

COLLECTION AND GRINDING OF ADSORBENTS AND CATALYSTS FOR ANALYSIS

UOP Method 918-92

SCOPE

This method is for collecting and grinding adsorbents and catalyst samples to be analyzed by UOP Methods. Individual methods should be consulted for specific instructions regarding details of the sample preparation for individual analyses.

OUTLINE OF THE METHOD

A representative bulk sample is selected from a larger lot in one of three ways depending on the origin of the adsorbent or catalyst: From production lots, a representative sample is obtained using established techniques. From research samples, a 200-mL sample, or at least 20% more than is required for the analysis, is obtained by riffing. From spent catalysts, the sample lot is screened to remove extraneous material prior to sampling by an established technique, mixed and reduced to a 200-mL sample by riffing. If grinding is not required, this representative bulk sample is riffled to obtain a representative analytical sample for the analysis. If grinding is required, a specified procedure is followed to produce a representative sample for analysis.

APPARATUS

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

Ammeter, in-line, 0-15 amp range

Bag, plastic, vapor impenetrable, sized appropriately for use

Balance, top loading, readability 0.01-g

Bottle, polypropylene, Mason jar, 500-, 1000-, 2000- and 3000-mL, 70-mm mouth, with closure, Fisher Scientific, Cat. Nos. 11-825A, -825B, -825C and -825D, respectively

Bottle, vapor impenetrable, sized appropriately for use

Brush, fine sieve brush, Gilson Company, Cat. No. TSA-170

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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UOP Methods are available through ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken PA 19428-2959, United States. The 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.

Cutter sampler, sample divider, with nitrogen purge, sized and constructed according to the specifications summarized in ASTM Method D 2234, Section 7

Cylinder, graduated, 100-mL

Drum-thief, pellet, three-zone powder sampler, 550-mm (22-inch) long, with three 76-mm (3-inch) cells, Scientific Products, Cat. No. S1032

Dust-box, constructed from 1/4-inch thick polycarbonate, having two hinged, front opening doors and openings for compressed air, vacuum and two accordion sleeves, see Figure. If hygroscopic catalysts are to be ground in the box, it must be air-tight and fitted with a nitrogen purge.

Pulverizer, laboratory, capable of grinding catalysts to 0.17-mm (80-mesh or finer), equipped with a stainless steel screen with herringbone slots of 0.25 mm, Mikro Samplemil, Hosokawa Micron. Modify the side door by covering the rubber gasket with the Teflon facing and connect the in-line ammeter to the power cable.

Regulator, air, two-stage, high purity, Matheson, Cat. No. 3104-590

Regulator, nitrogen, two-stage, high purity, Matheson, Cat. No. 3104-580

Riffler, manual sample splitter, with scoop and receiver pans. The scoop for adding sample to the riffler must have straight sides equal in length to the effective width of the riffler. The riffler must be fitted with an even number of dividing slots that are of equal width, at least three times the maximum diameter of the whole-pill sample. The slots must discharge alternately to each side of the riffler. The size of the riffler is governed by the sample size to be divided. It must be possible to distribute the sample evenly across the entire length of the scoop used to add the sample to the riffler. A riffler suitable for 200 mL of sample is available from Thomas Scientific, Cat. No. 8261-C10.

Riffler, rotary, micro, Quantachrome, Model RR-4

Spatula, scoop, 300-mm length, Fisher Scientific, Cat. No. 14-375-57

Teflon facing, surface protector, Bytac, general purpose, Baxter Diagnostics, Cat. No. L5590-1

Vacuum-cleaner, high efficiency, capable of filtering 99.9995% of the particulate with a diameter of 0.12 μm or greater, Nilfisk of America, Model GS 82

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified. References to water mean deionized or distilled water, except where noted.

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

Air, 207 kPa minimum delivered pressure, less than 2.0 ppm H_2O

Nitrogen, high purity, less than 1.0 ppm oxygen, less than 1.0 ppm water

PROCEDURE

Sample Collection

Catalyst Screening

All catalyst is screened at the plant prior to sampling to remove extraneous material. Fresh oxidized catalyst that will be reduced is passed directly through a screening unit from the product accumulation hopper. Reduced and sulfided catalysts are screened following reduction or sulfiding. Spent catalyst is screened to remove extraneous material such as ceramic spacers.

Fresh Catalyst from Production Lots

1. Purge an appropriately sized clean bottle or plastic bag with dry nitrogen and cap or seal.
2. Sample the entire production lot of fresh catalyst using an established technique.
 - Cutter sampling according to the procedure described in ASTM Method D 2234 (Note 1), Condition IB-1 is an example technique.
 - Withdrawing small regularly spaced portions from a moving sample stream is generally recognized as the easiest, most effective procedure for collecting representative samples of large lots.
 - If a cutter-sampler is used, the sample or increment size is controlled by the cutter-sampler size, the frequency of the pass and speed of transverse.
 - The number of increments should be selected so as to obtain a representative sample of the production lot; generally one per drum.
3. Layer the sample in the bottle or plastic bag as the samples are taken and cap the bottle or seal the plastic bag when the lot is complete.
 - This sample is often referred to as the gross sample.
4. Pour the contents of the bottle or plastic bag into a large plastic bag while maintaining a dry nitrogen purge.
5. Mix the contents of the bag to destroy the layering characteristics of the sample.
6. Divide the entire sample by successive riffing to obtain a 200-mL analytical sample plus an appropriate quantity of a retained sample, using the procedure described in ASTM Method D 346 (Notes 1, 2 and 3), Procedure A, and a properly sized riffler.
7. Store the retain of the sample appropriately.

Adsorbents from Production Lots

Obtain a representative sample by established techniques.

- Cutter sampling and riffing as described under *Fresh Catalyst from Production Lots* are examples of techniques.

Experimental Adsorbents and Catalyst

Divide the entire, well-mixed experimental sample to obtain a 200-mL sample by successive riffing, using the procedure described in ASTM Method D 346 (Notes 1, 2 and 3), Procedure A, and a properly sized riffler.

- If the lot volume of the sample is smaller than 200 mL, or if a limited quantity of sample is available for analysis, a representative sample that exceeds the minimum sample size actually required for the analysis by at least 20% is obtained from the entire, well-mixed experimental sample by riffing according to ASTM Method D 346, Procedure A (Notes 1, 2 and 3).

Spent Catalyst

Each spent catalyst shipment is screened in the catalyst receiving plant and 4 fractions are obtained: oversize, on-size, fines and dust. The on-size material, fines and dust may afford one or more analytical samples depending on the size of the shipment. The oversize material is not sampled for analysis. The analytical samples are obtained as follows:

- If a precursory inspection of the spent catalyst is required, it may be performed as follows: Count the number of drums in the spent catalyst shipment. Determine the square root of the total number of drums in the spent catalyst shipment, that is the number of drums that require sampling. Using the drum-thief, sample that number of drums of the shipment combining each increment in an appropriately sized bottle or plastic bag. Mix the gross sample well by randomly shaking for 5 minutes in a tightly capped or sealed container.
1. Collect the fines (broken pills and chips) into drums and sample by either using a cutter-sampler as described in Steps 1 through 7 of *Fresh Catalyst from a Production Lot* or by sampling each drum with a drum-thief, combining each increment in an appropriately sized container and mix well.
 - This sample is often referred to as the fines gross sample.
 2. Collect the dust collected by the dust collectors and sample using a drum-thief.
 3. Sample the on-size catalyst using a cutter sampler according to Steps 1 through 7 described under *Fresh Catalyst from a Production Lot*.
 - The on-size catalyst may be separated into parts depending on the size of the shipment.
 - This sample is often referred to as the on-size gross sample.

Sample Selection and Preparation

Grinding Not Required

Riffle the representative sample down to the sample size required for the determination using a properly sized riffle and riffing procedure as described in ASTM Method D 346 (Notes 1, 2 and 3), Procedure A.

Grinding of Hygroscopic Samples

Follow the procedure detailed in *Grinding of 200-mL Samples* or *Grinding of Samples When Less than 200 mL is Available*, in an air-tight dust box or glove box, respectively, with a dry nitrogen purge.

Grinding of 200-mL Samples

Pulverizer Set-up and Cleaning

1. Set up the pulverizer and install it in a dust box, glove box, fume hood or other appropriate location.
 - The pulverizer must be set up in such a manner as to prevent the release of dust to the environment during pulverizer cleaning and sample grinding.
2. Provide a high-efficiency vacuum and a pressurized air line for cleaning the grinder.

3. Clean the pulverizer thoroughly as follows to remove any material remaining from grinding of the previous sample.
 - The cleaning must be performed in such a manner to prevent inhalation of the dust.
4. Verify that the power to the pulverizer is off and lock out the power switch.
5. Remove the receiver bottle, if in place, and open the front feeder door.
6. Remove and clean the door gasket using the vacuum and air hose.
7. Remove the feeder screw, the side door and gasket, the screen and the screen support plate.
8. Vacuum, brush or blow all loose dust from the disassembled pulverizer and the removed pulverizer parts.
 - It is important to remove as much dust as possible from inside the mill housing, especially behind the grinder blades.
9. Reassemble the pulverizer. Examine the Teflon facing material on the side door prior to assembly. Replace the Teflon facing if it shows any sign of wear.
 - Check that the door gasket is in place.
10. Close and lock tight the front and side doors.

Sample Grinding

Grind the representative bulk sample as follows:

1. Verify that the pulverizer has been cleaned as described in *Pulverizer Set-up and Cleaning*.
 - Clean if necessary.
2. Riffle out a representative portion of 50 mL of the sample to be ground from the well mixed representative sample.
 - The optimal sample size for grinding is 200 mL. Larger samples may be ground if the equipment is sized appropriately.
 - This first 50-mL portion of sample is to be used as a “mill wash” and is intended to precoat the interior surfaces of the pulverizer and simultaneously serve as a test for any dust leaks.
3. Attach a clean, appropriately sized receiver to the pulverizer to receive the “mill wash”.
4. Turn on the properly assembled pulverizer.
 - Appropriate safety precautions must be taken when operating the pulverizer.
 - If a dust box is used, the doors are closed and the vacuum is attached to the dust box (see Figure) and turned on.
5. Feed a few particles of the “mill wash” through the mill and watch for any dust escaping around the gaskets on the frontal feeder door, the side door and the receiver.
6. Tighten or reassemble any section of the mill from which any dust was observed to escape.
7. Repeat Steps 5, 6 and 7 until no further dusting is observed.

8. Grind the balance of the “mill wash” sample to a consistency of less than 0.17 mm (80 mesh) by a single pass through the pulverizer. Maintain a minimum constant load on the mill as observed on the in-line ammeter by varying the rate at which the particles are added. Collect the portion of the “mill wash” that falls into the receiver, leaving the remainder in place as a coating inside the mill.
 - This procedure affords a powder that passes approximately 90% through 200 mesh.
 - Good laboratory practice suggests that the pulverizer mill be checked periodically to verify that it is producing the desired mesh size by grinding a sample and checking the mesh size by an appropriate technique such as wet sieving.
9. Turn off the mill, remove the receiver containing the “mill wash” and dispose of the “mill wash” sample.
10. Attach a clean, appropriately sized receiver to the pulverizer to receive the balance of the gross sample.
11. Turn on the pulverizer.
12. Feed a few particles of the balance of the gross sample through the mill and watch for any escaping dust from the gasket around the receiver. Tighten or reattach the receiver if any dust was observed leaking from the gasket and verify that no further dust is leaking.
13. Grind the balance of the sample by a single pass through the pulverizer. Maintain a minimum constant load on the mill as observed on the in-line ammeter by varying the rate at which the particles are added. Collect only that portion of the sample which freely collects in the receiver.
 - Approximately 98-100% of the balance of the sample should be recovered.
 - If a dust box is used, the vacuum is turned off when grinding is complete.
14. Mix the sample thoroughly by randomly shaking it for five minutes in either the tightly capped receiver or a sealed plastic bag to which it may be transferred.
 - A well shaken powdered sample will be uniform throughout but agitation will resegment the particles.
15. Open the receiver or plastic bag without agitating it and scoop out the portion required for the analysis.
16. Clean the pulverizer as described in *Pulverizer Set-up and Cleaning*.

Grinding of Samples When Less than 200 mL is Available

1. Select an alternative means of grinding that is capable of reducing the sample particle size to less than 0.17 mm (80 mesh).
 - Grinders that may be suitable include a mortar and pestle, a shaker mill or a shatter box, but the grinder used must meet the following requirements:
 - Grinding must be performed without dust loss.
 - For noble metal analysis the powder must pass through 80 mesh with approximately 90% through 200 mesh.
 - The grinding device must not contaminate the sample and the sample recovered must be representative.

2. Clean the alternative grinding device by blowing out old powdered samples with air, brushing and then thoroughly clean by grinding and discarding a portion of the catalyst not required for the analysis.
 - Care should be taken to avoid breathing the dust from ground catalysts.
3. Grind the portion of the sample to be used for the analysis, transfer the **entire** ground sample to a bottle and tightly cap it.
 - Care should be taken to avoid dust losses while transferring the ground sample into the bottle.
4. Shake the sample randomly in the tightly capped bottle for five minutes. Carefully set the bottle down without tapping it on the lab bench.
 - A well shaken powdered sample will be uniform throughout but tapping it will resegment the particles.
5. Remove the cap without tapping the bottle and scoop out the portion required for the analysis.

CALCULATIONS

No calculations are required for this method.

NOTES

1. In the wording of the ASTM standard, the bulk fresh, spent or experimental sample is identified by the phrases “crushed gross sample,” “crushed coke” or “coke”.
2. If desired, a rotary riffler may be substituted for the manual riffler for whole pill samples. Such devices are preferred for subdivision of small samples. Ground catalysts are not to be riffled because of dust loss, hygroscopic behavior and electrostatic problems.
3. Riffing minimizes sample variability but does not prevent it.

PRECISION

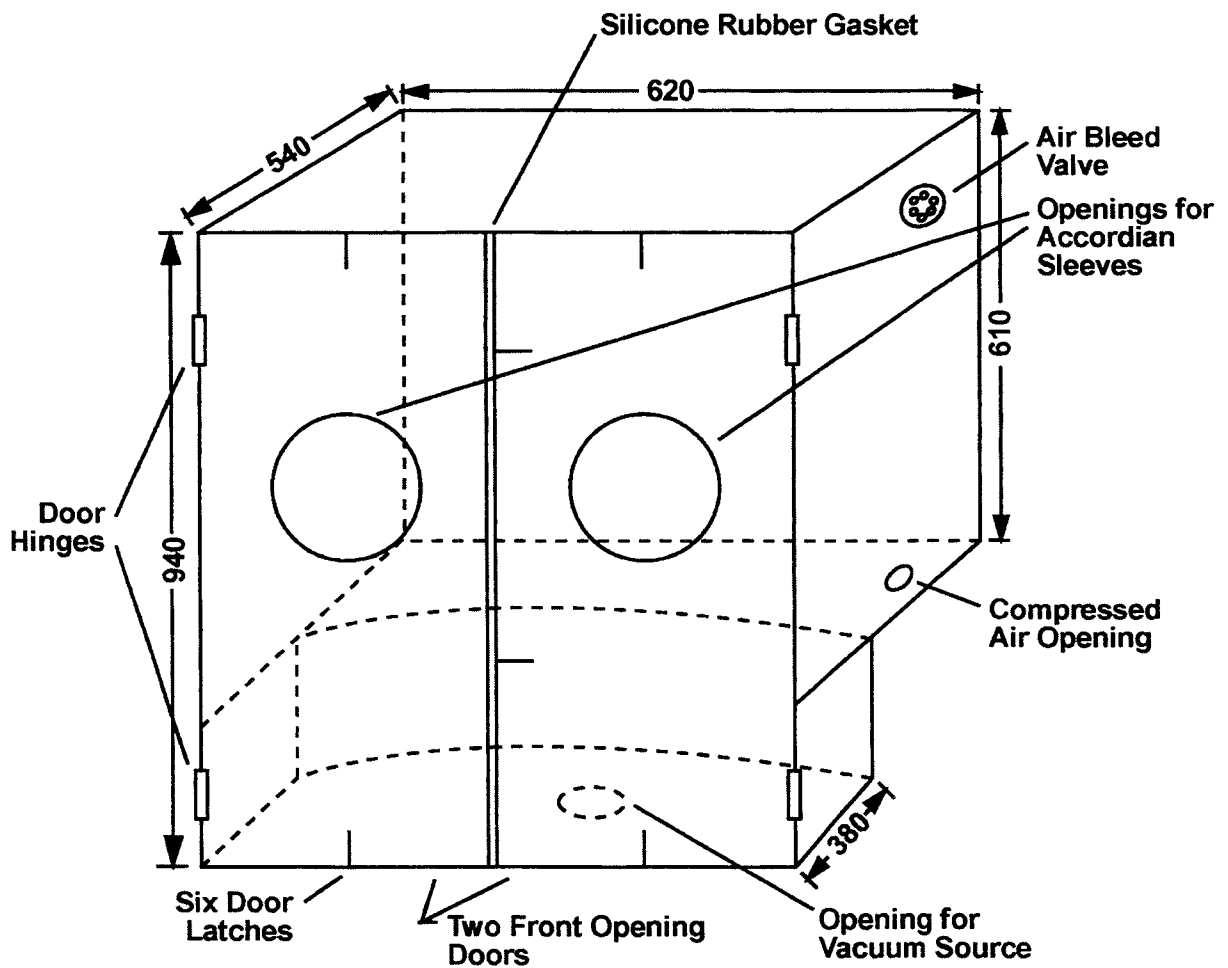
Precision cannot be determined for this method because a numerical result is not obtained.

REFERENCE

ASTM Methods D 346 and D 2234, www.astm.org

SUGGESTED SUPPLIERS

Baxter Diagnostics, Inc., 1430 Waukegan Rd., McGaw Park, IL 60085-6787 (708-689-8410)
 Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-9919 (412-562-8300)
 Gilson Company, Inc. P.O. Box 677, Worthington, OH 43085-0677 (614-548-7298)
 Hosokawa Micron Div., 10 Chatham Rd., Summit, NJ 07901 (201-273-6360)
 Matheson Gas Products, Inc., P.O. Box 96, Joliet, IL 60434 (815-727-4848)
 Nilfisk of America, Inc., 300 Technoloa Dr., Malvern, PA 19355 (215-647-6420)
 Quantachrome Corp., 5 Aerial Way, Syosset, NY 11791 (516-935-2240)
 Scientific Products, 1430 Waukegan Road, McGaw Park, IL 60085-6787 (708-689-8410)
 Thomas Scientific, P.O. Box 99, Swedesboro, NJ 08085-0099 (609-467-2000)



Constructed from
1/4-inch Thick Polycarbonate
All Dimensions in mm

Figure
Dust Box