



Standard Test Method for Testing Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test¹

This standard is issued under the fixed designation D 3907; 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 determining the activity of equilibrium or laboratory-deactivated fluid catalytic cracking (FCC) catalysts, or both. This is evaluated on the basis of weight percent conversion of gas oil in a microactivity unit. The selectivity of FCC catalysts can be determined using Test Method D 5154.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography

D 5154 Test Method for Determining the Activity and Selectivity of Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test³

E 105 Practice for Probability Sampling of Materials

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 456 Terminology Relating to Quality and Statistics

E 691 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:

¹ This test method is under the jurisdiction of ASTM Committee D32 on Catalysis and is the direct responsibility of Subcommittee D32.04 on Catalytic 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.

³ Withdrawn.

3.1.1 *ASTM consensus mean conversion*—each reference catalyst has a consensus mean conversion value assigned to it by Committee D32 (see 11.2).

3.1.2 *ASTM reference catalysts*—a set of equilibrium fluid cracking catalysts with conversions within the useful range of this test method is used to improve the reproducibility of test results between different laboratories. Samples of the ASTM reference catalysts can be obtained from NIST.

3.1.3 *ASTM standard feed*—a specific batch of gas oil that is used as feedstock in the described test method. This standard feed can be obtained from the National Institute of Standards and Technology (NIST).⁴

3.1.4 *conversion calibration curve*—a calibration curve can be obtained by plotting the consensus mean conversion values for the ASTM reference catalysts (see 11.2) versus the individual laboratory-measured conversion for the same catalysts.

3.1.5 *measured conversion*—is calculated as the difference between the weight of feed used and the weight of unconverted material, divided by the weight of feed used, times 100 %. The unconverted material is defined as all liquid product with a boiling point above 216°C (421°F).

4. Summary of Test Method

4.1 A sample of cracking catalyst in a fixed-bed reactor is contacted with gas oil (ASTM standard feed). Cracked liquid products are analyzed for unconverted material and the conversion is calculated.

4.2 A corrected conversion value can be obtained from the measured conversion and the conversion calibration curve.

5. Significance and Use

5.1 The microactivity test provides data to assess the relative performance of FCC catalyst. Because results are affected by catalyst pretreatment, feedstock characteristics, test equipment, and operating parameters, adherence to this test method is a prerequisite for correct interpretation of results. Apparatus, test conditions, and analytical procedures actually used should closely resemble those described in this test method.

5.2 Caution should be used in interpreting results above 80 weight % conversion due to the significance of overcracking.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

6. Apparatus

6.1 *Flow Chart*—The flow chart is given in Fig. 1. During 75 s, gas oil from a syringe is forced over 4 g of catalyst in a fixed-bed reactor. Liquid products are collected in a receiver and kept at a wet ice temperature.

6.2 *Syringe*—A syringe with 2.5 mL capacity is used for oil addition. It should be equipped with a multiport, high-pressure valve to allow nitrogen and oil entry to the reactor through a common feed line.

6.3 *Syringe Heater*—Heat syringe to $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$) using a heat lamp or resistance heater or any other suitable means.

6.4 *Syringe Pump*—A syringe pump that can deliver uniform flow of 1.33 ± 0.03 g of gas oil in 75 ± 1 s.

6.5 *Furnace*—A three-zone furnace is used: middle zone, 150-mm (6-in.) length, and top and bottom zones, 75-mm (3-in.) length. The catalyst bed is positioned in the middle zone. The temperature controllers of the three zones are calibrated to achieve a constant temperature $482 \pm 1^\circ\text{C}$ ($900 \pm 2^\circ\text{F}$) over the whole length of the catalyst bed (actual bed temperature).

6.6 *Reactor and Insert*—A glass or stainless steel reactor of 15.6 mm internal diameter is used. Dimensions are shown in Fig. 2. Details of the reactor insert are shown in Figs. 2 and 3.

NOTE 1—General dimensions are given in SI units. Dimensions given in SAE, U.S. Standard gage sizes for sheet, tubing, and wire are considered standard. In general, the closest metric equivalent should be

adequate for proper functioning.

6.7 *Liquid Product Collection System*—Liquid product is collected in the receiver shown in Fig. 4.

6.8 *Analytical Balance and Weights*—The balance used to weigh the sample, the receiver, and the syringe shall have a precision of 1 mg. Analytical weights shall be precision grade or calibrated against a set of certified standard weights.

6.9 *Chromatographic Equipment*—The gas chromatographic equipment specified in Test Method D 2887 is suggested for the analysis of liquid product. A flame ionization detector is recommended.

7. Sampling

7.1 If a sampling procedure is desired, Practice E 105 is recommended.

8. Sample Preparation

8.1 Dry samples or decoke, or both, by heating a shallow (less than 10 mm thick) bed of catalyst in a porcelain crucible as follows:

$120 \pm 20^\circ\text{C}$ ($248 \pm 36^\circ\text{F}$) for 1 h
 120°C (248°F) to 590°C (1094°F) for approximately 1 h
 $590 \pm 20^\circ\text{C}$ ($1094 \pm 36^\circ\text{F}$) for 3 h

8.2 Sufficient air should be available in the furnace to burn the sample free of coke. Insufficient decoking is indicated by a difference in color of the top and bottom layers. The hot crucible is cooled in a desiccator to prevent moisture pickup.

9. Procedure

9.1 Reactor Preparation:

9.1.1 Rinse the feed line with acetone or other suitable solvent and dry with air. Periodic cleaning of the insert is recommended by an air purge at 482°C (900°F) for 1 h, at least once every 12 tests.

9.1.2 Wash the reactor and product receiver thoroughly with acetone or other suitable solvent and dry. If necessary, burn out any coke deposited in the reactor by heating in air at 482°C (900°F) prior to washing.

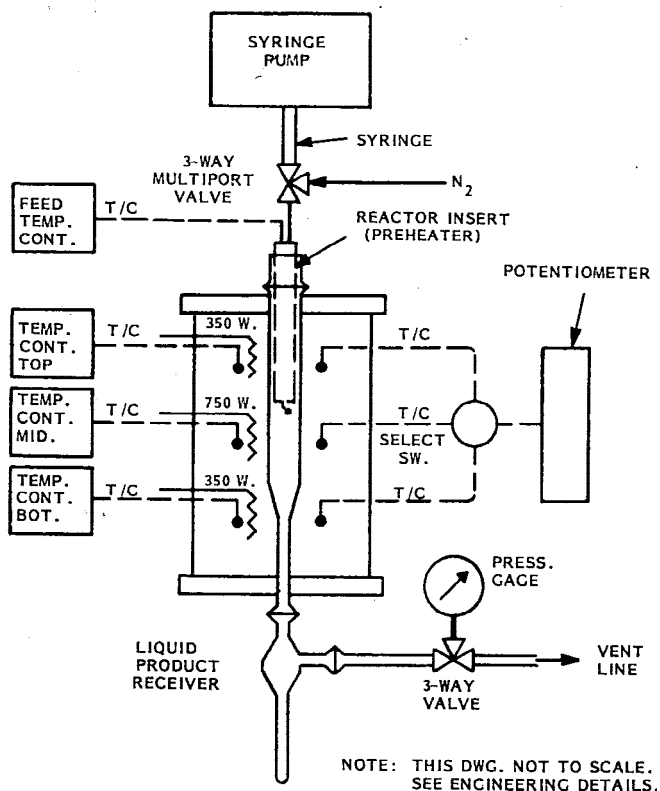
9.1.3 Insert a plug of quartz or borosilicate glass wool (about 20-mm length) into the reactor. Add 4.00 ± 0.05 g of catalyst in a free-flowing manner. Tap the reactor lightly to ensure good radial distribution (do not pack). Position another plug of quartz or glass wool (about 6-mm length) above the catalyst bed. Do not tamp wool plugs excessively.

9.1.4 Inspect the reactor feed tube insert to be sure it is free of deposits and the tip of the thermocouple (see Fig. 3, Detail 2) is bent under the tip of the syringe needle. (This is necessary to control the oil preheat temperature accurately.) Place the insert in the reactor and adjust, if necessary, so that the insert needle is between 10 to 50 mm above the catalyst bed. Place the reactor in the furnace that has been preheated to 482°C (900°F) and connect the nitrogen purge line directly to the reactor feed line. Purge with 30-mL/min of nitrogen for at least 30 min.

9.1.5 Make electrical connections on the integral oil feed preheater and connect the thermocouple to the recorder.

9.2 Preparation of Syringe and Liquid Product Receiver:

9.2.1 Fill the syringe with ASTM standard feed and invert to allow air to rise.



NOTE—This drawing is not to scale. For engineering details, see other drawings.

FIG. 1 Microactivity Flow Chart

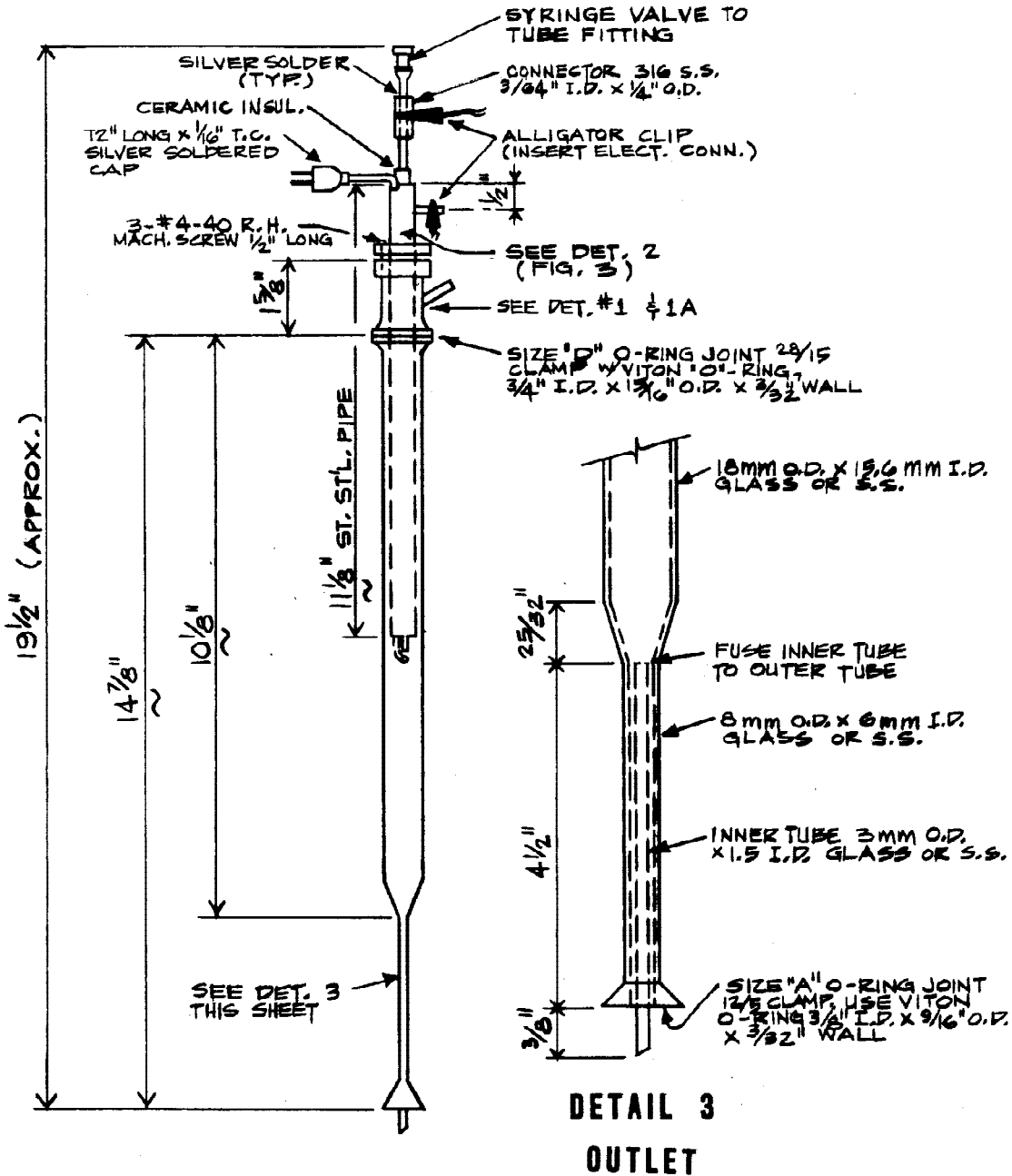


FIG. 2 Microactivity Reactor

NOTE 2—The ASTM standard feed is very viscous at 30°C; therefore, loading the syringe and removal of air bubbles can be facilitated by preheating the oil to $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$).

9.2.2 Remove the air bubbles. The syringe should contain a small amount of oil in excess of the nominal volume to be charged.

9.2.3 After the syringe is filled and the valve is in correct position, blow excess oil out of the valve and clean the outside of the syringe.

9.2.4 Weigh the syringe assembly and record the weight.

9.2.5 Disconnect the nitrogen from the reactor feed line, install the syringe, and connect the nitrogen to the syringe

valve. Make sure the valve is in the nitrogen flow position. Place a thermocouple on the syringe body to detect the syringe temperature. Set the syringe temperature at $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$).

9.2.6 Obtain the tare weight of the liquid product receiver and record.

9.2.7 Install the receiver and connect the gas line to the vent.

9.2.8 Prepare and install a wet ice bath for the liquid product receiver. Immerse the receiver to the level of the gas outlet line. Optionally, a controlled temperature circulating bath may be used in place of the wet ice bath.

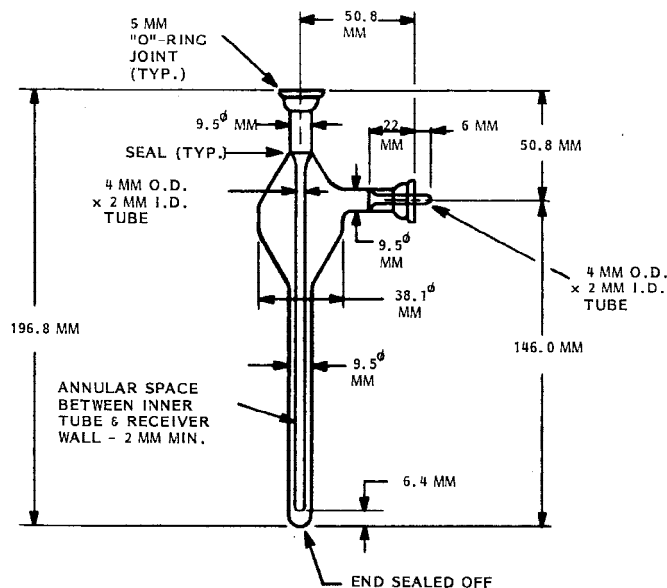


FIG. 4 Liquid Product Receiver

9.3.4 Switch the syringe multiport valve from the nitrogen flow to the oil feed position. Note that the position of the valve can be: joint-feed only, nitrogen flow, or pure-feed only.

9.3.5 Start the syringe pump and deliver 1.33 ± 0.03 g of feed over 75 ± 1 s to the reactor.

9.3.6 The oil preheat temperature, as measured by the thermocouple at the oil exit point above the bed, shall be $482 \pm 5^\circ\text{C}$ ($900 \pm 9^\circ\text{F}$).

9.3.7 Note that fine particle samples can cause excessive catalyst bed differential pressures, significantly affecting results. Back pressures up to 13.3 kPa (100 mm Hg) measured during oil addition (see Fig. 3, Detail 1A) are not detrimental. More typical back pressures are in the range from 4.0 to 8.0 kPa (30 to 60 mm Hg). If the catalyst bed differential pressure exceeds 13.3 kPa (100 mm Hg), it is recommended that the catalyst be screened free of particles finer than <325 mesh ($45 \mu\text{m}$) and the test be repeated with the larger-sized particles.

9.3.8 Immediately after the oil addition, restart the nitrogen-only flow at 30 mL/min by switching the multiport valve to purge the reactor and liquid product receiver.

9.3.9 Purge the reactor and product recovery system for 15 min at 30 mL/min nitrogen, with a wet ice bath. Optionally, a controlled temperature circulating bath may be used in place of the wet ice bath.

9.3.10 Remove the product receiver from the reactor, seal, dry, and weigh immediately. Record weight of liquid in receiver as W_r . If sample cannot be analyzed immediately, store in a sealed vial and refrigerate.

9.3.11 Collect the liquid holdup in the reactor exit line and around the reactor-receiver joint with a tared cotton swab or other suitable material. Determine the weight of this liquid and record as W_Q .

NOTE 3—Experience indicates this liquid material is predominantly unconverted material.

9.3.12 Remove the syringe from the reactor, reweigh it, and record the weight of oil feed as W_f .

9.3.13 Remove the reactor from the furnace. After cooling, the catalyst can be removed from the reactor for analysis or regeneration, or both.

9.4 Analysis of Liquid Product:

9.4.1 Analyze the liquid product by gas chromatography using a simulated distillation technique described by Test Method D 2887.

9.4.2 Use a retention time of a 216°C (421°F) material (that is, *n*-dodecane) as a standard for determining the end point of converted product.

9.4.3 Express integrated peak area of material boiling above 216°C (421°F) as a percentage of total peak area and record as *R*.

10. Calculation

10.1 Calculate the measured conversion as follows:

$$\text{Measured conversion, weight \%} = \frac{W_f - \frac{R \times W_L}{100} - W_Q}{W_f} \times 100 \quad (1)$$

where:

W_f = ASTM standard feed, g,

R = weight percentage of material boiling above 216°C (421°F) in liquid product receiver,

W_L = liquid product in receiver, g, and

W_Q = liquid holdup at reactor exit line and around reactor-receiver joint, g.

10.2 A corrected conversion value may be obtained by using the conversion calibration curve obtained in 3.1.4.

11. Precision and Bias

11.1 *Test Program*—An interlaboratory study was conducted in which the MAT conversion was measured in six separate test materials (RR-1 to RR-6) with ASTM standard feed in nine separate laboratories. Not all materials were

measured by all laboratories. Practice E 691, modified for nonuniform data sets, was followed for the data reduction.⁵

11.2 *Precision*—Pairs of test results obtained by the procedure described in the study arc 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 Terminology E 456 and Practice E 177, respectively.

RR-2	64.9	2.8	8.1
RR-3	76.1	2.6	8.7
RR-4	76.2	2.8	7.4
RR-5	80.9	2.8	7.2
RR-6	70.1	1.2	7.3

11.3 *Bias*—The procedure described is without known bias, since there is by definition no absolute standard for comparison.

ASTM Standard Sample No.	ASTM Consensus Mean Conversion, Weight %	95 % Repeatability Limit (Within-Laboratory), Weight %	95 % Reproducibility Limit (Between-Laboratories), Weight %
RR-1	59.4	2.5	5.2

12. Keywords

12.1 catalysts; FCC; fluid cracking catalyst; MAT; microactivity

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D32-1016.

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