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# METHODS OF SOIL ANALYSIS

## Part 1

## Physical and Mineralogical Methods

## Second Edition

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# 14

# Particle Density[1]

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## 14–1 INTRODUCTION

Particle density of soils refers to the density of the solid particles collectively. It is expressed as the ratio of the total mass of the solid particles to their total volume, excluding pore spaces between particles. Convenient units for particle density are megagrams per cubic meter (Mg $m^{-3}$), or the numerically equal grams per cubic centimeter (g $cm^3$).

Particle density is used in most mathematical expressions where volume or weight of a soil sample is being considered. Thus interrelationships of porosity, bulk density, air space, and rates of sedimentation of particles in fluids depend on particle density. Particle-size analyses that employ sedimentation rate, as well as calculations involving particle movement by wind and water, require information on particle density.

## 14–2 PRINCIPLES

Particle density of a soil sample is calculated from two measured quantities, namely, the mass and volume of the sample. The mass is determined by weighing; the volume, by calculation from the mass and density of water (or other fluid) displaced by the sample. The pycnometer and the submersion methods are based on the same principle. Both have long been in use. They are simple, direct, and accurate if done carefully.

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## 14–3 PYCNOMETER METHOD (ASTM, 1958, p. 80; U.S. Dep. Agric., 1954, p. 122)

### 14–3.1 Special Apparatus

A pycnometer (specific-gravity flask) is employed. A pycnometer is a glass flask fitted with a ground-glass stopper that is pierced lengthwise by a capillary opening. A thermometer is sometimes an integral part of the stopper, the glass-enclosed mercury reservoir being in contact with the fluid in the flask, with the stem extending above the ground joint. A 10-mL pycnometer has sufficient capacity.

A small volumetric flask (25, 50, or 100 mL) may be used in place of a pycnometer when the sample is large enough to compensate for the decrease in precision of measuring fluid volume.

### 14–3.2 Procedure

Weigh a clean, dry pycnometer in air. Add about 10 g of air-dry soil sieved through a 2-mm sieve. If a 100-mL volumetric flask is used, add 50 g of soil. Clean the outside and neck of the pycnometer of any soil that may have spilled during transfer. Weigh the pycnometer (including stopper) and its contents. Determine the water content of a duplicate soil sample by drying it at 105 °C.

Fill the pycnometer about one-half full with distilled water, washing into the flask any soil adhering to the inside of the neck. Remove entrapped air by gentle boiling of the water for several minutes, with frequent gentle agitation of the contents to prevent loss of soil by foaming.

Cool the pycnometer and its contents to room temperature, and then add enough boiled, cooled, distilled water at room temperature to fill the pycnometer. Insert the stopper and seat it carefully. Thoroughly dry and clean the outside of the flask with a dry cloth, using care to avoid drawing water out of the capillary. Weigh the pycnometer and its contents, and determine the temperature of the contents after they have cooled to room temperature.

Finally, remove the soil from the pycnometer and thoroughly wash it. Fill the pycnometer with boiled, cooled distilled water at the same temperature as before, insert the stopper, thoroughly dry the outside with a cloth, and weigh the pycnometer and contents, being careful that the temperature remains the same as before.

Calculate the particle density as follows:

$$\rho_p = \rho_w (W_s - W_a)/[(W_s - W_a) - (W_{sw} - W_w)] \qquad [1]$$

where

$\rho_w$ = density of water in grams per cubic centimeter at temperature observed,

$W_s$ = weight of pycnometer plus soil sample corrected to oven-dry water content,

$W_a$ = weight of pycnometer filled with air,

$W_{sw}$ = weight of pycnometer filled with soil and water, and

$W_w$ = weight of pycnometer filled with water at temperature observed.

## 14–4 SUBMERSION METHOD (Capek, 1933)

### 14–4.1 Special Apparatus

1. A laboratory balance with a thin wire attached to the weighing beam, to which a light frame can be suspended. The frame serves as a platform for placing a weighing dish so that both frame and dish can be immersed in a container of liquid during weighing (see also section 13–4.2.1).
2. Sample containers. Aluminum weighing dishes of about 5-cm diameter and 3-cm height are suitable.
3. A container for water or a nonpolar liquid such as xylene or toluene, into which the weighing dish and sample can be immersed. Surface diameter should be about three times that of the weighing dishes.

### 14–4.2 Procedure

Moisten about 25 g of soil to a plastic consistency and force it by hand through a 2-mm sieve to form spaghetti-like threads. Dry the soil in a tared weighing dish to 105 °C, cool it to room temperature in a desiccator with a drying agent, and weigh it.

Add water to the dish to cover the soil, place weighing dish and soil in a vacuum desiccator, and evacuate for about 10 min to eliminate entrapped air from between the threads. Transfer weighing dish and sample to the weighing frame attached with a wire to the balance. Submerge weighing dish, frame, and soil sample into container of water and carefully reweigh while they are suspended in the water. Remove and discard the sample, clean the weighing dish, and weigh it while it is submerged in water. Determine the temperature of the water, and from handbook tables, determine its density. Use the same technique when working with a nonpolar liquid instead of water.

When a series of samples is analyzed using the same organic liquid, it is convenient at this point to submerge and weigh a small piece of metal such as 30 to 50 g of brass in the same container of liquid. Constancy of its submerged weight after each soil sample weighing assures the analyst that the organic liquid is not contaminated and allows re-use of the same liquid.

Calculate particle density as follows:

$$\rho_p = \rho_l (\mathrm{W}_{sd} - W_d)/[(W_{sd} - W_d) - (W_{sdl} - W_{dl})] \qquad [2]$$

where

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$\rho_l$ = density of water or organic liquid used, g cm$^{-3}$,
$W_{sd}$ = oven-dried weight of soil with weighing dish,
$W_d$ = weight of weighing dish,
$W_{sdl}$ = weight of sample and dish submerged in liquid, and
$W_{dl}$ = weight of dish alone, submerged in liquid.

## 14–5 COMMENTS

The pycnometer method has the advantage of giving very precise densities if volumes and weights are carefully measured. The submersion method sacrifices some precision but offers ease of measurement, especially when measurements are made on a series of samples. It does not require a calibrated pycnometer, it avoids accurate drying and cleaning of containers during repeated measurements, and it is less laborious, since the care needed to obtain reproducible accuracy in filling the pycnometer or flask is unnecessary. These advantages of the submersion method are best realized when a nonpolar organic liquid is used. A disadvantage of the submersion method is that it cannot be used on sandy soils where coherence may be too small to allow one to make the spaghetti-like threads.

With the pycnometer or a flask, a weighing error of 1 mg on a 10-g soil sample gives an error in particle density of only 0.0003 g cm$^{-3}$. A weighing error of 10 mg on a 30-g sample gives a particle density error of 0.001 g cm$^{-3}$. Greater errors can result from lack of precision in the volume measurement. If $W_{sw}$ in Eq. [1] is based on a volume that exceeds the volumetric flask marking by 0.2 mL, and $W_w$ on a volume 0.2 mL deficient of the marking, the compounded particle density error is 0.05 g cm$^{-3}$ on a 40-g sample. The analyst should check the calibration marking on the flask as well as his or her ability to measure a reproducible volume, by making a number of preliminary weighings of water in the flask to be used for the analysis. The submersion method, if performed as described with 25 samples each between 20 and 30 g, gives a standard error of 0.005 g cm$^{-3}$ for homogenized material. If unmixed replicate samples are used from surface soils, standard error tends to be several times greater. In addition to weight and volume errors, one must assume some error due to nonrepresentative sampling in either method.

Particle density values for finely divided active soil obtained by weighing in water are greater than those obtained with nonpolar organic liquids. There appears to be little difference between organic liquids. Anderson and Mattson (1926) found the average specific gravity of the clay fractions of six soils to be greater in water than in toluene by 0.13, while Capek (1933), using xylol, benzol, petroleum ether, and benzene, found an increase averaging 0.001 for quartz and 0.01 to 0.1 or even more for loam and chernozem soils. Smith (1943) found that water gave higher values for five soils than xylene, tetralin, or dichloroethyl ether by 0.01 to 0.03; and Gradwell (1955) found that the value for the specific gravity increased as the content of minerals with expanding lattices increased, or as the presence of finely divided, amorphous minerals increased. As the internal surface of non-allophane minerals increased from 33 to 306 m$^2$ g$^{-1}$, the increase in specific gravity determined in water over that determined in toluene varied from 0.014 to 0.094. Allophane values were greater in water by 0.05 to 0.3.

Water density is known to be affected by surfaces of finely divided particles. Though interactions of nonpolar organic molecules with clay surfaces are incompletely understood, it seems evident that the more accurate particle densities of clays would be obtained by use of nonpolar inorganic liquids in a pycnometer. Nevertheless, as Gradwell (1955) pointed out, where finely divided amorphous minerals or minerals with expanding lattices are present, it may be undesirable to substitute other liquids for water in determinations of specific gravity if the measurements are to be applied in computing the volume of solids in a soil in contact with water. For many applications, however, densities inaccurate by 0.05 g cm$^{-3}$ will suffice. Whether to use water or organic liquids is thus largely a question of how the data are to be used.

An advantage of nonpolar organic liquids is that soil samples, especially those high in organic matter, are wetted more easily than they are with water. Boiling is unnecessary when the pycnometer or flask is used; gentle shaking or stirring lightly with a glass rod is sufficient. It is desirable, however, when using nonpolar liquids, to evacuate the half-filled container in a vacuum desiccator for 10 min to facilitate removal of air. Another advantage in using organic liquids, especially for organic soils and peat, is that the soil or organic particles sediment faster after stirring than they would in water. In the submersion method this is important in reducing buoyancy when one weighs the sample in a weighing dish submerged in the fluid. Disadvantages of using organic liquids are their high vapor pressure and their low heat capacities. Because of the former, work in a well-ventilated hood is necessary. Since the low heat capacity presents the hazard of thermal dilation, it is essential to use only tongs for handling the containers.

Both the pycnometer and the submersion methods give the weighted mean density of all particles in the sample. This is the value needed for calculations mentioned in the introduction. Densities of individual soil grains may vary widely from the weighted mean. For example, handbook densities of silt and sand-sized particles are 2.65 for quartz, 2.5 to 2.8 for feldspars, 2.7 to 3.3 for micas, and 3.1 to 3.3 for apatite. The density of humus is usually $< 1.5$ Mg m$^{-3}$.

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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 $\mu$m 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 $\mu$m), silts ($<$50–2 $\mu$m), 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

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[1] Prepared for the U.S. Department of Energy and the U.S. Nuclear Regulatory Commission under Contract DE-AC06-76RLO 1830.

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