



Designation: D3907/D3907M – 13

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

This standard is issued under the fixed designation D3907/D3907M; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 [D5154](#).

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D2887](#) Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography

[D5154](#) Test Method for Determining Activity and Selectivity of Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test

[E105](#) Practice 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

¹ This test method is under the jurisdiction of ASTM Committee [D32](#) on Catalysts 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. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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).³

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

³ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

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.

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 D2887 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 E105 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°C [248 \pm 36°F] for 1 h
- 120°C [248°F] to 590°C [1094°F] for approximately 1 h
- 590 \pm 20°C [1094 \pm 36°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

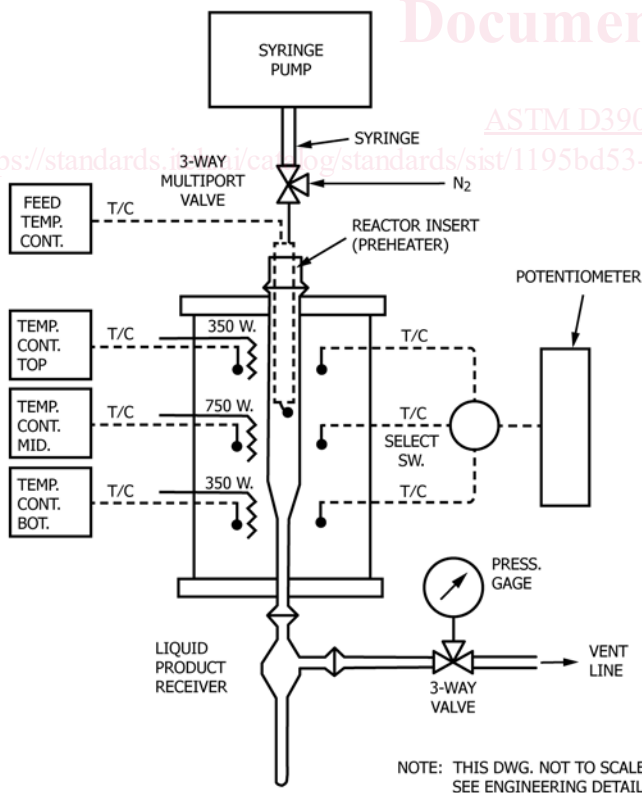
9.1 Reactor Preparation:

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



NOTE 1—This drawing is not to scale. For engineering details, see other drawings.

FIG. 1 Microactivity Flow Chart

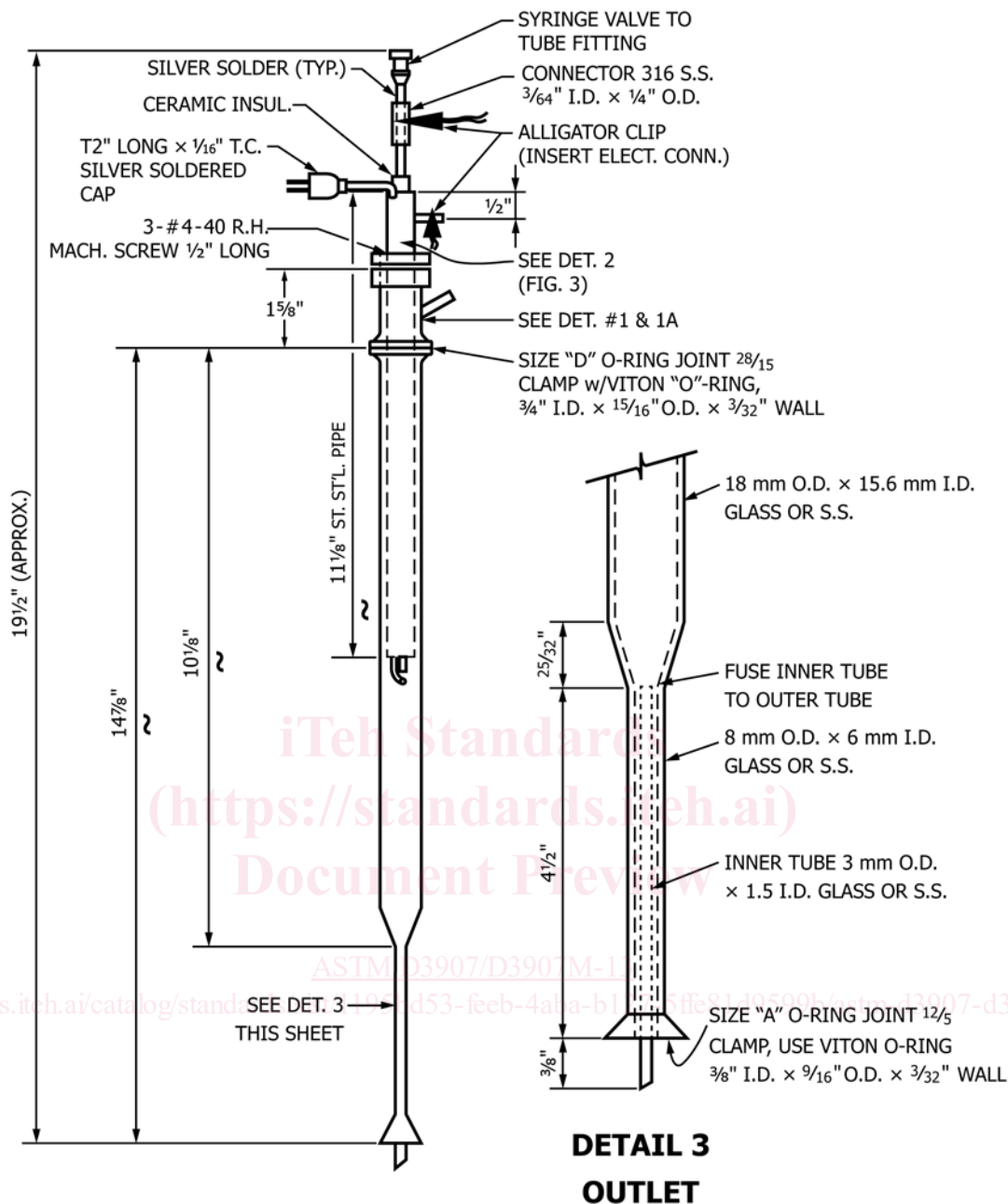


FIG. 2 Microactivity Reactor

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 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 ± 5°C [104 ± 9°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 ± 5°C [104 ± 9°F].