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## NOTE ON THE HYDROMETER METHOD OF PARTICLE-SIZE ANALYSIS

D. F. Grigal<sup>1/</sup>

Particle-size distribution is one of the most stable properties of a soil (Day 1965) and is closely related to both water and nutrient retention characteristics. The classification of soils in the United States is also partially based on particle-size distribution. Thus this distribution is widely used to characterize soils in both forestry and other ecological studies.

The most accurate method of particle-size analysis is considered to be the pipette method, a sedimentation procedure by which samples are removed by pipette at controlled depths and times (Day 1965; Soil Conservation Service 1967). However, the hydrometer method, a relatively simple and rapid sedimentation procedure, is sufficiently accurate to be widely used in research. This method, developed by Bouyoucos in 1927, was most recently modified by him in 1962 (Bouyoucos 1962).

The Bouyoucos method calls for two hydrometer readings of a settling soil suspension to be taken; one at 40 seconds after shaking for determination of total sand (particles 2.0 to 0.05mm), and one at two hours after shaking for determination of total clay (particles less than 0.002mm) (Bouyoucos 1962). Since Bouyoucos first developed the method, other workers have put it on a firmer theoretical basis (see Day 1965). This work has led to modifications of the procedure which provide greater accuracy (Day 1965). Unfortunately, though some modifications are apparently being used routinely (e.g., Miller 1969), much work continues to be reported using the standard Bouyoucos method. This may arise in part because many non-soil scientists' only introduction to particle-size analysis is as a student. Most laboratory exercises, probably because of time constraints, continue to use the Bouyoucos method (e.g., Jacobs *et al.* 1971).

Although the hydrometer method described by Day (1965) is more accurate than the Bouyoucos method, it requires development of a summation curve of particle-size distribution and interpolation off the curve for particular size classes. This method does not lend itself well to large numbers of routine analyses where concern is usually centered on the proportions of sand (2.0 to 0.05mm), silt (0.05 to 0.002mm), and clay (less than 0.002mm). In our laboratory we have attempted to draw a compromise between rapidity and accuracy.

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<sup>1/</sup>Assistant Professor, Department of Soil Science and College of Forestry, University of Minnesota.

## METHODS

We selected soil samples from the University of Minnesota Mineralogical Soil Survey Investigations Laboratory. Particle-size distribution of all samples had been determined using the standard pipette method. Our analytical procedure, detailed below, is only slightly modified from Day (1965). After samples had been dispersed and transferred to one liter settling jars, hydrometer readings were taken periodically according to the design of the experiment. Initially, analyses were carried out on 20 sub-surface soil samples, 10 surface samples (A1 horizons) whose organic matter had been digested with  $H_2O_2$  and 10 undigested surface samples. Hydrometer readings of the suspensions were taken at 2, 4, 6, 8 and 24 hours after shaking for all samples. Preliminary results led to an additional run of 10 sub-surface and five digested surface samples with readings taken at two and eight hours, and five undigested surface samples read at 2, 4 and 8 hours. All sedimentation temperatures were between 23° and 25°C. A settling jar with only water and dispersant was maintained at the same temperature as the soil suspensions to serve as a blank for adjustment of the hydrometer readings.

## RESULTS

The results of analyses of the sub-surface and the digested surface samples were similar and so they were combined in the statistical analysis. A paired t-test, comparing the percent clay determined by the pipette method and the percent of material in suspension at 2, 4, 6, 8 and 24 hours by the hydrometer method, was run on the initial group of 20 sub-surface and 10 digested surface samples. The t values for the 2, 4, 6, 8 and 24 hour readings were, respectively, 4.33\*\*, 2.05\*, 1.90\*, 1.51† and 4.05\*\* (N=30). Inclusion of the additional sub-surface and digested surface samples yielded t values of 5.36\*\* (2 hours) and 0.61† (8 hours) (N=45). These samples ranged from 0.4 to 77.5% clay (13 from 0 to 10%; 13 from 10 to 20%; 9 from 20 to 30%; 5 from 30 to 40%; and 5 greater than 40%). The eight-hour reading in both cases was not significantly different than the percent clay determined by the pipette method. This relationship can also be demonstrated by the correlation

$$c = 0.32 + 0.97 (e) \quad (r^2 = 0.98) \quad (1)$$

where c is the percent clay by the pipette method and e is the percent of material in suspension at eight hours. This compares with

$$c = 0.04 + 0.80 (t) \quad (r^2 = 0.93) \quad (2)$$

where c is as above and t is the percent of material in suspension at two hours. Both the slope of equation (1), which is nearly unity, and its high correlation coefficient demonstrate the superiority of the eight-hour to the two-hour reading in determining percent clay. As Bouyoucos (1951) states, in soils without sharp transitions between size classes, differences between the results of the hydrometer and pipette methods are more likely to occur. This appears to be especially true in the case of soils high in clay which may have a wide range of clay sizes.

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\*\* significant at 0.01 level

\* significant at 0.05 level

† nonsignificant

The undigested surface samples (total C range from 0.5 to 4.6% and clay from 3.2 to 63.4%) settled more rapidly. In this case, the paired t-test comparing material in suspension at 2, 4 and 8 hours with pipette-determined clay yielded t values of 4.45\*\*, 1.34† and 2.62\* (N=15), respectively. In the presence of organic matter, dispersion apparently is not as complete and clay-size particles settle more rapidly.

## DISCUSSION

These data indicate that an eight-hour hydrometer reading, as described below, is probably most accurate for routine use in determining percent clay by the hydrometer method. Samples of A1 horizons can either be digested with  $H_2O_2$  and a reading taken at eight hours, or can be read at four hours without digestion. Percent sand in our samples is usually determined by wet sieving. We have found, however, that the 40-second sand reading (Bouyoucos 1962), if carefully taken, does approximate the sieved sand. The use of an eight-hour hydrometer reading for routine laboratory work under an eight-hour day is possible by preparing samples on alternate days. Settling jars can then be shaken in the morning and the readings taken in the afternoon.

Two notes of caution should be made concerning these procedures. First, it is important that temperature changes within the cylinders during sedimentation be minimized (ideally with a sedimentation cabinet or constant temperature room). Temperature changes affect the rate of sedimentation, and in addition large changes in temperature may cause convection currents within the cylinders, invalidating the results of the analysis. Second, the procedure for surface samples (A1 horizons) is valid for mineral soils, not for organic soils. Soils such as those at the surface of swampy areas or in bogs are too high in organic matter for the procedure to work well. Samples with a sizable mineral component (about 75% or more) should be digested with  $H_2O_2$  at about 80°C and the eight-hour hydrometer reading used to determine the percent clay. Analyses of particle-size distribution of samples with low mineral content are not meaningful.

## PROCEDURE

### Equipment

Standard hydrometer, ASTM No. 152H with Bouyoucos scale in grams per liter.

Electric soil dispersion mixer with cups.

Settling jars, with 1000 ml calibration mark approximately 36cm from the bottom.

Sieve, 0.047mm (300 mesh), 5 or 8 inch frame.

### Reagent

Calgon solution (50 g/l)

### Procedure

1. Weigh 40.0 g of soil for analysis (55.0 or 60.0 g of coarse-textured soil may be desirable). The moisture content of an additional sample can be determined to make the appropriate oven-dry weight correction.
2. Add sample, 100 ml Calgon solution, and about 200 ml of distilled water to beaker or mixing cup and let stand for at least ten minutes. Longer times, even overnight, are better.

3. Transfer sample to mixing cup and fill cup to approximately two-thirds with distilled water.
4. Mix for exactly five minutes on mixer.
5. Transfer sample into settling jar and bring to 1000 ml volume with distilled water.
6. Prepare a calibration standard at same time with only Calgon solution and water in a settling jar.
7. Eight hours before readings are to be taken, place hand or large stopper over mouth of settling jar and mix thoroughly by rotating cylinder 180° a number of times. Minimize temperature variations during the eight-hour settling period.
8. Readings are taken eight hours after mixing. Lower the hydrometer into suspension carefully and read at upper edge of meniscus. At same time, determine appropriate hydrometer correction by suspending hydrometer in calibration standard.
- 9A. Mix the contents of the cylinder again and wash the soil suspension through the 300-mesh sieve with ordinary tap water. Agitate the sieve with continuous washing of clean water until fines appear to be washed out. Dry sand and weigh.
- 9B. Alternatively, a hydrometer reading of the suspension at 40 seconds after mixing may be taken to determine percent sand.
10. Compute percent sand as dry weight of sand divided by initial sample weight. Percent clay is corrected eight-hour hydrometer reading (corrected by calibration reading) divided by original sample weight. Percent silt by difference.

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