



A Honeywell Company

Piece Density by Mercury Displacement

UOP Method 766-13

Scope

This method is for determining the piece density (mass per unit volume) of particulate solids by mercury displacement. The method is applicable to catalyst supports and granular material ranging in size from 0.6 mm (30 mesh) to 5 mm (3 mesh) or powders in the range of 0.6 mm to 0.15 mm (100 mesh). Void space between particles is not included in the volume. Internal pores having diameters less than approximately 12 μm are included in the volume; larger pores are not. The piece density is reported as g/mL.

Reference

UOP Method 954, "Loss on Ignition (LOI) for Fresh, Regenerated, Used, and Spent Catalysts, Catalyst Supports, and Adsorbents," www.astm.org

Outline of Method

The particulate sample is placed in a tared and calibrated pycnometer. The pycnometer is weighed, evacuated, filled with mercury and weighed again. The piece density of the sample is calculated and reported on either an as-received or volatile-free basis.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Balance, readable to 0.001 g

Funnel, polypropylene, 65-mm top ID, 15-mm stem OD, Fisher Scientific, Cat. No. 10-348A

Piece density apparatus, constructed as shown in the attached Figures. The apparatus includes the following items and also requires additional items for the manifold that are commonly available.

Cap, glass, circular, pycnometer, Newport Scientific, Cat. No. 55005006800

Clamp, pycnometer filling device, Newport Scientific, Cat. No. P-1002015100

Flask, filtering, with sidearm, borosilicate glass, 500-mL, Fisher Scientific, Cat. No. 10-181E

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).
--

© COPYRIGHT 1991, 2013 UOP LLC. All rights reserved.

Nonconfidential UOP Methods are available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, USA. The UOP Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

Gauge, vacuum, 4 ½" NPT lower connected, Grainger, Cat. No. 1X583

Gauge, vacuum, digital, Grainger, Cat. No. 3KNP6

Holder, pycnometer, metal, Newport Scientific, Cat. No. 65024000400

Pycnometer, with top, solid, Newport Scientific, Cat. No. 5-7146

Pycnometer, with top, powder, Newport Scientific, Cat. No. 5-7147

Pycnometer filling apparatus. Assemble an apparatus similar to that shown in Figure 1 using valves and fittings locally sourced or from Swagelok.

Pycnometer filling device, Aminco, Newport Scientific, Cat. No. C2-65024

Pycnometer filling device top, with stopcock, Newport Scientific, Cat. No. 5-7164

Pump, vacuum, Welch Duo-Seal, Model 1400B01, Fisher Scientific, Cat. No. 01-096

Trap, vacuum, plain, 30-mm body diameter, 225-mm length, tubing connection 13-mm OD, Reliance Glass Division, Cat. No. R-7205-200

Screen, 0.15-mm (100-mesh) stainless steel, F.P. Smith Wire Cloth

Thermometer, -50 to +70°C, digital panel mounted, VWR, Cat. No. 82020-64

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Epoxy adhesive

Gloves, nitrile, disposable, VWR, Cat. No. 40101-344, -346, -348, -350 for sizes small, medium, large, and extra-large, respectively

Mercury, triple distilled, reagent grade, Bethlehem Apparatus

Mercury spill cleanup kit, VWR, Cat. No. JT4439-1

O-ring, pycnometer, Newport Scientific, Cat. No. T-1604018700

O-ring, pycnometer filling device, Newport Scientific, Cat. No. P-1604028200

Pipe cleaners, jumbo stem 12"x 6 mm, Runco Office Supply, Cat. No. CKC711001

Stopcock grease, high vacuum type, Cello Seal, Fisher Scientific, Cat. No. C602-100

Wipes, Kimwipes EX-L, laboratory wipers, lint free, Fisher Scientific, Cat. No. 06-666A

Preparation of Apparatus

The analyst is expected to be familiar with general laboratory practices and the equipment being used. Dispose of all reagents, materials, and samples in an environmentally safe manner according to local regulations.

Assemble the apparatus as shown in Figures 1 and 2 (see *Notes and Precautions 1*). Install the vacuum trap between the apparatus and the vacuum pump in order to prevent mercury from entering the vacuum pump.

System Vacuum Check

1. Add mercury to the pycnometer filling device until the level is about 12 mm below the bottom of the pycnometer stem when it is inserted into the filling device in the vertical position.
 - Nitrile gloves should be worn whenever there is a possibility of mercury contact.
2. Assemble the pycnometer by applying a thin coating of stopcock grease to the top edge of the pycnometer sample chamber. Place the circular glass cap on top of the chamber and then an O-ring. Tighten the metal retaining cap on the pycnometer sample chamber.
 - A vacuum tight seal with the glass cap is required, but an excessive application of grease may cause errors.
 - A Teflon washer and O-ring may be used in place of the glass cap, eliminating the possibility of chipping or breaking.
3. Insert the assembled pycnometer into the filling device.
 - The pycnometer is held in position with the pycnometer holder.
4. Attach the pycnometer filling device top using an O-ring seal and clamp. Close the atmospheric vent stopcock at the top.
5. Start the vacuum pump and begin taking readings with the vacuum gauge.
 - The apparatus should rapidly pump down to less than 0.0027 kPa (0.020 torr). If it does not, check for leaks and/or improper operation of the vacuum pump (see *Notes and Precautions 2*).

Filling Device Vacuum Check

1. Tilt the filling device so that the mercury level covers the bottom of the pycnometer stem, with the vacuum pump on and the vacuum below 0.0027 kPa (0.020 torr). Hold it in this position.
2. Isolate the filling device.
 - The valve is closed only after the pycnometer stem is immersed in the mercury to prevent the introduction of air into the system.
 - Do not shut off the vacuum pump before the valve is closed.
3. Consider the filling device section leak tight if the mercury does not rise in the pycnometer stem over a period of 10 minutes.

Solid Pycnometer Preparation

The powder pycnometers require no special preparation before use. Prepare each solid pycnometer assembly before initial use as follows:

1. Cover the stem opening to the pycnometer sample chamber with a previously prepared 6-mm diameter disk of 100-mesh screen (see Figure 2).
 - This prevents loss of sample into the stem.
2. Apply epoxy adhesive to this disk making sure the edges are completely sealed.
 - Take care not to plug the openings in the screen with the adhesive or the stem with adhesive.
3. Clean the pycnometer by washing with detergent after the epoxy is hard.

Procedure

Pycnometer Calibration

1. Assemble the pycnometer as described under *System Vacuum Check*, Step 2.
2. Weigh the assembled empty pycnometer to the nearest 0.001 g.

3. Insert the pycnometer into the filling device, attach the filling device top and clamp in place.
4. Evacuate the filling device to less than 0.0033 kPa (0.025 torr) of mercury for a minimum of one minute.
5. Tilt the filling device so that the mercury level covers the bottom of the pycnometer stem and hold it in this position.
 - Make sure the pressure is still below 0.0033 kPa (0.025 torr).
6. Close the valve to the vacuum.
7. Open the atmospheric vent stopcock at the top of the filling device very slowly, allowing mercury to be pressured up the pycnometer stem and into the sample chamber.
8. Allow the filling device to reach atmospheric pressure.
9. Remove the top portion of the filling device.
10. Lift out the pycnometer and invert the pycnometer so that no mercury will spill out.
11. Remove any mercury adhering to the outside of the pycnometer stem with the hose attached to the filtering flask.
12. Read the level of the mercury in the pycnometer stem to the nearest 0.001 mL.
 - This value becomes the calibration stem correction volume, S, in Equation 1.
13. Weigh the mercury-filled pycnometer to the nearest 0.001 g.
14. Record the temperature of the mercury (room temperature).
15. Determine the density of mercury from Table 1.

Table 1
Density of Mercury

<u>Temperature, °C</u>	<u>Density, g/mL</u>
13 through 16	13.56
17 through 20	13.55
21 through 24	13.54
25 through 28	13.53
29 through 32	13.52
33 through 36	13.51

16. Calculate the pycnometer volume to the nearest 0.001 mL using Equation 1:

$$V = \left(\frac{W - T}{D} \right) + S \quad (1)$$

where:

D = density of mercury at test temperature, see Table, g/mL

S = calibration stem correction volume, mL

T = mass of assembled empty pycnometer, with top and retaining cap, g

V = volume of pycnometer, mL

W = mass of assembled pycnometer with mercury, g

17. Make minimum of three separate determinations for each pycnometer and average the results.
18. Repeat the determination for any calibration that varies from the mean by more than 0.25%.

Sample Analysis

The solids pycnometer is used for samples having a particle size range of 0.6 to 5 mm. The powder pycnometer is used for samples having a particle size range of 0.6 to 0.15 mm (see *Notes and Precautions 3*).

1. Assemble the appropriate pycnometer by applying a thin coating of stopcock grease to the top edge of the pycnometer chamber. Place the circular glass cap on the top of the chamber and then an O-ring. Tighten the metal retaining cap on the pycnometer top.
2. Obtain the mass of the assembled pycnometer to the nearest 0.001 g.
3. Disassemble the pycnometer and insert a funnel into the pycnometer sample chamber.
 - The 15-mm OD stem funnel is used for solid particulates and the smaller stem funnel is used for powders.
4. Fill the sample chamber with the particulate sample.
 - A sample mass of approximately 2-5 g is typically required. When there is insufficient sample, a smaller amount may be used, but a smaller sample will increase the effect of weighing errors.
 - If the determination is to be reported on a volatile-free basis, weigh a sample portion for LOI at 900°C by UOP Method 954 at the same time.
 - Take care that powdered samples do not enter the pycnometer capillary.
5. Remove the funnel and make sure the sealing area is still clean.
6. Reassemble the pycnometer and tighten the retaining cap hand tight.
7. Weigh the sample-filled pycnometer to the nearest 0.001 g.
8. Insert the pycnometer into the filling device, attach the filling device top and clamp in place.
9. Evacuate and fill the pycnometer with mercury as described under *Pycnometer Calibration*, Steps 4 through 9.
10. Lift out the mercury-filled pycnometer and invert the pycnometer stem.
11. Remove any mercury adhering to the outside of the pycnometer stem with the vacuum hose and read the level of the mercury in the pycnometer stem to the nearest 0.001 mL.
 - This value becomes the procedure stem correction volume, C, in Equation 2.
12. Weigh the mercury-filled pycnometer to the nearest 0.001 g.
13. Insert the mercury filled pycnometer back into the filling device to drain any of the excess mercury out of the stem before emptying the pycnometer into a container that can be sealed.
 - This is saved for mercury reclamation or proper disposal.
14. Obtain the mercury temperature and determine the density of the mercury from Table 1.
15. Remove the remaining mercury and sample from the pycnometer using a pipe cleaner and vacuum.
 - This is saved for mercury reclamation or proper disposal.
16. Wipe the entire pycnometer with a wiper.

Calculations

Calculate the piece density of the as-received sample to the nearest 0.001 g/mL using Equation 2:

$$P = \frac{A - T}{V - \left(\frac{B - A}{D} \right) + C} \quad (2)$$

where:

- A** = mass of assembled pycnometer with sample, g
B = mass of assembled pycnometer with sample and mercury, g
C = procedure stem correction volume, mL
P = piece density, as-received, g/mL
T, V, and D = previously defined, Equation 1

Calculate the piece density of the sample on a volatile-free basis (900°C) using Equation 3:

$$F = PL \quad (3)$$

where:

- F** = piece density, volatile-free, g/mL
L = volatile-free factor ((100-LOI)/100)
P = previously defined, Equation 2

Notes and Precautions

1. Mercury vapor is poisonous. Assembly of the apparatus in a properly functioning fume hood is recommended. Clean up all mercury spills with the mercury spill kit and seal all the waste in a container. A tray under the apparatus can help confine spills. Follow proper industrial hygiene procedures and personnel medical requirements. Dispose of gloves, broken glassware, tissues, etc. in a manner consistent with applicable regulatory procedures for mercury-contaminated waste. This procedure should also be followed for non-reclaimed sample waste.
2. The apparatus is provided with a cold trap on the vacuum system to remove volatiles. Cooling is not normally required but can be used when the analyst knows that the sample contains volatiles that would affect the vacuum pump operation.
3. The accuracy and precision of the analysis is reduced as the particle size is reduced because of the increased volume of small voids at points of particle contact.

Precision

Precision statements were determined using UOP Method 999, "Precision Statements in UOP Methods."

Repeatability and Site Precision

A nested design was carried out for determining piece density on three samples. The samples were analyzed by two analysts, with each analyst performing two analyses on two separate days for a total of 24 analyses. Using a stepwise analysis of variance procedure, the within-day and within-lab estimated standard deviations (esd) were calculated at the concentration means listed in Table 3. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 3 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 3 with 95% confidence.

The data in Table 3 represent short-term estimates of the repeatability of the method. When the test is run routinely, use of a control standard and a control chart is recommended to generate an estimate of long-term repeatability.

Table 3
Repeatability and Site Precision, mass-%

<u>Sample</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>	
		<u>Within-Day esd</u>	<u>Allowable Difference</u>	<u>Within-Lab esd</u>	<u>Allowable Difference</u>
A	0.72	0.003	0.01	0.003	0.01
B	0.88	0.002	0.01	0.002	0.01
C	1.17	0.003	0.01	0.004	0.02

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Time for Analysis

The elapsed time and labor requirement for one analysis are identical, 0.25 hour. Pycnometer calibration requires an additional 0.25 hour.

Suggested Suppliers

Bethlehem Apparatus Co., Inc., 890 Front Street, Hellertown, PA 18055, USA 1-10-838-7034,
www.bethlehemapparatus.com

Circle Seal Controls, 2301 Wardlow Circle, Corona, CA 92880, USA, 1-951-270-6200,
www.circlesealcontrols.com

F. P. Smith Wire Cloth Co., 11700 West Grand Ave, Northlake, IL 60164, USA, 1-708-562-3344,
www.fpsmith.com

Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219, USA, 1-412-490-8300,
www.fishersci.com

Grainger, Lake Forest, IL, USA; find a local branch at www.grainger.com

Newport Scientific, 8246 E. Sandy Ct., Jessup, MD 20794, USA, 1-301-498-6700,
www.newportscientific.com

Reliance Glass Division, 1455 Elmhurst Rd., Elk Grove Village, IL 60007, USA, 1-847-228-2851,
www.wilmad-labglass.com

Runco Office Supply, 1655 Elmhurst Rd., Elk Grove Village, IL 60007, USA, 1-847-437-4300,
www.runcoonline.com

Swagelok Company, 29500 Solon Rd., Solon, OH 44139, USA, 1-440-248-4600,
www.swagelok.com

VWR International, 1310 Goshen Pkwy., West Chester, PA 19380, USA, 1-610-431-1700,
www.vwr.com

Walter Norris Co., 55530 Milton Pkwy., Rosemont, IL 60018, USA, 1-847-671-6570

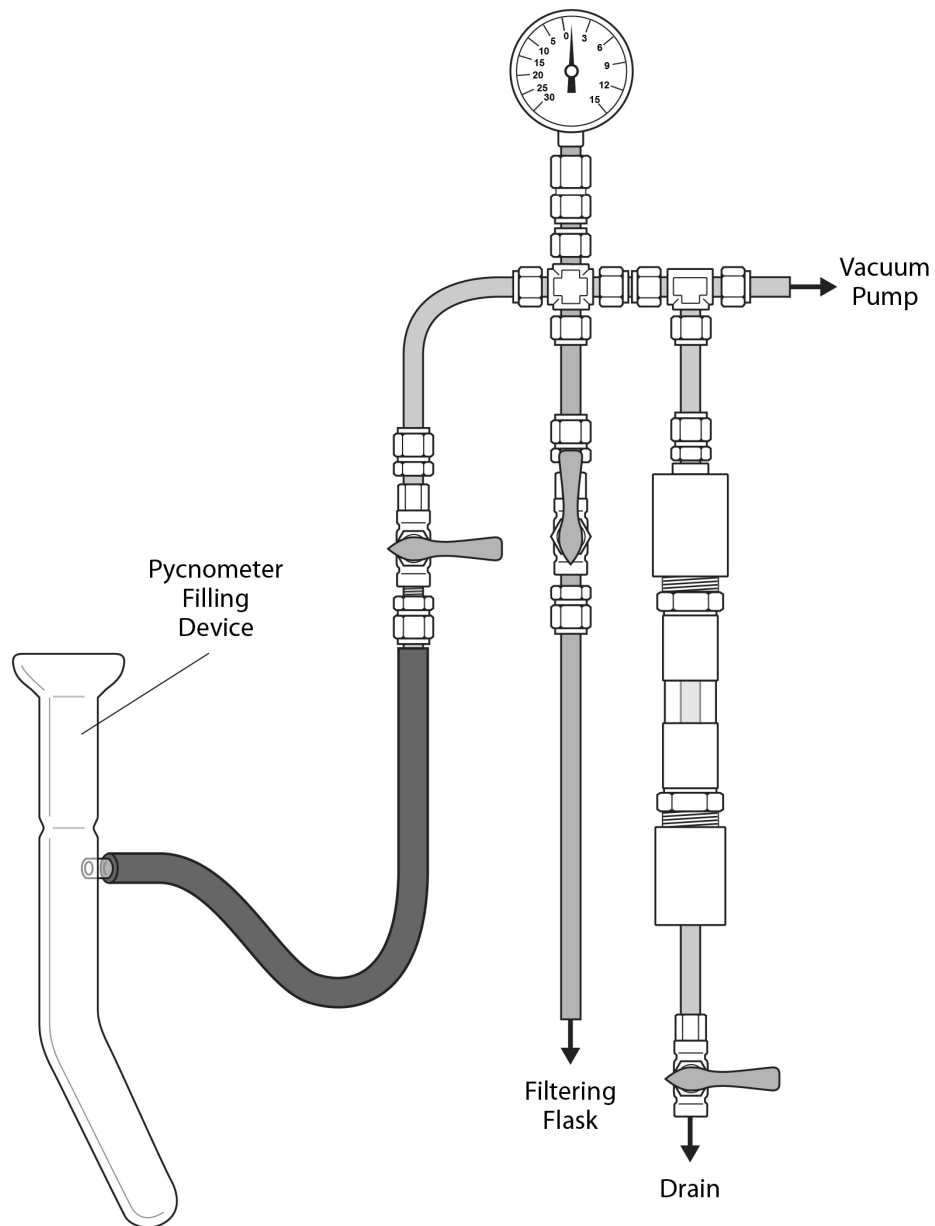


Figure 1
Pycnometer Filling Apparatus

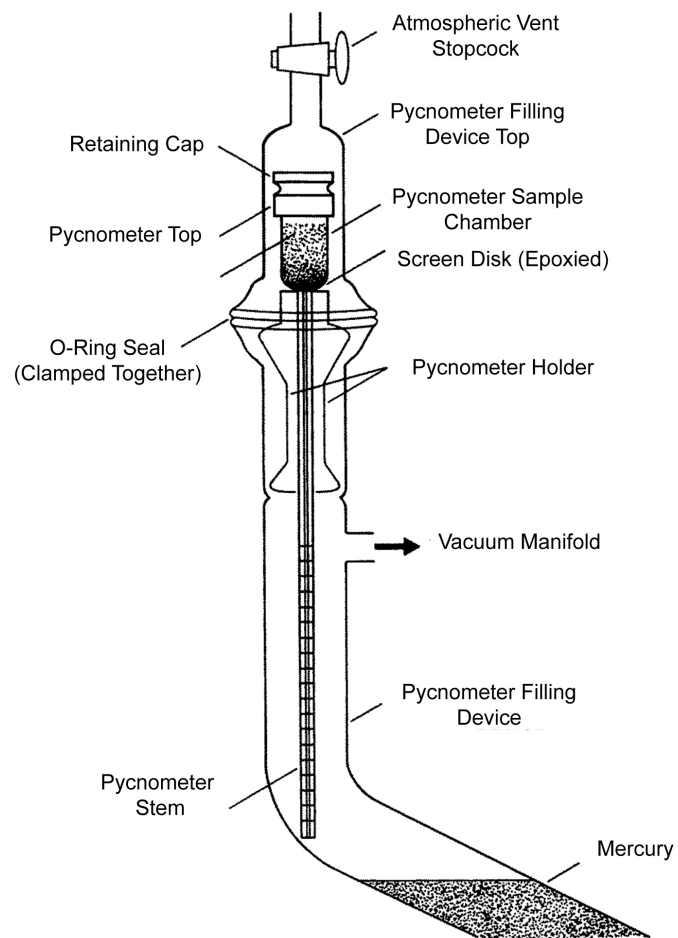


Figure 2
Assembled Pycnometer and Filling Device