



A Honeywell Company

Packed Apparent Bulk Density (ABD) of Extruded Catalyst

UOP Method 778-07

Scope

This method is for determining the packed apparent bulk density of extruded catalysts. The method is applicable to catalysts conforming to the following approximate dimensions: a maximum diameter of 3.2 mm (1/8 inch) and a maximum average length of 9.5 mm (3/8 inch), with a maximum individual length of 15.9 mm (5/8 inch). Apparent bulk density, referred to as ABD, is an empirical value defined as weight per unit volume. The packed ABD of molecular sieves can be determined using UOP Method 563, "Packed Apparent Bulk Density of Molecular Sieves," which addresses the hydrophilic nature of the material.

References

UOP Method 563, "Packed Apparent Bulk Density of Molecular Sieves," www.astm.org

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

Outline of Method

A 100-mL graduated cylinder is slowly filled with the sample at a uniform rate. During the entire filling process the cylinder is vibrated by a mechanical device. The net weight of sample divided by the observed sample volume is calculated. The resulting number is referred to as the packed ABD of the sample.

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, glass, 100-mL capacity, 1-mL subdivisions, Class A, serialized and certified, VWR, Cat. No. 89001-708

Funnel, powder, polypropylene, 65-mm top diameter, 16-mm stem diameter, VWR, Cat. No. 30252-951

Timer, 60-minute, GraLab, VWR, Cat. No. 62371-001

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).

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Variable autotransformer, VWR, Cat. No. 9461Q40

Vibrating table, Syntron Model J-1, FMC Technologies

Procedure

The analyst is expected to be familiar with general laboratory practices and the equipment being used.

Before performing quantitative analyses, perform the procedure several times with typical sample types to optimize the vibration intensity of the vibrating table. Adjust the control on the variable autotransformer and/or the vibrator to achieve a settling motion of the catalyst without excessive bouncing that could cause attrition or breakage. A setting of about 60-65 on the autotransformer may be suitable. Once the setting is determined, do not change it.

1. Plug the vibrating table into the variable autotransformer, and variable autotransformer into the timer. Set the timer for one minute and 45 seconds (105 seconds).
 - Leave the vibrating table and autotransformer switched on. The on-off function is controlled by the timer.
2. Weigh the dry empty 100-mL graduated cylinder to the nearest 0.01 g.
3. Place the cylinder on the vibrating table and insert a funnel into the top opening.
4. Turn on the vibrator (timer) and while firmly holding the base of the cylinder to the table, begin pouring the sample into the cylinder in a slow and uniform manner. Pour at a rate such that 95-100 mL of sample are added over a 60-90 second period.
5. When 95-100 mL of sample have been added to the cylinder, remove the funnel, and continue to vibrate the sample for an additional 15 seconds while slowly rotating and moving the cylinder across the top of the vibrator. Stop the vibrator after the additional 15 seconds have elapsed. The total vibration time should not exceed 105 seconds.
6. Remove the cylinder from the vibrator and record the volume of sample to the nearest 0.5 mL. Weigh the cylinder containing the sample to the nearest 0.01 g. The gross weight minus the tare weight is the net weight of catalyst.
7. Repeat the ABD determination two more times.

The average of the three determinations is reported as the packed apparent bulk density (ABD). If any of the values determined deviates from the average by more than one percent relative, discard that value and repeat the analysis to obtain an acceptable number.

Calculations

Calculate the packed ABD using the following formula:

$$ABD, \text{ g/mL} = \frac{W - T}{V} \quad (1)$$

where:

- T** = weight of graduated cylinder, g
- V** = volume of sample after vibration, mL
- W** = weight of sample and cylinder, g

Report the results to three significant figures.

Precision

The precision statement was determined using UOP Method 999.

Repeatability and Intermediate Precision

A nested design was carried out for determining packed ABD on one sample with three analysts in one laboratory. The analyst carried out two tests on two separate days. The total number of tests for the sample was 12. Using a stepwise analysis of variance procedure, the within-day estimated standard deviation (esd) was calculated and is listed in the Table. Two tests performed by the same analyst on the same day should not differ by more than the repeatability allowable difference 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 intermediate precision allowable difference shown in the Table with 95% confidence.

The data in the Table are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Reproducibility

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

Table
Repeatability and Intermediate Precision, g/mL

<u>Sample</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Intermediate Precision</u>	
		<u>Within-Day esd</u>	<u>Allowable Difference</u>	<u>Within-Lab esd</u>	<u>Allowable Difference</u>
Extruded catalyst	0.609	0.0022	0.008	0.0100	0.040

Time for Analysis

The elapsed time and labor requirement are identical, 0.3 hour.

Suggested Suppliers

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

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