Designation: D8414/D8414M – 21

Standard Test Method for Measurement of Jet Cup Attrition Index of Catalytic Materials[1]

This standard is issued under the fixed designation D8414/D8414M; 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 The jet cup attrition test is applicable to fluid catalytic cracking (FCC) catalysts, catalyst additives, and catalytic materials.

1.2 Applications for other powdered catalysts have been reported in the literature. The round robin test samples included two FCC catalysts and one powdered alumina.

1.3 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.4 *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.*

1.5 *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]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *control factor, CF, n*—unitless value determined by the repetitive analysis of a standard material; it is equal to the (initial value of the control standard)/ (current value of the control standard) = CF.

3.1.1.1 *Discussion*—Refer to 12.1 for details on the standard.

3.1.1.2 *Discussion*—The values used in the calculations are monthly averages.

3.1.2 *jet cup index, JCI, n*—unitless value equal to the "weight percent of 0- to 20-µm [0- to 787-µin.] fines generated per hour" under test conditions.

4. Summary of Test Method

4.1 Of the sample to be tested, 5 g [0.175 oz] is placed into the jet cup of the test apparatus where humidified air is passed through an orifice in the jet cup at high velocity.

4.2 The air impinges on the catalyst and causes particles to collide with each other and the walls of the jet cup. These particle-to-particle and particle-to-wall interactions cause fines to form via attrition. In addition, weaker particles will fracture and form fines.

4.3 The jet cup is attached to a fines disengagement chamber that allows particles less than 20 µm [787-µin.] to pass into a fines collection assembly.

NOTE 1—The fines disengagement chamber is based on the AMINCO-Roller air elutriation particle size analyzer, historically used to measure the 0-20 micron fraction in catalyst.

4.4 The JCI is calculated from the mass of fines collected and is numerically equivalent to the "weight percent of fines generated per hour" under test conditions.

5. Significance and Use

5.1 The jet cup attrition test will provide an estimate of the relative attrition resistance of fluid catalytic cracking (FCC) catalyst, catalyst additives, and catalytic materials.

5.2 The test is designed to simulate the attrition a catalyst or additive undergoes in a fluid catalytic cracking unit but at an accelerated rate.

[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.

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[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.

5.3 The data from this test can be used to rank catalyst according to attrition rate.

5.4 The test requires a relatively small sample size of 5 g [0.175 oz] and a relatively short analysis time of 40 min. This test should be useful to quality control facilities that require fast turnaround time and research and development (R&D) facilities that have limited sample material.

6. Interferences

6.1 There are no known interferences. Factors such as density, particle size distribution, morphology, and intrinsic hardness will affect the results. These and other factors must be considered.

7. Apparatus

7.1 *Pressure Regulator,* to reduce the Instrument grade air supply pressure to 206.8 kPa [30 psig].

7.2 *Mass Flow Controller,* capable of delivering 21.0 ± 0.05 slpm of air into a system with 34 kPa [5 psig] of backpressure. Mass flow controller should not be affected by small amounts of moisture in the air supply, changes in room temperature, or changes in barometric pressure.

7.3 *Humidification Equipment,* capable of humidifying 21 slpm of air to 55 % relative humidity (RH) in a continuous, uninterrupted operation. De-ionized water addition, 16 meg-Ω minimum, can be automatic or manual. The humidifier should have a 70-kPa [10-psi] pressure-relief valve installed.

7.4 *Backpressure Gauge,* 0-100 kPa [0-15 psig].

7.5 *Three-way Air Solenoid Valve.*

7.6 *Timer.*

7.7 *Jet Cup*—See Fig. A1.1 in Annex A1.

7.8 *Fines Disengagement Chamber*—See Fig. A1.2 in Annex A1. Can be welded steel or solid 2-piece with or without a fines release coating.

7.9 *Connector,* to attach the jet cup to the fines disengagement chamber. Commercially available equipment has the jet cup permanently mounted into a holder that forms a positive connection with the fines disengagement chamber. Lab built units use a machined plastic connector with o-rings to make a leak tight seal. The connector can be as simple as a piece of large diameter Tygon®[3] tubing and two hose clamps.

7.10 *Rack or Mounting Brackets,* to support the fines disengagement chamber in an upright position.

7.11 *Tubing,* pre-cleaned stainless steel or food grade tubing.

7.12 *Assorted Tubing Fittings.*

7.13 *Pipe Elbows,* 15 mm [½ in.] NPT × 10 mm [⅜ in.] Barb. Light weight plastic.

7.14 *Rubber Stoppers,* number 5.5 with a hole bored with a #7 cork borer.

7.15 *Ancillary Equipment:*

7.15.1 *Analytical Balance,* 4-place, readable to 0.0001 g, with a minimum range of 30 gms.

7.15.2 *Weigh Boats,* small, disposable plastic.

7.15.3 *Desiccator,* with desiccant.

7.15.4 *Humidity Probe,* with calibration traceable to the National Institutes of Standards and Technology (NIST).

7.15.5 *Oven,* 225 °C [437 °F], vented with 1 CFH (0.5 lpm) air flow.

7.15.6 *Furnace,* muffle, 565 °C [1049 °F], vented with 1 CFH (0.5 lpm) air flow.

7.15.7 *Assorted Borosilicate Glass or Ceramic Dishes and Crucibles.*

8. Reagents and Materials

8.1 *Instrument Grade Air.*

8.2 *De-ionized Water,* 16 meg-Ω, minimum.

8.3 *Collection Thimbles,* 19 by 90 mm [0.7 by 0.4 in.], Whatman #2814199®, or equivalent.[4]

9. Assembling the Apparatus

9.1 Refer to the block diagram, Fig. A1.3, in Annex A1.

9.2 Attach the shutoff valve and the pressure regulator to the source of instrument grade air. No air should be flowing at this time.

9.3 Attach the inlet of the mass flow controller to the exit of the pressure regulator.

9.4 The exit of the mass flow controller is attached to the inlet of the humidifier. The humidifier should have a 70-kPa [10-psi] pressure-relief valve installed. If not, install a tee fitting and a pressure-relief valve at the exit of the humidifier.

9.5 Install a tee fitting and the backpressure gauge, 0-100 kPa [0-15 psig].

9.6 Next, install the three-way air solenoid valve controlled by a timer. The inlet of the valve is attached to the tee fitting used to mount the backpressure gauge. With the timer deactivated, air should be flowing through the vent. Wire the solenoid to a timer capable of timing 20.0 min. Attach a 10-cm [4-in.] length of 1.6-mm [1⁄16-in.] outer diameter (OD) tubing to the vent.

9.7 Attach a hose to the exit of the solenoid valve. Attach the other end of the hose to the jet cup. Keep the hose as short as possible, usually less than 1 m [3 ft].

9.8 Mount the fines disengagement chamber in an upright position.

9.9 Assemble the thimble holders (14.3).

9.9.1 Insert the barb end of the pipe elbow into the rubber stopper.

9.9.2 Prepare a minimum of two thimble holders. Four or more will speed up analysis time.

[3] Tygon® a registered trademark of Saint-Gobain Corporation, 20 Moores Rd, Malvern, PA 19355.

[4] Whatman® is a registered trademark of Cytiva, 100 Results Wy, Marlborough, MA 01752.

10. Hazards

10.1 The main hazard in this test is the formation of respirable fines. All cleaning of the jet cup, fines disengagement chamber, thimbles, and thimble holders should be performed either in a hood rated for this type usage or with a high-efficiency particulate air (HEPA) vacuum cleaner.

11. Sampling

11.1 This test uses 5-g [0.175-oz] aliquots of catalyst material for testing. The main source of error in this test occurs in the sampling process. Fluid catalytic cracking catalyst, for example, is a non-homogeneous material in that it has a particle-size distribution.

11.2 Riffle the sample to obtain a 100-g [3.5-oz.] subsample. If a riffler is not available, use the following procedure:

11.2.1 Gently mix the original sample container to blend the sample. This blending shall not cause the sample to attrite or form fines, or both.

11.2.2 Remove a 100-g [3.5-oz] aliquot and place in a borosilicate glass or ceramic dish sized to provide a 1.2-cm [½-in.] bed depth.

11.3 If 100 g [3.5-oz] of material is not available, size the dish to maintain a 1.2-cm [½-in.] bed depth. Small aliquots such as 6 g [0.2 oz] are generally placed into a crucible.

11.4 Dry the material for 1 h at 225 °C [437 °F] followed by 2 h at 565 °C [1049 °F].

11.5 Cool the material in a desiccator. Place in a 125-ml [4-oz] glass bottle when cool.

11.6 Store the dried material in a desiccator.

11.7 Material not analyzed in 24 h requires reheating for 1 h at 565 °C [1049 °F].

12. Preparation of Apparatus

12.1 Turn on the instrument grade air. Set the mass flow controller to 21.0 slpm.

12.2 Air should be flowing through the vent of the solenoid valve. Verify the backpressure is between 28 to 34 kPa [4 to 5 psig]. If not, adjust the length of the tubing attached to the vent to obtain the proper backpressure.

12.3 Set the timer for 20.0 min. Depress the start button on the timer. The solenoid should switch and air should be flowing through the jet cup. The backpressure should be between 28 to 34 kPa [4 to 5 psig].

12.4 *Pressure Testing the System (see Note 2):*

12.4.1 Reset the timer so that air is flowing through the vent.

12.4.2 Insert a plug into the exit of the fines disengagement chamber. The plug can be made by screwing a large bolt into a one-hole stopper. Attach a safety cord to the bolt and the frame of the unit.

12.4.3 Reduce the mass flow controller setting to zero. Verify the flow reading is zero. The mass flow controller should read zero and the backpressure gauge should read zero.

12.4.4 Depress the timer so that air is directed through the jet cup.

12.4.5 Slowly increase the flow on the mass flow controller until the backpressure gauge reads 41 kPa [6 psig]. Above 48 kPa [7 psig], the plug will blow out of the chamber. Shut off the air flow at 41 kPa [6 psig].

12.4.6 Allow the pressure to stabilize and then begin taking readings on the backpressure gauge. A leakage rate of less than 0.7 kPa [0.1 psig] per minute is allowable.

12.4.7 Above that level, snoop the connections with a leak detector solution. Do not forget the rubber stopper in the top of the fines disengagement chamber. If your chamber is welded steel, snoop the welds.

12.4.8 If the leakage rate is still unacceptable, isolate sections of the flow assembly until the leak is found.

12.4.9 Once the leakage rate is below the acceptable level, slowly and carefully remove the plug from the top of the chamber.

12.4.10 Reset the timer.

12.4.11 Turn on the air supply and reset the mass flow controller to 21.0 slpm.

12.5 *Adjusting the Humidification:*

12.5.1 Turn on the power to the humidifier.

12.5.2 Adjust the set point to 55 % RH.

12.5.3 Wait until the humidity gauge has stabilized near 55 % RH.

12.5.4 Depress the timer to direct the airflow through the jet cup and into the fines disengagement chamber.

12.5.5 Insert a National Institute of Standards and Technology (NIST) traceable humidity probe into the center of the fines disengagement chamber.

12.5.6 Begin recording the % RH every 30 sec. Adjust the set point on the humidifier until a constant reading of 50 % RH is obtained at the fines disengagement chamber.

12.5.7 Remove the humidity probe and reset the timer.

NOTE 2—The pressure test is only run on initial startup of the system and whenever the control chart indicates there may be a problem with the system.

13. Calibration and Standardization

13.1 *Establishing a Control Standard and a Control Chart:*

13.1.1 Select a fresh fluid catalytic cracking (FCC) catalyst with a normal attrition rate of 3 to 6 %. Maintain a sufficient quantity for at least 300 sample preparations and analyses.

13.1.2 Dry 100 g [3.5-oz] of the material as directed in Section 11.

13.1.3 Perform a minimum of seven analyses of the control standard as directed in Section 13. Record these values to two decimal places. Calculate the average and the Relative Standard Deviation (RSD) for the JCI values. (See Note 3.)

13.1.4 Prepare a control chart with the average value from 13.1.3 as the center line. Draw in the 2-sigma and 3-sigma lines.

13.1.5 The control standard is analyzed daily and plotted on the chart. Any result outside 3-sigma is repeated immediately with a freshly prepared control standard. Whenever there are two consecutive values beyond 2-sigma, the test is repeated with a freshly prepared control standard.

13.2 *Calculating the Control Factor (CF):*

13.2.1 The initial CF is set at 1.000.

13.2.2 For example, let us say that the seven analyses had an average JCI of 5.00.

13.2.3 Monthly, all of the data on the control chart is averaged and the 1-sigma standard deviation is calculated for the monthly average. One way to record this data is with an Excel spreadsheet. It is easy to obtain the average and Relative Standard Deviation (RSD) needed to construct a new control chart. Control Charting software is also available. Let us say that the average JCI for the month was 4.95.

13.2.4 The CF is calculated:

$$CF = (\text{JCI-Initial}) / (\text{JCI-Monthly Average})$$
$$CF = 5.00 / 4.95$$
$$CF = 1.010,\ \text{for this example}$$

13.2.5 The CF corrects for wear in the jet cup but will also correct for long-term drift in the mass flow controller and other system components.

13.2.6 The CF allows you to replace a jet cup and correct the data back to the original jet cup.

13.2.7 The control chart will indicate when the process is "in control" and when there are operational problems, an "out-of-control" situation.

13.2.8 Correct the out-of-control situation before proceeding with any future analyses. A good place to start is to verify the operating conditions and performing a pressure test.

13.2.9 An example of a control chart, produced from actual Jet Cup lab data, is included in Annex A1 as Fig. A1.4.

NOTE 3—For a new system or a new Jet Cup, analyze three samples to "break in" the jet cup before recording any data.

14. Procedure—Analyzing a Sample

14.1 Verify the operating conditions as given in Section 11:

14.1.1 Mass flow controller is set to 21.0 slpm.

14.1.2 Humidifier is set to deliver 50 % RH at the fines disengagement chamber.

14.1.3 Timer is set for 20.0 min.

14.1.4 Timer is reset and air is flowing through the vent.

14.2 Prepare the sample according to Section 11.

14.3 Prepare two fines collection thimbles by threading the collection thimble onto the pipe threads of the NPT fitting. Gently press the open end of the thimble against the pipe fitting until it flares open. Keep a gentle pressure, pushing the thimble against the pipe fitting and rotate it to feed the thimble onto the fitting. Label the two thimble assemblies "A" and "B."

14.4 Tare the balance and obtain and record the initial weights of Thimble assemblies "A" and "B" as Aw-initial and Bw-initial, to the nearest 0.0001 g.

14.5 Select the sample to be analyzed. It should be in a 125 ml [4 oz] jar. Tumble the jar end over end to homogenize the sample.

14.6 Tare the balance. Obtain the tare weight for a weigh boat.

14.7 Add approximately 5.05 g [0.178 oz] of the catalyst sample to the weigh boat. Record the weight of the sample and weigh boat as Sw-initial, to the nearest 0.0001 g.

14.8 Be certain air is not flowing through the jet cup. Transfer the sample to the jet cup. A small amount of sample, approximately 0.05 g [0.02 oz] will remain in the weigh boat. That is acceptable.

14.9 Reweigh the weigh boat and record as Sw-final, to the nearest 0.0001 g.

14.10 Attach the jet cup to the fines disengagement chamber.

14.11 Attach Thimble A to the exit of the fines disengagement chamber.

14.12 Depress the start button.

14.13 After 20 min, the timer will reset. Remove Thimble A and replace with Thimble B.

14.14 Depress the start button.

14.15 After 20 min, the timer will reset. Remove Thimble B.

14.16 Reweigh Thimbles A and B and record the weights as Aw-final and Bw-final, to the nearest 0.0001 g.

14.17 Remove the jet cup, fines disengagement chamber, and the thimbles and holders to a hood.

14.18 Clean using a regulated air line or a HEPA vacuum cleaner or a combination (see Note 4).

14.18.1 An air line with 100 kPa [15 psig] pressure and a nozzle with a trigger is recommended. The nozzle should be fitted with a 30 cm [12 in.] length of plastic tubing. If you use metal tubing, cover the end with Tygon®[3] to prevent scratching the inside of the fines disengagement chamber.

14.18.2 In a hood, transfer the contents of the Jet Cup into a waste container. Vacuum out the remaining catalyst or alternately blow out the catalyst residue with the air line. Direct the air flow from the air nozzle through the orifice of the Jet Cup to be sure it is free of catalyst residue.

14.18.3 Attach the hose from the HEPA vacuum cleaner to the inlet (jet cup connector) of the fines disengagement chamber.

14.18.4 Turn on the vacuum cleaner and insert the air line into the other end of the chamber. Turn on the air and use the air flow to sweep the fines into the vacuum cleaner. Slowly move the length of tubing into the chamber, directing the air flow at the walls of the chamber.

14.18.5 Turn off the vacuum cleaner and remove the hose.

14.18.6 Do not beat on the chamber to remove fines. This will damage the welds in welded steel chambers and cause leaks. (See Note 5.)

14.19 Reinstall the jet cup and fines disengagement chamber. Reload the thimble holders with fresh thimbles.

NOTE 4—The recommended cleaning procedure is designed to minimize the amount of catalyst powder placed into the air. Any cleaning procedure that removes all of the catalyst powder from the Jet Cup and Chamber without damage and meets your safety requirements is acceptable.

NOTE 5—Leaks in welded steel chambers are best repaired with epoxy resin. Re-welding causes spalling on the inside of the chamber. This traps fines and renders the chamber useless.

15. Calculation

15.1 Thimble A is for information purposes only (see the addendum for additional information).

15.2 Calculate the mass of fines in Thimble A:

$$A\ Yield = Aw\text{-}final - Aw\text{-}initial \quad (1)$$

15.3 The JCI is calculated from Thimble B data:

$$JCI = \left[\frac{((CF) \quad (Bw\text{-}final - Bw\text{-}initial) \times 3 \times 100)}{(Sw\text{-}final - Sw\text{-}initial)} \right] \quad (2)$$

NOTE 6—The initial CF = 1.000.

16. Report

16.1 Report the mass of fines in Thimble A as "A Yield."

16.2 Report the JCI.

16.3 Report any deviations from the standard procedure.

17. Precision and Bias

17.1 The precision of this test method is based on an inter-laboratory study of Test Method D8414/D8414M conducted in 2020. Each of ten volunteer laboratories were asked to test three different materials. Every "test result" represents an individual determination, and all participants were instructed to report two replicate test results for each material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D32-2001.[5]

17.1.1 *Repeatability Limit (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

17.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

17.1.1.2 Repeatability limits are listed in Table 1.

17.1.2 *Reproducibility Limit (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

17.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

17.1.2.2 Reproducibility limits are listed in Table 1.

17.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

17.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

17.3 The precision statement was determined through statistical examination of 59 results, from 10 laboratories, on 3 materials.

18. Keywords

18.1 attrition; catalyst additives; catalytic materials; FCC catalyst; fluid catalytic cracking catalyst; JCI; jet cup; jet cup index; powdered catalyst

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D32-2001. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Jet Cup Attrition Index (Weight Percent of Fines Generated per Hour Under Test Conditions)

Material	Number of Laboratories	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	n	$\bar{x}$	s_r	s_R	r	R
Sample 1	10	3.129618	0.191396	0.357341	0.535908	1.000556
Sample 2	10	9.942578	0.233231	0.652281	0.653046	1.826386
Sample 5	10	14.972884	0.292734	1.806104	0.819655	5.057090

[A] The average of the laboratories' calculated averages.