Designation: D3907/D3907M – 19

Standard Test Method for Testing Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test[1]

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 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 mass percent conversion of gas oil feed 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 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.

1.3 *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]

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

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[3] with conversions within the useful range of this test method is used to improve the reproducibility of test results between different laboratories.

3.1.3 *ASTM standard feed*—a specific batch of gas oil that is used as feedstock in the described test method.[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) or the known conversion values for other suitable reference catalysts versus the individual laboratory-measured conversion for the same catalysts.

3.1.5 *measured conversion*—calculated as the difference between the mass of feed used and the mass of unconverted material divided by the mass 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 feed (ASTM standard feed or other suitable feedstock). 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.

[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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D32-1016. Contact ASTM Customer Service at service@astm.org.

5. Significance and Use

5.1 The microactivity test provides data to assess the relative performance of FCC catalysts. 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 mass % conversion due to the significance of overcracking.

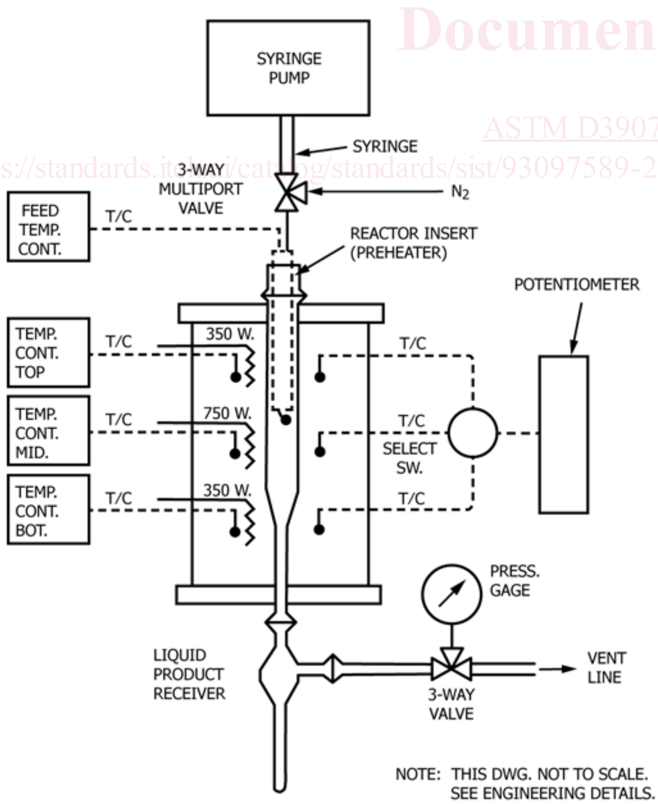
6. Apparatus

6.1 *Flow Chart*—The flow chart is given in Fig. 1. During 75 s, gas oil feed 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 ± 5 °C [104 ± 9 °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.



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

FIG. 1 Microactivity Flow Chart

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 ± 1 °C [900 ± 2 °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 gauge 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 and remove coke by heating a shallow (less than 10 mm thick) bed of catalyst in a porcelain crucible as follows:

120 ± 20 °C [248 ± 36 °F] for 1 h
120 °C [248 °F] to 590 °C [1094 °F] for approximately 1 h
590 ± 20 °C [1094 ± 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) until it reaches the constricted region of 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). Insert another plug of quartz or glass wool and add approximately 10 cc of 80 to 100 mesh glass microspheres or crushed glass particles. These act as a preheat bed and aid in feed dispersion, especially at higher WHSV's.

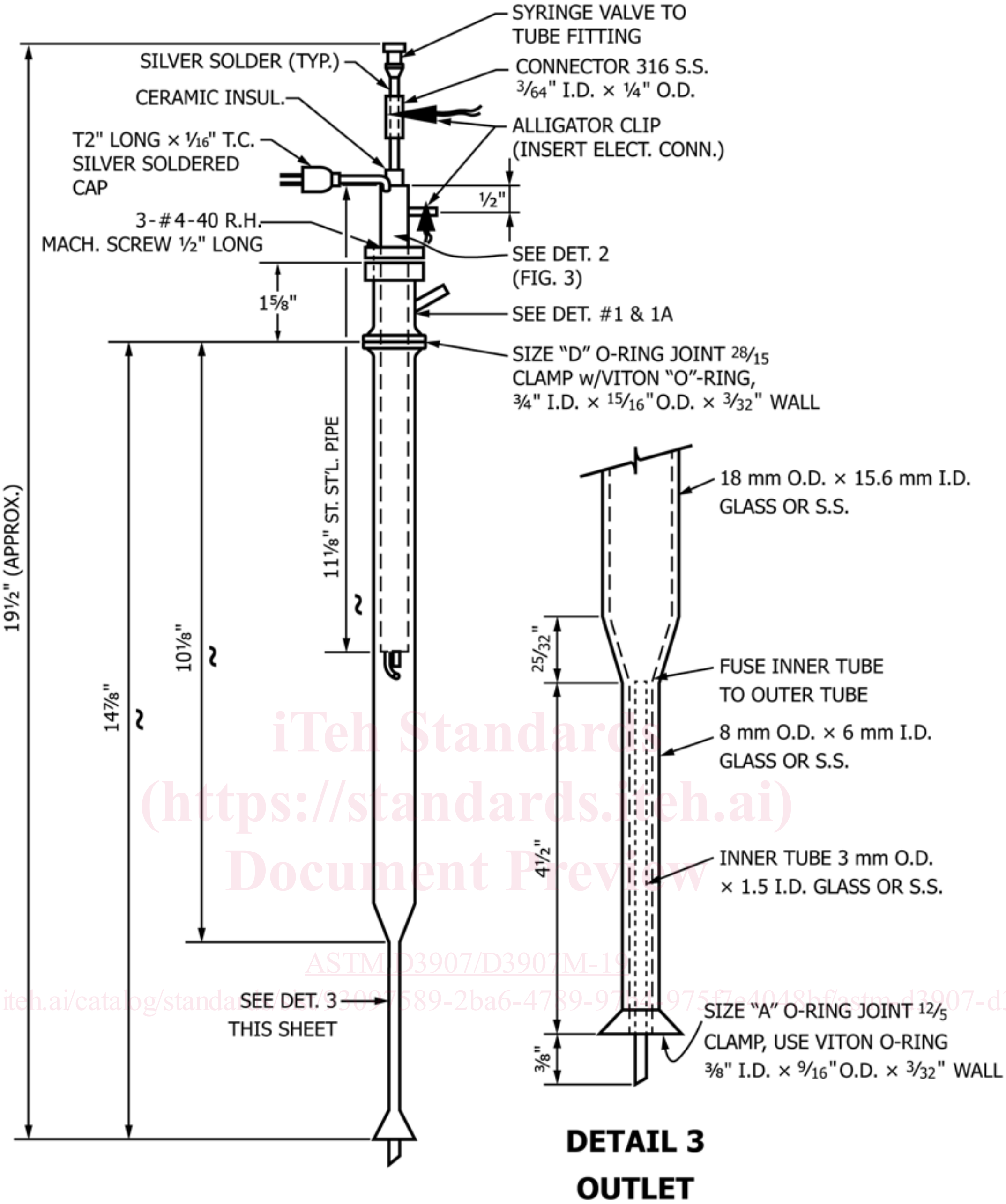


FIG. 2 Microactivity Reactor

9.1.4 Inspect the oil insert needle to ensure it is clear of deposits. Place insert in reactor and adjust if necessary so that the bottom of the oil insert needle is just touching the top of the glass microsphere preheat bed (about 20 mm above the catalyst bed). Inspect the reactor insert before using to be sure 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 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 [30 sccm] 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 Preheat the gas oil feedstock to 40 ± 5 °C [104 ± 9 °F] to allow filling of the syringe. Before testing, calibrate the syringe pump to the correct feed rate by collecting the oil, preheated to 40 ± 5 °C [104 ± 9 °F], outside the reactor and weighing the oil or by weighing the syringe before and after delivery to assure the appropriate mass of oil delivered over unit time.