



Designation: D 5154 - 91

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determining the Activity and Selectivity of Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test¹

This standard is issued under the fixed designation D 5154; 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 and selectivity of either equilibrium or laboratory deactivated fluid catalytic cracking (FCC) catalysts. The activity is evaluated on the basis of weight percent conversion of gas oil in a microactivity unit. The selectivities are evaluated on the basis of weight percent yields of specifically defined products resulting from the catalytic cracking of gas oil.

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 problems, 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:

- D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography²
- D 3907 Method for Testing Fluid Cracking Catalysts by Microactivity Test³
- D 4463 Test Method for Steam Deactivation of Fresh Fluid Cracking Catalysts³
- E 105 Practice for Probability Sampling of Materials⁴
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴
- E 456 Terminology Relating to Quality and Statistics⁴
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Terminology

3.1 Description of Terms:

3.1.1 *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 through the National Institute of Standards and Technology (NIST) (formerly the National Bureau of Standards) in Gaithersburg, MD, 20899.

3.2 *ASTM Reference Catalysts*—a set of equilibrium FCC catalysts within the useful range of this test method is used to improve the reproducibility of test results between different

laboratories. Each catalyst has a consensus mean conversion value assigned to it by Committee D-32. Samples of the ASTM reference catalysts can be obtained through NIST.

3.3 *Measured Conversion*—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).

3.4 *Activity*—calculated as conversion divided by the difference of 100 minus conversion.

3.5 *Catalyst/Oil (C/O) Ratio*—the weight of catalyst used in the test divided by the weight of feed fed to the reactor. In practice, the weight of catalyst is usually maintained at a constant value and the total weight of feed is varied.

3.6 *Weight Hourly Space Velocity (WHSV)*—the oil feed rate in grams per hour divided by the weight of catalyst in grams. Units are hr⁻¹.

3.7 *Reaction Severity*—an indication of the severity of the cracking reaction which allows a range of conversions to be obtained from any particular catalyst without changing reactor temperature. Changing reaction severity is achieved by changing WHSV or C/O ratio or both.

3.8 *Contact time*—in seconds is calculated as 3600/(WHSV * C/O). This is the delivery time during which feed is introduced to the reactor.

3.9 *Product Yield*—one hundred times the weight of a specific product divided by the weight of feed used in the test.

3.10 *Normalized Product Yield*—the result obtained when each product yield has been corrected for non-perfect weight balances. For a run to be judged acceptable, the total recovery, wt % of feed, should be in the range of 96 to 101 % (see 10.2). If the recovery is outside this range the test data should be discarded.

3.11 *LCO*—the light cycle oil product defined to have a boiling point range of 216–343°C (421–650°F).

3.12 *HCO*—the heavy cycle oil product defined to have a minimum boiling point of 343°C (650°F).

4. Summary of Test Method

4.1 At least two samples of FCC catalysts, one of which is chosen as a reference, are separately contacted with gas oil (ASTM Standard Feed or other suitable feedstock) in a fixed bed reactor at a specified reaction zone temperature and at more than one reaction severity. Reaction products (liquid, gaseous and coke on catalyst) are analyzed. Conversion, activity and product yields are calculated for each experiment.

4.2 Following analysis of the products, the total recovery (that is, weight balance) of the feed as converted and unconverted products is determined. If the recovery is less

¹ This test method is under the jurisdiction of ASTM Committee D-32 on Catalysts and is the direct responsibility of Subcommittee D32.04 on Catalytic Properties.

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² Annual Book of ASTM Standards, Vol 05.02.

³ Annual Book of ASTM Standards, Vol 05.03.

⁴ Annual Book of ASTM Standards, Vol 14.02.

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than 96 % or greater than 101 %, the test is rejected as unsatisfactory.

4.3 For each catalyst tested, normalized product yields are plotted against conversion or activity to generate a yield curve. The data comprising the yield curve may be used to obtain the parameters of an appropriate mathematical expression for the curve. Comparisons among catalysts can be made by interpolating the yield curves to obtain the product yields at some specified conversion.

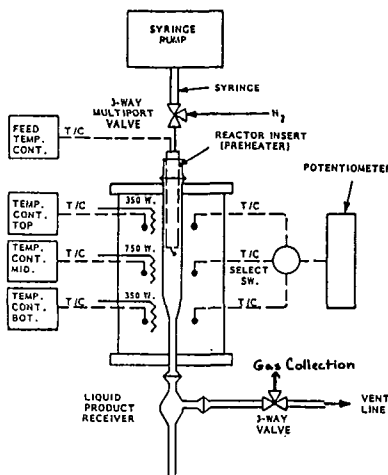
5. Significance and Use

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. Significant variations in apparatus, test conditions and/or analytical procedures may result in activity and selectivity data which do not correlate with data developed by other laboratories on identical catalyst/feedstock samples.

5.2 The standard method reaction temperature is 516°C (960°F). Other reaction temperatures can be used; however, catalyst selectivity data developed at temperatures other than 516°C may not correlate with selectivity data developed at 516°C. Also, precision at other reaction temperatures may change compared to data obtained at 516°C.

6. Apparatus

6.1 The apparatus of this test method is essentially that described in Method D 3907 - 87, as shown in Figures 1-4, with the addition of a gas collection system on the effluent gas vent line. In a typical gas collection system, a fluid is displaced by the collected gas and the volume of displaced liquid provides a quantitative measurement of the amount of gas collected. To minimize the solubility of gases like H₂ and H₂S in the fluid, a saturated brine (NaCl) solution is recommended. Some publications have appeared which give example flow schematics and more detailed descriptions of



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

FIG. 1 Micro-Activity Flow Chart

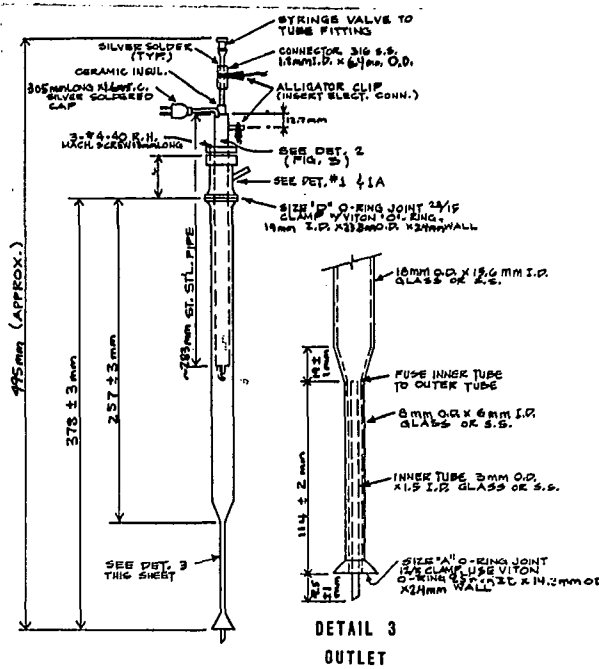


FIG. 2 Microactivity Reactor

typical apparatus.⁵ Alternatively, there are several vendors as listed in RR: D32-1030 who can provide specific equipment for performing this test. However, the D-32 Committee can only suggest and will not recommend nor certify any specific vendor. Significant variations from the test apparatus of this method most likely will result in significantly different activity and selectivity data from identical catalyst samples.

6.2 Chromatographic Equipment:

6.2.1 Liquid product analyses should be performed using Test Method D 2887 on a gas chromatograph (G.C.) equipped with a flame ionization detector.

6.2.2 Gas product analyses may be accomplished in two parts. First, an analysis using a G.C. equipped with a thermal conductivity detector, employing N₂ as a carrier gas, and using a G.C. column that permits the separation and quantitative identification of H₂. Second, an analysis using a G.C. equipped with a FID or TCD, employing He as a carrier gas and using a column that permits the separation and quantitative identification of methane, ethane, ethylene, propane, propylene, n-butane, iso-butane, 1-butene and iso-butene, cis-2-butene, and trans-2-butene. Hydrocarbon components of C₃ and greater are lumped together and reported as a C₅⁺ group in the gas phase. The C₅⁺ group is included as part of the gasoline fraction.

6.3 Carbon analysis of a representative sample of the spent catalyst (that is, after the cracking reaction has been completed) may be accomplished using a commercially available carbon analyzer.

7. Sampling

7.1 A sampling procedure is needed. Practice E 105 is appropriate.

⁵ Campagna, R. J., Wick, J. P., Brady, M. F., and Fort, D. L., "Fresh FCC Catalyst Tests Predict Performance," OGI, March 24, 1986, p. 85.

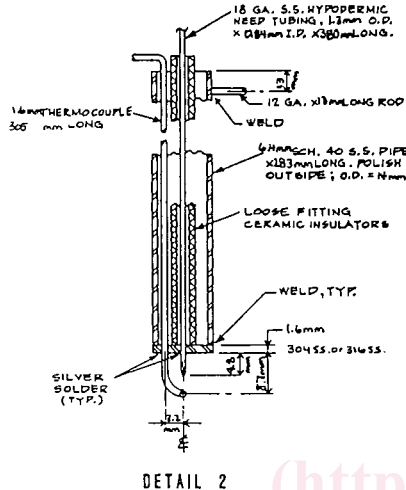
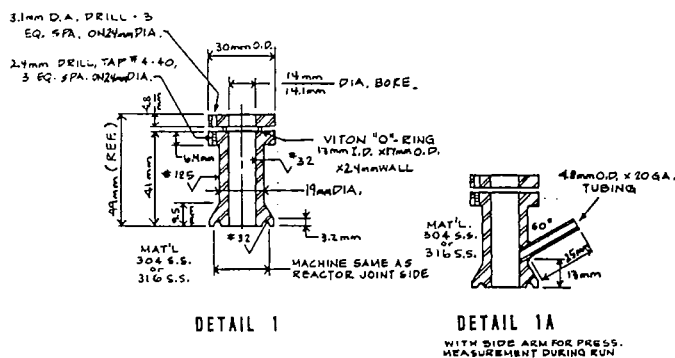


FIG. 3 Reactor Feed Tube Insert

8. Sample Preparation

8.1 *Equilibrium Catalysts*—dry samples or decoke, or both, by heating a shallow (less than 10 mm thick) bed of catalyst in a porcelain crucible at $590 \pm 20^\circ\text{C}$ ($1094 \pm 36^\circ\text{F}$) for a length of time sufficient to remove any coke. This typically requires approximately three hours. 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.

NOTE 1—Heavily coked samples may be damaged by sintering or deactivation if oxidation is allowed to occur too rapidly, leading to artificially low catalytic activity and surface area.

8.2 *Fresh Catalysts*—fresh catalyst samples should be steam treated prior to selectivity testing. Steaming procedures such as those specified in Test Method D 4463 may be used. However, specific conditions (temperature, partial pressure of steam, and time) should be chosen such that the steamed catalyst properties (activity, zeolite and matrix surface areas, and unit cell size) approximate those found in equilibrium FCC catalysts of the same type.

9. Procedure

9.1 Reactor Preparation:

9.1.1 Rinse feed line with acetone or other suitable solvent and dry with air. Periodic cleaning of the insert is recommended by air purge at 550°C (1022°F) for one hour at least once every 12 tests.

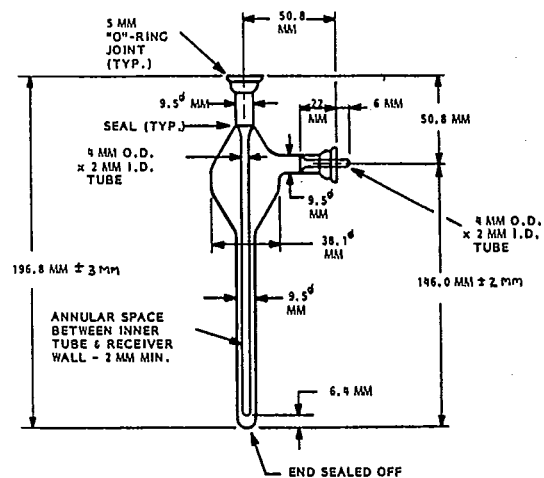


FIG. 4 Liquid Product Receiver

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 516°C (960°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. Optionally, a reactor with a fritted disc may be used to position the catalyst. Add 4.00 to 4.05 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.

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 516°C (960°F) and connect the nitrogen purge line directly to the reactor feed line. Purge with 30 sccm (30 ml/minute) of nitrogen for at least 30 minutes.

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 $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$) to allow filling of the syringe. Before testing, calibrate the syringe pump to the correct feed rate by collecting the oil [preheated to $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$)] before it enters the reactor and weighing it or by weighing the syringe before and after delivery to assure the appropriate weight of oil delivered over unit time.

NOTE 2—If heavier feedstocks are used, a syringe temperature of $70 \pm 5^\circ\text{C}$ ($158 \pm 9^\circ\text{F}$) should be used for obtaining accurate feed rates.

9.2.2 Fill the syringe with preheated feedstock and invert to allow air to rise. Remove the air bubbles. The syringe should contain a small amount of oil in excess of the nominal volume to be charged.