



A Honeywell Company

Apparent Bulk Density of Catalyst Particles

UOP Method 294-14

Scope

This method is for determining the apparent bulk density (ABD) of loosely packed spherical catalyst particles with an average particle diameter of 1.6- to 3.2-mm ID. It is also applicable to extruded catalyst particles having a maximum diameter of 3.2 mm, a maximum average length of 9.5 mm, and a maximum individual length of 16 mm. Apparent bulk density is the mass per unit volume of the particulate material.

Reference

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The sample is poured into a weighed and volume-calibrated cylinder. The catalyst is leveled to the top of the cylinder and weighed. ABD is calculated by dividing the mass of the catalyst by the volume of the cylinder.

Apparatus

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

Balance, readable to 0.01-g

Beaker, 250-mL, VWR, Cat. No. 89000-204

Cover, cylinder, flat plastic, glass, or other transparent and non-porous material, approximately 50-mm (2-inches) square, local supply

Cylinder, graduated, glass, 250-mL, 38-mm ID, Fisher Scientific, Cat. No. 08-555E, cut down to the 100-mL mark and fire polished, "weighing cylinder." A cylindrical crucible, 100-mL, 39-mm diameter, 91-mm height, CoorsTek, Cat. No. 65539, is also suitable

Funnel, powder, polypropylene, 75 to 80-mm top diameter, Fisher Scientific, Cat. No. 10-500-1

Ring, support, iron, with screw clamp, slotted, VWR, Cat. No. 60130-004

Spatula, 152-mm straight-edge blade, VWR, Cat. No. 82027-462

Support, rectangular base, steel rod, VWR, Cat. No. 60110-222

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Thermometer, -1 to 51 °C range, 0.1 °C subdivisions, partial immersion, spirit filled, VWR, Cat. No. 61222-624

Timer/stopwatch, VWR, Cat. No. 98000-186

Tray, polypropylene, medium-deep form to catch scattered particles, large enough to accommodate ring support, Fisher Scientific, Cat. No. 13-359-20A

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. References to water mean deionized or distilled.

Tissues, Kimwipes™, Fisher Scientific, Cat. No. 06-666A

Water, deionized or distilled

Procedure

The analyst is expected to be familiar with general laboratory procedures and the equipment being used. Dispose of used supplies and samples in an environmentally safe manner according to applicable regulations.

Volume Calibration of the Weighing Cylinder

The weighing cylinder must be calibrated for volume initially and on a periodic basis thereafter to ensure accurate analyses. For a certified calibration, the weighing cylinder is sent out to a certified calibration laboratory, e.g., The Calibration Solution, for calibration.

Alternatively, if a certified calibration is not necessary, the calibration may be done in house by filling the cylinder with water and weighing it as described below. For in house calibration only:

1. Weigh the dry cylinder and cylinder cover to the nearest 0.01 g.
2. Fill the cylinder to the brim with water. Dislodge any bubbles adhering to the cylinder walls by gently tapping the sides, being careful not to spill the water. Add additional water, if necessary, without introducing bubbles.
3. Touch one edge of the cover to the rim of the cylinder. Lower the cover onto the cylinder while carefully excluding all air bubbles from below the cover. Dry the outside of the cylinder carefully.
4. Weigh the filled cylinder and cover to the nearest 0.01 g.
5. Remove the cylinder from the balance and determine the temperature of the water to the nearest 0.1 °C. Determine the density of the water at the measured temperature from the Figure or other source.

Calculate the exact volume of the cylinder, using Equation 1:

$$C = \frac{W - E}{D} \quad (1)$$

where:

C = calibrated volume, mL

E = mass of empty cylinder and cover, g

D = density of water at measured temperature, g/mL

W = mass of cylinder and cover containing water, g

Determination of ABD

1. Record the mass of the dry, empty calibrated weighing cylinder without the cover.
2. Place the ringstand with the ring in the tray to catch bouncing particles.
3. Support the funnel on the ringstand so the bottom of the stem is approximately 115 mm above the top of the cylinder.
 - If at least two thirds of the particles do not enter the cylinder from this height, the distance may be reduced until greater than two thirds of the catalyst particles enter the cylinder.
4. Over a time period of 45 to 60 seconds, pour 150 mL of a representative sample, at a uniform rate, onto the sides of the funnel using a circular motion. Allow the catalyst to fill the cylinder until it overflows.
5. Without tapping, shaking or otherwise disturbing the cylinder, scrape off the excess catalyst with a straight-edged spatula. Use a sawing motion to prevent packing of the catalyst in the cylinder and to avoid removing catalyst below the plane of the cylinder rim.
6. Weigh the full cylinder to the nearest 0.01 g.
7. Pour the catalyst collected in the cylinder and tray into a beaker and repeat the analysis two more times.
8. If the highest and lowest values differ by more than 1%, repeat the analysis twice more and discard the one suspected result.

Calculations

Calculate the average apparent bulk density (ABD) for the three replicates and report to 3 decimal places, using Equation 2:

$$\text{ABD, g/mL} = \frac{\sum_{i=1}^n (M - T)}{nC} \quad (2)$$

where:

- C** = calibrated volume of cylinder, from Equation 1, mL
M = mass of cylinder plus catalyst for each individual test, g
n = number of individual tests
T = tare mass of empty cylinder, g

Precision

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

Repeatability, Site Precision, and Reproducibility

A nested design was carried out for determining ABD in five laboratories, by two analysts in each laboratory, with each analyst performing analyses on two separate days, performing two analyses each day for a total of 40 analyses. Using a stepwise analysis of variance procedure, the within-day, within-lab, and between-lab estimated standard deviations (esd) were calculated at the concentration means listed in the Table. 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 the Table 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 the Table with 95% confidence. Two analyses performed in different laboratories by different analysts on different days

should not differ by more than the reproducibility allowable differences shown in the Table with 95% confidence.

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

Table
Repeatability, Site Precision, and Reproducibility, g/mL

<u>Catalyst</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>		<u>Reproducibility</u>	
		<u>Within- Day esd</u>	<u>Allowable Difference</u>	<u>Within- Lab esd</u>	<u>Allowable Difference</u>	<u>Between- Lab esd</u>	<u>Allowable Difference</u>
Extruded A	0.669	0.0024	0.007	0.0061	0.019	0.0072	0.022
Extruded B	0.808	0.0039	0.011	0.0051	0.016	0.0113	0.051
Extruded C	1.037	0.0072	0.021	0.0156	0.050	0.0195	0.068
Spherical A	0.319	0.0004	0.001	0.0016	0.006	0.0024	0.010
Spherical B	0.554	0.0009	0.003	0.0014	0.005	0.0030	0.014
Spherical C	0.714	0.0010	0.003	0.0028	0.009	0.0044	0.017

Time For Analysis

The elapsed time and labor requirements for both spherical and extruded catalyst particles are identical, 0.6 hour.

Suggested Suppliers

CoorsTek, 4545 McIntyre St., Golden, CO 80403, USA, 1-303-277-4038, www.usalabware.com

Fisher Scientific, 300 Industry Dr., Pittsburgh, PA 15275, USA, 1-412-490-8300,
www.fishersci.com

The Calibration Solution, 9865 N. Alpine Rd., Machesney Park, IL 61115, USA, 1-815-877-0080,
www.thecalibrationsolution.com

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

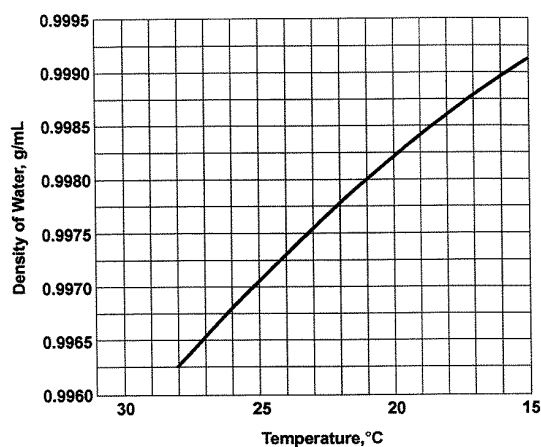


Figure
Temperature, °C vs. Density of Water, g/mL
Water Density = 1.000 g/mL at 3.98 °C