

METHOD OF DETERMINING DENSITY OF SOLIDS

1. Scope and Definition

1 This method covers procedures for determining the density of solids. The density of solids is the ratio of the mass in air of a given volume of crushed solids to the total volume of solids.

2. Apparatus

1 The apparatus shall consist of the following:

- (a) Volumetric flask, 500-mL capacity.
- (b) Vacuum pump or aspirator connected to vacuum line.
- (c) Oven of the forced draft type, automatically controlled to maintain a uniform temperature of 110 ± 5 °C throughout the oven.
- (d) Balance, sensitive and accurate to 0.01 g, capacity 500 g or more.
- (e) Thermometer, range 0 to 50 °C, graduated in 0.1 °C.
- (f) Evaporating dish.
- (g) Water dish.
- (h) Sieves, U.S. Standard 4.75-mm (No. 4) and 600- μ m (No. 30) conforming to ASTM Designation E 11, "Specifications for Wire-Cloth Sieves for Testing Purposes."
- (i) Sample splitter suitable for splitting material passing 4.75-mm (No. 4) and 600- μ m (No. 30) sieves.

3. Calibration of Volumetric Flask

1 The volumetric flask shall be calibrated for the mass of the flask and water at various temperatures. The flask and water are calibrated by direct determination of mass at the range of temperatures likely to be encountered in the laboratory. The calibration procedure is as follows.

2 Fill the flask with de-aired, distilled, and demineralized water to slightly below the calibration mark and place in a water bath which is at a temperature between 30 and 35 °C. Allow the flask to remain in the bath until the water in the flask reaches the temperature of the water bath. This may take several hours. Remove the flask from the water bath and adjust the water

level in the flask so that the bottom of meniscus is even with the calibration mark on the neck of the flask. Thoroughly dry the outside of the flask and remove any water adhering to the inside of the neck above the graduation, then determine the mass of the flask and water to the nearest 0.01 g. Immediately after determination of mass, shake the flask gently and determine the temperature of the water to the nearest 0.1 °C by immersing a thermometer to the middepth of the flask. Repeat the procedure outlined above at approximately the same temperature, than make two more determinations, one at room temperature and the other at approximately 5 °C less than room temperature. Draw a calibration curve showing the relation between temperature and corresponding values of mass of the flask plus water. Prepare a calibration curve for each flask used for density determination and maintain the curves as a permanent record. A typical calibration curve is shown in Fig. 1.

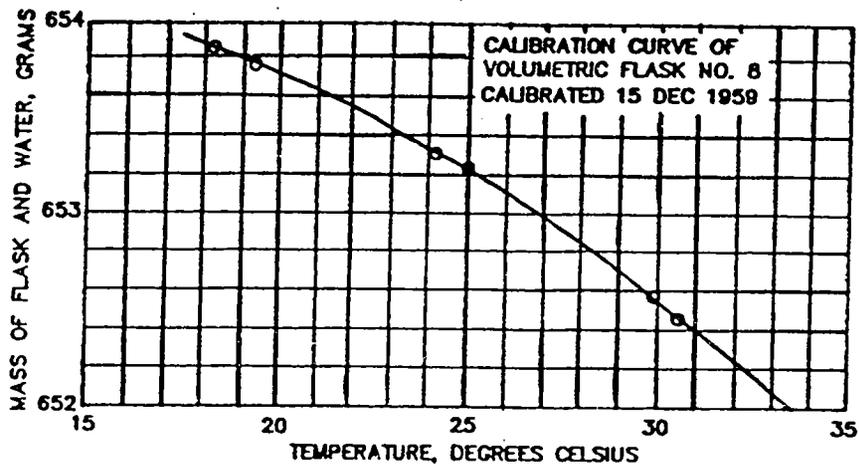


Fig. 1. Typical calibration curve of volumetric flask.

4. Sample

1 Crush the sample until it all passes a 4.75-mm (No. 4) sieve. With a sample splitter, separate out 120 to 150 g of representative crushed material. Pulverize this material to pass a 600- μ m (No. 30) sieve. Oven-dry the crushed material to constant mass, determine the mass of the material to the nearest 0.01 g, and record the mass.

5. Procedure

1 After determination of mass, transfer the crushed material to a volumetric flask, taking care not to lose any material during this operation. To reduce possible error due to loss of material of known mass, the sample may have its mass determined after transfer to the flask. Fill the flask approximately half full with de-aired, distilled water. Shake the mixture well and allow it to stand overnight.

2 Then connect the flask to the vacuum line and apply a vacuum of approximately 99.99 Pa (750 mm of mercury) for approximately 4 to 6 h, agitating the flask at intervals during the evacuation process. Again, allow the flask to stand overnight. Finally, fill the flask with de-aired, distilled water to about 3/4 in. (19 mm) below the 500-mL graduation and again apply a vacuum to the flask until the suspension is de-aired, slowly and carefully remove the stopper from the flask, and observe the lowering of the water surface in the neck. If the water surface is lowered less than 1/8 in. (3.2 mm), the suspension can be considered sufficiently de-aired. Fill the flask until the bottom of the meniscus is coincident with the calibration line of the neck of the flask. Thoroughly dry the outside of the flask and remove the moisture on the inside of the neck by wiping with a paper towel. Determine the mass of the flask and contents to the nearest 0.01 g. Immediately after determination of mass, stir the suspension to assure uniform temperature, and determine the temperature of the suspension to the nearest 0.1 °C by immersing a thermometer to the middepth of the flask. Record the mass and temperature.

3 Compute the density of the solid, P_s , from the following formula:

$$P_s = \frac{W_s \gamma_w}{W_s + W_{fw} - W_{fW_s}}$$

where

W_s = the oven-dry mass of the crushed rock sample, g
 γ_w = density of water at test temperature, g/cc

W_{fw} = mass of flask plus water at test temperature, g
(from calibration curve, Fig. 1)

W_{fws} = mass of flask plus water plus solids at test
temperature, g

6. Report

1 The report shall include the following:

- (a) The density of the solid.
- (b) Owendry mass of test sample.
- (c) Water temperature during test.