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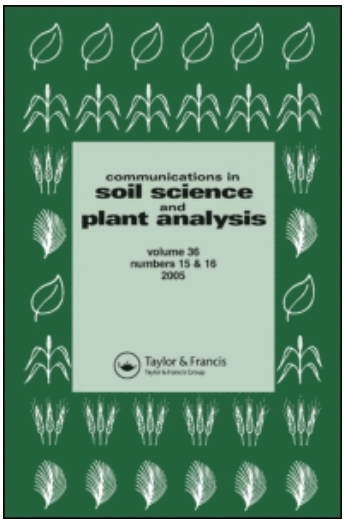

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STANDARD PROCEDURE IN THE HYDROMETER METHOD FOR PARTICLE SIZE ANALYSIS

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STANDARD PROCEDURE IN THE HYDROMETER METHOD FOR PARTICLE SIZE ANALYSIS

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ABSTRACT

In a widely-used method for particle size analysis of soils, the weight percentages of sand, silt, and clay are calculated from the density of an aqueous soil suspension measured by hydrometer. There are many versions of the procedure, differing in the type of dispersing solution, the volume of the suspension, the time of settling before taking hydrometer readings, or in the method of correcting the raw readings. Our procedure avoids errors inherent in some versions of the method, which can cause discrepancies from expected values. The details of our procedure should interest those concerned with minimizing confidence limits in inter-laboratory surveys and with providing reliable particle-size distribution data to laboratory clients.

INTRODUCTION

A knowledge of particle size distribution is useful for characterizing soils in terms of their suitability for a range of agricultural, engineering, landscaping or reclamation purposes.

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The hydrometer method for particle size analysis of soils (1) is still in widespread use. It requires inexpensive equipment and only basic technical expertise. One version of the procedure can be used to construct soil particle size summation curves (2, 3, 4,), but we are concerned here with the original Bouyoucos (1) method for assessing weight percentages of the sand, silt and clay fractions.

In this procedure a density reading made 40 s after agitating a soil (<2 mm) suspension in a measuring cylinder is used for calculating % sand (defined as particles >50 μ), and a later (2 h) reading is used to calculate % clay (<2 μ). These particle size thresholds conform to the classification systems of both the United States Department of Agriculture and the Canadian Society of Soil Science. The % silt is the balance required for 100% of the sample weight.

Procedural details have altered over the many years during which this method has been in general use, possibly accounting for variations in results of inter-laboratory studies of particle size analysis using replicate samples [e.g., USU (5)]. There are many factors which can affect the results obtained.

Volume of Suspension

Bouyoucos (1, 6, 7) specified that, with the hydrometer inserted, a suspension with 50 g of soil was to be made up to a final level corresponding to 1,130 mL. Manufactured glass cylinders are available with this mark inscribed. The hydrometer (ASTM type 152H) displaces approximately 60 mL and so the volume of suspension is approximately 1,070 mL. However, the hydrometer reading increases as the volume is reduced, and other versions of the procedure specify a suspension volume of 1,000 mL [e.g., Sheldrick and Wang (8)].

Adjustments to Raw Hydrometer Readings

This step alters the estimated density by several grams L^{-1}. If it is not done correctly, large discrepancies in the calculated particle size distribution can be caused. Ways of adjusting for the density of a blank dispersing solution, and for laboratory temperature, are discussed below.

Time of Hydrometer Readings

The validity of Bouyoucos's (7) procedure was questioned by Gee and Bauder (9) on the grounds that the settling times which he had empirically adopted are inconsistent with the requirements of sedimentation theory. They found that % sand values calculated from a 40 s hydrometer reading often differed from

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weights retained on a 53 μ sieve, by more than 5% of the sample weight. They showed (Gee and Bauder, 10) that a reading made after 2 h leads to an estimate of % clay higher the theoretical amount (2). These issues are also discussed below.

Cylinder Dimensions

Some documents (2, 4, 8) describe a measuring cylinder with the 1,000 mL mark near to 36 cm above the floor of the cylinder. Bouyoucos-type cylinders (with the engraved line corresponding to 1,130 mL) have an internal diameter near 62 mm, so that 1,000 mL reaches 33 cm above the floor, as is the case with the 1 L plastic graduated cylinders that we use. The effects of cylinder dimensions, which are minor, are discussed below.

Dispersing Agent

Heavy clay soils and soils contaminated with sodium salts may not disperse completely if insufficient dispersing chemical is used. In terms of efficacy, Bouyoucos (7) said there is little to choose between sodium pyrophosphate and metaphosphate. The final concentration of reagent determines the density and viscosity of the solution.

In view of all the above possible sources of discrepancy, there is a clear need for a unified, validated procedure.

MATERIALS AND METHODS

In our procedure, a stock dispersing solution is prepared weekly from tetrasodium pyrophosphate decahydrate ($Na_4P_2O_7 \cdot 10H_2O$, 500 g) made up to 10 L in deionized water. Soils are dried in a current of warm air (45°C) and pulverized to pass a 2-mm sieve. A subsample (50 g) is treated in a 1 L plastic cup with 100 mL of the stock solution. The mixture is made up to about 250 mL and left overnight (16 h). It is then transferred to a metal cup with indentations (milk-shake cup), mixed with a high speed blender for 5 minutes and rinsed into a measuring cylinder. The suspension is made up exactly to the 1,000 mL mark with deionized water from a large stock at laboratory temperature. The temperature of the suspensions is not controlled, but it never drifts more than 2°C at most in the course of the day.

Homogenizing the suspension by repeatedly inverting the capped cylinder (4, 7) may be impractical when many samples are processed daily. An alternative is to rake the suspension from top to bottom with a plunger (2). In cursory tests, we found that this method was as effective as end-over-end mixing. After mixing

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by either method, a standard ASTM type 152H hydrometer (9) is carefully inserted 20 s ahead of the reading time. The stem scale is read at the top of the meniscus. The stem of the hydrometer is kept free of grease so that a meniscus forms properly. We estimate the 40 s reading to the nearest 0.5 g L^{-1} and the later, far more stable reading (6 h after mixing) to the nearest 0.1 g L^{-1} under good light with the aid of a magnifying glass.

The above procedure was verified using a batch of 22 soils provided by the University of Alberta. These surface and sub-surface materials, taken from various locations in Alberta, varied widely in particle size distribution, organic matter (2 to 10%) and lime content (0 to 25%).

Verifying the 40 s Sand Settling Time

Subsamples (50 g) of dried soil (<2 mm) were prepared and analyzed by the procedure given above. Replicate subsamples (50 g) were also wet-sieved through 53 μ (270 mesh) and the dry weight of retained sand was measured, thus obtaining also the weight of silt and clay passing the sieve.

Verifying the Hydrometer Reading

Suspensions of fine silt and clay were obtained by allowing suspensions of whole soil, prepared as described, to settle for about 0.2 h in order to separate all sand and coarser silt. The upper portion, about 600 mL, was then removed by siphoning. It was made up again to 1 L with 0.5% sodium pyrophosphate decahydrate solution. The suspension was raked with the plunger and the hydrometer inserted. Readings were stable for several minutes and were estimated to the nearest 0.1 g L^{-1}. These suspensions were finally transferred to a pre-weighed pan and dried to constant weight in a current of air at 45°C, in order to obtain the weight of suspended solids. A correction was made to these weights for the dry weight of $Na_4P_2O_7$ (mean 3.1 g) in a 1 L blank suspension.

Temperature-Dependence of Hydrometer Readings

The above density measurements were made in the range 22–24°C, as indicated by a mercury in glass thermometer calibrated against an NBS standard. Before being dried, the suspensions of fine silt and clay were left overnight in a separate room maintained near 15.0°C. Each was then thoroughly agitated and homogenized using the plunger; the hydrometer readings were repeated, and the actual suspension temperature measured.

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RESULTS AND DISCUSSION

Correcting the Raw Hydrometer Reading

Two approaches have previously been used in correcting the raw hydrometer reading (R). One way is to subtract an actual blank reading (R_L) made in the dispersing solution (with no soil) at the same temperature as that of the soil suspensions (8, 9). The other way is to subtract a fixed, standard blank reading, whose value corresponds to a standard temperature, and then to make a correction for the difference between that standard and the actual temperature (7).

Using the first approach, we found a direct linear relationship (with $r^2=0.998$) between the weights of fine silt and clay in 1000 mL suspensions, and blank-adjusted hydrometer readings taken at 22–24°C soon after homogenizing them (Figure 1). This finding supports the view that blank-adjusted readings indicate with sufficient accuracy the density D (in g L^{-1}) of suspended solids (8, 9), according to the equation:

$$D = R - R_L \quad [1]$$

Judging from hydrometer readings made in the 1000 mL suspensions and in dispersing solution, at various temperatures in the range 13–24°C, the average value of dR/dT (approximately 0.3 g L^{-1} °C^{-1}) scarcely differs from dR_L/dT (approximately 0.4 g L^{-1} °C^{-1}). Therefore, dD/dT is very small. Having subtracted an actual blank reading R_L, further correction, to a standard temperature within several °C of the actual laboratory temperature, would have little effect on D.

The alternative of correcting to a standard 20°C and subtracting a fixed blank reading (7) performs the same function as subtracting R_L. It avoids the need for making an actual R_L, with its associated blank reading error. However, it unfortunately also introduces the risk (11) of inadvertently subtracting an actual R_L which, depending on the laboratory temperature, can differ by several g L^{-1} from the true standard blank reading appropriate to 20°C.

We therefore favor using equation [1]. The practice of subtracting an actual R_L from R also offsets any discrepancies among different hydrometers in the positioning of the scale in the stem during manufacture.

Time of Clay Reading

Equation [1] can be used to estimate the proportion of any particle size fraction, if the hydrometer is read after the appropriate settling time. In Gee and Bauder's (9) equations [13] and [14] [based on the theory of Day (2)], setting the difference between particle and solution densities at 1.6 g mL^{-1}, and using 0.95×10^{-3} kg m^{-1} s^{-1} as the solution viscosity at 22–24°C (by interpolation

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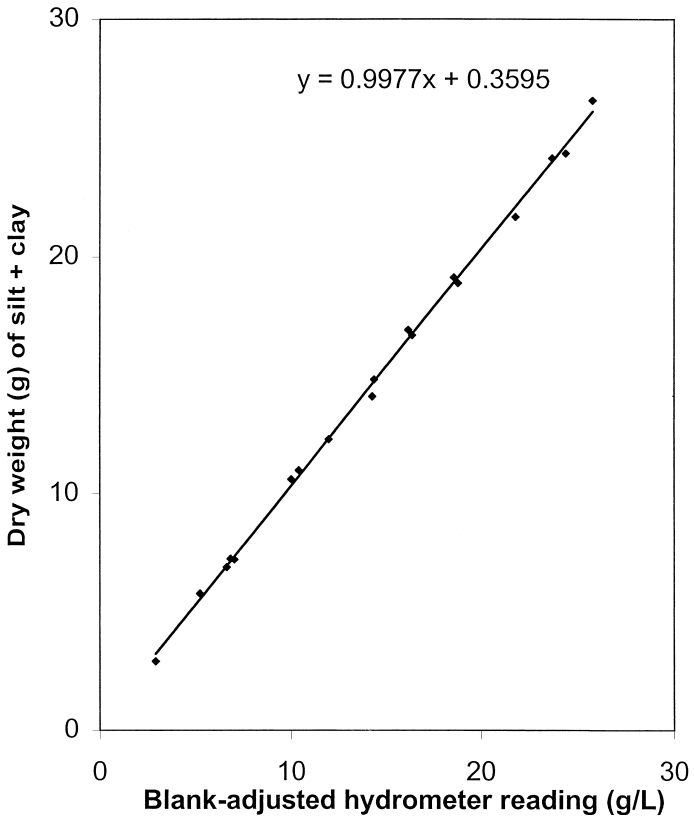

Figure 1. Weight of suspended solids versus blank-adjusted reading.

from their Table 15-1), a 6 h settling time in the cylinders that we use corresponds to an equivalent particle size diameter of 2.0±0.1 μ, for hydrometer readings in the range 10–30 g L^{-1}.

Using a 6 h reading (R_{6h}) therefore removes the high bias in % clay associated with a 2 h reading, noted by Gee and Bauder (10), while still allowing preparation and readings to be done in a regular working day by the same technician.

Cylinder Dimensions

The effective hydrometer settling depth is insensitive to the distance from the surface to the floor of the cylinder, and is scarcely affected by small changes to the cross-sectional area of the cylinder [using equation [10] of Gee and Bauder (9)].

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Sand Reading

We also found a linear relationship for the batch of 22 soils (Figure 2) between weights of silt and clay particles passing 53 μ, and values calculated using equation [1] from blank-adjusted 40 s hydrometer readings (R_{40s}) in 1000 mL suspensions at 23°C. The near 1:1 relationship obtained (with $r^2=0.989$) supports the empirical choice of 40 s for the sand reading time (1).

Calculations

The following equations are therefore considered suitable:

$$\% \text{ clay} = (100/w)\ (R_{6h} - R_L) \quad [2]$$

in which w is the weight of dry soil in 1000 mL of suspension;

$$\% \text{ sand} = 100 - (100/w)\ (R_{40s} - R_L) \quad [3]$$

and

$$\% \text{ silt} = 100 - \% \text{ sand} - \% \text{ clay} \quad [4]$$

The blank readings R_L in equations [2] and [3] are actual readings, which will not have the same value if the suspension temperature drifts.

Calcium Carbonate and Organic Matter

Many workers [e.g., Sheldrick and Wang (8)] have pointed out that lime and organic matter must first be removed, if a true assessment of size fractions of mineral particles is required. Even though these components had not been taken out of the soils we tested, there were no seriously outlying points in Figures 1 and 2. Suspended lime and organic matter fractions seem in effect to have conformed to an equivalent size distribution of mineral particles. Bouyoucos (6, 7) suggested that particle size distribution may for practical purposes be characterized by analysis done on whole soil and that, if desired, lime and organic matter can be quantified separately.

Hydrometer Consistency

The possibility that individual standard ASTM type 152H hydrometers might vary significantly in their density readings R is not explicitly covered in the literature cited below, and needed to be considered. To check variability, we tested

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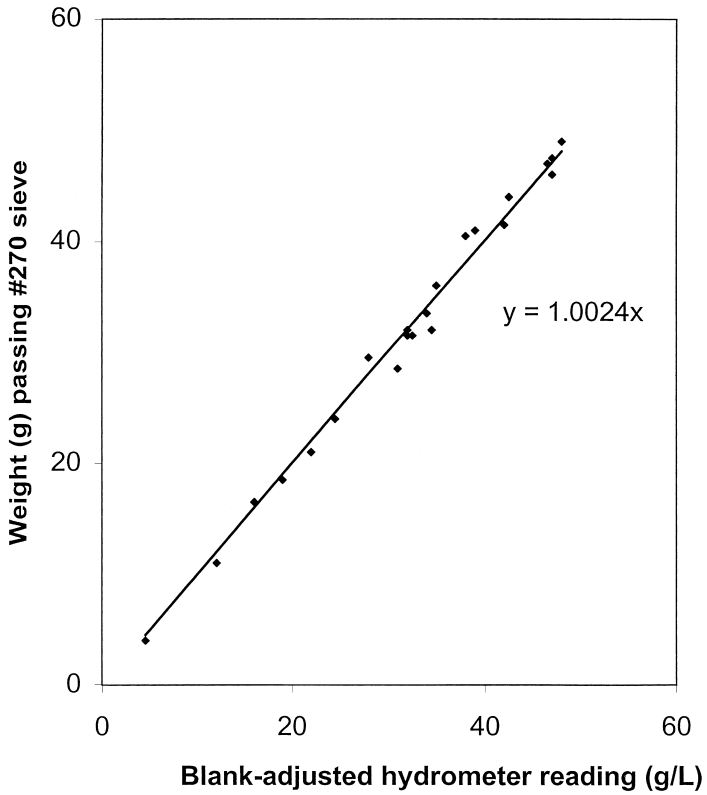

Figure 2. Sieve analysis versus blank-adjusted readings (40 s).

salt solutions of known density, using ASTM type 152H hydrometers already in use in our laboratory and new ones purchased from different suppliers. The results indicated that blank-adjusted readings made with hydrometers of this type generally agree within ± 1 g L^{-1}. Equations [1] through [4] can therefore be expected to apply for all standard hydrometers of this type.

Sample Preparation

Sample preparation procedures could be a further source of inter-laboratory discrepancy. Ball mills or other grinders which could break sand particles into smaller fragments should be avoided. The risk of damaging soil particles is considered low with the flail-type pulverizing equipment we use, though we have not investigated this possibility. The advantage of analyzing subsamples of dried, pul-

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verized material lies in avoiding both the difficulty of preparing representative subsamples of moist, as-received soils and the need for a separate moisture content determination on another such subsample (2, 8).

CONCLUSIONS

For the soils we studied, a blank-corrected hydrometer reading taken 40 s after mixing a suspension of 50 g soil in exactly 1 L of solution gave an accurate estimate of the weight percentage of material passing a 53 μ sieve, and hence of sand content.

The clay content was calculated from a blank-adjusted 6 h reading, thus eliminating the high bias in results obtained with a 2 h reading (10), while still allowing the sand and the clay readings both to be made by one technician conveniently in the course of a working day. To guard against variability among hydrometers and in readings made by different technicians, it is advisable for one technician to measure R and R_L, using the same hydrometer.

Over a wide range in the temperature of measurement, blank-adjusted readings were in good agreement with dry weights of suspended solids. Having subtracted an actual blank reading from suspension densities measured at nearly the same temperature, an additional temperature-standardization of the adjusted reading is not required.

The validity of equations [2] and [3] is unlikely to be affected by small differences in design of measuring cylinders, or in the amount or type of dispersing chemical used. However, errors in estimates of particle fractions of the order of 5–10% of sample weight can be caused by using suspension volumes other than 1,000 mL, by using inappropriate reading times, or applying a temperature-correction to the hydrometer reading in addition to an actual blank adjustment.

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