STANDARD OPERATING PROCEDURE

Procedure for Determining Soil Particle Size Using the Hydrometer Method

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KEY WORDS

Particle-size analysis, soil texture, hydrometer, sodium hexametaphosphate, Stoke's law, sedimentation time

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1.0 PURPOSE

This document provides the procedures that are used by staff of the Department of Pesticide Regulation (DPR) of the California Environmental Protection Agency for the analysis of particle size in soil samples.

2.0 SUMMARY OF THE METHOD

Particle size analysis (PSA) refers to the determination of the size distribution of individual particles in a soil sample. There are various systems of size classification that arbitrarily adopt different limits and ranges of soil-particle size. PSA is used to define soil texture, which is identified by the combination of the three soil fractions sand, silt, and clay that make up particle size distribution. The U.S. Department of Agriculture classification system identifies different soil textural classes based on the percentage by weight of the soil fractions < 2 mm, i.e., sand (2.0–0.05 mm), silt (0.05–0.002 mm), and clay (0.002 mm) (Gee and Bauder, 1986).

PSA performed using the hydrometer method is based on the Stoke's law, which expresses the velocity of sedimentation of a particle in a continuous viscous fluid, such as an aqueous solution, as a function of particle diameter, particle and suspension densities, and suspension viscosity. Temperature is also an important variable to be accounted for because of its effect on suspension density and viscosity (Gee and Bauder, 1986).

An American Society for Testing and Materials (ASTM)-certified hydrometer is used to nondestructively measure the concentration (grams of soil per liter of suspension) at different points in time of a soil suspension undergoing settling. By obtaining information about the suspension concentration at different points in time, it is possible to determine a soil's particle size distribution with minimum effort based on the Stoke's law. A series of equations based on the Stoke's law and adopted by the Soil Science Society of America has been implemented into the Excel file named "meth004templt.xls" (California EPA DPR, http://www.cdpr.ca.gov/docs/emon/pubs/sopmethdta.htm). Because the ASTM-certified hydrometers are calibrated at 20 ºC in terms of soil suspension concentration, it is necessary to correct for the effect of temperature on water characteristics by taking periodic readings in a blank suspension (BLK), i.e., without any soil.

Standard PSA methods require the soil particles be dispersed in an aqueous solution by both chemical and physical means. Chemical dispersion can be achieved using a number of dispersing chemicals, including Na-hexametaphosphate (HMP), which helps to maintain the soil particles in a dispersed state until sedimentation measurements are completed. Physical dispersion of particles can be achieved by shearing action or turbulent mixing, using mechanical shakers, electrical mixers, or ultrasonic probes.

3.0 APPLICABILITY

Pretreatment of samples is necessary at certain times to enhance the separation or dispersion of aggregates by eliminating binding agents of soil particles. These agents include organic matter, Fe oxides, carbonates, soluble salts, and gypsum. Different chemical pretreatment methods may be employed to remove these binding agents prior to dispersion as described in Gee and Or (2002, p. 257–263). The removal is performed when samples contain high amounts of soil organic matter (> 3.5%), iron oxides, and/or soluble salts.

4.0 SAFETY

All possible health hazards of each compound or reagent used in DPR standard operating procedures have not been precisely determined. However, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory maintains Material Safety Data Sheets (MSDS) which contain information regarding the safe handling of chemicals used at DPR facilities and their proper disposal. All personnel involved with these materials must read the reference file of MSDS available in the laboratory. All laboratory personnel should direct any questions regarding safety issues to their supervisors or the Safety Officer.

5.0 APPARATUS AND REAGENTS

- Standard hydrometer, ASTM no. 152 H, with scale 0–50 g L^{-1} (mass of soil/ volume of suspension)
- Electric stirrer (malted-milk-type) or horizontal shaker
- Half liter bottles with rubber stopper if using horizontal shaker
- Sedimentation cylinders with 1000-mL mark
- Rubber stoppers or plunger for 1000-mL sedimentation cylinders
- 600-mL beakers
- Amyl alcohol
- Na-HMP solution (50 g L^{-1})

- Set of sieves with the following openings: 2 mm and 53 µm
- Balance with a resolution of ± 0.01 g
- Stopwatch and digital clock
- One-liter or two-liter volumetric flasks
- Rubber-tipped pestle
- Wooden or rubber rolling pin
- ASTM Precision Thermometer

6.0 QUALITY CONTROL REQUIREMENTS

The quality control samples routinely used in this procedure include two sample duplicates (DUP) and a standard reference soil (SRS) per batch of 20 or less samples. In addition, the separation of sand fraction through a 53-µm (270-mesh) sieve is performed on three selected samples (SDUP) per batch of 20 or less samples. The number and type of QC samples may be modified to satisfy study objectives and sample availability.

7.0 SAMPLE PREPARATION

All soil samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. Subsampling of the primary samples is necessary to obtain the desired soil amount for the analysis. Homogenization of the primary samples or composited, multiple primary samples is performed in the field or in the laboratory, by placing the samples into a glass or stainless steel homogenization container, or plastic bags, and mixing thoroughly.

7.1 Homogenization and air-drying

7.1.1 Air dry samples collected in plastic bags until constant air-dry weight (i.e., approximately three or four weeks) prior to analysis start and store them away from possible contamination sources. While drying, leave the bag open to maximize exposure to air. During this phase, periodically seal the bag, mix, and homogenize the sample inside the bag, and reopen the bag to facilitate mixing and drying.

7.1.2 If samples are in containers, spread the bulk sample thinly (2- to 3-cm thick layers) on trays and let it air dry. Thoroughly mix and roll the sample with wooden or rubber rolling pin or rubber-tipped pestle to break up the main, large (> 2 mm) aggregates.

7.1.3 When samples have been air dried, transfer sample in a round bowl and stir the soil sample to break up the smaller soil aggregates (< 2 mm). Lightly crush the aggregates using a rubber-tipped pestle in a circular fashion, reversing direction and occasionally turning the sample over. (The use of a rubber-tipped pestle will prevent any grinding of the actual soil particles that make up soil structure.)

8.0 SEPARATION OF DIFFERENT SIZE FRACTIONS

- Use a 2-mm mesh sieve to separate the soil fraction > 2-mm, record weights of both < 2mm and > 2mm fractions to calculate their percentages.
- As needed, weigh a subsample (~10 g) of the < 2 mm soil fraction to determine the oven-dry weight and moisture correction factor following SOP# MET 001.00.

9.0 METHOD

9.1 Dispersion of soil

9.1.1 Preparation of soil samples will include a DUP and SRS sample with every batch of 20 or less soil samples.

9.1.2 For each sample, including the DUP and SRS, weigh approximately 50 g of air-dried soil into a 600-mL beaker and record the weight to a resolution of ± 0.01 g. (The amount of sample needed depends on soil texture. Ten grams to 20 g may be enough for fine clays but 60 g to 100 g may be needed for coarse sands.)

9.1.3 For each batch of 20 or less samples prepare one empty beaker (without soil sample) that will be used as the blank (BLK) sample.

9.1.4 For each sample, including the DUP, SRS, and BLK, transfer 250 mL deionized (DI) water and 100 mL of the HMP solution into the 600-mL beaker containing the sample, gently swirl the soil slurry for five seconds, and allow the sample to soak overnight.

9.1.5 Transfer quantitatively (i.e., without any loss of sample material) the soil-HMP slurry for each sample, including the HMP solution for the BLK sample, to a dispersing cup and mix for 5 min with an electric mixer. During the transfer, minimize the amount of DI water added to avoid splashing.

9.1.6 If using the horizontal shaker, transfer quantitatively the suspension to 500-mL shaker bottles with rubber stopper and shake overnight (for 15 hours) on a standard horizontal shaker table, using a 5-cm stroke and a 2-s cycle.

9.1.7 Transfer quantitatively the soil slurry for each sample into a sedimentation cylinder and add DI water at room temperature to adjust volume to 1 L.

9.1.8 Add the BLK to a cylinder and adjust the volume to 1 L with room-temperature DI water. Insert the thermometer into the BLK cylinder and attach it at the top of the cylinder using its rubber fitting.

9.2 Readings for samples and blanks

9.2.1 Once the suspensions in the cylinders have reached ambient temperature, the series of hydrometer readings begins with three replicate readings at 30 s since completion of mixing. This requires the preparation, for prompt and swift use when necessary, of the amyl alcohol pipette/dispenser and the stop watch and/or clock. The time information to be recorded for each sample is the (a) start date and time (e.g., May 19 at 10:30 AM), (b) three readings at 30 s since start, (c) one reading at 60 s, (d) one reading at 90 min, and (e) one 24-hour reading since start (e.g., May 20 at 10:30 AM). Time information is also recorded when taking periodic hydrometer and temperature readings for the BLK.

9.2.2 Start by inserting the plunger into cylinder and mixing thoroughly. Use upward strokes to remove sediment from bottom and finish with five or six smooth strokes. (An alternative to plunging is to seal each cylinder using a rubber stopper, and invert the cylinder 15–20 times to achieve complete mixing—number of inversions should be always consistent.)

9.2.3 READING AT THIRTY SECONDS: As soon as mixing is complete swiftly perform the following sequence: (1) start the timer; (2) gently place hydrometer into the suspension at an appropriate depth of the water column; if the surface of suspension is too foamy to read the hydrometer, (3) add one or two drops of amyl alcohol (if not go directly to next step); and (4) take an accurate reading at exactly 30 s from start.

9.2.4 Repeat previous steps (9.2.2–9.2.3) two more times. At the end of the third reading, leave the hydrometer in the cylinder and take another reading at 60 s instead. Remove, rinse/clean, and dry hydrometer with Kimwipes®. From this step on, do not shake or move the cylinder until the 24-hour reading.

9.2.5 Repeat previous steps as described in 9.2.4 for remaining samples and also include PERIODIC BLK READINGS (See next step).

9.2.6 PERIODIC BLK READINGS: Take a hydrometer and temperature reading, and record the time of reading, for the BLK after the 60-s reading for the first sample, and periodically every other 5 samples in each batch of 20 samples or less, to monitor variations in room temperature and hydrometer corrections (Mixing of the BLK is unnecessary).

9.2.7 Immediately after completion of all 60-s readings, as well as the BLK hydrometer and temperature readings, for the entire batch of 20 or less samples, wait for the next set of readings at 90 min.

9.2.8 READING AT 90 MINUTES: Starting from the first sample of the batch and approximately 40–60 s before the 90 min reading time, reinsert hydrometer——time of hydrometer insertion should always be consistent—and take the 90 min reading.

9.2.9 Repeat previous step (9.2.8) for the remaining samples and also include PERIODIC BLK READINGS (See next step).

9.2.10 PERIODIC BLK READINGS: Take a hydrometer and temperature reading, as well as record time of reading, for the BLK after the 90 min reading for the first sample of the batch, and periodically every 5 samples in each batch.

9.2.11 READING AT 24 HOURS: Repeat previous steps (9.2.8–9.2.9) to take the final, 24-hr reading.

10.0 QUALITY ASSURANCE (QA) AND QUALITY CONTROL (QC) CALCULATIONS

The QA-QC parameters include the percentage differences between the original and duplicate sample relative to their average, percentage of sand, silt, and clay within the 95% confidence interval for the SRS, and the percentage differences between the sand fraction and SDUP relative to their average. The QA–QC samples are processed and analyzed with every batch.

11.0 COMMENTS

- It is important to perform any appropriate pretreatments when necessary. If samples are not pretreated when needed, there can be large measurement errors particularly for the clay and silt fractions.
- The major source of error is the hydrometer reading. An error of ±1 g L^{-1} hydrometer reading results in an error of about ± 2 % (w/w) for clay-sized particles (Gee and Bauder, 1979).
- The hydrometer method is generally in agreement with the pipette method. The latter is recommended for when more accurate and precise measurements of the silt and clay fractions are necessary. However, Coates and Hulse (1985) commented that with the pipette and hydrometer methods “it is likely that the operator is the greatest source of imprecision”.

12.0 REFERENCES

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13.0 APPENDIX: CALCULATIONS

The following sections list the equations implemented in the Calculations Template (SOP_meth004_calculations_template.xls) following Gee and Bauder (1986) and Gee and Or (2002), to determine particle diameter in suspension at each time of hydrometer or temperature readings. Equation numbers refer to the column heading numbers of the worksheet titled "Soil Texture Template" that is contained in meth004templt.xls.

13.1 Calculation of mean particle diameter *X*

It is calculated at each reading time t (i.e., 30 s, 60 s, 1.5 h, or 24 h) as

$$X = \theta t^{-1/2} \qquad \text{Eq. 1}$$

Where

X is the mean particle diameter in suspension (μm),

t the time (minutes), and

$$\theta = 1000\left(B\,h'\right)^{1/2} \qquad \text{Eq. 2}$$

Where

θ is the sedimentation parameter (μm $min^{1/2}$) and

h′ is the effective hydrometer depth (cm)

$$h' = -0.164\,R + 16.3 \qquad \text{Eq. 3}$$

Where

R is the uncorrected hydrometer reading (g L^{-1}) at time *t* (average for the 30 s or individual reading for all other times) and

B (Eq. 6) the correction for HMP solution (see section 13.3).

13.2 Calculation of soil concentration in suspension *C* and summation percentage *P*

In a suspension at any given time, *C* is calculated as

$$C = R - R_L \qquad \text{Eq. 4}$$

Where

C is the concentrations (g L^{-1}) of soil in suspension,

R the uncorrected hydrometer reading, and

R_L the BLK hydrometer reading.

The summation percentage is calculated as

$$P_x = \frac{C}{C_0} \times 100 \qquad \text{Eq. 5}$$

Where

P_x is the summation percentage at a specific mean particle diameter x (µm), i.e., the 50 µm- or the 2 µm-summation percentage, in suspension at time t;

C the concentration of soil in suspension (g L^{-1}) (Eq. 4); and

C_o the sample mass of soil in 1 L suspension.

13.3 Calculation of the correction for HMP solution B

In the equation to calculate the sedimentation parameter θ (Eq. 2), the term B is calculated as

$$B = \frac{30\eta}{g(\rho_s - \rho_l)} \qquad \text{Eq. 6}$$

Where

η, the fluid HMP solution viscosity (g $cm^{-1}s^{-1}$) at temperature T, is calculated as

$$\eta = \eta^{\circ}(1 + 4.25\ C_S) \qquad \text{Eq. 7}$$

Where

C_S = 0.05 g cm^{-3} is the concentration of HMP,

η° the water viscosity (g $cm^{-1}s^{-1}$) at temperature T,

g = 980 cm s^{-2} the gravitational constant,

ρ_s the soil particle density = 2.65 g cm^{-3}, and

ρ_l the HMP solution density (g cm^{-3}) calculated as

$$\rho_l = \rho^{\circ}(1 + 0.630\ C_S) \qquad \text{Eq. 8}$$

Where

ρ° is the water density (g cm^{-3}) at temperature T.

13.4 Simplified clay fraction determination

Using both R and R_L values at 1.5 h and 24 h only, determine the mean particle diameter X and summation percentage P for 1.5 and 24 h readings, respectively, as described in the previous sections 13.1 and 13.2.

Calculate percent clay, $P_{2\mu m}$, as

$$P_{2\mu m} = m \ln\left(\frac{2}{X_{24}}\right) + P_{24} \qquad \text{Eq. 9}$$

Where

X_{24} is the mean particle diameter in suspension at 24 h (Eq. 1),

P_{24} the summation percentage at 24h (Eq. 5),

$m = (P_{1.5} - P_{24}) / \ln (X_{1.5} / X_{24})$ corresponds to the slope of the summation percentage curve between X at 1.5 h and X at 24 h,

$X_{1.5}$ the mean particle diameter in suspension at 1.5 h (Eq. 1), and

$P_{1.5}$ the summation percentage at 1.5 h (Eq. 5).

13.5 Simplified sand fraction determination

Use both hydrometer readings, R and R_L, at 30 s and 60 s.

Determine the effective particle diameter X and summation percentage P for the 30 s and 60 s readings as described in previous sections 13.1 and 13.2, respectively.

Calculate $P_{50\mu m}$ using

$$P_{50\mu m} = m \ln\left(\frac{50}{X_{60}}\right) + P_{60} \qquad \text{Eq. 10}$$

Where

X_{60} is the mean particle diameter in suspension at 60 s (Eq. 1),

P_{60} the summation percentage at 60 s (Eq. 5),

$m = (P_{30} - P_{60}) / \ln (X_{30} / X_{60})$ the slope of the summation percentage curve between X at 30 s and X at 60 s,

X_{30} the mean particle diameter in suspension at 30 s (Eq. 1), and

P_{30} the summation percentage at 30 s (Eq. 5).

Subtract the computed $P_{50\mu m}$ value from 100 to obtain sand percentage.

Eq. 11

13.6 Simplified silt fraction determination

It is calculated as

$$\% \text{ silt} = 100 - (\% \text{ sand} + \% \text{ clay}) \qquad \text{Eq. 12}$$