

PARTICLE SIZE DISTRIBUTION BY MICROMESH SIEVES

UOP Method 422-66

SCOPE

This method is for the determination of particle sizes of fluid cracking catalysts by means of calibrated sieves having uniform precise square openings. It may also be used for determining particle sizes of other powdered materials. The method provides for the classification of particles in a range of sizes from about 20 to 149 microns.

The method is a modification of Shell Development Co. Method EMS 5Z2/61.

OUTLINE OF METHOD

A representative sample is appropriately humidified and then placed on the top sieve of a calibrated set of precision sieves. The set is mounted on a specified shaking apparatus and shaken for 20 minutes. The weight of catalyst retained on each sieve and in the pan is determined, and the percentage of the sample retained on each sieve and in the pan is calculated on the basis of the sum of weights of the recovered fractions.

APPARATUS

Acoustic barrier. A hood or small box of acoustical material is advantageous in reducing the slight amount of noise caused by the vibration of the shaker.

Balance, analytical, with pan of 3.5-inch or larger diameter capable of weighing to ± 1 mg. A single-pan, semi-automatic, constant-load balance is recommended.

Brush, sable, 0.5-inch wide

Log probability paper, No. 3128, Codex Book Co., Inc., Norwood, Mass., or equivalent

Precision micromesh sieves, 3-inch diameter, made of electroformed mesh and conforming to "Tentative Specifications for Precision Micromesh Sieves," ASTM E 161 - 60T, except that sieve frames can be fabricated from either brass or aluminum. The sieve series includes 20-, 30-, 45-, 60-, 75-, 90-, 105-, and 149- μ sieves, a pan and cover. The sieves are available only from Buckbee Mears Co., St. Paul, Minn.

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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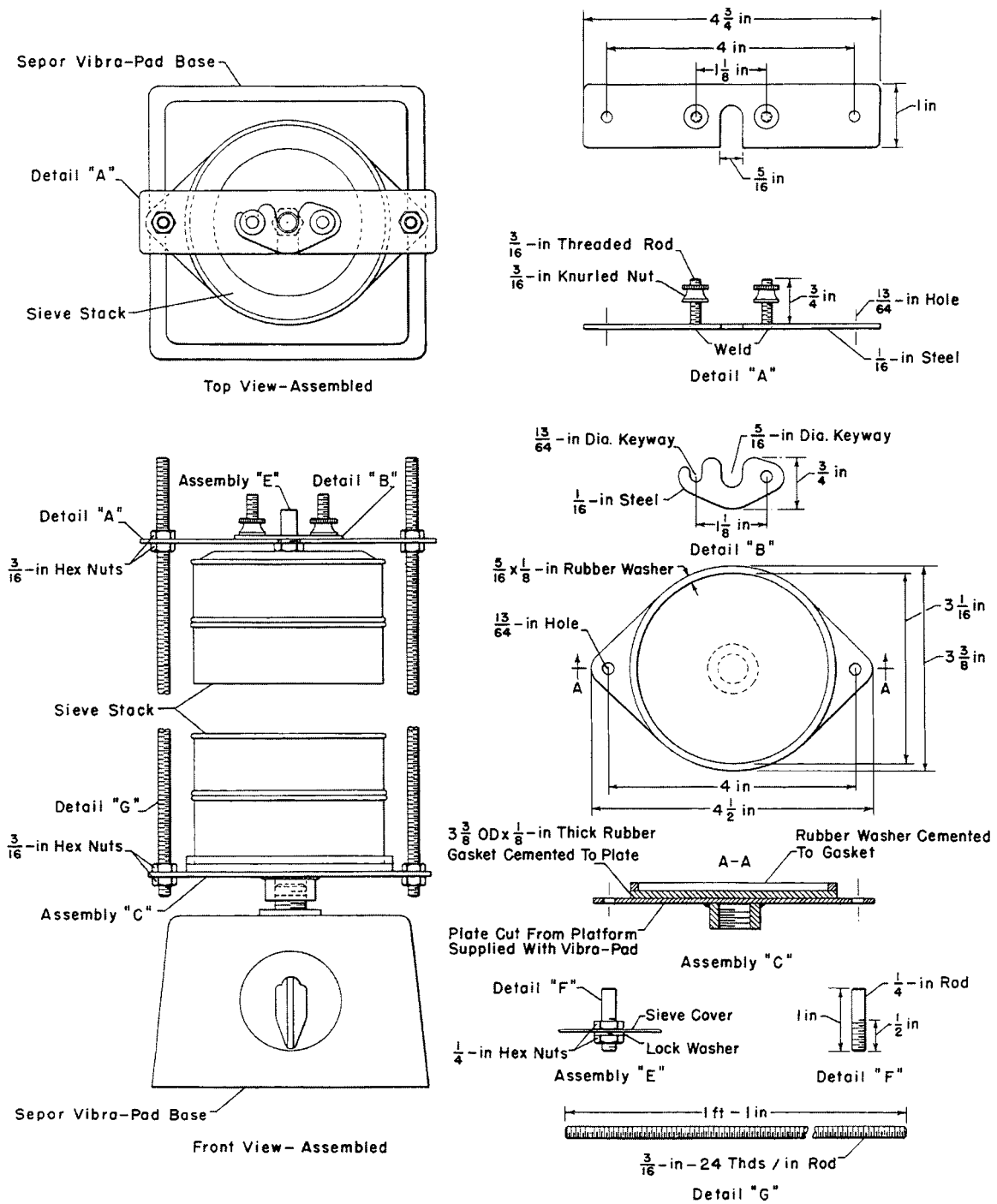


Figure 1

Sieve Yoke For Sepor Vibra - Pad Base

Riffler. A Sepor “micro-splitter,” obtainable from Sepor Laboratory Supply, Torrance, Calif. 90505, is recommended to reduce quantities of approximately 1 qt or less to samples of a size suitable for directly obtaining the amount necessary for each determination.

Sampler. A cylindrical scoop of appropriate capacity to hold the required amount of sample. Alternatively, use any suitable type of scoop such as a Fisher “Scoopula”.

Sieve shaker. A Sepor “Vibra-Pad”, No. 412, modified for use with 3-inch diameter sieves in accordance with Fig. 1. Obtainable from Sepor Laboratory Supply.

MICROMESH SIEVE CALIBRATION

Calibrate or check the sieves for accuracy of openings by one of the following procedures:

Microscopic measurement. Measure the sieve openings by the microscopic method given under “Method of Calibrating Precision Micromesh Sieves”, appended to “Tentative Specifications for Precision Micromesh Sieves”, ASTM E 161 - 60T. This is considered the primary method of calibration. However, for most practical purposes in testing catalysts for conformance to specifications, the alternative method of using standard samples of catalyst will suffice.

Standard sample measurement. Obtain at least 3 samples of fluid cracking catalyst that have been previously analyzed according to this method, using a set of sieves calibrated microscopically. The several samples should include at least one having a minimum of 5% of material finer than 20 μ and another sample having a minimum of 5% of material coarser than 105 μ . Prepare curves representing the particle size distribution of these samples. Use these samples as “standards” in calibrating other sets of sieves.

Using the set of sieves to be calibrated, determine the particle size distribution according to the procedure of this method in duplicate on at least 3 “standard” samples. Convert the fractions to “cumulative weight-percent” finer than each nominal size. Using the values so obtained, determine the effective sieve sizes from the curves representing the practical size distribution of the standard samples.

Calculate the calibrated size of each sieve by averaging the effective sizes obtained from the standard samples. Use the calibrated sizes in determining the particle size distribution of samples submitted for analysis.

SAMPLING AND SAMPLE PREPARATION

Obtain a representative portion of the catalyst by riffing to produce a sample of approximately 10 g, making sure that the amount of fines lost in the process is kept at a minimum. Place the 10-g sample in a container having 2 or 3 times the volume of the sample and tumble it by hand to assure an even distribution of particles. Withdraw at one time 1 to 1.5 g of sample, using a scoop that will hold this quantity of catalyst. More than one sampling to get the required amount of sample may disturb the normal size distribution. Place this sample in a container and add 2 drops of water. Close the container and let it stand for 5 to 10 minutes, then tumble and shake until there are no signs of agglomeration.

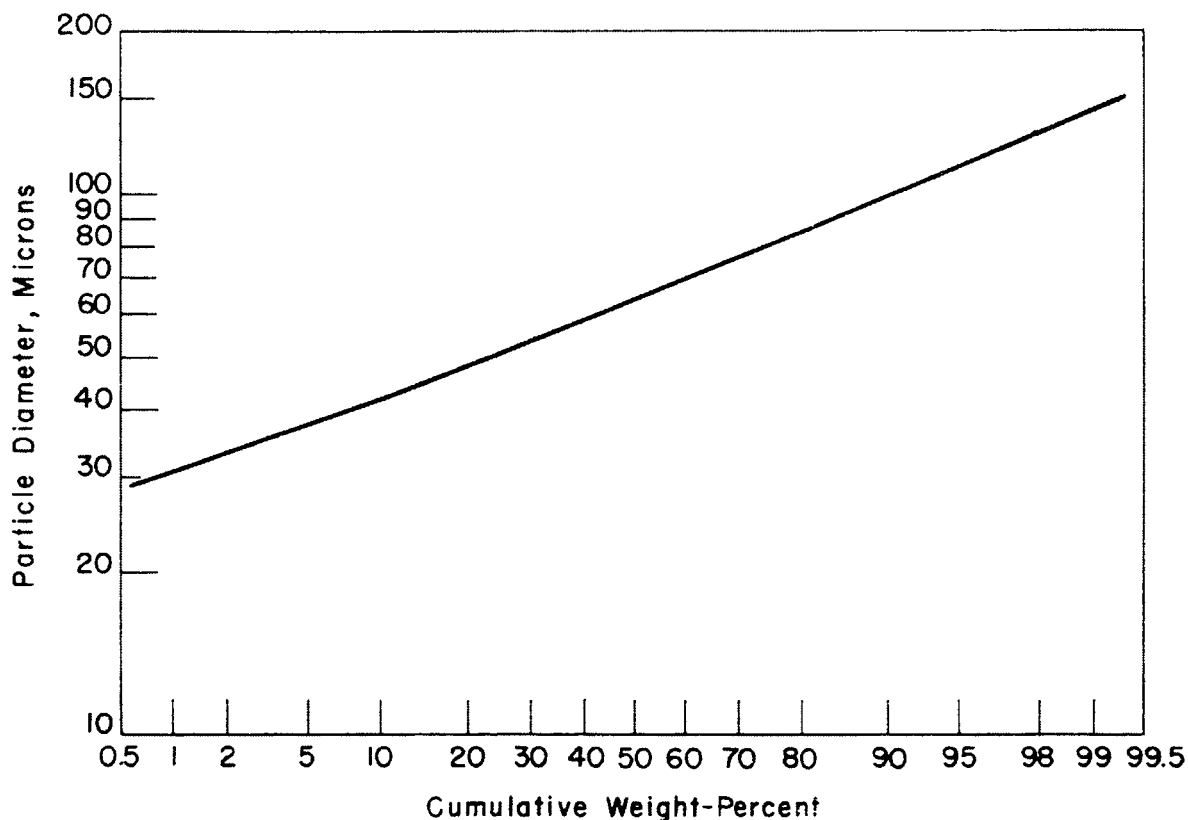


Figure 2

Typical Particle Size Distribution Curve

PROCEDURE

For the convenience of not having to adjust the height of the sieve yoke between determinations, use the entire series of sieves for all determinations. Weigh each of the sieves and the pan to the nearest milligram. Assemble the sieves and pan in proper order and place the entire sample on the top sieve. Place the cover on the top sieve, place the stack of sieves on the shaker and tighten the sieve retainer strip sufficiently to hold the sieves firmly in place. Use the medium setting for sieves with aluminum frames, and the high setting for sieves with brass frames. Attach a ground wire from the stack to a suitable electrical ground. Shake the stack of sieves for 15 minutes, then tilt and tap the stack in several directions and shake it for another 5 minutes. Remove the stack from the shaker and weigh each sieve and pan to the nearest milligram.

Clean each sieve by holding it in an inverted position, removing the contents by carefully brushing the bottom with the sable brush and gently blowing away any particles clinging to the sieve.

CALCULATIONS

Calculate the weight-percent of catalyst retained on each sieve and pan to the nearest 0.1% as follows:

$$\text{Retained, wt - \%} = \frac{100A}{W}$$

where:

A = weight of catalyst retained, g

W = summation of the weights of the fractions, g

Convert the fractions to a cumulative basis as “percent finer than” the calibrated mesh size of the sieve and plot these values on log probability paper in such a manner that sizes may be estimated to less than 0.5 μ and proportions to less than 0.5%. Draw a smooth curve through the points. For equilibrium catalysts report the percent finer than 20, 30, 40, 50, 60, 70, 80 and 90 μ , as determined from the curve. For fresh catalysts also report the percent finer than 105 and 149 μ . A typical curve is shown in Fig. 2.

Plotted points will generally form a nearly straight line on log probability paper for samples which have not been subjected to mechanical separation or classification. A point to the left of such a line, resulting from an excessive amount on that particular sieve, indicates a “blinding” of the sieve or incomplete sieving through the mesh. A point to the right of the line, resulting from too small an amount on that sieve, indicates a broken mesh. Inaccurate calibration values may also cause either condition to exist.

NOTES AND PRECAUTIONS

Take care to avoid excessive tightening of the sieves. A slight rotary action of the sieves relative to each other during shaking has been found to be a suitable criterion of tightness.

No “blinding” of micromesh sieves has been experienced using this method. However, if excessive “blinding” (more than about 15%) does occur, clean the sieves by the use of ultrasonic cleaning equipment. If it is necessary to use sieves which are blinded by more than about 15%, extend the sieving time by additional 5-minute shaking periods followed by weighings until the change in weight on any sieve or the pan is no greater than 4 mg.

Micromesh sieves should be considered as delicate precision instruments and handled accordingly. Do not tap the sieve on the bench or other surface in attempting to dislodge the particles, since such action will spring and tear the fine mesh from the supporting grid. Micromesh sieves are not intended for bulk preparations or approximate analysis because of their size and construction.

If a sieve does become damaged, repair it with an epoxy cement applied with the point of a fine needle. Use a low-power magnifier to assist in these repairs.

PRECISION

Based on the mean range for 10 duplicate determinations at 10 levels, the estimated standard deviation (esd) is 0.33%.

Duplicate results on any sieve or the pan, expressed as percent of the summation of the fractions, by the same operator should be considered suspect if they differ by more than 1.1% (95% probability).

TIME FOR ANALYSIS

Elapsed time per test is 1.35 hours. Man-hours per test is 1.19.

REFERENCES

- Daeschner, H. W., Siebert, E. E., and Peters, E. D., "Application of Electroformed Precision Micromesh Sieves to the Determination of Particle Size Distribution", *ASTM Special Technical Publication No. 234*, 27, American Society for Testing and Materials, Philadelphia, Pa. (1959)
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- Anon., "Method EMS 5Z2/61, Particle Size Distribution of Cracking Catalysts, Micromesh Sieve Method," *Shell Emeryville Method Series*, Analytical Department, Shell Development Co., Emeryville, Calif.