STANDARD OPERATING PROCEDURE

Procedure for Determining Soil Particle Density Using Gay-Lussac Specific-Gravity Bottles

KEY WORDS
Particle density analysis, Pycnometer, Deggassing membrane, de-aired water,

APPROVALS

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1.0 INTRODUCTION

1.1 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 density in soil samples.

1.2 PRINCIPLE OF THE METHOD

Soil Particle Density Analysis (SPDA) refers to the determination of the average density of individual particles that comprises a soil sample. The principle of determining the particle density of a soil relies on two measured quantities, namely, the mass and volume of the sample (Blake and Hartge, 1986; Flint and Flint, 2002). Mass of the particle is determined by weighing, using a high precision analytical balance. The volume of the soil particles is determined using displacement of liquid by the soil particles. Based on this concept, the volume is calculated by the mass and density of water displaced by the particles. The method is a liquid-displacement method where a pycnometer (glass flask) is fitted with a glass stopper that contains a capillary opening at the center that allows excess water to flow out of the unit when the stopper is inserted (Blake and Hartge, 1986; Flint and Flint, 2002). This method is basically a compilation of a pycnometer methods based on Blake and Hartge (1986) and Flint and Flint (2002).

2.0 APPLICABILITY

Entrapment of any air between particles after filling the pycnometer with de-aired distilled water can be removed by boiling the water or applying a vacuum. In this procedure, the vacuum chamber will be used for removing entrapped air in the pycnometer. In the case of unavailability of distilled water, we de-air de-ionized (DI) water using a vacuum application. This method can be applied to gravel or rock fragments in addition to soil samples and can provide higher degree of accuracy (≥ three significant figure) in determining particle density if it is needed.

3.0 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 MSDS reference file available in the laboratory. All laboratory personnel should direct any questions regarding safety issues to their supervisors or the Safety Officer.

4.0 APPARATUS AND REAGENTS

4.1  100 mL ± 0.10 mL tolerance Gay-Lussac specific gravity bottles that meet American Society for Testing and Materials (ASTM) no. D 369 and D 854 standard (Photo 2)
4.2  De-aired distilled water. If distilled water is unavailable, we use de-aired de-ionized (DI) water
4.3  MINIMODULE® or PermSelect® Degassing Membrane contactor systems for de-airing distilled or de-ionized water
4.4  A vacuum pump with regulator
4.5  Universal 185 L/h water pump
4.6  A vacuum chamber
4.7  2 mm opening sieve
4.8  Balance with a resolution of ± 0.01 g
4.9  Small funnel for adding soil samples and de-aired water
4.10  ASTM precision thermometer

5.0 SOIL SAMPLE PREPARATION

5.1  Air dry soil samples collected in plastic bags until constant air-dry weight (i.e., approximately three or four weeks depending on the wetness of the sample). While drying, leave each bag open to maximize exposure to air and ensure they are stored away from possible contamination sources such as dust. During this phase, periodically seal each bag, mix and homogenize the sample inside the bag, and reopen the bag to facilitate mixing and drying.

5.2  If samples are in containers, spread each bulk sample thinly (2- to 3-cm thick layers) onto trays and let 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. When samples have been air dried, transfer each sample to a porcelain mortar and gently stir the soil sample with a pestle to break up the smaller soil aggregates (< 2 mm).
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5.3 Use a 2-mm sieve to sieve through crushed soil sample. Transfer the sieved soil material into a plastic bag or an air-tight container for analysis.

6.0 DE-AIRING DE-IONIZED WATER

6.1 Connect a vacuum pump and vacuum regulator to the vacuum outlet of the PermSelect® Degassing Membrane contactors as shown in APPENDIX I (Figure 1 or Figure 2). The set up will depend on the applied vacuum as explained in the captions of Figures 1 and 2. The set up in Figure 1 is more suitable for our purpose. A schematic of set up for MiniModule® membrane, which will be used as a backup degasser, is in Figure 3.

6.2 Connect a water pump to the water inlet or outlet of PermSelect® degassing membrane contactor. The flow rate of the water pump should be adjusted to approximately 5 gallons per hour (~ 19 L/h) using the water flow valve as shown in APPENDIX II (Photo 1).

6.3 Connect a water outlet of the degassing membrane to a water storage unit (2-3 liters) and to the degassing reservoir (1-2 liters). Start the water pump. The water will enter the center port of the PermSelect® membrane contactor, then flows on the outside of the hollow fibers (shell side) and exits at side ports (shown on top of the module). Ensure that the water carrying system is initially primed full of water to operate at an optimum level.

6.4 Using a vacuum regulator, set the vacuum pressure of the PermSelect Membrane contactor to the desired operational range (4-20 kPa or 0.6-3 psi). A vacuum pressure of 7 kPa is sufficient for the degassing of water using the MiniModule® and PermSelect® membranes. The vacuum is applied at the ports on the end caps, which creates a vacuum inside (lumen side) the hollow fibers. As liquid flows through the hollow fiber bundle within the unit, dissolved gases permeate the hollow fiber walls driven by the vacuum in the lumens. Extracted gases flow toward the vacuum pump and the degassed liquid exits the side ports of the degasser.

6.5 Degas the de-ionized water until the water storage unit collects 2-3 liters of degassed (de-aired) water. Then close the 2-way valve and turn off the water pump by unplugging it from the electrical outlet. Keep the storage unit closed to minimize exposure of degassed water to the air.

7.0 PARTICLE DENSITY DETERMINATION
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7.1 Weigh a clean, empty and dry pycnometer (100 ml Gay-Lussac specific gravity bottle) with its stopper \((W_a)\) (Photos 2 and 3). Using a funnel to prevent the sample spillage, add 50 g of air-dry soil sieved through a 2-mm sieve. Write down the measured parameters on a default sheet given in APPENDIX III Form 1 for record keeping.

7.2 Clean the outside and neck of the pycnometer of any soil that may have spilled during transfer.

7.3 Weigh the pycnometer including the stopper with the added air-dried soil sample \((W_s)\) (Photo 4) and record the results in Form 1.

7.4 Weigh another 10 g air-dried soil and determine the water content \((w)\) of that soil sample by drying it at 105 °C for 24 hours in the oven (Photo 5).

7.5 Fill the pycnometer that contains the soil sample about one-half full with de-aired distilled or de-ionized water, making sure to wash into the pycnometer any soil that adhered to the inside of the neck of bottle (Photo 6).

7.6 Place the half full pycnometer with the stopper sealing its contents on the electrical shaker at speed 4 for 10 minutes (Photo 7). The shaking helps to disperse soil particles in the water and allows air bubbles entrapped between soil particles to escape. Avoid faster shaking speeds to prevent the soil solution from spilling and adhering to the inside of the neck of bottle.

7.7 After 10 minutes of shaking, remove the stopper and transfer the pycnometer into a vacuum chamber and slowly apply a vacuum until the pressure gauge of the vacuum chamber reaches to -0.08 MPa (= -80 kPa) (Photos 8 and 9). This should help in removing entrapped air inside the pycnometer. Be careful not to let bubbles remove any soil from the bottle. Keep the pycnometer in the vacuum chamber for one hour, then slowly release the vacuum from the chamber.

7.8 Add enough de-aired water to fill the pycnometer, and insert the stopper slowly. Be sure to insert the correct stopper. The stopper and pycnometer have corresponding serial numbers etched on their surfaces.

7.9 Seating the stopper carefully causes the capillary tube pierced lengthwise to overflow with excess water.
7.10  Dry and clean the outside of the bottle. Care needs to be taken to avoid drawing water out of the capillary tube and to make sure that the capillary tube is full of water (Photo 10).

7.11  Weigh the pycnometer and its contents \((W_{gw})\) and record the result (Form 1). After weighing, remove the capillary stopper and determine the temperature \((T_{gw})\) of the water using a thermometer (Photo 11). Record the temperature in Form 1.

7.12  Remove the soil and water from the pycnometer and clean it thoroughly. After ensuring cleanness of the pycnometer, fill it with de-aired water at the same temperature.

7.13  Carefully insert the stopper again, observing overflow of excess water from the tip of capillary outlet.

7.14  Dry and clean the outside of the pycnometer, avoiding drawing water out of the capillary tube. Weigh the pycnometer filled with water \((W_w)\) and check the temperature of the water by removing the stopper (Photos 12 and 13). Record the results in Form 1.

8.0  CALCULATION OF PARTICLE DENSITY

8.1  The particle density \((g \text{ cm}^{-3})\) can be calculated as:

\[
\rho_p = \frac{\rho_w (W_s - W_a)}{(W_s - W_a) - (W_{sw} - W_w)}
\]

where

\(\rho_w\) = density of water at observed temperature \((g \text{ cm}^{-3})\)
\(W_s\) = weight of pycnometer plus soil sample corrected to oven-dry water content \((g)\)
\(W_a\) = weight of pycnometer filled with air \((g)\)
\(W_{sw}\) = weight of pycnometer filled with soil and water at observed temperature \((g)\)
\(W_w\) = weight of pycnometer filled with water at observed temperature \((g)\)
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Before calculating particle density, determine $W_s^*$ based on soil sample corrected to oven-dry water content. First, determine oven-dry water content ($w$) (g water g$^{-1}$ soil) from the duplicated soil sample as follows:

$$w = \frac{M_{airsoil} - M_{oven}}{M_{oven}} = \frac{M_w}{M_{oven}}$$

where

- $M_{airsoil} = \text{mass of air-dry soil (g)}$
- $M_{oven} = \text{mass of oven-dry soil (g)}$
- $M_w = \text{mass of water (g)}$

The weight of the pycnometer plus soil sample corrected to oven-dry water content, $W_s$, can be calculated with these measured variables as follows:

$$W_s = W_s^* - \left((W_s^* - W_d) \cdot w\right)$$

where

- $W_s^* = \text{weight of pycnometer including stopper with added air-dried soil sample (g)}$
- $W_d = \text{oven-dry water content (g g}^{-1})$

9.0 REFERENCES


APPENDIX I – The Set-up Scheme

APPENDIX II – Photos

APPENDIX III – Form for Recording Data
APPENDIX I

Figure 1. Liquid degassing set up using *PermSelect* membrane modules and degassers with the liquid on the shell side. (Figure taken from Degassing Liquids-MedArray, Inc.). The maximum recommended trans-membrane pressure (TMP) for shell side water flow is 15 psi (103 kPa).
Figure 2. Liquid degassing set up using *Perm*Select membrane modules and degassers with the liquid on the lumen side (Figure taken from Degassing Liquids-MedArray, Inc.). The maximum recommended trans-membrane pressure (TMP) for Lumen side water flow is 45 psi (310 kPa).
Figure 3. Liquid degassing set up using MiniModule® membrane degassers. The recommended vacuum mode should be within the range of 4-20 kPa.
APPENDIX II

Photo 1. *PermSelect®* degassing membrane setup with 7 kPa applied vacuum pressure for the shell side water flow. This setup is a similar to the setup depicted in Figure 1.
APPENDIX III

Form 1. Form for recording data during analysis.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Water Content Determination</th>
<th>Soil Particle Density Determination</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mass of air dry soil</td>
<td>Mass of oven dry soil</td>
</tr>
<tr>
<td>$M_{\text{Airsoil}}$</td>
<td>(g)</td>
<td>$M_{\text{Oven}}$</td>
</tr>
</tbody>
</table>