Designation: D4463/D4463M – 19

Standard Guide for Metals Free Steam Deactivation of Fresh Fluid Cracking Catalysts[1]

This standard is issued under the fixed designation D4463/D4463M; 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 guide covers the deactivation of fresh fluid catalytic cracking (FCC) catalyst by hydrothermal treatment prior to the determination of the catalytic cracking activity in the microactivity test (MAT) or the Advanced Cracking Evaluation (ACE) test.

1.2 The hydrothermal treatment of fresh FCC catalyst, prior to the MAT or the ACE test, is important because the catalytic activity of the catalyst in its fresh state is an inadequate measure of its true commercial performance. During operation in a commercial cracking unit, the catalyst is deactivated by thermal, hydrothermal, and chemical degradation. Therefore, to maintain catalytic activity, fresh catalyst is added (semi) continuously to the cracking unit, to replace catalyst lost through the stack or by withdrawal, or both. Under steady state conditions, the catalyst inventory of the unit is called equilibrium catalyst. This catalyst has an activity level substantially below that of fresh catalyst. Therefore, artificially deactivating a fresh catalyst prior to determination of its cracking activity should provide more meaningful catalyst performance data.

1.3 Due to the large variations in properties among fresh FCC catalyst types as well as between commercial cracking unit designs or operating conditions, or both, no single set of steam deactivation conditions is adequate to artificially simulate the equilibrium catalyst for all purposes.

1.3.1 In addition, there are many other factors that will influence the properties and performance of the equilibrium catalyst. These include, but are not limited to: deposition of heavy metals such as Ni, V, and Cu; deposition of light metals such as Na; and contamination from attrited refractory linings of vessel walls. Furthermore, commercially derived equilibrium catalyst represents a distribution of catalysts of different ages (from fresh to >300 days). Despite these apparent problems, it is possible to obtain reasonably close agreement between the performances of steam deactivated and equilibrium catalysts. It is also recognized that it is possible to steam deactivate a catalyst so that its properties and performance poorly represent the equilibrium. It is therefore recommended that when assessing the performance of different catalyst types, a common steaming condition be used. Catalyst deactivation by metals deposition is not addressed in this guide, but is addressed in Guide D7206/D7206M.

1.4 This guide offers two approaches to steam deactivate fresh catalysts. The first part provides specific sets of conditions (time, temperature, and steam pressure) that can be used as general pre-treatments prior to comparison of fresh FCC catalyst MAT activities (Test Method D3907) or activities plus selectivities (Test Methods D5154 and D7964).

1.4.1 The second part provides guidance on how to pretreat catalysts to simulate their deactivation in a specific FCCU and suggests catalyst properties which can be used to judge adequacy of the simulation. This technique is especially useful when examining how different types of catalyst may perform in a specific FCCU, provided no other changes (catalyst addition rate, regenerator temperature, contaminant metals levels, etc.) occur. This approach covers catalyst physical properties that can be used as monitors to indicate the closeness to equilibrium catalyst properties.

1.5 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.

1.6 *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.*

1.7 *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.*

[1] This guide is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.04 on Catalytic Properties.

Current edition approved April 1, 2019. Published April 2019. Originally approved in 1985. Last previous edition approved in 2013 as D4463/D4463M–96(2013)ε1. DOI: 10.1520/D4463_D4463M-19.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3663 Test Method for Surface Area of Catalysts and Catalyst Carriers

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

D3942 Test Method for Determination of the Unit Cell Dimension of a Faujasite-Type Zeolite

D4365 Test Method for Determining Micropore Volume and Zeolite Area of a Catalyst

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

D7206/D7206M Guide for Cyclic Deactivation of Fluid Catalytic Cracking (FCC) Catalysts with Metals

D7964 Test Method for Determining Activity of Fluid Catalytic Cracking (FCC) Catalysts in a Fluidized Bed

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. Summary of Guide

3.1 A sample of fresh fluid cracking catalyst is placed in a reactor, either fixed bed or preferably fluid bed, and is contacted with steam at elevated temperature. This treatment causes partial deactivation of the catalyst.

NOTE 1—In a fixed bed reactor, material containing sulfates, chlorides, etc. can result in significant additional chemical deactivation.

3.2 The catalyst is withdrawn from the reactor and may be subjected to an activity or activity plus selectivity determination, by using the microactivity test (Test Methods D3907 or D5154) or ACE test (Test Method D7964).

4. Significance and Use

4.1 In general, steam treatment of FCC catalyst can be used either to compare a series of cracking catalysts at a simulated equilibrium condition or conditions, or to simulate the equilibrium condition of a specific cracking unit and a specific catalyst. This guide gives an example for the first purpose and an approach for the latter purpose.

5. Apparatus

5.1 Fixed bed or fluid bed steaming reactors can be used for the hydrothermal treatment of FCC catalyst.

5.2 In the steaming reactor, temperatures of the catalyst can be maintained at selected constant mean levels between 700 °C [1292 °F] and 850 °C [1562 °F] ±2 °C [±3.6 °F] during the steam treatment.

5.3 Temperature control during steam treatment is critical, as temperature variations of ±2 °C [±3.6 °F] can lead to ±1 mass % conversion changes or more, especially at higher temperatures.

5.4 In fixed bed steaming, the temperature gradient through the catalyst bed should be kept as small as possible and should not exceed 4 °C [7.2 °F]. In fluid bed steaming the bed temperature must be homogeneous.

5.5 Heating and cooling of the catalyst must be performed in the reactor under a flow of dry nitrogen.

5.6 Precautions must be taken to achieve uniform contact of the steam with the bed.

6. Sampling

6.1 A suitable sampling procedure is needed. Practice E105 is appropriate.

7. Sample Preparation

7.1 No sample preparation is necessary if the catalyst is heated slowly during preheating (non-shock steaming).

7.2 If the sample is introduced directly into a preheated steaming reactor (shock-steaming), it is desirable to predry the sample for about 1 h at about 550 °C [1022 °F] to prevent excessive catalyst loss.

8. Procedure

8.1 *Procedure for Fluid Bed and Fixed Bed Steam Treatment (Non-shock Steaming):*

8.1.1 With the reactor heated to 300 °C [572 °F] or lower, load the reactor with catalyst.

8.1.2 Start nitrogen flow to the reactor at a flow velocity of 3 cm/s [0.1 ft/s].

8.1.3 Heat the reactor at the maximum rate until a temperature of 600 °C [1112 °F] is reached.

8.1.4 Keep the temperature constant at 600 °C [1112 °F] for 30 min in order to remove volatile material from the catalyst.

8.1.5 Heat the reactor at the maximum rate until the desired steaming temperature is reached; for example, at 760, 788, or 800 °C [1400, 1450, or 1472 °F] ±2 °C [±3.6 °F].

8.1.6 Stop the nitrogen flow and start a flow of undiluted steam at atmospheric pressure and at constant temperature of 760, 788, or 800 °C [1400, 1450, or 1472 °F]. Continue this steam flow for 5 h. For fixed bed operation, keep the steam flow velocity at 5 ± 1 cm/s [0.16 ± 0.03 ft/s] at the desired deactivation temperature. For fluid bed operation, keep the steam velocity at 3 ± 1 cm/s [0.10 ± 0.03 ft/s].

8.1.7 After 5 h, stop the steam flow and start nitrogen flowing at 3 cm/s [0.10 ft/s] through the reactor.

8.1.8 Cool down the reactor to less than 300 °C [572 °F]. The rate of cooling is not critical.

8.1.9 Remove the catalyst from the reactor and store in a sealed bottle.

8.2 Variations in this procedure in which predried catalyst is added to a steaming reactor preheated to the desired steaming temperature (shock steaming) are also permissible provided a consistent procedure is used.

[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.