

Standard Test Method for Determining Activity and Selectivity of Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test¹

This standard is issued under the fixed designation D5154; 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 mass percent conversion of gas oil in a microactivity unit. The selectivities are evaluated on the basis of mass percent yields of specifically defined products resulting from the catalytic cracking of gas oil.
- 1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.
- 1.2.1 *Exception*—SI units have been retained in some of the figures.
- 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

D3907 Test Method for Testing Fluid Catalytic Cracking (FCC) Catalysts by Microactivity Test

D4463 Guide for Metals Free Steam Deactivation of Fresh Fluid Cracking Catalysts

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 *activity*—calculated as conversion divided by the difference of 100 minus conversion.
- 3.1.2 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 D32. Samples of the ASTM reference catalysts can be obtained through NIST.
- 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 catalyst/oil (C/O) ratio—the mass of catalyst used in the test divided by the mass of feed fed to the reactor. In practice, the mass of catalyst is usually maintained at a constant value and the total mass of feed is varied.
- 3.1.5 contact time—calculated as $3600/(WHSV \cdot C/O)$. This is the delivery time, in seconds, during which feed is introduced to the reactor.
- 3.1.6 *HCO*—the heavy cycle oil product defined to have a minimum boiling point of 650°F (343°C).
- 3.1.7 *LCO*—the light cycle oil product defined to have a boiling point range of 421 to 650°F (216 to 343°C).
- 3.1.8 *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 42°F (216°C).
- 3.1.9 normalized product yield—the result obtained when each product yield has been corrected for non-perfect mass balances. For a run to be judged acceptable, the total recovery, wt % of feed, should be in the range of 96 to 101 % prior to normalization. If the recovery is outside this range the test data should be discarded.

 $^{^{\}rm 1}\,\rm This$ 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 Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

- 3.1.10 *product yield*—one hundred times the mass of a specific product divided by the mass of feed used in the test.
- 3.1.11 *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.
- 3.1.12 *weight hourly space velocity (WHSV)*—the oil feed rate in grams per hour divided by the mass of catalyst in grams. Units are hr⁻¹.

4. Summary of Test Method

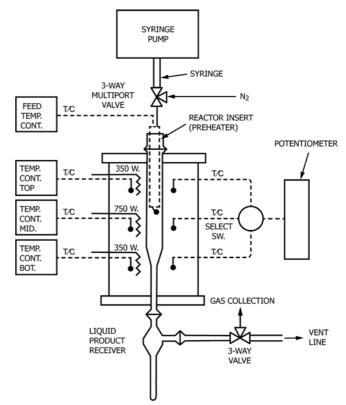
- 4.1 At least two samples of FCC catalysts, one of which is chosen as a reference, are separately contacted with gas oil (ASTM Standard Feed or other suitable feedstock) in a fixed bed reactor at a specified reaction temperature and at more than one reaction severity. Reaction products (liquid, gas, 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, mass balance) of the feed as converted and unconverted products is determined. If the recovery is less 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 960°F (516°C). Other reaction temperatures can be used; however, catalyst selectivity data developed at temperatures other than 960°F (516°C) may not correlate with selectivity data developed at 960°F (516°C). Also, precision at other reaction temperatures may change compared to data obtained at 960°F (516°C).

6. Apparatus

6.1 The apparatus of this test method is essentially that described in Test Method D3907, as shown in Figs. 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



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

FIG. 1 Microactivity Flow Chart

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 typical apparatus.⁴ Alternatively, there are several vendors as listed in Research Report RR:D32-1030⁵ who can provide specific equipment for performing this test. However, Committee D32 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 D2887 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 N_2 as a carrier gas, and using a G.C. column that permits the separation and quantitative identification of H_2 and H_2S . 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,

⁴ Campagna, R. J., Wick, J. P., Brady, M. F., and Fort, D. L., "Fresh FCC Catalyst Tests Predict Performance," *OGJ*, March 24, 1986, p. 85.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D32-1030.



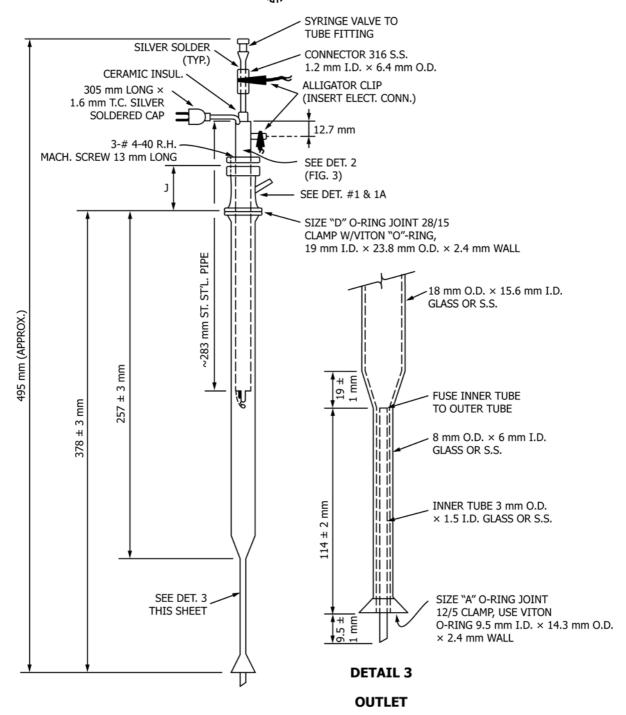


FIG. 2 Microactivity Reactor

propylene, n-butane, iso-butane, 1-butene and iso-butene, cis-2-butene, and trans-2-butene, C_5 's and a C_5 ⁺ lump that is not resolved. The C_5 's and the C_5 ⁺ group are included as part of the gasoline fraction.

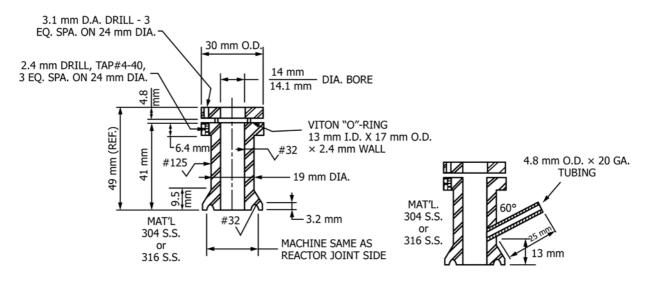
6.3 Carbon analysis of a representative sample of the spent catalyst (that is, after the cracking reaction has been completed) may be accomplished using a commercially available carbon analyzer.

7. Sampling

7.1 A sampling procedure is needed. Practice E105 is appropriate.

8. Sample Preparation

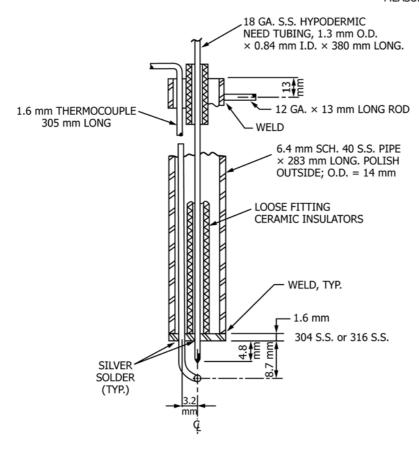
8.1 Equilibrium Catalysts—Dry samples or decoke, or both, by heating a shallow (less than 10 mm thick) bed of catalyst in a porcelain crucible at $1094 \pm 36^{\circ}F$ ($590 \pm 20^{\circ}C$) for a length



DETAIL 1

DETAIL 1A

WITH SIDE ARM FOR PRESS, MEASUREMENT DURING RUN



DETAIL 2

FIG. 3 Reactor Feed Tube Insert

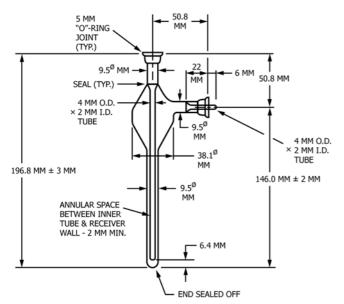


FIG. 4 Liquid Product Receiver

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 Guide D4463 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 1022°F (550°C) for one hour 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 960°F (516°C) 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 960°F (516°C) and connect the nitrogen purge line directly to the reactor feed line. Purge with 30 seem (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 Preheat the gas oil feedstock to $104 \pm 9^{\circ}F$ ($40 \pm 5^{\circ}C$) to allow filling of the syringe. Before testing, calibrate the syringe pump to the correct feed rate by collecting the oil, preheated to $104 \pm 9^{\circ}F$ ($40 \pm 5^{\circ}C$), 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.

Note 2—If heavier feedstocks are used, a syringe temperature of 158 \pm 9°F (70 \pm 5°C) 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.
- 9.2.3 After the syringe is filled and the valve is in the 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 mass.
- 9.2.5 Attach the syringe and syringe valve to the reactor making sure the valve is in the nitrogen flow position. A thermocouple may be placed on the syringe body to monitor the syringe temperature during oil injection.
- 9.2.6 Obtain the initial mass of the liquid product receiver(s). If necessary, a second liquid receiver cooled by dry ice may be added in-line to aid collection of the lighter liquid products (mainly C_5 and C_6 's). If such a receiver is used, it should also be weighed. Optionally, a small piece of glass wool in the receiver sidearm may be used to reduce the carryover of C_5^+ products into the gas collection vessel.
- 9.2.7 Install receiver(s) and connect the gas line to the gas collection system. Open the gas vent valve to prevent premature collection of purge gas.
- 9.2.8 Prepare and install wet ice (and dry ice) bath(s) for the receiver(s). Immerse the receiver(s) to the level of the gas outlet line(s). Optionally, a controlled temperature circulating bath may be used in place of the wet ice bath.
- 9.2.9 Pressurize the reactor to 150 mm Hg (20 kPa) nitrogen and close the gas vent and nitrogen supply valves. Watch the pressure gages for 2 min to determine if any significant leaks occur. If no leaks are apparent, open the gas vent valve carefully to release pressure.

9.3 Run Conditions:

9.3.1 Check syringe temperature, $104 \pm 9^{\circ}F$ ($40 \pm 5^{\circ}C$), and reactor insert temperature, $960 \pm 2^{\circ}F$ ($516 \pm 1^{\circ}C$).

- 9.3.2 Set syringe pump to deliver the appropriate amount of oil per minute as determined in 9.2.1.
- 9.3.3 Bring the syringe drive head flush with the syringe plunger.
- 9.3.4 Switch the syringe valve from the nitrogen flow to the oil feed position and the gas vent valve to the gas collection position.
- 9.3.5 Start the pump and deliver the appropriate amount of oil at the specified feed rate. Collect the displaced brine in a calibrated volumetric cylinder.
- 9.3.6 The oil preheat temperature, as measured by the T/C at the oil exit, shall be maintained at $960 \pm 9^{\circ}F$ ($516 \pm 5^{\circ}C$) by the temperature controller assembly during oil feeding.

Note 3—Fine particle samples can cause excessive catalyst bed differential pressures, significantly affecting results. Differential pressures up to 100 mm Hg (13.3 kPa) measured during oil addition (see Fig. 3, Detail 1 A) are not detrimental. More typical pressures are in the range from 30 to 60 mm Hg (4.0 to 8.0 kPa). If the catalyst bed differential pressure exceeds 100 mm Hg (13.3 kPa), it is recommended that the catalyst be screened free of particles finer than 400 mesh (37 μm) and the test be repeated with the larger-sized particles.

- 9.3.7 Immediately after the oil addition, restart the nitrogen flow at 30 sccm (30 mL/min.) by switching the multiport valve to purge the reactor and product recovery system.
- 9.3.8 Remove the syringe from the reactor and reweigh it. Record the mass of oil feed (W_F). W_F should vary no more than ± 0.03 g from the desired feed mass.
- 9.3.9 After 4 \pm 0.25 min of nitrogen purge, remove the cooling bath(s), from the liquid product receiver(s) and replace it (them) with a water bath(s) at about 77°F (25°C). Continue purging for at least another 3 min. This will permit the liquid products to warm up and allow most of the C_5 and lighter products to be purged from the liquids for proper identification in the gas analyses. Stop the N_2 purge and close the valves on the gas collection system when a desired volume of brine (such as 500 mL) has been displaced.
- 9.3.10 Remove the liquid product receiver(s) and immediately seal; dry the outside of the receiver(s) and weigh. Record the total mass of liquid product ($W_{L1} + W_{L2}$). Equalize the pressure of the product gases to atmospheric by eliminating the liquid head; then close the line discharge valve. Record the volume of displaced brine (V_B), the gas temperature (T_R), and atmospheric pressure (P_R). The collected gases may then be analyzed immediately by syringe sampling or may be forced from the gas collection system into a gas sampling loop for analysis or a sealable gas sample bag for storage and later analysis.
- 9.3.11 Collect the liquid holdup in the reactor exit line and around the reactor-receiver joint with a tared cotton wool swab or other suitable material. Determine the mass of this liquid and record (W_Q). Experience indicates this liquid material is predominantly unconverted products.
- 9.3.12 Remove the reactor from the furnace. After cooling, the catalyst can be removed from the reactor for carbon analysis as described in 9.4.4.
 - 9.4 Analyses of Products:
- 9.4.1 If two liquid product receivers are used, the products should be combined by adding the major amount to the minor amount, using a syringe with needle of suitable length, and

thoroughly mixing. If needed, a suitable solvent such as CS_2 may be used to rinse out residues. (CS_2 may be used to rinse out residues, since it does not interfere with the G.C. analysis when an FID is used.)

9.4.2 Using Test Method D2887, simulated distillation, determine the mass percentage of the liquid products boiling in the ranges of gasoline, IBP-421°F (216°C) and LCO, 421-650°F (216-343°C). The remaining product is the HCO, 650+°F (343+°C).

9.4.3 Analyze the gas product collected using a single G.C. or a combination of G.C.'s that will permit quantitative volume (mole) % determination of H_2 , H_2S , C_1 , C_2 's, C_3 's, C_4 's, C_5 saturates and a C_5^+ lump that is not resolved, such that, separation of the individual components, C_5 and lighter, is obtained.

9.4.4 Carbon analysis of a representative sample of the spent catalyst may be done by using equipment such as that supplied by LECO (Model #CR12) or Coulometrics (Model CM-190). Alternatively, carbon analysis may be done by passing an oxygen stream over a known amount of spent catalyst at high temperature and determining the amounts of CO and $\rm CO_2$ formed.

10. Calculation

10.1 Gas Product:

10.1.1 The total volume of gas collected (including purge gas) is determined by:

$$V_t^o = V_B \cdot \frac{273 \text{ K}}{273 \text{ K} + T_R} \cdot \frac{P_R}{101.3 \text{ kPa}} \text{ cm}^3 STP$$
 (1)

where:

101.3 kPa = 760 mm Hg,

 V_t^o = standard volume of gas collected, cm³,

 V_B = volume of brine displaced during the test and is equivalent to the actual volume of gas collected, cm³,

 T_R = room temperature, °C, and P_R = atmospheric pressure, kPa.

10.1.2 To find the volume of each gas component:

$$V_i = N_i \ V_i^o / 100 \tag{2}$$

where:

 V_i = volume of the *i*th gaseous product, and N_i = mole % of the *i*th component in the gas.

10.1.3 To find the mass (absolute) of each component:

$$W_i = \frac{V_i \cdot M_i}{22412} \,\mathrm{g} \tag{3}$$

where:

 W_i = mass of the *i*th gaseous product, g, and M_i = molecular weight of the *i*th gaseous product.

10.1.4 The molecular weight of the C_5^+ unresolved lump is assumed to be 75.

10.1.5 The total mass of products collected in the gas phase is:

$$W_p = \sum W_i$$
 (sum over all components) (4)

where:

 W_g = total mass of all gaseous products, g.

10.1.6 Coke:

10.1.6.1 The total mass of coke (in grams) on the catalyst $W_{\rm c}$ is:

$$W_{K} \cdot C_{c} \cdot 1.07/100$$
 (5)

where:

 W_K = mass of the catalyst used in grams, and

 C_c = mass percent of carbon on the spent catalyst as determined in 9.4.4.

Note 4—The 1.07 factor is empirically found to account for a 6.5 mass percent hydrogen content of a typical coke.

10.2 Mass Balance (Recovery):

10.2.1 To determine the quality of the test, the mass balance can be calculated as follows:

$$R = \frac{\left(W_g + W_{L1} + W_{L2} + W_c + W_Q\right)}{W_F} \cdot 100\%$$
 (6)

where:

R = recovery, wt % of feed.

10.2.2 For a run to be judged acceptable, the *R* should be in the range 96 to 101 %. If the recovery is outside this range, the test data should be discarded.

10.3 Determination of Raw Yields:

10.3.1 Each measured product can be expressed as an actual yield, wt % of feed as follows:

10.3.1.1 Gas Components H_2-C_4 's:

$$Y_i = W_i / W_F \cdot 100 \tag{7}$$

where:

 Y_i = raw yield of the *i*th gaseous product expressed as wt % of feed.

10.3.1.2 *Gasoline*:

$$Y_{g} = \frac{\left[X_{g}(W_{L1} + W_{L2}) + W_{i}(C_{5}^{+})\right]}{W_{F}} \cdot 100\%$$
 (8)

where:

 Y_o = raw yield of the gasoline, wt % of feed,

 X_g = mass fraction of gasoline in the combined liquid products, and

 $W_i(C_5^+)$ = mass of all C_5 's plus the unresolved C_5^+ lump in the gas sample.

10.3.1.3 LCO:

$$Y_{1} = \frac{X_{1}(W_{L1} + W_{L2})}{W_{T}} \cdot 100\%$$
 (9)

where:

 Y_I = raw yield of the LCO products, wt % of feed, and X_I = mass fraction of LCO products in the combined liquid products.

10.3.1.4 HCO:

$$Y_{h} = \frac{\left[X_{h}(W_{L1} + W_{L2}) + W_{Q}\right]}{W_{E}} \cdot 100\%$$
 (10)

where:

 Y_h = the raw yield of the HCO products, wt % of feed, and

 X_h = the mass fraction of HCO products in the combined liquid products.

10.3.1.5 Coke:

$$Y_c = \frac{W_c}{W_c} \cdot 100\% \tag{11}$$

where:

 Y_c = raw yield of coke, wt % of feed.

10.3.1.6 Conversion:

Conversion (raw), wt % =
$$100 \% - (Y_1 + Y_h)$$
 (12)

10.4 *Yield Normalization*—Unless the mass balance is 100 %, the raw yield will require normalization. The simplest way is normalization of all yields assuming loss (or gain) is proportional to the actual yield.

$$Y_i^o = \frac{Y_i}{R} \cdot 100\% \tag{13}$$

where:

 Y_i^o = normalized product yield of the *i*th product, wt % of feed

Conversion (Normalized), wt % =
$$100\% - (Y_i^o + Y_h^o)$$
 (14)

10.5 Correction of C_4 Gas Yields for C_4 's in the Liquid Product—It is assumed that the amount of C_4 and lighter in the liquid products are negligible. Experience has shown that if the liquids are warmed and purged at room temperature, the contribution of C_4 and lighter products to the gasoline is less than 0.5 wt % of the feed.

10.6 Selectivity Curve—To determine the selectivity curve for any catalyst, a range of reaction severities is chosen so that conversions in the range of 50 to 80 wt % are obtained. It is recommended that a minimum of three severities, tested in triplicate, be used to characterize the yield curve of any particular catalyst.

10.6.1 Selection of Reaction Severity—The coke build up that occurs on cracking catalysts during use is thought to have a significant influence on catalyst activity and selectivity. Therefore, it may be important to test catalysts at equal catalyst contact times. This may be accomplished by adjusting WHSV and C/O simultaneously as determined from the equation in 3.1.5, which still permits wide ranges in conversions to be observed. Catalyst/oil ratios in the range of 2.0 to 6.0 are recommended.

10.6.2 Comparison of Catalyst Performance—By themselves, the absolute yields obtained in microactivity testing of any catalyst are of little value. Rather, it is the comparison of yields between two (or more) catalysts at equal conversion(s) that provides a means for distinguishing them. Using the selectivity curves developed for each catalyst, yield ratios of selected components (dry gas, gasoline, coke, and so forth.) for the catalysts can be determined at specific conversions to truly distinguish the performance characteristics of different catalysts.

11. Precision and Bias⁵

11.1 Test Program—An interlaboratory study was conducted in which MAT selectivities were compared on two

TABLE 1 Repeatability and Reproducibility Limits

Catalyst	RR1			RR5		
Catalyst/Oil	3	4	6	1	2	3
Number of Labs	7	7	6	6	7	7
ASTM Consensus Mean Conversion, mass %	59.07	64.42	70.40	61.55	74.92	79.82
95 % Repeatability Limit (Within Laboratory), mass %	1.97	2.00	1.54	2.03	1.01	1.33
95 % Reproducibility Limit (Between Laboratories), mass %	14.68	14.78	13.10	13.88	6.15	7.90
ASTM Consensus Coke, mass %	2.17	2.79	3.46	2.11	3.69	4.91
95 % Repeatability Limit, mass %	0.59	0.20	1.08	0.15	0.32	0.68
95 % Reproducibility Limit, mass %	1.28	1.57	1.66	0.96	1.59	1.58
ASTM Consensus Gasoline, mass %	41.89	44.52	46.46	47.35	53.36	52.77
95 % Repeatability Limit, mass %	1.45	2.79	1.35	1.72	1.33	1.31
95 % Reproducibility Limit, mass %	8.01	7.61	7.16	11.77	6.19	7.87
ASTM Consensus Dry Gas, mass % (H ₂ + C ₁ + C ₂ 's)	1.35	1.50	1.90	1.31	1.95	2.77
95 % Repeatability Limit, mass %	0.29	0.14	0.13	0.12	0.24	0.25
95 % Reproducibility Limit, mass %	1.33	1.39	1.62	0.98	1.37	2.80
ASTM Consensus LPG, mass % (C ₃ 's + C ₄ 's)	13.43	15.11	18.28	10.77	15.87	19.20
95 % Repeatability Limit, mass %	1.54	2.08	0.96	0.55	0.92	1.46
95 % Reproducibility Limit, mass %	5.81	5.36	6.26	5.86	5.05	6.60
ASTM Consensus LCO, mass %	24.94	23.25	20.62	23.08	17.28	14.42
95 % Repeatability Limit, mass %	1.09	1.05	0.82	0.82	0.56	0.88
95 % Reproducibility Limit, mass %	7.23	7.59	7.16	8.27	3.87	4.50
ASTM Consensus HCO, mass %	16.02	12.31	8.98	15.31	7.48	5.76
95 % Repeatability Limit, mass %	1.49	1.32	1.10	1.83	2.37	1.25
95 % Reproducibility Limit, mass %	9.99	8.70	6.97	7.47	5.80	5.72

separate test materials in seven different laboratories. Practice E691, modified for non-uniform data sets, was followed for the data reduction. Since each laboratory used their own apparatus design, and it is well known that equipment design affects MAT product selectivities, absolute product selectivities could not be compared. Therefore, the selectivity ratios between the two catalysts at a fixed conversion value were used for comparison and found to be directionally consistent for all laboratories.

11.2 *Precision*—Pairs of test results obtained by a procedure similar to that described in the study are 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 E456 and Practice E177, respectively.

11.2.1 The good repeatability within laboratories as described here, is consistent with that achieved by experienced laboratories using similar technology and procedures. It reflects the ability to consistently implement the essence of the test method, although often deviating from it significantly according to individual laboratory resources and capabilities.

11.2.2 The poor reproducibilities obtained on four of six consensus mean conversions and many selectivity values for both catalysts at all test conditions used are a consequence of the round robin participants varying implementation of the test method as written. These interlaboratory differences suggest that precise implementation of the test method is required for more acceptable reproducibility to be obtained. As presented, the reproducibility data do not reflect the true potential of the method, but rather its limitations resulting from inconsistent implementation (see Table 1).

11.3 *Bias*—The procedure described is without known bias since there is by definition no absolute standard for comparison. However, if apparatus, test conditions and analytical procedures actually used do not closely resemble those described in this method, results may not be comparable to those from other laboratories.

12. Keywords

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

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