Designation: D5757 – 22

Standard Test Method for Determination of Attrition of FCC Catalysts by Air Jets[1]

This standard is issued under the fixed designation D5757; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the relative attrition characteristics of FCC catalysts by means of air jet attrition. Other fine powder catalysts can be analyzed by this test method, but the precision of this test method has been determined only for FCC catalysts. It is applicable to spherically or irregularly shaped particles which range in size between 10 and 180 µm, have skeletal densities between 2.4 and 3.0 g/cm^3 (2400 and 3000 kg/m^3) (see IEEE/ASTM SI-10) and are insoluble in water. Particles less than 20 µm are considered fines. (See Terminology D3766.)

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3766 Terminology Relating to Catalysts and Catalysis
E105 Guide for Probability Sampling of Materials
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Air Jet Index (AJI), n*—a unitless value numerically equal to the percent attrition loss at 5 h.

4. Summary of Test Method

4.1 A sample of dried powder is humidified and attrited by means of three high velocity jets of humidified air. The fines are continuously removed from the attrition zone by elutriation into a fines collection assembly.

4.2 The AJI is calculated from the elutriated fines to give a relative estimate of the attrition resistance of the powdered catalyst as may be observed in commercial use.

5. Significance and Use

5.1 This test method is intended to provide information concerning the ability of a powdered catalyst to resist particle size reduction during use in a fluidized environment.

5.2 This test method is suitable for specification acceptance, manufacturing control, and research and development purposes.

6. Apparatus

6.1 The air jet attrition system consists of the following:

6.1.1 *Attrition Tube,* a stainless steel tube 710 mm long with a 35 mm inside diameter.

NOTE 1—NPS 1¼ in. pipe, Schedule 40 has the appropriate inside diameter.

6.1.2 *Three 2 mm Long Drilled Sapphire Square Edged Nozzles,* precision drilled 0.381 ± 0.005 mm in diameter. They are mounted equidistant from each other, 10 mm from center and flush with the top surface in a circular orifice plate 6.4 mm thick. The plate is designed to be attached to the bottom of the vertical attrition tube within an air delivery manifold.

6.1.3 *Settling Chamber,* a 300 mm long cylinder with a 110 mm inside diameter and with conical ends reducing to 30 mm inside diameter. The upper cone is approximately

[1] This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.02 on Physical-Mechanical Properties.

Current edition approved Oct. 1, 2022. Published October 2022. Originally approved in 1995. Last previous edition approved in 2017 as D5757 – 11(2017). DOI: 10.1520/D5757-22.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

100 mm long and the lower cone is approximately 230 mm long. The chamber is mounted to the top of the attrition tube.

6.1.4 *Fines Collection Assembly,* made up of a 250 mL filtering flask, an extraction thimble connected to the side arms of the flask, and a 200 by 13 mm diameter metal tubing bent to an angle of 125° connecting the top of the flask to the top of the settling chamber.

NOTE 2—The flask may be eliminated and the thimble connected directly to the tubing if the attrition is expected to be low enough to avoid clogging the thimble and creating a backpressure in the settling chamber.

6.1.5 *Rubber Couplers and Seals,* appropriately sized to ensure tight and leak free connections of the system.

6.2 *Air Supply Source,* controlled and capable of maintaining an air flow rate of 10.00 L/min stable to 0.05 L/min at a pressure up to 200 kPa. The air must be at a relative humidity of 30 to 40 % to minimize electrostatic effects.

NOTE 3—The air may be bubbled through a 0.25 m column of deionized water at ambient temperature to obtain the required humidity.

6.3 *Diaphragm-Type Test Meter (Dry Test Meter) or Liquid-Sealed Rotating Drum Meter (Wet Test Meter),* minimum capacity of 30 L/min and maximum scale subdivision of 0.1 L or electronic mass flow controllers may be used.

6.4 *Balance,* 400 g capacity open pan with 0.01 g sensitivity.

6.5 *Desiccator,* with a desiccant grade molecular sieve such as 4A.

6.6 *Muffle Furnace.*

6.7 *Relative Humidity Gage.*

7. Sampling

7.1 Obtain a representative sample of approximately 65 g of material from larger composites by riffling or splitting in accordance with subsection 5.12 of STP 447A[3] or some other suitable means with the aim of obtaining a sample that represents the size distribution of the larger composite. The analyst should also consult Guide E105 to help develop a sampling plan.

7.2 Gently screen the sample through a No. 80 (180 µm) ASTM sieve to remove any particles larger than 180 µm.

7.3 The step in 7.3.1 is followed for all samples except fresh FCC catalysts for proper equilibration at 35 % humidity to avoid absorption of water during the test.

7.3.1 Transfer the presieved sample to a shallow wide dish and place in a humidifier over a saturated calcium chloride solution for 16 h.

7.4 The steps in 7.4.1 – 7.4.4 are followed for fresh FCC catalysts to ensure a proper moisture level that will not change during the test.

7.4.1 Transfer the presieved sample to a shallow dish and dry it for 1 h in a muffle furnace preheated to 565 °C.

7.4.2 Cool the sample to room temperature in a desiccator.

7.4.3 Mix 45 g of the dried and cooled material thoroughly with 5 g of demineralized water ensuring that the water is well dispersed and that there are no lumps of material present.

7.4.4 Allow the sample to stand over a saturated calcium chloride solution in a humidifier for 1 h.

8. Preparation of Apparatus

8.1 Thoroughly clean any residual material from the apparatus by tapping it loose and blowing or vacuuming the dust. Reassemble the system except for the fines collection assembly.

8.2 Turn on the air supply and adjust the relative humidity of the air exiting the settling chamber to a range of 30 to 40 %.

8.3 Connect the inlet of the wet test gas meter to the top of the settling chamber and adjust the humidified air flow to 10.00 ± 0.05 L/min at standard temperature and pressure (STP) (273.15 K and 101.325 kPa). The back pressure should be in the range of 130 to 180 kPa; if it is not, check that the air jet nozzles are clean and within specifications and that there are no leaks in the apparatus connections.

NOTE 4—The back pressure of 130 to 180 kPa should be determined by a gauge or transducer installed relatively near the chamber.

8.4 Prepare two fines collection assemblies and condition the thimbles by installing them on the apparatus in succession and passing the humidified air through the apparatus for 30 min each.

9. Procedure

9.1 Weigh the first conditioned fines collection assembly to the nearest 0.01 g and record its mass.

9.2 With the air flowing at the prescribed 10.00 L/min and the fines collection assembly off, charge 50 g of water equilibrated sample to the apparatus through the top of the settling chamber, quickly secure the first fines collection assembly to the apparatus and start the timekeeping.

9.3 Weigh the second conditioned fines collection assembly to the nearest 0.01 g and record its mass.

9.4 After exactly 1 h from the start, replace the first fines collection assembly with the second one. Weigh and record the mass of the first fines collection assembly.

9.5 After exactly 5 h from the start, remove the fines collection assembly, weigh it, and record its mass.

9.6 Turn off the apparatus and disassemble.

9.7 Recover the sample from the attrition tube and settling chamber and weigh to the nearest 0.01 g.

9.8 Clean the apparatus.

10. Calculations

10.1 Calculate the percent fines lost in the first hour as follows:

$$\text{fines loss, \%} = (m_1 - m_0)/m_s \times 100 \quad (1)$$

where:

m_0 = mass of the first empty fines collection assembly at the start of the test, g

[3] STP 447A, *Manual on Test Sieving Methods*, ASTM International, West

m_1 = mass of the first fines collection assembly at 1 h, g, and
m_s = mass of the sample charged to apparatus (nominally 50 g).

10.2 Calculate the percent fines lost from attrition at the fifth hour as follows:

$$\text{attrition loss, } \% = (m_1 - m_0 + m_5 - m'_0)/m_s \times 100 \quad (2)$$

where:

m'_0 = mass of the second empty fines collection assembly, g, and
m_5 = mass of the second fines collection assembly at 5 h, g.

10.3 Calculate the percent sample recovery after the test as follows:

$$\text{recovery, } \% = (m_1 + m_5 + m_r - m_0 - m'_0)/m_s \times 100 \quad (3)$$

where:

m_r = mass of the sample recovered from the attrition tube and the settling chamber.

11. Report

11.1 Report the following information:
11.1.1 The AJI, that is, the percent attrition loss at 5 h.
11.1.2 The first hour fines loss as percent.
11.1.3 The recovery as percent.

12. Precision and Bias

12.1 *Test Program*—An interlaboratory study was conducted in which the named property was measured in four separate test materials in four to seven separate laboratories. Practice E691, modified for nonuniform data sets, was followed for the data reduction. Analysis details are in the research report.

TABLE 1 Repeatability and Reproducibility

Test Result–AJI (consensus mean)	95 % Repeatability Interval (within laboratory)	95 % Reproducibility Interval (between laboratories)
2.530	0.226 (8.95 % of mean)	0.907 (35.9 % of mean)
4.209	0.279 (6.64 % of mean)	1.939 (46.1 % of mean)
20.353	1.121 (5.51 % of mean)	11.370 (55.9 % of mean)
39.945	1.414 (3.54 % of mean)	12.029 (30.1 % of mean)

12.2 *Precision*—Pairs of test results obtained by a procedure similar to that described in the study are expected to differ in absolute value by less than 2.772 S, where 2.772 S is the 95 % probability interval limit on the difference between two test results, and S is the appropriate estimate of standard deviation. Definitions and usage are given in Practices E456 and E177, respectively. See Table 1.

12.3 *Bias*—The procedure in this test method for measuring attrition has no known bias because the value of the attrition loss is defined only in terms of this test method.

13. Keywords

13.1 abrasion; air jet; attrition; fines; powdered catalysts