence of flocculation, even though acid pretreatment was omitted. Since acid treatment appears to serve no essential



purpose, and may be objectionable, it is omitted in the method in routine analysis.

#### WASHING

After the soil sample has been pretreated it is washed to remove soluble organic matter, dissolved lime, replaced ions (especially bivalent and trivalent cations), hydrogen peroxide, and excess acid if hydrochloric or acetic acid is used. Experiments by Wiegner (29) indicate that the removal of electrolytes by thorough washing is necessary in order to obtain a high degree of dispersion by any subsequent treatment. Soils from humid regions are low in soluble salts and usually do not require much washing to prevent flocculation in the dispersion cylinder. For soils subject to low rainfall, however, thorough washing is essential to good dispersion.

As washing proceeds the soil, flocculated by acid treatment, gradually deflocculates again, slowing the filtering rate. Occasionally, however, a soil such as the Cecil of Georgia does not deflocculate on washing. For this reason it was chosen for the following test. Eight duplicate 10-gm. samples were given hydrogen peroxide and hydrochloric-acid treatment. The samples were then washed 1, 2, 4, 5, 6, 7, 9, and 15 times, respectively, by the method described later, and the amount of clay was determined by the pipette method. There was no correlation between the number of washings and the percentage of clay, the results agreeing within the limits of experimental error. The Cecil, a soil developed in a humid climate, is low in soluble salts and does not require much washing. The routine treatment is 6 washings of about 125 c. c. each, removing the solution completely each time. The entire operation requires about one day. When acid treatment is omitted only a fraction of 1 per cent of soluble matter is removed by washing, and no chemical analysis of it is made.

#### DISPERSING AGENTS

The dispersing agents most used in mechanical analysis are ammonia, sodium hydroxide, and sodium carbonate. In the centrifuge method, in which the soil after dispersion is repeatedly washed and the percentage of clay determined by difference, any of the dispersing agents may be used. When ammonia is used in the pipette method, the amount of ammonia held by the dried colloid is somewhat uncertain. The pipetted solutions are alkaline and consequently absorb carbon dioxide from the air, giving an uncertain increase of weight of the aliquot. If sodium oxalate is used as a dispersing agent this difficulty disappears.

Ammonium and sodium hydroxides give good dispersion when calcium and magnesium carbonates have been removed from the soil. This removal requires a thorough acid pretreatment and washing of the sample. If calcium carbonate remains in the soil after pretreatment, sodium carbonate is a better dispersing agent than either sodium or ammonium hydroxide because the carbonate decreases the solubility of the calcium carbonate while the hydroxides may produce large amounts of the flocculating calcium ions. Sodium oxalate is even better as a dispersing agent than sodium carbonate. Unlike the hydroxides, sodium oxalate frees no alkaline earth hydroxides as a result of chemical equilibriums established. Also, the solubility of the oxalates of calcium and magnesium is much lower than that of the

corresponding carbonates. The oxalate does not react with carbon dioxide from the air.

Surface layers of three soil types were treated with hydrogen peroxide but no acid, and shaken with the four dispersing agents, equimolecular concentrations ( $10 \text{ c. c. } \frac{N}{2}$ ) being used. This is a smaller amount of ammonia than is generally used but is an adequate quantity of the other deflocculents. Table 6 gives the quantities of  $5 \mu$  and  $2 \mu$  clay obtained by the pipette method. The total lime computed as  $\text{CaCO}_3$  was less than 2 per cent for the Carrington and Wabash soils and 13.9 per cent for the Houston. The results indicate that ammonium and sodium hydroxides are not suitable deflocculents for soils which have not had acid pretreatment, and that, of the other two, sodium oxalate gives consistently higher results. The activity of the oxalate ion may cause this increased dispersion. Oxalic acid is often used to clean minerals for petrographic examination. Tests are soon to be made upon the efficiency of lithium oxalate as a deflocculent.

TABLE 6.—Yield of clay obtained with various dispersing agents <sup>1</sup>

Soil type and source	Percentage of clay obtained from treatment with—							
	Ammonia		Sodium hydroxide		Sodium carbonate		Sodium oxalate	
	$5 \mu$	$2 \mu$	$5 \mu$	$2 \mu$	$5 \mu$	$2 \mu$	$5 \mu$	$2 \mu$
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
Wabash silt loam (Nebraska)-----	30.7	24.1	33.2	28.5	33.1	28.3	34.5	29.6
Houston black clay (Texas)-----	38.4	26.4	44.5	30.6	61.3	53.5	63.8	53.8
Carrington loam (Iowa)-----	7.8	6.4	24.9	21.8	23.4	19.9	24.7	22.9

<sup>1</sup> Average of duplicate determinations.

After it was determined that sodium oxalate gave the best dispersion of soils containing calcium carbonate, tests were made on soils containing gypsum, which is much more soluble in water. Table 7 gives the results of comparative analyses of some special samples of gypsum soils unclassified as to soil type. Nos. 28742 and 28743 are from South Dakota, Nos. 28655 and 28668 from Montana, No. 29735 from Hungary, and No. 5218 from Kansas. Analyses are made by the international method, dispersing with sodium carbonate and by our method, described later, in which acid treatment is omitted and sodium oxalate is used as the dispersing agent. With the exception of sample No. 5218, which contains only 1 per cent of gypsum, the routine washing was insufficient to remove all the gypsum from the soils not given acid treatment. The routine amount of sodium oxalate ( $10 \text{ c. c. } \frac{N}{2}$ ) was sufficient to remove from solution the calcium ions formed during the three days in which the suspension stood in the sedimentation chamber. There was no indication of flocculation. The amount of clay and colloid obtained was, in each case, greater by method 2 than by method 1. It is likely that in soils containing larger quantities of a soluble calcium salt, such as gypsum, more thorough washing, acid treatment, or electro dialysis may be required for dispersion unless larger quantities of sodium oxalate are used.

TABLE 7.—Mechanical analysis of soils containing gypsum

Sample No.	Gypsum <sup>1</sup>	Method	Sand, 2 to 0.05 mm.	Silt, 0.05 to 0.005 mm.	Clay <5 μ	Colloid <2 μ	Solution loss
	<i>Per cent</i>		<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
28742	2.52	<sup>2</sup> 1	37.9	14.5	42.9	40.1	4.7
28742	2.52	<sup>3</sup> 2	37.3	16.8	43.8	40.8	2.0
28743	2.28	1	55.1	10.6	26.3	24.9	8.2
28743	2.28	2	56.2	10.9	31.7	26.3	1.3
28655	1.61	1	14.8	21.1	51.2	38.9	12.9
28655	1.61	2	15.5	23.5	60.0	44.3	1.2
28668	5.29	1	31.6	31.0	31.0	27.1	6.5
28668	5.29	2	31.7	30.7	32.8	28.6	4.8
29735	2.82	2	3.3	46.1	48.2	38.4	2.4
5218	1.04	2	3.6	34.0	60.9	52.7	1.6

<sup>1</sup> Computed as CaSO<sub>4</sub>·2H<sub>2</sub>O from analysis of total SO<sub>3</sub>.

<sup>2</sup> Hydrogen peroxide and hydrochloric acid treatment; dispersed with sodium carbonate.

<sup>3</sup> Hydrogen peroxide treatment only; dispersed with sodium oxalate.

Occasionally a sample flocculates in the sedimentation chambers. This may occur with any of the dispersing agents and with or without a mild acid treatment. The difficulty may be due to the presence of some soluble soil material or of an excess or deficiency of dispersing agent. It is impossible to determine accurately from inspection which is the cause. No investigation of the electrokinetic potentials of these suspensions has been made, but a simple remedy has been found which usually restores dispersion. A Pasteur-Chamberland suction filter is placed in the suspension, and the liquid volume is reduced from 1,000 c. c. to some smaller definite volume, say 200 or 250 c. c. Distilled water is then added to make up the original volume. The suspension is thoroughly stirred and allowed to stand. If flocculation again takes place measured amounts of deflocculent are added until the suspension is stable. This procedure leaves some uncertainty as to the weight of dispersing agent to be deducted from the pipetted aliquots. It is assumed that the filtrate removed has the same concentration as the material left in the cylinder.

#### ELECTRODIALYSIS

Another means of effecting dispersion is electro dialysis. For samples dispersed in this way the Mattson cell (17) was used. By this method carbonates and "exchange bases" as well as the chlorides, sulphates, and phosphates can be removed; so that, in addition to giving an excellent pretreatment for dispersion, electro dialysis enables one to determine the amount of "exchange bases" in the soil. The advantage of electro dialysis over hydrochloric acid treatment is that it does not introduce into the soil a foreign ion which must be washed out later. No washing is required after electro dialysis. The method requires considerable extra equipment, extra time, additional transfers of the sample with attendant possibilities of loss, and, where determination of exchangeable bases is not desired, yields no results that can not more easily be obtained by acid treatment.

#### SHAKING

After the hydrogen peroxide treatment, the sample is washed, dried at 105° C., and weighed. It is this weight which is later made the basis for the determination of the percentage of material in each size class when the results are used for textural classification. The

dried sample is transferred to a 250 c. c. nursing bottle, the dispersing agent is added, and the material is shaken overnight. The shaker has a horizontal movement of 10 cm. and a rate of 120 complete oscillations per minute. Joseph and Snow (14) find 2 hours' shaking to be sufficient. In the experiments of Davis and Middleton (8) the material was shaken 7 hours. Since the samples used in the laboratory were first dried at 105° C. it is possible that longer shaking is required. Although 16 hours is longer shaking than necessary, it is convenient to let the shaker run overnight. The amount of grinding of soil grains is negligible.

## SEPARATION INTO SIZE CLASSES

### SIZE CLASSES EMPLOYED

Early in the work of the former Bureau of Soils, the soil sample for mechanical analysis was separated into the following seven size classes:

Diameter, mm.	Conventional name
2.0 to 1.0-----	Fine gravel.
1.0 to 0.5-----	Coarse sand.
0.5 to 0.25-----	Medium sand.
0.25 to 0.10-----	Fine sand.
0.10 to 0.05-----	Very fine sand.
0.05 to 0.005-----	Silt.
<0.005-----	Clay.

Analyses based on the percentage by weight of these size classes are used by the bureau for the textural classification of soils (?). The bureau's records contain some 50,000 analyses based on these size classes, and for this reason no change has been made in these classes except to report also the amount of material below 2  $\mu$  effective diameter. This is called the "colloid" fraction. The 5  $\mu$  clay and the 2  $\mu$  "colloid" both contain all particles below diameters of 5  $\mu$  and 2  $\mu$ , respectively. Usually less than 10 per cent of the clay is between 5  $\mu$  and 2  $\mu$  in effective diameter. Most of the material below 2  $\mu$  has a diameter of only a few tenths of a micron or less, so this fraction may be regarded as essentially colloidal in character. In addition to the eight size classes, this laboratory reports the loss by pretreatment of organic matter and soluble material. When acid treatment is omitted the solution loss represents roughly the amount of organic matter. (See Table 4.) Neither the solution loss nor the amount of 2  $\mu$  colloid is used in textural classification. They are reported for information only.

The number of sand fractions may be greater than necessary, but these separations are quickly and easily made. The decimal system of grading, with separations made at 2.0, 0.2, 0.02, and 0.002 mm., would not furnish an adequate number of size classes for the extensive system of textural classification of soils used in the United States. Although the coarse clay is silty in character and the larger silt might be classed as very fine sand it now seems desirable to maintain the present size classification. The simplest changes would be to omit the 5  $\mu$  separation, place the material between 5  $\mu$  and 2  $\mu$  in the silt class, and also omit the 1 mm. sand separation, throwing the material between 2 mm. and 0.5 mm. into one class.

## METHOD OF SEPARATION

After the sample has been shaken with dispersing agent it is wet sieved through a Tyler 300-mesh phosphor bronze twilled wire-cloth screen, which passes material finer than  $50 \mu$  equivalent diameter. All the clay and the finer portion of the silt is washed through the sieve into a 1-liter sedimentation chamber. The silt remaining with the sands is dry sifted through another 300-mesh sieve added to the nest of sand sieves.

The sedimentation chamber contains only fine material with low settling velocities where the accuracy of pipette sampling is greatest. The suspension in the sedimentation chamber is stirred, and an aliquot is quickly taken with a 25 c. c. Lowy automatic pipette. After the suspension has stood 77 minutes (at  $20^\circ \text{C}$ .), and again after standing 8 hours, aliquots are taken at a depth of 10 cm., the material in the chamber being stirred thoroughly each time at the beginning of the sedimentation period.

## SEDIMENTATION VELOCITY AND PARTICLE SIZE

All decantation or pipette methods of determining size classes depend on the relation of settling velocity to particle size—and this relationship is not accurately known. The relationship most used is Stokes's, formula (28) for the fall of a solid sphere in a viscous fluid,

$$V = \frac{2}{9} g \frac{(d-d')}{\eta} r^2$$

where  $V$  is the settling velocity of a sphere of radius  $r$  and density  $d$  in a medium of density  $d'$  and viscosity  $\eta$ , under a gravitational acceleration or centrifugal force,  $g$ .

The international method arbitrarily fixed 8 hours as the time for a particle  $2 \mu$  in diameter to fall 10 cm. in water at  $20^\circ \text{C}$ . This is the value determined experimentally by Atterberg (2), although he does not state the temperature. If Stokes's law holds, as it is assumed in determining settling velocities in other sizes, then fixing the relation between settling velocity and particle size is equivalent to fixing the density of the soil particles. Assuming  $d' = 1$ , taking the values of the viscosity of water given in the international critical tables, and the above values for  $V$  and  $r$ , Stokes's formula gives 2.61 for the density of the soil particle  $2 \mu$  in diameter. That this settling velocity may not be very accurate is indicated by the fact that densities of larger particles, computed from Atterberg's settling velocities and Stokes's formula, range from 1.64 to 2.03.

Joseph and Snow (14) use a settling velocity of 10 cm. in 7.5 hours, which corresponds to an average particle density of 2.71. Most specific-gravity measurements of clay give values above 2.7. Robinson and Holmes (26) have shown that the average chemical composition of soil colloid is quite different from the composition of the sand fraction. The higher specific gravity of the colloid may be due, then, either to difference in composition or to the change in density of water in the micropores and in the surface films of high curvature and large total area, surrounding the particles. For purposes of mechanical analysis it is important to know not only the average density of the soil particle of diameter  $2 \mu$ , but also the character of the particle, whether it is a solid mineral grain or an aggregate, or

whether it is a gel or has a gel coating. The particles are too small to permit the application of the usual petrographic methods.

There is also some uncertainty as to the validity of Stokes's law for such a large range of particle shapes and sizes. Millikan (18) found it necessary to add a correction to Stokes's formula for spheres falling in a gas when the mean free path of the gas molecules exceeded a small fraction of the diameter of the sphere. In applying Stokes's law to the settling of sludges, C. S. Robinson (21) used the viscosity and density of the suspension instead of the solution and a coefficient other than  $2/9$  for the particle shape factor. It is proposed to study the relationship of settling velocity and particle size in the near future. In the meantime it seems best to conform to the relationship used in the international method.

The average density of soil particles at any given size is chiefly a matter of academic interest if size fractions are expressed in terms of settling velocities, as recommended by G. W. Robinson (24) rather than in terms of equivalent diameters. But in a method in which five separations are made by sieving and only two by settling velocities, and the results are for the use of field investigators, it seems preferable to hold to the concept of particle size.

Joseph and Snow (14) have shown that, for five soils, considerable variation in temperature, time, or depth of pipetting produces a negligible error in the determination of  $2\mu$  clay but that a higher relative accuracy is required for larger particles. It has been noticed in this laboratory that the higher the sedimentation velocity the more difficult it is to secure good checks from pipette sampling. One reason is that, after the suspension has been stirred, several minutes are required for the turbulent motion to die down and permit settling to proceed undisturbed.

Formerly when all material smaller than  $100\mu$  was sieved into the sedimentation chamber, a pipette determination was made at  $50\mu$ . The pipette was then fitted with a metal tip, closed at the bottom, and provided with six small equally spaced holes drilled horizontally through the wall of the tip near its lower end. This tip assisted greatly in securing good duplicates, but it was found preferable to make this separation with a finer sieve. The tip was then used in the determinations of clay and colloid in an effort to secure a cross section of the liquid at the desired depth. Observations on particles of lampblack indicated that a pear-shaped volume, with the tip near the bottom of the pear, was removed by this means. The results thus obtained were no more consistent than those obtained with the plain straight glass tip so that the removable tip was abandoned. According to Kohn (15), who has worked out the hydrodynamic principles involved, a pipette with any kind of tip, straight or bent, should remove a sphere of liquid whose center is the opening of the pipette tip. It is necessary that the rate of filling the pipette be high in comparison with the settling velocity of the largest particles.

## OUTLINE OF METHOD

### GENERAL STATEMENT

In view of the foregoing facts the writers desire to present a detailed method of procedure for carrying out the mechanical analysis of soils. This procedure is the one now in use in the Bureau of Chemistry and

Soils. It differs from the international method in certain particulars, viz, the omission of hydrochloric acid treatment, the method of filtering and washing, the drying and weighing of the sample after pre-treatment, the use of sodium oxalate as dispersing agent, the use of a finer sieve for wet sieving, and the fact that there is only a single transfer of the sample before separation into size classes. This procedure is more rapid and, it is believed, more accurate than the international method.

#### PREPARATION AND DISPERSION OF SAMPLE

The sample of soil for analysis is mixed with a large spatula, and is then quartered. The quarter reserved for analysis is rolled with a wooden rolling-pin to break up clods and then is passed through a sieve with 2-mm. round holes. Care is taken in all mixing and quartering to see that the fine material on the paper is properly distributed. Samples are run in sets of eight.

Two samples of each soil are weighed out at the same time, a 10-gm. sample for analysis and a 5-gm. sample for moisture determination. The latter is dried at 105° C. for 16 hours, cooled in a desiccator, and weighed. The 10-gm. sample is put into a 250 c. c. Pyrex electrolytic beaker.

To the 10-gm. sample is added 40 c. c. of 6 per cent hydrogen peroxide, and the beaker is covered with a watch glass. The beaker is frequently shaken in order to bring all the organic matter in contact with the peroxide. When the reaction has quieted down, more peroxide may be added where experience indicates that it is needed, and the beaker is placed on a slow steam bath overnight. Then if all organic matter does not appear to have been removed, more hydrogen peroxide of full strength (about 30 per cent) is added. If manganese dioxide is known to be present or its presence is indicated by decomposition of the hydrogen peroxide without removal of organic matter, then a small quantity of acetic acid is added. About 50 mg. of glacial acetic acid is usually sufficient for a soil containing 0.5 per cent  $MnO_2$ . Repeated additions of small amounts of hydrogen peroxide appear to give best results. After decomposition of the organic matter the excess peroxide is boiled off.

If acid treatment is desired the sample is cooled and then treated with 10 c. c. of N HCl, made to a volume of 100 c. c. and allowed to stand overnight. If it is desired to remove all the carbonates, enough acid is added so that the concentration of acid in the beaker, after the carbonates are dissolved, will be 0.1 N. This really involves a prior determination of carbonates. In this laboratory, method B of the Association of Official Agricultural Chemists (1) is used for carbonate determination.

The sample with or without acid treatment is now ready for washing. This is done by removing as completely as possible the solution from the sample in the beaker with a short Pasteur-Chamberland suction filter. The lower 12 cm. of the filter is sawed off, fitted with a removable stopper, and used for this purpose. The liquid is then removed from the hollow filter core and replaced with distilled water. By means of a rubber bulb back pressure is applied to the filter in order to remove adhering soil material. The sample is well stirred with 125 c. c. of distilled water and the solution again removed by

filtration. Six such washings are usually sufficient. If it is desired to determine the quantity of dissolved oxides of iron and aluminum the washings from each sample are saved separately, made slightly alkaline with ammonia, and boiled. The precipitate is washed on a filter paper, dried, ignited, and weighed.

After cleaning and removing the suction filter, the soil sample is evaporated to dryness on a steam bath and then dried in an electric oven at 105° C. for 16 hours (overnight). Upon cooling in a desiccator the sample, still in the beaker, is quickly weighed on a Chainomatic balance. If this weighing is done rapidly the sample will not take up more than 1 mg. of moisture. The sample is then soaked for a few minutes with about 25 c. c. of water, stirred with a rubber policeman, and transferred to a 250 c. c. nursing bottle. The beaker is again dried and weighed, the difference in weight being the portion of the sample left after organic matter and solution loss. This is the portion of the sample upon which the percentage of size classes is based, when the results are used for textural classification. To the shaking bottle is added 10 c. c. of 0.5 N sodium oxalate, and the volume is made up to 150 c. c. It is shaken overnight on the reciprocating shaker described in Bureau of Soils Bulletin 84 (9).

#### SEPARATION INTO SIZE CLASSES

A sieve fitted with Tyler 300-mesh wire screen cloth is clamped above a 1-liter graduated glass sedimentation cylinder on foot, and the clay and finer silt are decanted from the nursing bottle, through the sieve, into the sedimentation cylinder. After a few pourings the clay is completely removed from the shaking bottle. The entire contents are then transferred to the sieve by means of a stream of distilled water. The cylinder is filled to the 1-liter mark with distilled water and set aside for sedimentation. Economical use of water is necessary in decantation and washing on the sieve, because the total volume of the silt and clay suspension is limited to 1 liter.

The sands and remaining silt may then be washed into a platinum dish and dried, but the preferred procedure is to set the wet sieve in a shallow aluminum dish, place in an oven to dry, and then transfer the dry sand and silt into the platinum dish. While the sands are drying, another sieve is used on the next sample. After drying for two hours at 105° C. the sands are cooled in a desiccator, weighed, separated by a nest of graded sieves into size classes, and the weight of each class determined. The summation method of weighing the silt and sands is used. The first sand is weighed, the second sand is added, the total weight determined, and so on. If the sum of the weights of the fractions is equal to the total weight it is assumed that no error in weighing or recording weights has been made.

The material in the sedimentation cylinder is stirred with a motor-driven propeller, shown in Figure 1, and immediately the pipette is filled, before the coarser silt has had time to settle out. The pipette is emptied into a low-form glass-covered weighing dish, and the washings from the pipette added. The time at which this stirring was made is noted. The time required for a particle 5  $\mu$  in diameter to settle 10 cm. is computed from Stokes's formula using a density of 2.61 and the viscosity of pure water for various temperatures (77 minutes at 20° C.). A curve is plotted with temperatures as ordinates



and time as abscissae. At the time shown on the graph for the temperature, as read from a thermometer hanging in air near the cylinder, the suspension is again pipetted for clay. This time the pipette, clamped vertically to a support (fig. 1), and with stopcock closed, is carefully lowered by a rack and pinion until the tip is just 10 cm. below the liquid surface, as shown by a pointer moving over a fixed scale. In case pipetting can not be done at the correct time the additional depth required for any delay can easily be computed, time

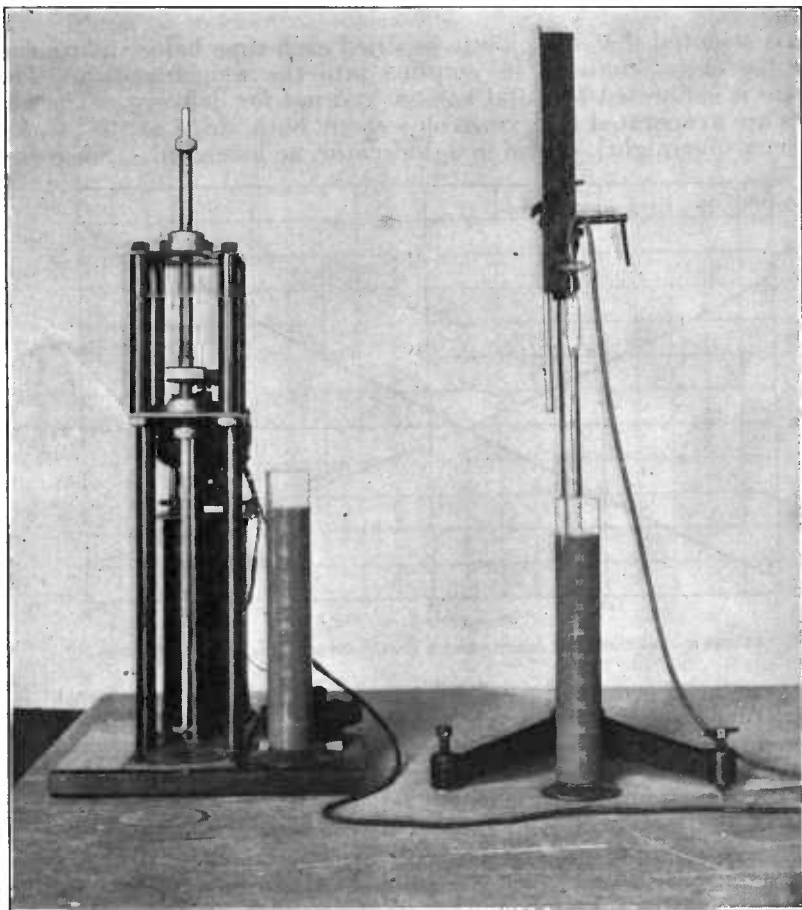


FIGURE 1.—At left, motor and propeller used to stir suspensions; at right, pipette and stand

and depth being directly proportional. This time the pipette is filled with an even suction in about 40 seconds, and the entire contents run into a weighing dish. The pipette is washed two or three times and the washings added to the dish.

Pipetting of the colloid is similar to that of clay except in this case settling time is fixed at six and a half hours, and a graph (fig. 2), computed from Stokes's law, giving the relation of depth to temperature is used.

In ordinary routine suspensions from eight soil samples are placed in a set of sedimentation cylinders, which are covered to prevent evaporation, and are allowed to stand until a convenient time for pipetting. Each suspension is stirred and the first aliquot taken at once. For the second aliquot the suspensions are stirred at 6-minute intervals and samples taken at the end of the required time interval, as read from the time-temperature chart. (Fig. 2.) For the third aliquot the suspensions are stirred early in the day and the samples taken after six and one-half hours at the correct depth as read from the depth-temperature chart. (Fig. 2.) If desired, both charts may be prepared for pipetting at constant time intervals or at constant depth.

It is essential that the pipette be dried each time before filling and that the entire contents be emptied into the weighing dish. The pipette is calibrated for total volume and not for delivery. The aliquots are evaporated to dryness on a steam bath, dried at 105° C. for 16 hours (overnight), cooled in a desiccator, and weighed. Since any

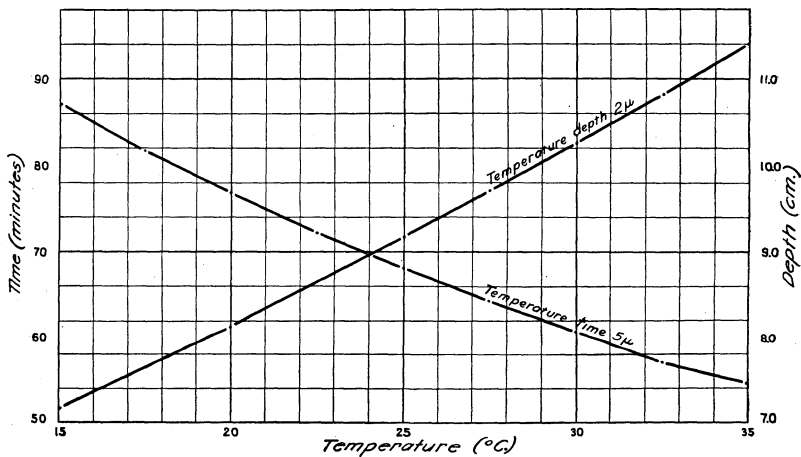


FIGURE 2.—Sedimentation velocity graphs used in the pipetting of clay and colloid

loss of material or error in weighing is magnified nearly fortyfold in the final result, it is necessary to use care in the determination of the pipetted fractions. Weighings are carried to 0.1 mg. and the weight of the dishes is determined each time they are used.

If the room temperature changes rapidly the cylinders are covered with bakelite jackets, but in general room-temperature changes produce only negligible variations. If there is a temperature difference between opposite sides of the cylinder there will be a circulatory movement of the suspension, a condition to be avoided.

Occasionally sets of separates are submitted to William H. Fry of this laboratory for microscopic examination by his methods (10). He usually finds the sands practically free from adhering colloid and the silt relatively free from colloidal aggregates.

#### CALCULATION OF RESULTS

From the moisture determination of the 5-gm. sample, the dry weight of the sample for analysis is computed. After peroxide treatment, and acid treatment if used, followed by washing, the sample is

dried and weighed. The difference between the two weights is the hydrogen peroxide-solution loss. The first dry weight, which includes organic matter and solution loss, is taken as the base for calculation of results except when the analysis is to be used for textural classification only. In the latter case the dry weight, after washing, is taken as the base.

From the weight of the first pipetted aliquot, after deducting the weight of the dispersing agent, the dry weight of silt and clay in the sedimentation chamber can be determined directly. It can also be determined by taking the difference between the total weight of the sample before dispersion and the weight of the dry sifted silt and sands. These weights should check, but in every case the weight directly determined by pipetting is slightly greater, varying from a small fraction to as much as 1 per cent or more. The reason for this discrepancy has not yet been found. In routine analyses the dry weight of material in the sedimentation chamber is obtained by difference, but the amount is also determined by pipetting, and if the two results fail to check within 2 per cent the analysis is repeated.

From the weight of the clay and colloid aliquots is deducted the weight of added sodium oxalate and the total amount of material smaller than  $5\mu$  and  $2\mu$  is computed. No size class between these two limits is established. If the weight of clay is deducted from the total dry weight in the sedimentation cylinder the difference is silt. This is added to the amount of silt sifted from the sands to give total silt.

Below are given the data obtained and the results computed from the analysis of a sample of soil:

Sample No. 561860.

Weight of sample, air-dry, 10 gm.

Moisture (determined on duplicate sample) = 4.89 per cent.

Dry weight of sample at  $105^{\circ}$  C. =  $10.000 - 0.489 = 9.511$  gm.

Dry weight of sample (after pretreatment and washing) 9.366 gm.

Solution loss =  $9.511 - 9.366 = 0.145$  gm. = 1.6 per cent.

Weight of sand (+ some silt) = 2.116 gm.

Silt = 0.046

	Size mm.	Weight gm.	Per cent
Sand	2.070 gm.	0.249	2.7
	2.0 — 1.0	.394	4.2
	1.0 — .5	.312	3.3
	.5 — .25	.567	6.1
	.25 — .1	.557	5.9

Weight of silt + clay aliquot 0.1905 gm.

Weight of clay aliquot 0.1127 gm.

Volume of pipette = 25.12 c. c. =  $1/39.8$  liter.

Weight of  $\text{Na}_2\text{C}_2\text{O}_4$  in 25.12 c. c. = 0.0065 gm.

Weight of clay =  $(0.1127 - 0.0065) \times 39.8$  ..... 4.227 45.1

Weight of silt<sup>4</sup> =  $9.356 - (2.070 + 4.227)$  ..... 3.059 32.7

Total except solution loss ..... 9.365 100.0

Weight of colloid aliquot = 0.0897

Weight of colloid =  $(0.0897 - 0.0065) \times 39.8$  ..... 3.311 35.4

## APPARATUS

A few special pieces of apparatus used in mechanical analysis that have not previously been described are the weighing dishes, pipette, stirrer, sieves, and shaker. The pipette (fig. 1) is a Lowy automatic,

<sup>4</sup> A determination of silt may be made as follows:

Weight of silt aliquot =  $0.1905 - 0.1127 = 0.0778$  gm.

Weight of silt =  $0.0778 \times 39.8 + 0.046 = 3.142$  gm. = 33.6 per cent.

equipped with a stopcock having an air vent. When the pipette is full a 90° rotation of the stopper will close the stopcock, and an additional 90° rotation will permit delivery. The house vacuum is connected through a fine capillary and a small sulphuric acid trap to the pipette. The capillary permits the desired slow and uniform filling of the pipette, and the sulphuric acid trap keeps moisture from interfering with the air flow through the capillary. The pipette has a delivery volume of 25 c. c. but is calibrated with mercury for total volume.

The stirrer shown in Figure 1 is a motor-driven 4-blade propeller on a sliding shaft which can be clamped at any height to the driving pulley. The variable speed motor is run as fast as possible without whipping air into the suspension. After a suspension has stood for two or three days it is sometimes difficult, with hand stirring, to break up the flakes of silt and clay aggregates which have settled on the bottom of the chamber.

The weighing dishes are of low, flat form in two sizes, one 65 mm. in diameter and 30 mm. high and the other 50 mm. by 30 mm., with correspondingly numbered glass lids ground to fit over the outside of the dish. The small dish is used for the clay and colloid pipetting, and the larger dish is used for the first or silt pipetting, because of the extra washing required to remove the coarser particles from the pipette.

The sieve frames, made from 2½-inch extra heavy brass pipe, are fitted with removable rings to permit the easy change of screens. The two larger sieves have round holes of 1 and 0.5 mm., respectively. The next two screens conform to the Bureau of Standards specifications for 60-mesh and 140-mesh sieves. They have square openings of 0.25 and 0.105 mm., respectively. The finest sieve is 300 mesh and holds particles larger than silt size. The screen wire cloth on the 300-mesh sieve is frequently renewed, as the wire is so fine that it is easily stretched, worn, or broken.

#### SUMMARY

The method of mechanical soil analysis developed for use in the soil physics laboratory of the Bureau of Chemistry and Soils is described. In pretreatment the organic matter is removed with hydrogen peroxide, but the hydrochloric acid treatment, used in the international method, ordinarily is omitted. A method of removing organic matter in the presence of manganese dioxide is described. Soluble matter is removed by washing and filtering with Pasteur-Chamberland suction filters. The sample is dried and weighed, and this weight is the basis of calculation of percentages of material in each size class, when the results are for use in textural classification. In all operations up to dispersion the sample remains in an extra tall form beaker. The sample is deflocculated by shaking in a dilute sodium oxalate solution. The colloid, clay, and fine silt are separated from the sands by means of a 300-mesh sieve. The clay and colloid are determined by sedimentation, the pipette method being used. The procedure is designed for accurate and rapid analysis.

Investigation of dispersion aids, incidental to this method, discloses the fact that acid treatment introduces undesirable solution losses and is not necessary for dispersion, even in calcareous soils, particularly if sodium oxalate is used as the dispersing agent.

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<i>Soil Investigations</i> .....	A. G. MCCALL, <i>Chief.</i>
<i>Division of Soil Chemistry and Physics.</i>	H. G. BYERS, <i>Principal Chemist, in Charge.</i>