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DETERMINATION OF PARTICLE SIZE DISTRIBUTION IN SOILS: Comparison of Results Obtained by the Hydrometer vs. the Pipette Method

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RESEARCH REPORT

Determination of Particle Size Distribution in Soils: Comparison of Results Obtained by the Hydrometer vs the Pipette Method

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ABSTRACT

To determine the particle size distribution in the soil, researchers have mainly used pipette method (the reference method, internationally adopted) or the hydrometer method, which is more rapid and less laborious. Scientists using both methods, however, have reported that readings taken after preparation of the soil suspension as suggested by Bouyoucos, overestimate the clay fraction of soils when compared with the pipette method.

In the present study, 94 soil samples were collected from the main agricultural areas of Syria and subjected to analysis by pipette and hydrometer methods. The samples represented different soil depths from irrigated and rainfed soils, collected from low, medium, and high rainfall areas. Calcium carbonate content in the soils ranged from 2% to 47%, the clay content from 4% to 85%, organic matter from 0.32% to 2.33%. The results from the two methods of analysis were highly correlated (R^2=0.83), although the hydrometer readings after 2h (H2) indicated higher values for clay content than did the pipette method. The differrences occurred because the hydrometer readings represented all soil particles with diameter less than 3.88±0.12 (microns) μ, whereas the clay fraction is represented by particles <2 (microns) μ in diameter. From these readings, correction factor was derived better results:

$$\%clay\ (pipette) = -13.787 + 1.136 \times (H2)$$

The hydrometer readings taken after 7 hours (H7) provided a closer fit with results from the pipette methods (R^2=0.87) because they represented all soil particles with diameter less than 2.1±0.07 (microns) μ. The regression equation was:

$$\%clay\ (pipette) = -1.213 + 1.090 \times (H7)$$

Hydrometer readings at 4 hours (H4) were found to be highly correlated with the 7-hours readings (R^2=0.97) and provided a closer fit with the pipette method than did 2-hours readings:

$$\%clay\ (pipette\ method) = -0.955 + 1.062 \times (H4)$$

Thus, the hydrometer method, with readings taken at 4 hours, could be used as an alternative for the pipette method to determine clay content for all practical purposes, no correction would be needed.

INTRODUCTION

Soil particles have diverse composition, structure, size and shapes. They are amorphous or crystalline and although normally bound together, can be separated by chemical and mechanical means. The "particle size distribution" refers to the distribution of soil particles into distinct classes according to size (Day 1965). Soil fractionation and particle size analysis are done by sieving and sedimentation, and Stoke's Law of sedimentation governs all methods used. The two main methods for determining particle size distribution are:

- The pipette method, which utilizes samplings of soil suspension at controlled depth and time, (this is the official method of the International Society of Soil Science, adopted in 1929, and considered the reference method); and

- The hydrometer method, which was first introduced by Boyoucos in 1926 and later improved by Casagrande (1934), (this method was given a solid theoretical basis by Day in 1965 and was assessed in detail for particle size analysis by Kaddah in 1973).

There is general agreement on the reproducibility and consistency of the pipette method, and as a result it has been adopted by soil scientists. However, the hydrometer method has been used extensively for routine analysis of soils, because it is easy and quick. Although the hydrometer apparatus was designed to give direct readings of a soil suspension, many workers found it overestimated the clay contents.

Hesse (1974) found that accurate determination of clay content in the nongypsiferous soils of the Euphrates could be obtained from hydrometer readings taken at 9h after the soil was put in suspension, and he suggested a correction factor for use with readings taken at 2h. We extended Hesse's work to determine, for a large variety of Syrian soils, the relationships between hydrometer readings and pipette determinations of clay content in the soil and to determine the optimal time for hydrometer readings, i.e., the time at which hydrometer readings most closely fit the results of the international pipette method.

THEORY

The principle involved in the particle size analysis of soils by the hydrometer method is similar to that of the pipette method. It depends fundamentally on Stoke's equation, which can be written (Day 1965):

$$X = \theta/t^{1/2} \qquad (1)$$

where: X = soil particle diameter in microns (u)

θ = sedimentation parameter equivalent to $[1000\ (30\ \eta h)/g\ (P_S - P_L)]^{1/2}$

where: η = viscosity in poise

h = hydrometer settling depth at time of reading (cm)

P_S = soil particle density (g/ml)

P_L = liquid density (g/ml)

g = acceleration of gravity (980.7 cm/s)

For the hydrometer method the sedimentation parameter θ is not constant during sedimentation because the depth of immersion of the hydrometer bulb is not constant. It changes with time and with the texture of soil to be measured. As a result θ could have different values. Consequently, one can calculate the average diameter (X) of soil particles measured by the hydrometer method from equation (1) by using time and the values of θ estimated from hydrometer readings (R) and referring to the conversion table prepared by Day (1965).

The value of θ should be constant for the pipette method because (h) is kept constant; and the clay particle diameters are all particles less than 2 microns (μ). But for the hydrometer method, h and θ are not constant, and the diameters measured could be quite different from 2 microns (μ).

MATERIALS AND METHODS

Ninety-four soil samples were collected from main agricultural areas of Syria (nine provinces), where ICARDA, in cooperation with the Syrian Ministry of Agriculture and Agrarian Reform, has been conducting on-farm agronomic and verification trials. The samples represented irrigated areas and areas where mean annual rainfall is high (>350 mm), medium (250-350 mm, with >350 mm in two-thirds of years), and low (≤250 mm, with >250 mm in half the years). Sampling depths varied, as did the contents of calcium carbonate and organic matter and the texture.

The soil suspension for both the pipette and the hydrometer method consisted of soil, water, hydrogen peroxide (to destroy the organic matter), and a mixture of sodium hexametaphosphate (calgon) and sodium carbonate, as a dispersing agent.

The procedures for the pipette and hydrometer methods are detailed in the Appendix.

Hydrometer readings for clay content determination were taken after 2 and 7 hours on all 94 soil samples for comparison with the pipette method. A second comparison (for 28 samples) was made with hydrometer readings taken after 4 and 7 hours.

RESULTS AND DISCUSSION

All soils were calcareous, with $CaCO_3$ content ranging from 2% to 47% (Table 1). Organic matter content was between 0.21% and 2.33%, and texture of soils varied from loam to heavy clay. Obviously, the soils were diverse representing a good sample for comparison of the methods for soil particle analysis.

The values for clay content, as determined by hydrometer readings, 2 hours after preparation of the suspension (as suggested by the original method, Boyoucos), were closely correlated (R^2=0.83) with, but had significantly higher value than those by the pipette method (Figure 1). The regression equation relating the methods was:

$$\%clay\ (pipette) = -13.787 + 1.136 \times (H2) \qquad (2)$$

The slope of the regression was significantly higher than 1, and the intercept large. The discrepancies between the pipette and hydrometer readings could be easily explained by the calculation from equation (1) of the average diameter of particles measured by the hydrometer method: all the soil particle diameters less than 3.88 microns (μ) (SE=0.12 μ). These particles included not only the clay fraction (< 2 μ) but some of the fine silt fractions.

The readings obtained by the hydrometer method after 7 hours more closely corresponded to the estimates by the pipette method (Fig. 2):

$$\%clay\ (pipette) = -1.213 + 1.090 \times (H7)$$

$$(R^2 = 0.87) \qquad (3)$$

The diameter of soil particles contributing to the hydrometer readings at 7 hours was calculated and was $\leq$(2.13$\pm$0.07) microns, a size that is more in line with the fraction measured by the pipette method, and is in keeping with observation by Hesse (1974).

Unfortunately, readings taken after 7 hours are difficult to accommodate in routine soil laboratories because of the time needed for preparation of soil suspensions. However, hydrometer readings taken after 4 and 7 hours were highly correlated (R^2=0.98), and the slope of regression was practically equal to one (Fig. 3). The regression equation was:

$$\%clay\ (H7) = -1.484 + 0.999 \times (H4) \qquad (4)$$

The particle diameter included in the hydrometer readings after 4 hours represented all particles $\leq$(2.78$\pm$0.08) microns.

Although hydrometer readings taken after 2 and 7 hours were also highly correlated (R^2=0.97), the slope of the regression line was greater than one (=1.05) and the intercept large (=-12.079). The regression equation was:

$$\%clay\ (H7) = -12.079 + 1.051 \times (H2) \qquad (5)$$

CONCLUSIONS AND RECOMMENDATIONS

As a result of the present study, we concluded that the clay content in soils could be estimated with a good degree of accuracy by the hydrometer method by taking readings after 2 hours and correcting the values using equation (1). A better approach would be to take readings after 4 hours, with no need for correction for all practical purposes. An equation that corrects the discrepancies is:

$$\%clay\ (pipette) = -0.955 + 1.062 \times (H4) \qquad (6)$$

ACKNOWLEDGEMENTS

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TABLE 1 Soil sample characteristics and geographical distribution

Province	Site	Zone[a]	Farming systems	Depth (cm)	$CaCO_3$ (%)[3]	Organic matter (%)
Hama	Lattamneh	-	Irrigated	0-20	29	1.24
	Lattamneh	-	Irrigated	20-40	30	0.97
	Skelbieh	A	Rainfed	0-20	15	1.61
	Skelbieh	A	Rainfed	20-40	18	-
	El-Ghab	A	Rainfed	0-20	25	2.32
	El-Ghab	A	Rainfed	20-40	25	2.23
	Res. Station	-	Irrigated	0-20	31	1.54
	Res. Station	-	Irrigated	20-40	31	-
	Suran	B	Rainfed	0-20	35	1.04
	Suran	B	Rainfed	20-40	36	0.92
	Bari Sharqi	C	Rainfed	0-20	2.6	1.60
	Bari Sharqi	C	Rainfed	20-40	-	-
	Bari Sharqi	C	Rainfed	40-60	-	-
	Bari Sharqi	C	Rainfed	60-80	-	-
HASSAKEH	Hannawieh	A	Rainfed	0-20	20	1.22
	Hannawieh	A	Rainfed	20-40	21	0.87
	Mjarjah	C	Rainfed	0-20	29	1.24
	Mjarjah	C	Rainfed	20-40	32	0.81
	Himo	A	Rainfed	0-20	28	1.30
	Himo	A	Rainfed	20-40	30	-
	Tweine	C	Rainfed	0-20	34	-
	Tweine	C	Rainfed	20-40	35	-
HASSAKEH	Tel-Khadr	C	Rainfed	0-20	30	1.54
	Tel-Khadr	C	Rainfed	20-40	35	0.94
	Arisha	B	Rainfed	0-20	35	1.04
	Arisha	B	Rainfed	20-40	35	0.55
	Kherbet-Jamal	B	Rainfed	0-20	17	1.80
	Kherbet-Jamal	B	Rainfed	20-40	18	-
	Tel Tishreen	A	Rainfed	0-20	-	0.90
	Abu-Hjera	B	Rainfed	0-20	-1-	
				20-40	-	-
				40-60	-	-
				60-80	-	-
				80-100	-	-

TABLE 1 Continued

Province	Site	Zone[a]	Farming systems	Depth (cm)	$CaCO_3$ (%)[3]	Organic matter (%)
	El-Areesha	C	Rainfed	0-20	35	1.08
				20-40	35	0.62
				40-60	-	-
ALEPPO	Tel Hadya	B	Rainfed	0-20	25	0.85
				20-40	26	0.71
				0-15	-	-
				15-30	-	-
				30-45	-	-
				45-60	-	-
				60-75	-	-
				75-90	-	-
				90-105	-	-
	Sfireh	C	Rainfed	0-20	22	0.80
				20-40	22	
	Yahmool	A	Rainfed	0-20	5.9	0.81
				20-40	6.6	0.78
	Oweina Saghira	C		0-20	32	-
				20-40	34	-
	Homaima	C	Rainfed	0-20	25	2.14
				20-40	27	1.61
	Breda	C	Rainfed	0-20	30	1.43
				20-40	29	1.22
	Atareb	A	Rainfed	0-20	18	1.04
				20-40	19	0.87
	Akhtarin	B	Rainfed	0-20	20	0.87
				20-40	21	0.76
	Abteen	B	Rainfed	0-20	28	0.76
				20-40	35	0.64
				0-20	-	-
				20-40	-	-
				40-60	-	-
				60-80	-	-
				80-100	-	-
IDLIB	Sarakeb	B	Rainfed	0-20	28	1.20
				20-40	31	0.83
	Khan Shekhoon	A	Rainfed	0-20	18	0.92
				20-40	19	0.78

TABLE 1 Continued

Province	Site	Zone[a]	Farming systems	Depth (cm)	$CaCO_3$ (%)	Organic matter (%)
	Tawahina	C	Rainfed	0-20	46	1.61
				20-40	47	-
	Taoum	B	Rainfed	0-20	-	0.70
				0-20	-	-
	Afes	B	Rainfed	0.20	0	0.70
RAQQA	Res. Station	-	Irrigated	0-20	19	0.76
				20-40	20	0.75
	Ras el Ain	B	Rainfed	0-20	35	1.06
			Rainfed	20-40	36	0.74
	Hamam el Turkmen	C	Rainfed	0-20	32	1.43
				20-40	35	0.67
	Ghzeilan	B	Rainfed	0-20	24	1.22
				20-40	26	0.64
HOMS	Res. Station	A	Rainfed	0-20	2.0	1.20
				20-40	3.0	0.94
DARAA	Gleen	A	Rainfed	0-20	4.0	0.78
				20-40	4.0	0.69
	Izreh	B	Rainfed	0-20	13.0	0.67
				20-40	14.0	0.62
	Khabab	C	Rainfed	0-20	22	0.39
				20-40	22	0.32
DER EL ZOR	Saalo	-	Irrigated	0-20	19	1.17
				20-40	20	-

a = Zones are divided according to annual rainfall: A = >350 mm; B = 250-350 mm, with two-thirds of years having >250 mm; and C = 250 mm, with one-half of years having >250 mm.

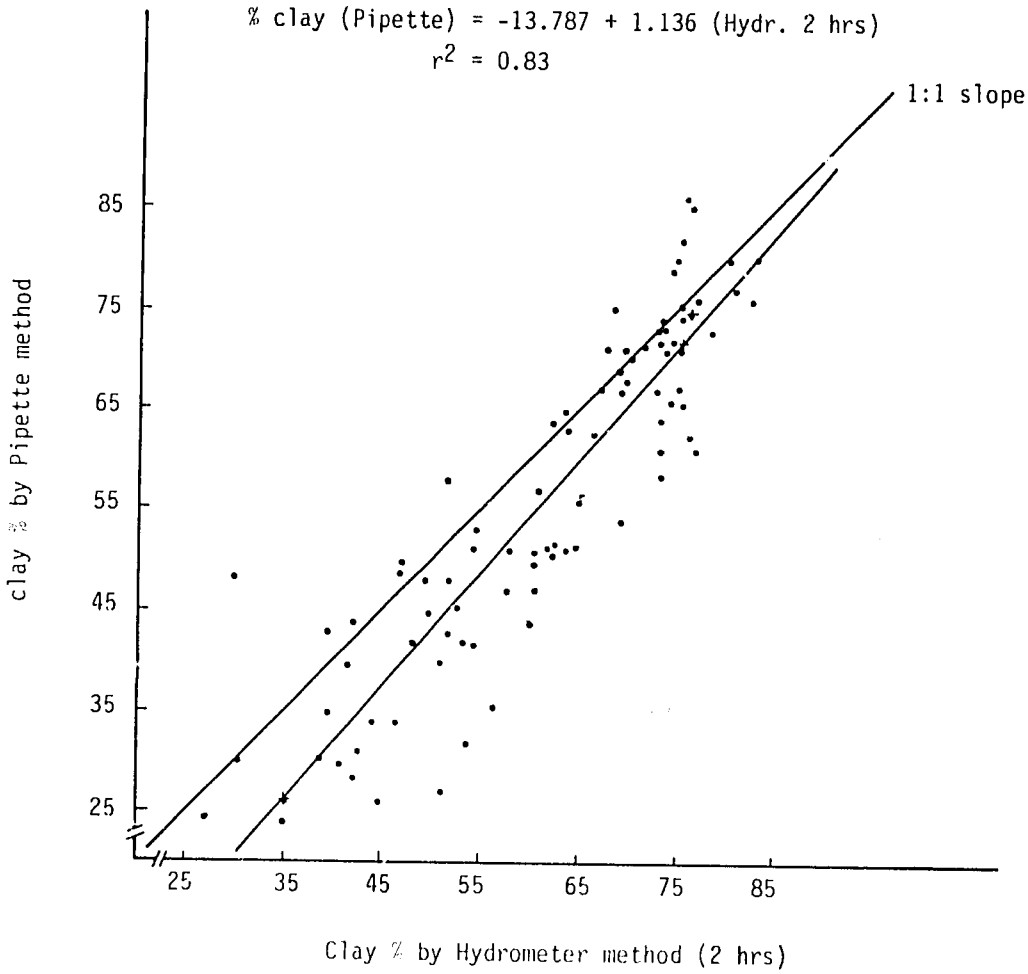

FIGURE 1 Relationships between values obtained for clay content of soils by the Hydrometer (2 hrs readings) and the Pipette method

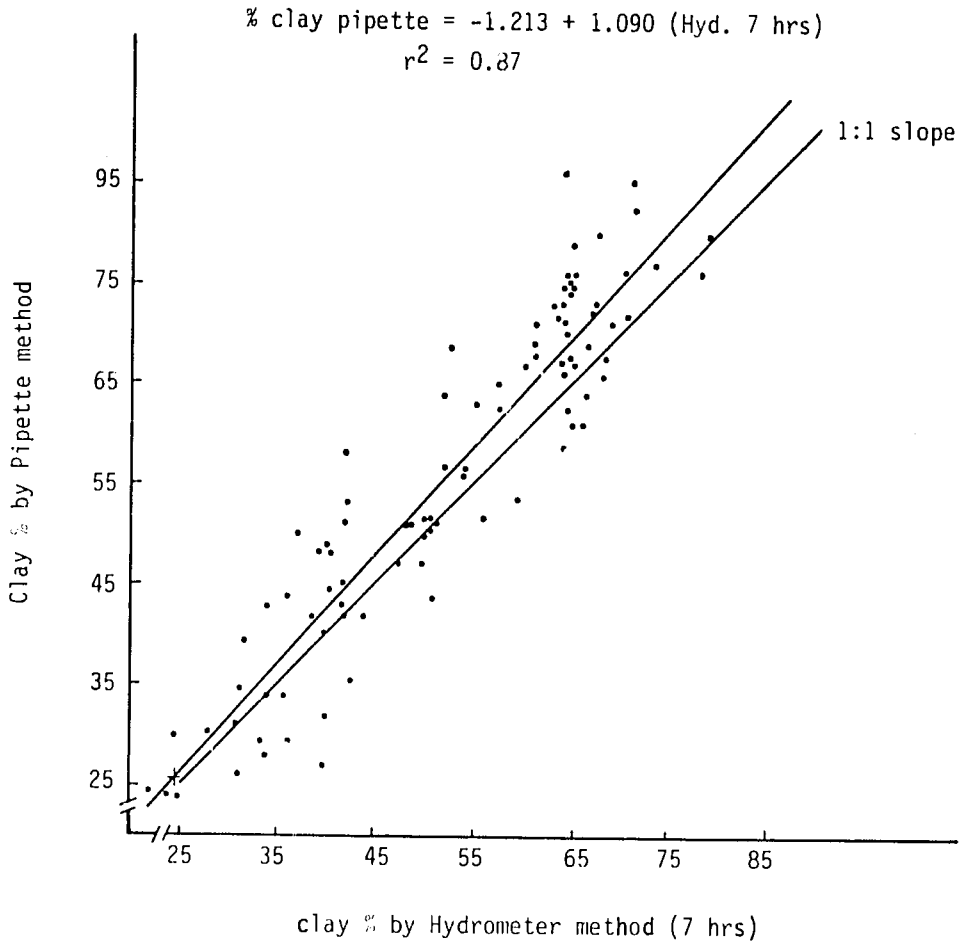

FIGURE 2 Relationships between values obtained for clay content of soils by the Hydrometer (7 hrs readings) and the Pipette method.

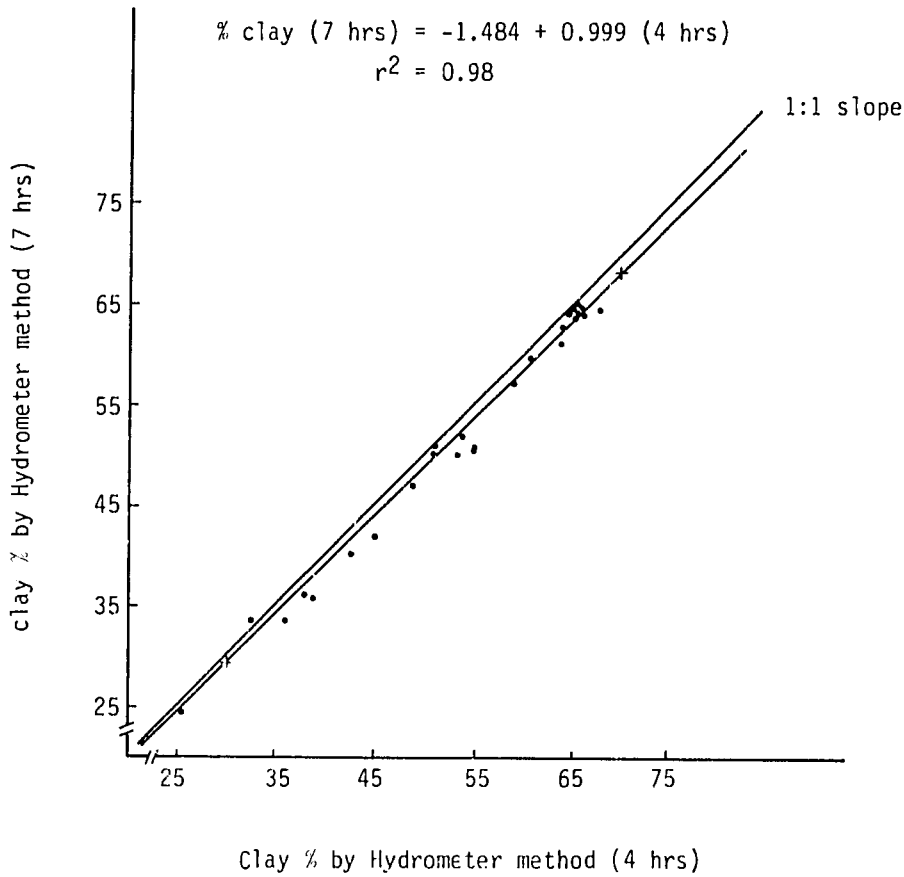

FIGURE 3 Relationships between values obtained for clay content of soils by Hydrometer method taken readings after 4 and 7 hours

APPENDIX

Procedures used for particle size analysis

Hydrometer Method

Dispersing solution:

40 g of sodium hexametaphosphate and 10 g of anhydrous sodium carbonate was dissolved in water, filtered, and diluted to 1 l. Fresh solution of this reagent was prepared with every batch of soils (solution should not be older than 2 weeks).

Procedure

- Weigh 40 g of 2 mm soil into a 600 ml beaker and add 60 ml of dispersing reagent with few drops of (H_2O_2) hydrogen peroxide 30% and mix. Cover the beaker and leave overnight.

- Quantitatively transfer the contents of the beaker to the soil stirring cup (Soil Test Inc. Cat No. Cl. 274) and fill the cup to about three-quarters with water. Stir the suspension at high speed for 2 minutes using the special stirrer (Soil Test Inc. Cat. No. Cl. 273).

- Rinse the stirring paddle into the cup, allow to stand for 1 min. and pour the suspension into a 1 l cylinder (Soil Test Inc. Cat. No. Cl. 271).

Determination of Silt and Clay

- Bring the volume of the cylinders to 1 l.

- Mix the suspension well using the special paddle; immediately after withdrawing the paddle insert the hydrometer. Disperse any froth with one drop of amylalcohol and take the hydrometer reading at once but not later than 40 s after withdrawing the paddle. Reading R_{SC}. The same hydrometer reading is taken for the blank RB.

$$\% \text{ (silt + clay)} = (R_{SC} - R_B) \times (100/40)$$

Determination of Clay

Remix the suspension in the cylinder with the paddle and leave undisturbed. Insert the hydrometer and take readings (R_C) at various times: 2, 4, 7 h.

$$\%clay = (R_C - R_B)\ (100/40)$$

Determination of Sand

Pour the suspension through a 0.053 mm sieve, very sandy soils tend to block the sieve and light stirring with a rubber tipped rod may be necessary.

When all liquid has passed, the residue on the sieve is thoroughly washed with a jet of water from a wash-bottle. Washing should be continued until the water passing through the sieve is clear.

Transfer the sand from the sieve to a small, tared dish using a jet of water: the traces of sand that stick to the sieve mesh can be transferred by washing from the underside of the sieve. Leave the dish for a few minutes to let the sand settle, and then decant the excess water. The dish plus sand is then dried at 110°C, after cooling it is reweighed and the weight of sand determined.

$$\%\ sand = (wt.\ of\ residue)\ (100/40)$$

Determination of Silt

Silt can be measured directly by subtracting the percent clay from the measured percent clay plus silt or it can be estimated by difference, i.e.

$$\%\ silt = 100 - (\%\ sand + \%\ clay)$$

in which case the totals will always be exactly 100. The former, direct method, is recommended as the actual total obtained serves as a check on the accuracy of the work.

NB: All results should be corrected for moisture content.

Pipette Method

Materials and reagents:

- Andreasen Sedimentation Pipettes (550 ml)
- Sodium hexametaphosphate solution, prepared as in hydrometer method
- Stirrer (Soil Test Inc. Cat. No. Cl. 273)

Procedure

- Weigh sufficient soil so that, on dilution in the sedimentation vessel to the 20 cm graduation mark (550 ml), the soil concentration will be about 1% by volume (5.5 g soil).
- Using a separate soil sample, determine the moisture content to correct the true soil weight.
- Add 9 ml of dispersing solution to the 5.5 g soil and mix.
- Leave overnight.
- On the second day, transfer the dispersed sample into the stirring cups and stir for 3 min.
- Transfer the suspension into the sedimentation vessel and adjust the volume with distilled water to the 20 cm, mark.
- Stopper the vessel and shake for 2 min.
- Note the exact time that shaking was stopped and leave undisturbed for 4 h.
- A 10-ml aliquot is slowly drawn after 4 h into the pipette bulb with aspirator suction. (A reasonable sampling time would be 20 s).
- Drain the sample rapidly into a weighing vessel and rinse the pipette into the vessel with a little distilled water. Dry and weigh the sample.

- Dilute 9 ml of dispersing reagent to 550 ml and add a similar 10 ml removed and dried and weighed, this is the blank

% clay = (wt of clay - blank) [5500/wt of dry soil (g)]

NB: The depth of sampling in our tests was corrected according to suspension temperature:

Temperature °C	Depth (cm)
16	4.7
17	4.8
18	4.9
19	5.0
20	5.2
21	5.3
22	5.4
23	5.5
24	5.7
25	5.8
26	5.9
27	6.1
28	6.2

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