



A Honeywell Company

Packed Apparent Bulk Density of Molecular Sieves

UOP Method 563-14

Scope

This method is for determining the packed apparent bulk density (ABD) of molecular sieves on an as-received basis. Apparent bulk density is an empirical value defined as mass per unit volume, which includes the void spaces between the particles, and is reported as grams per milliliter (g/mL).

Reference

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

Outline of Method

A 100-mL graduated cylinder is slowly filled with a representative sample in a dry nitrogen atmosphere. While it is being filled, the cylinder is agitated with a mechanical vibrator. After additional vibration time, the sample mass and volume are recorded. The mass per unit volume of the sample is calculated.

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

Cylinder, graduated, borosilicate glass, 100-mL capacity, $\frac{1}{8}$ 24/40 joint, one-milliliter subdivisions, calibrated "to contain," with TFE stopper, Fisher Scientific, Cat. Nos. 08-567-1B and 14-642-17C, respectively

Flow meter, 0-1200 mL/min (0-2.5 SCFH), Omega Engineering, Cat. No. FL-3402C

Funnel, powder, glass, 65-mm ID, 15-mm stem OD, Fisher Scientific, Cat. No. 10-346A

Rag, shop wiping, local supply

Regulator, nitrogen, two-stage, 0-345 kPa (0-50 psi), Matheson, Model No. 81-580

Timer, 60-minute, Fisher Scientific, Cat. No. 06-656-1E

Transformer, variable, 120 V, 10 A, VWR Scientific, Cat. No. 62546-251

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Tubing, Tygon™, approximately one-meter long, 4.8-mm ID, by 7.9-mm OD, Fisher Scientific, Cat. No. 14-169-3B

Vibrating table, Syntron Model J-1, FMC Technologies

Reagents and Materials

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

Nitrogen, extra dry, 99.9% minimum purity

Procedure

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

Apparatus Set Up

1. Plug the timer into a 120 V outlet.
2. Set the timer outlet switch to off and plug the transformer into the timer outlet.
3. Plug the vibrator into the transformer.
 - This configuration automatically starts the vibrator when the timer is turned on and shuts it off when the timer reaches zero.
4. Place a shop wiping rag or similar cloth across the top of the vibrator.
 - The cloth, which reduces the noise, can be held in place with rubber bands.
5. Attach the nitrogen regulator to the nitrogen source.
6. Connect the inlet side of the flow meter to the low pressure side of the regulator and attach approximately one meter of Tygon tubing to the discharge side of the flow meter.

Analysis

Molecular sieves absorb moisture from the air very quickly. The measured ABD varies according to the moisture content of the sieve and, therefore, exposure of the sample to air must be minimized.

1. Weigh the dry, empty, 100-mL graduated cylinder with stopper to the nearest 0.01 g.
2. Set the nitrogen regulator to 140-280 kPa (20-40 psi) and adjust the flow meter to approximately 900 mL/min (2 SCFH).
3. Insert the free end of the Tygon tubing all the way down to the bottom of the graduated cylinder and purge for at least one minute.
4. Adjust the transformer to a setting of approximately 65% so that approximately 75 volts are supplied to the vibrator.
5. Set the timer to five minutes. Place the cylinder containing the purge tubing on the vibrator and insert the funnel into the top of the cylinder.
6. Pull the purge tube up until it is just below the bottom of the funnel.
 - This position maintains a nitrogen blanket during the test.

7. Turn on the timer, which activates the vibrator, and begin pouring the sample into the funnel using a slow circular motion so that 95-100 mL of the sample are added uniformly over a one-minute period.
8. Remove the nitrogen purge and funnel, stopper the cylinder and quickly adjust the transformer so that the sieves at the surface are moving in a rapid manner but not jumping wildly.
 - This usually is a small $\pm 3\%$ adjustment.
9. Continue to vibrate the sample until the timer shuts off the vibrator.
 - The total vibration time must be 5 ± 0.1 minutes.
10. Remove the cylinder from the vibrator, record the volume of sample to the nearest 0.5 mL and obtain the mass of the stoppered cylinder containing the sample to the nearest 0.01 g.

Calculations

Calculate the packed ABD, as received, to three decimal places using the following formula:

$$\text{ABD, as received, g/mL} = \frac{W - T}{V}$$

where:

T = mass of empty graduated cylinder, g
V = volume of sample in the cylinder after vibration, mL
W = mass of sample and 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 three 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 24 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 tests 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 tests 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 at the concentrations listed (95% probability). Two tests performed in different laboratories by different analysts, on different days should not differ by more than the reproducibility allowable differences shown in the Table at the concentrations listed (95% probability).

Table
Repeatability, Site Precision, and Reproducibility, mass-%

<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>		<u>Reproducibility</u>	
	<u>Within- Day</u>	<u>Allowable esd Difference</u>	<u>Within- Lab</u>	<u>Allowable esd Difference</u>	<u>Between- Lab</u>	<u>Allowable esd Difference</u>
0.784	0.0047	0.014	0.0072	0.025	0.0126	0.044

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.

Time For Analysis

The elapsed time and labor requirement are identical, 0.6 hour.

Suggested Suppliers

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

FMC Technologies, Inc., 57 Cooper Ave., Homer City, PA 15748, USA, 1-724-479-4500,
www.fmcsyntron.com

Matheson Tri-Gas, 166 Keystone Dr., Montgomeryville, PA 18936, USA, 1-215-641-2700,
www.mathesontrigas.com

Omega Engineering, Inc., One Omega Dr., P.O. Box 4047, Stamford, CT 06907, USA, 1-203-359-1660, www.omega.com

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