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METHODS OF SOIL ANALYSIS

Part 1

Physical and Mineralogical Methods

Second Edition

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15 Particle-size Analysis[1]

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15–1 INTRODUCTION

Particle-size analysis (PSA) is a measurement of the size distribution of individual particles in a soil sample. The major features of PSA are the destruction or dispersion of soil aggregates into discrete units by chemical, mechanical, or ultrasonic means and the separation of particles according to size limits by sieving and sedimentation.

Soil particles cover an extreme size range, varying from stones and rocks (exceeding 0.25 m in size) down to submicron clays ($< 1 \mu$m). Various systems of size classification have been used to define arbitrary limits and ranges of soil particle size. Soil particles smaller than 2000 μm are generally divided into three major size groups: sands, silts and clays. These groups are sometimes called soil separates and can be subdivided into smaller size classes. Figure 15-1 shows the particle size, sieve dimension, and defined size class for the system of classification used by the U. S. Department of Agriculture (USDA), the Canadian Soil Survey Committee (CSSC), the International Soil Science Society (ISSS) and the American Society for Testing and Materials (ASTM). The American Society of Agronomy has adopted the USDA classification [i.e., sands (<2000–50 μm), silts (<50–2 μm), and clays ($<2 \mu$m)]. Although the USDA classification scheme will be emphasized in the following methods, it should be recognized that other systems are frequently cited, particularly in engineering literature, hence, care should be taken to specify clearly which system is being used when reporting results.

Particle-size analysis data can be presented and used in several ways, the most common being a particle-size distribution curve. An example of this type of curve is shown in Figure 15-2. The percentage of particles

PARTICLE SIZE LIMIT CLASSIFICATION

PARTICLE SIZE (mm)	ASTM SIEVE NUMBER OR SIZE (OPENINGS/INCH)	USDA	CSSC	ISSS	ASTM (UNIFIED)
0.0002			FINE CLAY		
0.001		CLAY	COARSE CLAY	CLAY	
0.002					FINES (SILT AND CLAY)
0.003			FINE SILT		
0.004				SILT	
0.006					
0.008		SILT	MEDIUM SILT		
0.01					
0.02					
0.03			COARSE SILT		
0.04	300				
0.06	270	VERY FINE SAND	VERY FINE SAND	FINE SAND	
0.08	200				
0.1	140	FINE SAND	FINE SAND		FINE SAND
0.2	60	MEDIUM SAND	MEDIUM SAND		
0.3					
0.4	40			COARSE SAND	MEDIUM SAND
0.6		COARSE SAND	COARSE SAND		
0.8	20				
1.0		VERY COARSE SAND	VERY COARSE SAND		
2.0	10				COARSE SAND
3.0		FINE GRAVEL			
4.0	4				
6.0					FINE GRAVEL
8.0			GRAVEL		
10	1/2 IN.			GRAVEL	
20	3/4 IN.				
30		COARSE GRAVEL			COARSE GRAVEL
40					
60					
80	3 IN.				
		COBBLES	COBBLES		COBBLES

USDA—U.S. DEPARTMENT OF AGRICULTURE, (SOIL SURVEY STAFF, 1975)
CSSC—CANADA SOIL SURVEY COMMITTEE, (McKEAGUE, 1978)
ISSS—INTERNATIONAL SOIL SCI. SOC. (YONG AND WARKENTIN, 1966)
ASTM (UNIFIED)—AMERICAN SOCIETY FOR TESTING & MATERIALS (ASTM, D-2487, 1985a)

Fig. 15–1. Particle-size limits according to several current classification schemes.

less than a given particle size is plotted against the logarithm of the "effective" particle diameter. Particle-size distribution curves, when differentiated graphically, produce frequency distribution curves for various particle sizes. Frequency curves usually exhibit a peak or peaks representing the most prevalent particle sizes.

Particle-size distribution curves are used extensively by geologists in geomorphological studies to evaluate sedimentation and alluvial pro-
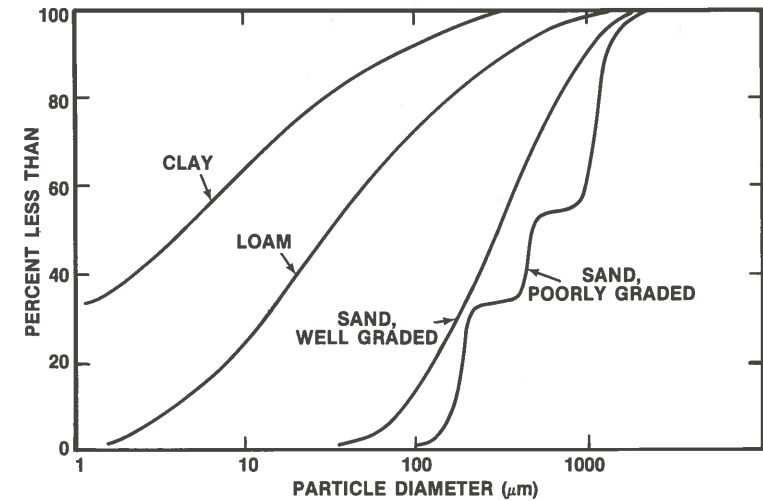
Fig. 15–2. Particle-size distribution curves for several soil materials (after Hillel, 1982).

cesses, and by civil engineers to evaluate materials used for foundations, road fills, and other construction purposes. Details of the use of these curves are given by Krumbein and Pettijohn (1938) and Irani and Callas (1963).

Particle-size analysis is often used in soil science to evaluate soil texture. Soils rarely consist entirely of one size range. Soil texture is based on different combinations of sand, silt, and clay separates that make up the particle-size distribution of a soil sample. Figure 15-3 shows the USDA defined limits for the basic soil textural classes. Details for interpretation of the textural triangle for soil classification purposes are given by the Soil Survey Staff (1975). The ASTM (Unified) engineering classification system is used widely for delineating soil types for construction purposes (Fig. 15-4). In this system, liquid limits and plasticity indexes must be known in order to properly classify the soil type (ASTM, 1985a,b).[2]

Hydrologists often use PSA as a means of predicting hydraulic properties, particularly for sands (Todd, 1964). Recently, Bloemen (1980) and Arya and Paris (1981) have used PSA as a means to predict water retention and unsaturated hydraulic conductivity of soils. These predictive methods appear to work best on sands or structureless soil materials.

15–2 PRINCIPLES

15–2.1 Pretreatment and Dispersion Techniques

Pretreatment of samples to enhance separation or dispersion of aggregates is a key step in PSA and is generally recommended, since many

[2]Stevens (1982) has published a BASIC program for computing the Unified (ASTM) classification for a tested soil. A BASIC program for computing the USDA textural classes is available upon request from the authors.
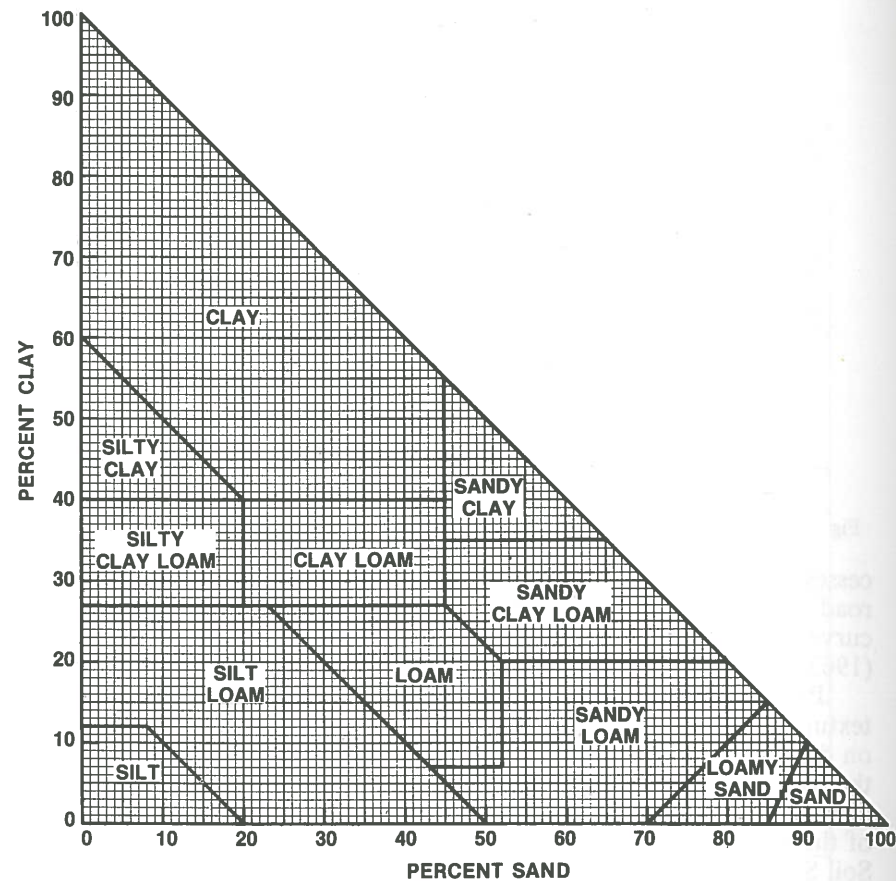
Fig. 15–3. Textural triangle for soil textural analysis using the USDA classification scheme.

soils contain aggregates that are not readily dispersed. Soils generally contain organic matter and often contain iron oxides and carbonate coatings that bind particles together. Chemical pretreatments are used for removal of these coatings; however, chemical treatment can result in destruction and dissolution of some soil minerals. Physical treatments are also used, but standardization of treatment and adequate testing of specific methods are needed, since the very process of separation by mechanical or ultrasonic means can fragment the individual particles into further subunits. Procedures should clearly specify the sample pretreatment, the separation method, and the purpose for which the size analysis is intended for a particular soil.

Standard PSA methods require that soil particles be dispersed in an aqueous solution by both chemical and physical means. After pretreatment, chemical dispersion is often accomplished using a dilute alkaline solution of sodium polyphosphate. The effectiveness of the chemical dispersing agent depends on its ability to create and maintain repulsive

forces between soil particles. Some soils (e.g., those of volcanic ash origin) that have been highly weathered disperse more readily in acid media; hence, some pretesting may be required to determine effects of soil mineralogy and other factors on soil dispersibility and to select an appropriate method to achieve complete dispersion. Physical dispersion of particles is accomplished by shearing action or turbulent mixing, using mechanical shakers, electrical mixers, or ultrasonic probes.

Dispersibility of soils low in organic matter depends primarily on soil mineralogy. Highly oxidized soils are particularly difficult to disperse. Examples include the "subplastic" soils of Australia (McIntyre, 1976; Brewer & Blackmore, 1976; Walker & Hutka, 1976; Blackmore, 1976; Norrish & Tiller, 1976). Depending on the method of chemical treatment and physical dispersion used, measured clay content for an individual soil sample can vary by factors of two to four or more.

Volcanic ash soils are high in amorphous (noncrystalline) clay-sized materials and have great resistance to dispersion, particularly after air or oven drying (Kubota, 1972; Schalscha et al., 1965; Espinoza et al., 1975; Maeda et al., 1977). Kubota (1972) reported clay contents ranging from 1 to 56 wt% for one volcanic ash soil, depending on pretreatment. Maximum clay content was obtained when the soil was retained at field moisture prior to ultrasonic dispersion. Warkentin and Maeda (1980) recommend that volcanic ash soils be left at field moisture and dispersed at either pH 3 or above pH 9. Tama and El-Swaify (1978) and El-Swaify (1980) have observed that soils with variable charge are particularly difficult to disperse unless the dispersant solution is well below or above the zero-point of charge.

Highly aggregated, stable clay soils may behave like coarse sands in terms of water infiltration; hence they may be identified in the field as sands or coarse loams. These same soils, having significant microporosity and high exchange capacities, retain water and nutrients much better than sands. For agricultural purposes, these soils should be texturally classed in a much finer category than they appear in the field. For soils where these uncertainties are known to exist, measurements such as a simple dispersive index (Sherard et al., 1976), ASTM dispersion test (ASTM, 1985c), or the water-stability of aggregates (see chapter 17) would be necessary and useful information. Also, a calculated clay content, determined from a ratio of the cation exchange capacity (CEC) of the total soil to the CEC of the clay-size material (Norrish & Tiller, 1976), can be used to estimate the theoretical maximum clay fraction of the soil material.

The method that produces the most complete dispersion of a soil sample is generally the more acceptable method. However, the chemical treatment and mechanical work done on the soil are dictated by somewhat arbitrary decisions, so there is no "absolute" size-distribution for a given sample. Intense mechanical or ultrasonic dispersion, coupled with appropriate chemical treatment, should yield a sample with most of the

Unified Soil Classification (ASTM—D2487)

Criteria for Assigning Group Symbols and Group Names Using Laboratory Tests[A]				Soil Classification	
				Group Symbol	Group Name[B]
Coarse-Grained Soils More than 50% retained on No. 200 sieve	Gravels More than 50% of coarse fraction retained on No. 4 sieve	Clean Gravels Less than 5% fines[C]	$Cu \geq 4$ and $1 \leq Cc \leq 3$[E]	GW	Well-graded gravel[F]
			$Cu < 4$ and/or $1 > Cc > 3$[E]	GP	Poorly graded gravel[F]
		Gravels with Fines More than 12% fines[C]	Fines classify as ML or MH	GM	Silty gravel[F,G,H]
			Fines classify as CL or CH	GC	Clayey gravel[F,G,H]
	Sands 50% or more of coarse fraction passes No. 4 sieve	Clean Sands Less than 5% fines[D]	$Cu \geq 6$ and $1 \leq Cc \leq 3$[E]	SW	Well-graded sand[I]
			$Cu < 6$ and/or $1 > Cc > 3$[E]	SP	Poorly graded sand[I]
		Sands with Fines More than 12% fines[D]	Fines classify as ML or MH	SM	Silty sand[G,H,I]
			Fines classify as CL or CH	SC	Clayey sand[G,H,I]
Fine-Grained Soils 50% or more passes the No. 200 sieve	Silts and Clays Liquid limit less than 50	Inorganic	PI > 7 and plots on or above "A" line[J]	CL	Lean clay[K,L,M]
			PI < 4 or plots below "A" line[J]	ML	Silt[K,L,M]
		Organic	Liquid limit - oven dried / Liquid limit - not dried < 0.75	OL	Organic clay[K,L,M,N] Organic silt[K,L,M,O]
	Silts and Clays Liquid limit 50 or more	Inorganic	PI plots on or above "A" line	CH	Fat clay[K,L,M]
			PI plots below "A" line	MH	Elastic silt[K,L,M]
		Organic	Liquid limit - oven dried / Liquid limit - not dried < 0.75	OH	Organic clay[K,L,M,P] Organic silt[K,L,M,Q]
Highly organic soils	Primarily organic matter, dark in color, and organic odor			PT	Peat

Fig. 15–4. Unified soil classification system including plasticity chart (ASTM, 1985a). Continued on p. 389.

[A] Based on the material passing the 3-in. (75-mm) sieve.
[B] If field sample contained cobbles or boulders, or both, add "with cobbles or boulders, or both" to group name.
[C] Gravels with 5 to 12% fines require dual symbols:
GW-GM well-graded gravel with silt
GW-GC well-graded gravel with clay
GP-GM poorly graded gravel with silt
GP-GC poorly graded gravel with clay
[D] Sands with 5 to 12% fines require dual symbols:
SW-SM well-graded sand with silt
SW-SC well-graded sand with clay
SP-SM poorly graded sand with silt
SP-SC poorly graded sand with clay
[E] $Cu = D_{60}/D_{10}$ and $Cc = \dfrac{(D_{30})^2}{D_{10} \times D_{60}}$
[F] If soil contains $\geq$ 15% sand, add "with sand" to group name.

[G] If fines classify as CL-ML, use dual symbol GC-GM, or SC-SM.
[H] If fines are organic, add "with organic fines" to group name.
[I] If soil contains $\geq$ 15% gravel, add "with gravel" to group name.
[J] If Atterberg limits plot in hatched area, soil is a CL-ML, silty clay.
[K] If soil contains 15 to 29% plus No. 200, add "with sand" or "with gravel," whichever is predominant.
[L] If soil contains $\geq$ 30% plus No. 200, predominantly sand, add "sandy" to group name.
[M] If soil contains $\leq$ 30% plus No. 200, predominantly gravel, add "gravelly" to group name.
[N] PI $\geq$ 4 and plots on or above "A" line.
[O] PI < 4 or plots below "A" line.
[P] PI plots on or above "A" line.
[Q] PI plots below "A" line.

For classification of fine-grained soils and fine-grained fraction of coarse-grained soils.

Equation of "A"-line
Horizontal at PI = 4 to LL = 25.5, then PI = 0.73 (LL-20)

Fig. 15–4. Continued.
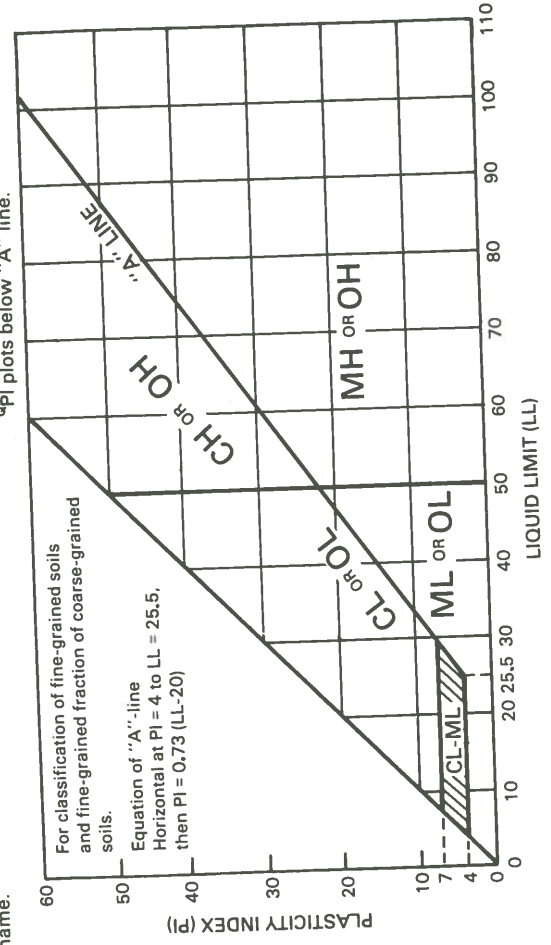
clay minerals in the measured clay fraction. In contrast, a less drastic chemical treatment and/or little mechanical dispersion may reflect the more "natural" particle-size distribution of the soil. Comparisons of PSA results should always include comparisons of the pretreatment and dispersion methods used.

15–2.1.1 ORGANIC MATTER REMOVAL

Removal of organic matter is often a first step in the chemical pretreatment of many soils. The necessity and difficulty of organic matter removal depends on the intended use of the analytical results of the PSA, the nature and concentration of organic matter in the sample to be analyzed, the pH of the soil, and the associated presence in the soil of free carbonates, gypsum, oxides, and soluble salts. A variety of reagents have been used in the past to successfully remove organic matter. Notable among these are hydrogen peroxide (H_2O_2), sodium hypochlorite, sodium hypobromite, and potassium permanganate. Hydrogen peroxide has been recommended as the standard oxidant for most soils (Day, 1965).

15–2.1.2 REMOVAL OF IRON OXIDE

Coatings and crystals of various iron oxides, such as hematite and goethite, often act as cementing and binding agents in soils. Removal of these cementing agents aids in dispersion of the silicate portion of the soil and is often necessary for accurate mineralogical analysis. Mehra and Jackson (1960) recommend the use of a bicarbonate-buffered, sodium dithionite–citrate system for iron oxide removal. This method, compared with several other methods for removal of free iron oxides from latosolic soils, was found to be the most effective. In addition, this method was the least destructive of iron silicate clays, as indicated by least loss of cation exchange capacity. Mehra and Jackson (1960) indicated that the optimum pH for maximum iron oxide removal was approximately 7.3. Since considerable OH^- is expended in the sodium dithionite–citrate reaction with iron oxide, a buffer is needed to hold the pH at the optimum level. Sodium bicarbonate has proven to be an effective buffer. This procedure minimizes the formation of sulfide, iron sulfide, zinc oxalate or other unwanted precipitates during iron oxide removal.

In soils where iron oxides are part of the dominant mineralogy, it is not recommended that iron oxides be removed, since many of the primary mineral grains in the clay fraction could be destroyed (El-Swaify, 1980).

15–2.1.3 REMOVAL OF CARBONATES

Removal of carbonate from soils prior to dispersion and sedimentation can be accomplished relatively easily by acidification of the sample. Heating accelerates the reaction. Samples that are acidified before organic matter removal with H_2O_2 will usually be free of carbonates. Hydrogen

chloride (HCl) treatment can cause destruction of crystalline lattice of clay minerals; therefore, acid treatment with 1 M NaOAC at pH 5 is preferred.

15–2.1.4 REMOVAL OF SOLUBLE SALTS

A variety of soluble salts including sodium, calcium, and magnesium chlorides and carbonates are commonly found in alkaline soils. High concentrations of soluble salts can cause flocculation of soil suspensions. Alkaline salts can cause decomposition of H_2O_2, decreasing its effectiveness as an oxidizing agent for soil organic matter. In addition, many soluble salts interfere with saturation of the exchange complex. Calcium and magnesium salts, commonly occurring as carbonates, are relatively unstable and are often measured as part of the clay and silt fractions.

The most common procedure for removal of soluble salts is to leach the salts with distilled water. Sample washing with distilled water can be accomplished by use of a filter candle or by centrifuging. The procedure should be repeated until the leachate salt concentration drops below 10 mM. The washing treatment is then followed by chemical and physical dispersion.

15–2.1.5 SAMPLE DISPERSION

Dispersion of soils is accomplished by a combination of methods. The methods for dispersion can be classified as either chemical or physical. Numerous methods of chemical dispersion have been investigated and reported (Theisen et al., 1968; Norrish & Tiller, 1976). Soils are chemically dispersed after oxidation of organic matter and removal of carbonates and iron oxides. Chemical dispersion is based primarily on the concept of particle repulsion, as a result of elevation of the particle zeta potential. This process is usually accomplished by saturating the exchange complex with sodium. Physical or mechanical methods of dispersion involve separation of the individual particles by means of some mechanical or physical process, such as rubbing, rolling, shaking, or vibrating. During the past 20 years, electronic dispersion, primarily by the use of ultrasonics, has become increasingly popular. Most researchers have found that a combination of chemical and physical or electronic methods provides the most complete and stable dispersion (Maeda et al., 1977; Mikhail & Briner, 1978).

15-2.1.5.1 Chemical Dispersion. Following removal of cementing and flocculating agents, samples must be dispersed and maintained in a dispersed state until sedimentation measurements are completed. A number of dispersing chemicals have been used. These include Na-hexametaphosphate (HMP), Na_2PO_7, NaOH, Na_2CO_3, and NaOBr. Of these, HMP appears to be the most commonly used dispersant. Commercial detergents contain quantities of HMP and other soluble phosphates, but uncertainty exists as to the exact amounts (Yaalon, 1976; Veneman, 1977).

For this reason, reagent grade HMP, which is commercially available, is the recommended chemical dispersant for the pipet and hydrometer tests described later in this chapter.

The exact amount of chemical dispersant needed to prevent flocculation is dependent on soil type (mineralogy, etc.). Flocculation often can be prevented by increasing the concentration of the dispersant solution. It should be noted that the pipet analysis requires only 0.5 g/L HMP, compared to a 5 g/L HMP solution for the hydrometer analysis. The lower amount needed for pipet analysis is likely due to pretreatment (organic matter, iron oxide, and soluble salt removal). Specific amounts used in these analyses have been established by empirical methods.

15–2.1.5.2 Physical Dispersion. Several methods of physical dispersion have been used in conjunction with pretreatment and chemical dispersion. The ASTM (1985d) recommends either an electric mixer with specially designed stirring paddles or an air-jet stirrer (Chu & Davidson, 1953; Theisen et al., 1968). For the hydrometer method, Day (1965) recommends a 5 min mixing with a standard electrical mixer (malted milk style), but cautions that the mixer blades deteriorate rapidly by abrasion and should be replaced after 1 or 2 h of use or when showing signs of wear. Reciprocating shakers have also been used. Overnight shaking is prescribed in the pipet procedure and can be used in the hydrometer method. However, the larger sample (40 g) used in the hydrometer method will pack to the bottom of 250 mL bottles; hence, larger (>500 mL) shaking bottles are recommended for the larger samples to avoid this problem. High-speed reciprocating shakers have been used effectively on small samples of 10 g or less (El-Swaify, 1980). These high-speed shakers optimize dispersion when the liquid-to-solid ratio is about 5:1.

15–2.1.5.3 Ultrasonic Dispersion. The principle behind ultrasonic dispersion is the transmission of vibrating sound waves in the soil solution. The sound waves produce microscopic bubbles, which collapse, producing *cavitation*. The release of intense energy of cavitation literally blasts the soil aggregates apart, causing dispersion even in highly aggregated soils.

Much work has been done in testing the use of ultrasonic dispersion of soils, but no standard procedures have been adopted (Edwards & Bremner, 1964, 1967; Saly, 1967; Bourget, 1968; Watson, 1971; Kubota, 1972; Mikhail & Briner, 1978). An initial concern with this method of dispersion was the possible destruction of primary particles, but Saly (1967) reported that ultrasonic vibration did not cause destruction of the crystalline lattice or breakdown of primary grains. Edwards and Bremner (1964, 1967) investigated the use of ultrasonic dispersion in the absence of a dispersing agent. For mineralogical analysis, ultrasonic dispersion was preferred, since dispersion was achieved without soil pretreatment or addition of a dispersing agent. Edwards and Bremner summarized the following advantages of ultrasonic dispersion: (i) the resultant suspension is stable, hence flocculation does not occur during sedimentation; (ii) the method works well for dispersing calcareous soils, organic soils, and soils with high clay content; (iii) ultrasonic dispersion does not cause destruction of organic matter; and (iv) ultrasonic dispersion does not alter the soil pH, electrical conductivity, or cation exchange capacity. In contrast to the work of Edwards and Bremner, Mikhail and Briner (1978) reported that the most satisfactory method of pretreatment and dispersion involved the following steps; oxidation of organic matter, removal of carbonates and acid washing, and sodium saturation followed by ultrasonic dispersion. Their results indicated that the highest degree of dispersion was achieved by this technique. Kubota (1972) reported that a sonic dispersion at low pH was effective in dispersing peroxide-treated volcanic ash soils. Each of the above authors used a different ultrasonic power and dispersion time, indicating that effective dispersion with ultrasonics is soil dependent.

For routine PSA, there is no standard method for ultrasonic mixing proposed at this time. Much additional research is needed to determine the effectiveness or limitations of ultrasonic dispersion for a wide range of soil materials.

15–2.2 Sieving

The typical particle size range for sieving is 2000 to 50 μm. Several limitations of sieving have been noted in the past. Day (1965) indicated that the probability of a particle passing through a sieve in a given time of shaking depends on the nature of the particle, the number of particles of that size, and the properties of the sieve. Particle shape and sieve opening shape affect probability of passage. For example, a particle whose shape permits its passage only in one orientation has a limited chance of getting through, except after prolonged shaking. Sieve openings are generally unequal in size, and extensive shaking is required before all particles have had the opportunity of approaching the largest openings. In fact, it is rare that complete sorting of a given size range can be achieved. Good reproducibility requires careful standardization of procedure.

15–2.3 Sedimentation

Sedimentation analysis relies on the relationship that exists between settling velocity and particle diameter. Settling velocity is related to the diameter of a spherical particle in the following way. The force acting downward on each particle due to its weight in water is

$$F_{down} = 4/3 \; \pi \; (X^3/8) \; (\rho_s - \rho_l)g \qquad [1]$$

where X = particle diameter, ρ_s = particle density, ρ_l = liquid density, and g = acceleration due to gravity. Because of the viscous resistance of the water, the opposing upward force is

$$F_{up} = 3 \pi X \eta v \qquad [2]$$

where η = fluid viscosity and v = velocity of fall. The resisting force is zero where velocity, v, is zero at time $t = 0$, and it increases with increasing v until it is equal to the downward force. For sedimenting particles in a dilute dispersent solution, it can be shown that the terminal velocity for silt- and clay-size particles is reached in a relatively short time (a few seconds).

Equating F_{down} and F_{up} relates the terminal velocity to the particle diameter as follows:

$$v = g (\rho_s - \rho_l) X^2/(18 \eta) . \qquad [3]$$

A form of this relationship was first developed by Stokes (1851) and is now known as Stokes' Law. Basic assumptions used in applying Stokes' Law to sedimenting soil suspensions are:
1. Terminal velocity is attained as soon as settling begins.
2. Settling and resistance are entirely due to the viscosity of the fluid.
3. Particles are smooth and spherical.
4. There is no interaction between individual particles in the solution. Gibbs et al. (1971) have shown that assumptions (1) and (2) are met by soil particles < 80 μm in diameter. Since soil particles are not smooth and spherical, X must be regarded as an "equivalent" rather than actual diameter. The assumptions of Stokes' Law as applied to soils are discussed fully by Krumbein and Pettijohn (1938).

In mineralogical analysis there is often a need to separate various clay fractions for specific analysis. The removal of the clay fraction by sedimentation can be accomplished by homogenizing a soil suspension and decanting all that remains above the plane $z = -h$ after time, t, where

$$t = 18 \eta h/[g (\rho_s - \rho_l) X^2] . \qquad [4]$$

Quantitative separation by decantation requires that the residue be resuspended and decanted repeatedly to salvage those particles that were not previously at the top of the suspension at the start of the sedimentation period.

15–2.3.1 PRINCIPLE OF THE PIPET METHOD

The pipet method is a direct sampling procedure. It depends on taking a small subsample by a pipet at a depth h, at time t, in which all particles coarser than X have been eliminated. Using Stokes' Law in the form of Eq. [4], settling times for the clay fraction (<2 μm) can be calculated for sampling at a given temperature. Table 15–1 lists sampling times for the clay fraction for a 10-cm sampling depth at selected temperatures for the pipet technique. Tables 15–2 and 15–3 list sampling

depths and times for various selected size fractions and specified settling times.

Experimental measurements with HMP solutions (Gee, unpublished data) show the following relationships for solution viscosity and density:

$$\rho_l = \rho°(1 + 0.630 \, C_s) \qquad [5]$$

where
ρ_l = solution density at temperature t, g/mL,
$\rho°$ = water density at temperature t, g/mL,
C_s = concentration of HMP, g/mL,
and

$$\eta = \eta°(1 + 4.25 \, C_s) \qquad [6]$$

Table 15–1. Settling times for 2-μm clay at various temperatures. Calculated for a 10-cm sampling depth in distilled water, 0.5 g/L, and 5 g/L HMP solutions; with a particle density equal to 2.60 Mg/m³.

Tempera-ture	Viscosity			Settling time		
	Distilled H₂O	0.5 g/L HMP	5.0 g/L HMP	Distilled H₂O	0.5 g/L HMP	5.0 g/L HMP
°C	—10^{-3} kg m⁻¹ s⁻¹—			— h —		
18	1.0530	1.0553	1.0759	8.39	8.41	8.58
20	1.0020	1.0042	1.0238	7.99	8.00	8.16
22	0.9548	0.9569	0.9756	7.61	7.63	7.78
24	0.9111	0.9131	0.9310	7.26	7.28	7.42
26	0.8705	0.8724	0.8895	6.94	6.95	7.09
28	0.8327	0.8345	0.8508	6.64	6.65	6.78
30	0.7975	0.7992	0.8149	6.36	6.37	6.50

Table 15–2. Selected depths for 2-μm clay at specified times and temperatures, assuming a particle density of 2.60 Mg/m³ and dispersion with 0.5 g/L HMP solution.

Temperature	Viscosity	Sampling depth			
		4.5 h	5.0 h	5.5 h	6.0 h
°C	10^{-3} kg m⁻¹ s⁻¹	— cm —			
20	1.0042	5.6	6.2	6.9	7.5
21	0.9800	5.8	6.4	7.0	7.7
22	0.9569	5.9	6.5	7.2	7.9
23	0.9345	6.0	6.7	7.4	8.1
24	0.9131	6.2	6.9	7.6	8.2
25	0.8923	6.3	7.0	7.7	8.4
26	0.8724	6.5	7.2	7.9	8.6
27	0.8532	6.6	7.4	8.1	8.8
28	0.8345	6.8	7.5	8.3	9.0
29	0.8166	6.9	7.7	8.4	9.2
30	0.7992	7.1	7.8	8.6	9.4

Table 15–3. Sampling times for 5-μm and 20-μm size fractions at a 10-cm sampling depth for pipet in 0.5 g/L HMP solution, over the temperature range 20 to 30°C for selected particle densities.

Tempera-ture	5-μm Particle size Particle density (Mg/m³)			20-μm Particle size Particle density (Mg/m³)		
	2.4	2.6	2.8	2.4	2.6	2.8
°C	time (min)					
20	87.7	76.8	68.3	5.5	4.8	4.3
21	85.7	75.0	66.7	5.4	4.7	4.2
22	83.7	73.2	65.1	5.2	4.6	4.1
23	81.7	71.5	63.6	5.1	4.5	4.0
24	79.9	69.9	62.1	5.0	4.4	3.9
25	78.0	68.3	60.7	4.9	4.3	3.8
26	76.3	66.8	59.3	4.8	4.2	3.7
27	74.6	65.3	58.0	4.7	4.1	3.6
28	73.0	63.9	56.8	4.6	4.0	3.5
29	71.4	62.5	55.6	4.5	3.9	3.5
30	69.9	61.2	54.4	4.4	3.8	3.4

where

η = solution viscosity at temperature t, 10^{-3} kg m^{-1}s^{-1} (cpoise), and
$\eta°$ = water viscosity at temperature t, 10^{-3} kg m^{-1}s^{-1} (cpoise).

Equations [5] and [6] apply to HMP solutions in the range of 0 to 50 g/L. For tests with HMP solution concentrations in the range 0 to 0.5 g/L, < 0.3% error in settling time results when the solution density is assumed to be that of pure water. Most settling-time calculations for pipet analysis (e.g., Day, 1965; Green, 1981) assume the dispersant solution has the viscosity of pure water. However, settling-time errors as great as 2% result from not correcting for increased viscosity when using 5 g/L HMP solutions. Water densities and viscosities at various temperatures are available from Weast (1983).[3]

Particle densities should be known with a precision of at least ± 0.05 Mg/m³. Settling-time errors in excess of 2% occur if particle densities are not known with at least this precision (see Table 15–3).

15–2.3.2 THEORY OF THE HYDROMETER METHOD

The hydrometer method, like the pipet method, depends fundamentally upon Stokes' Law, which for the hydrometer may be written as

$$X = \theta t^{-1/2} \qquad [7]$$

where θ is the sedimentation parameter and is a function of the hydrometer settling depth, solution viscosity, and particle and solution density. This relationship follows from Eq. [4] by rearranging terms such that

$$X = (18\eta h'/[g(\rho_s - \rho_l)])^{1/2} \, t^{-1/2} . \qquad [8]$$

[3]Note that Weast (1983) reports viscosity in centipoise (cpoise). For conversion to SI units, 1 cpoise = 10^{-3} kg m^{-1}s^{-1}.

Hence

$$\theta = (18\eta h'/[g(\rho_s - \rho_l)])^{1/2} \qquad [9]$$

where h' = hydrometer settling depth, cm.

The hydrometer settling depth, h', is a measure of the effective depth of settlement for particles with diameter X. It can be related to the hydrometer stem reading, R, by considering the specific design and shape of the hydrometer (Kaddah, 1974; ASTM, 1983d). The relationship of the settling depth to the hydrometer dimensions can be approximated by

$$h' = L_1 + 1/2 \, (L_2 - V_B/A) \qquad [10]$$

where

L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
L_2 = overall length of the hydrometer bulb, cm,
V_B = volume of hydrometer bulb, cm³, and
A = cross sectional area of the sedimentation cylinder, cm².

For the ASTM 152H hydrometer and a standard sedimentation cylinder: L_1 = 10.5 cm for a reading, R, of 0 g/L and 2.3 cm for a reading, R, of 50 g/L; L_2 = 14.0 cm; V_B = 67.0 cm³; and A = 27.8 cm². Substitution of these values into Eq. [10] and solving in terms of R yields

$$h' = -0.164 \, R + 16.3 \qquad [11]$$

where R is the uncorrected hydrometer reading. The use of Eq. [11] and [8] to calculate particle diameter is detailed in section 15–5.2.5.

Sedimentation parameter values, θ, as a function of hydrometer readings, R, have been tabulated for the ASTM 152H hydrometer for temperatures of 30 °C by Day (1965) and for 20 to 25 °C by Green (1981). Correction factors for other temperatures and for particle densities other than 2.65 g/cm³ are given by Day (1965). However, the use of Eq. [9] and [11] provides a straight-forward method to determine θ for any given temperature and particle density; hence tabulated θ values are not reported here.

ASTM 152H hydrometers are calibrated at 20 °C directly in terms of soil solution concentration, expressed as grams of soil per liter of solution (ASTM, 1985d). Correction of hydrometer readings for other temperatures and for solution viscosity and density effects is made by taking a hydrometer reading, R_L, in a blank (no soil) solution. This reading should be taken immediately after the uncorrected reading, R, is taken. The corrected concentration of soil in suspension at any given time is $C = R - R_L$, where C is expressed in g/L.

Differences in particle density for different soils affect particle settlement time, hence requires the correction of hydrometer readings and sedimentation parameter values. However, Gee and Bauder (1979) and

ASTM (1985d) show that moderate changes in particle density have only small effects on a given size determination. For example, errors in particle density of ± 0.1 g/cm^3 result in errors of $< \pm 0.5$ wt% clay for soils with clay contents up to 50 wt%.

15–3   SAMPLE PREPARATION

15–3.1   Apparatus

1. Drying trays
2. Wooden rolling pin
3. Sodium hexametaphosphate (HMP) solution (50 g/L)
4. Sieves. Large 20.5 cm (8 in.) diameter, with a 2 mm (2000 μm) square hole screen.
   Other screen sizes needed include: 5, 20, and 75 mm (USDA 1982); 5 mm (#4), 13 mm (1/2 in.), 20 mm (3/4 in.), 25 mm (1 in.), 50 mm (2 in.), and 75 mm (3 in.) (ASTM, 1985d).
5. Ruler or caliper capable of measuring to 250 mm (10 in.).

15–3.2   Method

Spread the bulk sample thinly (in 2 to 3 cm thick layers, maximum) on trays and air-dry. Thoroughly mix and roll the sample with a wooden rolling pin to break up clods to pass a 2-mm sieve. Sieve out the >2-mm size fractions. Continue rolling and sieving until only coarse fragments that do not slake in water or HMP solution remain on the 2-mm screen. Use a rubber roller for samples with easily crushed coarse fragments. Sieve larger size fractions, record weights, and use total sample weight to calculate the percentage of total sample < 2 mm.

15–3.3   Comments

Sometimes it is desirable to keep the sample at field moist conditions. If this is determined appropriate, force the field moist sample through the 2-mm screen by hand, using a large rubber stopper, double bag the sample in plastic, and store for further use. From a separate subsample determine the water content, so that a check can be made on possible drying effects during storage.

Whether material over 2 mm in diameter is sieved depends on the purpose for the data set. For soil survey purposes, methods specified by the USDA (1982) may be used. For engineering purposes, the material >2 mm can be sieved according to requirements specified by ASTM method D-2487 (ASTM, 1985a).

Sample size depends upon the maximum size fragments present. Suggested sample sizes are:
1. Particles up to 20 mm diameter—use 5 kg or more

2. Particles up to 75 mm diameter—use 20 kg or more
3. Particles up to 250 mm diameter—use 100 kg or more.

Because of the large samples required, the volume percent of particles coarser than about 20 mm is usually estimated. A suggested procedure for handling coarser fragments follows.

Weigh and sieve the entire sample through 75- and 20-mm screens. Weigh the >75-mm and the 75- to 20-mm fractions. Take a subsample of the <20-mm fraction for laboratory processing. Weigh the <20-mm sample before and after air-drying and correct the total sample weight for the loss of water from field conditions. Separate and weigh the 2- to 5-mm and the 5- to 20-mm fractions. If fine earth adheres to the coarse fraction, wash the coarse material, dry, reweigh, and apply the appropriate corrections. Calculate the coarse fractions as a percentage of the <20 mm material (or the <75 mm or the <250 mm depending upon the size limit involved in sampling). Note that for taxonomic (classification) purpose, stones or rock fragments >250 mm (10 in.) are separated and used to estimate the volume of coarse fragments for family placement of soils. A large caliper or ruler can be used to check the dimensions of the >250-mm material. In addition, weight measurements and volume displacement techniques can be used to evaluate coarse fragment volume.

15–4   PIPET METHOD

The pipet method is often used as a standard method from which other PSA methods are compared. This procedure has been adapted from Day (1965) and Green (1981).

15–4.1   Apparatus and Reagents

1. Beakers—100 mL to 1000 mL; centrifuge bottles, both glass and plastic—250 mL.
2. Centrifuges—low speed, about 1500 rpm, and high speed, about 12 000 rpm, with 250-mL bottles.
3. Filter candle—Porus ceramic tube, 0.05 MPa (0.5 bar) pressure rated.
4. Shakers—horizontal reciprocating shaker, sieve shaker, wrist action shaker, holders for 250-mL centrifuge bottles on paint shaker.
5. Cylinders—1000 mL (height of 1000-mL mark, 36 $\pm$ 2 cm).
6. Large (no. 13) rubber stoppers for 1000-mL cylinder.
7. Stirrers—electric stirrers for mechanical mixing (available from Soil Test, Inc., Evanston, IL, or other source),[4] hand stirrer made by joining a brass rod about 50 cm long to the center of a thin circular piece of perforated brass or plastic sheeting. The circular plate should

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[4]Trade names are used in this chapter soley for the purpose of providing specific information. Mention of a trade name does not constitute a guarantee of the product, nor does it imply an endorsement over other products not mentioned.

be cut to fit easily into the sedimentation cylinder. A 6-cm-diameter plate is normally adequate. If brass is used, place a wide rubber band around the edge of the brass sheeting to prevent scratching of the cylinder.

8. pH meter.
9. Pipet rack—device to permit sliding the pipet laterally and lowering the pipet to a percise depth in the sedimentation cylinder (Clark, 1962; Day, 1965; see also Fig. 15–4).
10. Lowy pipets—25 mL capacity (available from Sargent-Welch Co., Skokie, IL, or similar source).
11. Weighing bottles—(beakers can be used).
12. Set of sieves—square mesh with bronze wire cloth, 7.6 cm (3 in.) diameter with the following openings: 1000, 500, 250, 106, 53, or 47 μm.
13. Reagents—hydrogen peroxide ($\sim$30%); 1 M NaOAc (adjusted to pH 5); citrate–bicarbonate buffer: prepare 0.3 M sodium citrate (88.4 g/L) and add 125 mL of 1 M sodium bicarbonate (84 g/L) to each liter of citrate solution; sodium dithionite (hydrosulphite); saturated NaCl solution; 10% NaCl solution; 1 M AgNO$_3$; 1 M BaCl$_2$; acetone; Na-hexametaphosphate (HMP), 50 g/L stock solution; 1 M CaCl$_2$; 1 M HCl.

15–4.2   Procedures

15–4.2.1   PRETREATMENT

15–4.2.1.1   Removal of Carbonates and Soluble Salts. Weigh a small portion of the <2-mm fraction of air-dry soil into a 250 mL centrifuge bottle (10 g for clays, 20 g for loams, 40 g for sandy loams and loamy sands, and 80 g for sands). Weights are optional, but these are generally suitable if clay samples are required for mineralogy. Add approximately 100 mL of water, mix, and add 10 mL 1 M NaOAc (adjusted to pH 5). Centrifuge (about 10 min at 1500 rpm) until the supernatant is clear, then pour it off. Wash the soil twice by shaking with 50 mL of water, centrifuging and discarding the centrifugate if it is clear. If the centrifugate is not clear (as is often the case for soils containing high amounts of soluble salts and soils containing gypsum), further washing may be necessary. Washing through a filter candle to remove salts is a permissible substitute for centrifugation, but this procedure takes considerably longer than centrifugation. Check for salts by testing with AgNO$_3$ for Cl$^-$ and BaCl$_2$ for SO$_4^{2-}$.

15–4.2.1.2   Removal of Organic Matter. After carbonate removal, add 25 mL of water to the soil in the centrifuge bottle, and shake on a wrist action shaker. Transfer samples containing high amounts of organic matter (>5%) to 1000 mL beakers. Add 5 mL of (H$_2$O$_2$) to the soil suspension, stir, cover, and observe closely for several minutes. If ex-

cessive frothing occurs, cool the container in cold water. Add more H$_2$O$_2$ when the reaction subsides. Note that MnO$_2$ decomposes H$_2$O$_2$, so if present in measurable amounts, steps should be taken to complex or remove before peroxide treatment. Heat to 90 °C when frothing has ceased, remove cover, and evaporate excess water (do not take to dryness). Continue peroxide and heat treatment until most of the organic matter has been destroyed (as judged by the rate of reaction and the bleached color of the sample). Rinse down the sides of the reaction vessel occasionally. Heat for about an hour after the final addition of peroxide to destroy excess peroxide. Transfer the sample to a 250-mL glass centrifuge bottle.

15–4.2.1.3   Removal of Iron Oxides. Add citrate-bicarbonate buffer to the peroxide treated sample in the centrifuge bottle to bring the total volume of solution to approximately 150 mL. Shake to disperse the soil. Add 3 g of sodium dithionite (Na$_2$S$_2$O$_4$) gradually, as the sample may froth. Put the bottle into a water bath at 80° C and stir the suspension intermittently for 20 min. Remove the sample from the bath, add 10 mL of saturated NaCl, mix, centrifuge, and decant off the centrifugate. It may be combined with subsequent centrifugates, if any, and analyzed for dithionite-extractable Fe, Al, Mn, etc. If the sample is completely gray (gleyed), proceed to the next step. If brownish color remains, repeat the previous step. Wash the sample once with 50 mL of citrate–bicarbonate buffer plus 20 mL of saturated NaCl (shake, centrifuge, and decant). Wash the sample twice with 50 mL of 10% NaCl, then twice with 50 mL of distilled water. If the wash solution is not clear, transfer the sample to a plastic centrifuge bottle and centrifuge at high speed. If this fails to yield clear centrifugate, add acetone, warm the sample, and re-centrifuge. Add 150 mL of water, shake the sample, and check the pH. It should be above pH 8 if the soil is Na-saturated. Transfer the suspension to a 1-L shaker bottle, add 400 mL of distilled water and 10 mL of HMP (dispersant) stock solution, and shake overnight on a horizontal shaker.

15–4.2.2   SEPARATION OF THE SAND FRACTIONS

Pour the suspension through a 270-mesh (53 μm) sieve into a 1-L sedimentation cylinder. A 20-cm-diameter (8-in.) sieve is placed in a large funnel held by a stand above the cylinder. Tap the funnel gently and wash the sand thoroughly on the sieve. A soap solution placed on the sieve will aid in wetting the fine screen. Collect the washings in the cylinder. Transfer the sand to a tared beaker or aluminum weighing dish, dry (105 °C), and weigh.

Transfer the dried sand to the nest of sieves arranged from top to bottom with decreasing size in the following order: 1000-, 500-, 250-, 106-, 53-μm, and pan. Shake the sieves on a sieve shaker. A 3-min shaking time is usually adequate. Weigh each sand fraction and the residual silt and clay that passed through the 53-μm (270-mesh) sieve. Weighing precision of 0.01 g is adequate.

15–4.2.3 DETERMINATION OF SILT FRACTIONS

The 20 and 5 μm fractions can be determined by pipet by following the procedure outlined in the next section for clay and using Eq. [4] or Table 15–3 for determining the required settling times.

15–4.2.4 DETERMINATION OF CLAY (< 2 μm)

Place the cylinder containing the silt and clay suspension in a water bath; add 10 mL of HMP solution and make up to 1 L volume with distilled water; cover with a watch glass. Let the suspension stand at least several hours to equilibrate.

After equilibration, stir the suspension thoroughly with a hand stirrer for at least 30 s using an up-and-down motion. Note the time at completion of stirring and the temperature of the water bath. It is convenient to complete stirring of adjacent suspensions at intervals of about 3 min. An alternative to hand stirring is stoppering the sedimentation cylinder and shaking end-over-end for 1 min.

After the appropriate time interval (see Tables 15–1 through 15–3), lower the closed Lowy pipet carefully to the appropriate depth, turn on the vacuum, and withdraw a 25-mL sample in about 12 s (see Fig. 15–5). A device for controlling the vacuum is required.

Discharge the sample into a tared and numbered weighing bottle, beaker, or aluminum dish. Rinse the pipet with distilled water and add the rinse water to the clay suspension in the weighing bottle. Evaporate the water, dry the clay at 105 °C, cool in a desiccator, and weigh.
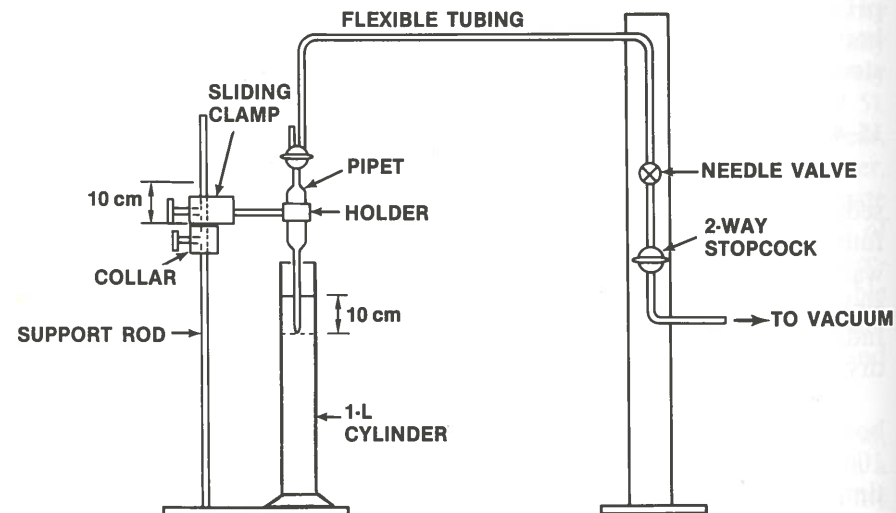

Fig. 15–5. Schematic diagram of pipet stand and apparatus for sedimentation analysis.

15–4.2.5 DETERMINING THE WEIGHT OF TREATED SOIL

Add 10 mL of 1 M CaCl$_2$ and 1 mL of 1 M HCl to the suspension remaining in the cylinder to prevent CaCO$_3$ formation. Siphon off the clear solution after flocculation has occurred. Transfer the soil from the cylinder to a tared beaker, evaporate, dry at 105 °C, and weigh.

Differences between original soil weight and weight found in the cylinder are attributed to pretreatment soil loss, solution loss, sieving loss, and sample removal for pipet sieving analysis. The total oven-dry weight of the treated sample is used as the basis for calculating the size fraction. The total oven-dry weight can be expressed as:

$$W_s + W_p + W_r = W_t \qquad [12]$$

where
W_s = oven dry weight of sand fraction,
W_p = corrected oven dry weights of pipet samples,
W_r = corrected oven dry weight of residual silt and clay, and
W_t = total weight of treated sample.
W_r and W_p are corrected by subtracting the weight of the dispersing agent (Table 15–4).

15–4.3.6 Calculations

Table 15–4 shows how the pipet method is used to determine size-fraction percentages using a 25-mL pipet.

15–4.4 Comments

Flocculation of clay from suspension has been observed in soils containing large amounts of gypsum (Kaddah, 1975; Hesse, 1976; Rivers et al., 1982). Flocculation is recognized by a distinct separation of clear liquid and suspended clay (flocculated clay often has the appearance of a cloudy gel-like precipitate). Removal of soluble salts (Section 15–4.2.1.1) helps prevent flocculation in most soils. However gypsum, having a low but measurable solubility, can cause flocculation by replacement of Na

Table 15–4. Example calculations of three particle-size percentages using a 25-mL pipet.

Particle size	Sample weight	Concentration	Corrected concentration†	Percent less than‡
mm	g	g/L	g/L	%
0.020	0.114	4.56	4.06	39.8
0.005	0.073	2.92	2.42	23.7
0.002	0.057	2.28	1.78	17.4

† Dispersing agent concentration = 0.5 g/L.
‡ Based on oven-dry weight of treated sample, W_t = 10.21 g.

with Ca. Procedures for removal of gypsum are available (Rivers et al., 1982). Options for removal of gypsum include adding barium (Hesse, 1976) or increasing the concentration of HMP dispersant (Kaddah, 1975). Flocculation must be prevented for sedimentation analysis (pipet, hydrometer, etc.) to provide meaningful results.

Errors in PSA values using the pipet analysis are mainly associated with sampling and weighing. With care, clay fractions can be determined with a precision of ± 1 wt% using pipet procedures.

15–5 HYDROMETER METHOD

Particle-size analysis can be done conveniently with a hydrometer which allows for nondestructive sampling of suspensions undergoing settling. The hydrometer method provides for multiple measurements on the same suspension so that detailed particle-size distributions can be obtained with minimum effort. The hydrometer method outlined is that modified from Day (1965) and ASTM (1985d).

15–5.1 Apparatus and Reagents

1. Standard hydrometer, ASTM no. 152 H, with Bouyoucos scale in g/L (Fig. 15–6).
2. Electric stirrer (malted-milk-mixer type, with 10 000-rpm motor).
3. Plunger or rubber stoppers for 1000-mL sedimentation cylinders.
4. Sedimentation cylinders with 1-L mark 36 ± 2 cm from the bottom of the inside.
5. Metal dispersing cups and 600-mL beakers.
6. Amyl alcohol.
7. Sodium-hexametaphosphate (HMP) solution (50 g/L).
8. Set of sieves—7.6-cm (3 in.) diameter square mesh woven bronze wire cloth, with the following openings: 1000, 500, 250, 106, 75, and 53 μm.
9. Electric oven and weighing jars.

15–5.2 Procedure

15–5.2.1 CALIBRATION OF HYDROMETER

Add 100 mL of the HMP solution to a cylinder and make the volume to 1 L with room temperature distilled water. Mix thoroughly with plunger
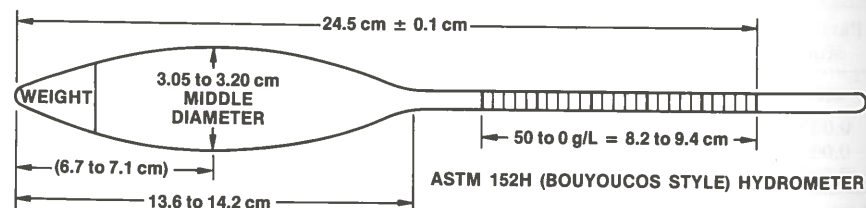

Fig. 15–6. Schematic diagram of ASTM 152 H-type hydrometer.

and record temperature. Lower the hydrometer into the solution and determine R_L, the hydrometer-scale reading of the (blank) solution. Read the upper edge of the meniscus surrounding the stem. Periodically recheck R_L during the course of the hydrometer tests (section 15–5.2.3). The calibration value R_L is used in the analysis to correct for solution viscosity and to correct the soil solution concentration, C.

15–5.2.2 DISPERSION OF SOIL

Weigh 40.0 g of soil into a 600-mL beaker, add 250 mL of distilled water and 100 mL of HMP solution, and allow the sample to soak overnight. The exact sample size depends upon soil texture. For fine-textured soils—silts or clays—10 to 20 g may be adequate. For coarse sands, 60 to 100 g will be needed in order to obtain reproducible results. Most temperate zone soils can be air dried prior to testing. However, for many tropical soils and soils of volcanic origin, samples must be stored at field moisture. Weigh another sample of the soil (about 10 g) for determination of oven-dry weight. Dry overnight at 105 °C, cool, and weigh.

Transfer the HMP-treated sample to a dispersing cup and mix for 5 min with the electric mixer, or transfer the suspension to shaker bottles and shake overnight on a horizontal shaker. Transfer the suspension to a sedimentation cylinder and add distilled water to bring the volume to 1 L.

15–5.2.3 HYDROMETER MEASUREMENTS

Allow time for the suspension to equilibrate thermally and record temperature. Insert plunger into cylinder and mix the contents thoroughly. Hold bottom of cylinder to prevent tipping. Dislodge sediment from the bottom using strong upward strokes of plunger. Finish stirring with two or three slow, smooth strokes. An alternative mixing procedure is to stopper the cylinder and use end-over-end shaking for 1 min. Add a drop of amyl alcohol if the surface of the suspension is covered with foam. As soon as mixing is completed, lower the hydrometer into the suspension and take readings after 30 s and again at the end of 1 min. Remove the hydrometer, rinse, and wipe it dry. Reinsert the hydrometer carefully about 10 s before each reading and take readings at 3, 10, 30, 60, 90, 120, and 1440 min. Times of reading can be modified according to need. Remove and clean the hydrometer after each reading. Record the reading R at each time. Read the hydrometer after placing it in the blank solution (containing no soil), and record the blank reading as R_L and the temperature at each time.

15–5.2.4 SEPARATION OF SAND FRACTIONS

Quantitatively transfer the sediment and suspension from the 1-L sedimentation cylinder through a 270-mesh (53-μm) sieve. A 20-cm-diameter (8 in.) sieve is placed over a sink. The sediment is washed onto

the 53-μm screen using a wash bottle or gentle stream of water. The 53-μm screen can be dipped in a soap solution to improve the wettability of the screen and speed the flow. Transfer the sand to a tared beaker or aluminum weighing dish, dry (105 °C), and weigh.

Transfer the dried sand to the nest of sieves arranged from top to bottom in the following order: 1000, 500, 250, 106, and 53 μm. Shake on a sieve shaker for 3 min. Weigh each sand fraction and the residual silt and clay that has passed through the 53-μm sieve.

15–5.2.5 CALCULATION OF PARTICLE SIZE

Determine C, the concentration of soil in suspension in g/L, where $C = R - R_L$, with R, the uncorrected hydrometer reading in g/L, and R_L, the hydrometer reading of a blank solution. R and R_L are taken at each time interval. Determine P, the summation percentage for the given time interval, where $P = C/C_o \times 100$ and $C_o =$ oven-dry weight of the soil sample.

Determine X, the mean particle diameter in suspension in μm at time t, using Eq. [7], [9], and [11]:

$$X = \theta t^{-1/2} . \qquad [13]$$

For the special case that X and t are reported in μm and min, respectively, and all other terms expressed in cgs units, the sedimentation parameter is commonly written as

$$\theta = 1000(Bh')^{1/2} , \qquad [14]$$

where $B = 30\eta/[g\,(\rho_s - \rho_l)]$, and $h' = -0.164R + 16.3$ (Eq. [11]), and with each term expressed in the following units:[5]
   $\theta =$ sedimentation parameter, μm min$^{1/2}$,
   $h' =$ effective hydrometer depth, cm,
   $\eta =$ fluid viscosity in poise, g cm^{-1}s^{-1},
   $g =$ gravitational constant, cm/s^2,
   $\rho_s =$ soil particle density, g/cm^3, and
   $\rho_l =$ solution density, g/cm^3.
Equations [5] and [6] can be used to provide approximate corrections for density and viscosity variations for HMP solutions.

Plot a summation percentage curve (P vs. log X) using hydrometer readings taken over a time period from 0.5 min to 24 h coupled with sieve data. From this curve determine sand, silt, and clay percentages.

For routine textural analysis a summation percentage curve has more detail than is required; hence, the following procedure may be used.

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[5] The sedimentation parameter and associated terms have not been expressed in standard SI units in order to maintain consistency with reported tables (Day, 1965; Weast, 1984).

15–5.2.5.1 Simplified Clay Fraction Procedure.

1. Take hydrometer readings at 1.5 and 24 h only (record both R and R_L values).
2. Determine effective particle diameter X and summation percentage P for 1.5- and 24-h readings using Eq. [7] and [13].
3. Compute $P_{2\mu m}$ (summation percentage at 2 μm) as follows:

$$P_{2\mu m} = m \ln (2/X_{24}) + P_{24} \qquad [15]$$

where

$X_{24} =$ mean particle diameter in suspension at 24 h (from Eq. [7]),
$P_{24} =$ summation percentage at 24 h,
$m = (P_{1.5} - P_{24})/\ln (X_{1.5}/X_{24}) =$ slope of the summation percentage curve between X at 1.5 h and X at 24 h,
$X_{1.5} =$ Mean particle diameter in suspension at 1.5 h, and
$P_{1.5} =$ summation percentage at 1.5 h.

15–5.2.5.2 Sand Fraction Calculation.
Compute the 50-μm summation percentage, using the same procedure as for $P_{2\mu m}$, but use the 30- and 60-s hydrometer readings rather than the 1.5- and 24-h readings, respectively, and subtract the computed $P_{50\mu m}$ value from 100 to obtain the sand percentages. A standard sieve analysis should be run for comparison, using a 53- or 47-μm screen (section 15-5.2.4).

15–5.2.5.3 Silt Fraction Calculation.
Determine the percent silt by difference as

$$\% \text{ silt} = 100 - (\% \text{ sand} + \% \text{ clay}) . \qquad [16]$$

Calculations for sand, silt, and clay are conveniently made with a programmable desk calculator or microcomputer. BASIC and FORTRAN programs for clay fraction and textural determinations are available from the authors upon request.

15–5.2.6 COMMENTS

Flocculation of clay by soluble salts or gypsum during sedimentation may cause significant errors in the hydrometer method, since no pretreatment is used. Kaddah (1975) recommends increasing the concentration of HMP to levels high enough to maintain dispersion. If higher concentrations are used, the blank solution must contain the same concentration of HMP as that used in the soil solution so that the blank reading, R_L, corrects for the increased solution viscosity and density. If soil is high in soluble salts or gypsum, pretreatment procedures (section

15-4.2.1.1), removal techniques (Rivers et al., 1982), or chemical treatment (Hesse, 1976) may be needed.

The Bouyoucos procedure (Bouyoucos, 1962) has been used by a number of laboratories to estimate sand, silt, and clay from hydrometer measurements. Readings at 40 s and 2 h are used to estimate sand and clay percentages, respectively. From basic sedimentation theory, the 2-hr reading cannot yield correct estimates of the 2-μm clay fraction. Based on theoretical considerations, the 2-h hydrometer reading is a closer estimate of the 5-μm silt fraction than it is of the 2-μm clay fraction, and errors in clay contents using the 2-h reading often exceed 10 wt% for clay soils (Gee & Bauder, 1979). Similar problems arise when using the 40-s hydrometer reading to estimate the sand fraction. Differences between sieve and 40-s hydrometer measurement often exceed 5 wt%. The correlations between silt and clay and the 40-s and 2-h readings are empirical. In some cases, they seem adequate for textural class identification, but cannot be used to accurately define the particle size, hence, the Bouyoucos procedure is not recommended.

Walter et al. (1978) compared pipet and hydrometer measurements of 2μm size fraction in glacial till soils and found agreement well within 5%. Liu et al. (1966) also found generally good agreement between pipet and hydrometer analysis. Calculated correlation coefficients (r values) varied between 0.90 and 0.99 for 155 samples of soils from eleven states. These and other results suggest that pipet and hydrometer can give comparable results, with major differences arising largely from differences in pretreatment techniques.

A detailed error analysis for the hydrometer has been made by Gee and Bauder (1979). They indicate that the major source of error is in the hydrometer reading. An error of ± 1 g/L hydrometer reading results in an error of about ± 2 wt% for clay-size particles.

15-6 OTHER METHODS

In addition to sieving and sedimentation procedures, there are numerous techniques for measurement of particle-size distribution that have been developed for powder technology and other applications. These techniques include optical microscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM), electrical sensory zone (Coulter counter) methods, and light-scattering methods such as laser-light scattering, turbidimeters, holography, and x-ray centrifuges. An excellent discussion of these and other methods for particle-size distribution is given by Allen (1981).

Pennington and Lewis (1979) and Lewis et al. (1984) describe a procedure for using Coulter counters for particle-size distribution and textural analysis. Tama and El-Swaify (1978) have used turbidimeters to qualitatively assess clay contents in tropical soils. Weiss and Frock (1976) and Cooper et al. (1984) detail the use of laser light scattering methods

for PSA and textural analysis. Laser-light instruments normally do not operate into the clay range; hence, a correction factor is used to estimate clay-size materials (Cooper et al., 1984). Soil mineralogy, particle shape, and density all affect this correction factor.

Standard procedures for PSA using Coulter counters, turbidimeters, or laser-light techniques are not proposed at this time. High cost of instrumentation coupled with uncertainties in correction factors make these methods less attractive than the pipet or hydrometer methods for most routine applications. However, in such applications as the analysis of runoff sediments, where great numbers of tests are required, the speed of these methods has encouraged their use, particularly when only relative values of particle size are considered adequate.

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