



Designation: ~~D3943-04~~ Designation: D3943 - 10

## Standard Test Method for Total Molybdenum in Fresh Alumina-Base Catalysts<sup>1</sup>

This standard is issued under the fixed designation D3943; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of molybdenum in alumina-base catalysts and has been cooperatively tested at molybdenum concentrations from 8 to 18 weight %, expressed as  $\text{MoO}_3$ . Any component of the catalyst other than molybdenum such as iron, tungsten, etc., which is capable of being oxidized by either ferric or ceric ions after being passed through a zinc-amalgam reductor column (Jones reductor) will interfere.

~~1.2~~

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:*<sup>2</sup>

D1193 [Specification for Reagent Water](#)

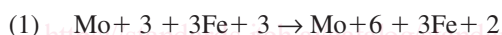
D7442 [Practice for Sample Preparation of Fluid Catalytic Cracking Catalysts and Zeolites for Elemental Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy](#)

E173 [Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals](#)

E507 [Test Method for Determination of Aluminum in Iron Ores by Flame Atomic Absorption Spectrometry](#)

### 3. Summary of Test Method

3.1 The catalyst is digested in hot sulfuric acid to dissolve molybdenum, destroy organic matter, and to expel nitrates if present. The acid solution is diluted and passed through an amalgamated zinc column (Jones reductor) into a receiving flask containing an excess of ferric ammonium sulfate. The column quantitatively reduces the molybdenum ( $\text{Mo}^{+6}$  to  $\text{Mo}^{+3}$ ) which in turn is reoxidized in the receiving flask according to the reaction:



3.2 The ferrous ion produced (three per  $\text{Mo}^{+3}$  oxidized) is titrated with standard ceric sulfate solution in the presence of ferroin indicator. The indicator undergoes a color change (pink to colorless or very faint blue-green) in the presence of excess ceric ion. The amount of  $\text{Fe}^{+2}$  titrated in the ~~reaction~~ reaction:

is a direct measure of  $\text{Mo}^{+6}$  concentration.

### 4. Significance and Use

4.1 This test method sets forth a procedure by which catalyst samples can be compared either on an interlaboratory or intralaboratory basis. It is anticipated that catalyst producers and users will find this method of value.

### 5. Interferences

5.1 Elements such as As, Sb, Cr, Fe, Ti, V, U, or W can interfere in this analysis. If serious interference occurs, it will be necessary to isolate the molybdenum from the interfering species prior to measurement.

5.2 Organics and nitrates can interfere or react with the zinc-amalgam if the sulfuric acid treatment is not properly carried out as described in 9.1.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.03 on Chemical Composition. Current edition approved April 1, 2004; 2010. Published April 2004; May 2010. Originally approved in 1980. Last previous edition approved in 2000; 2004 as ~~D3943-00; D3943-04~~. DOI: 10.1520/D3943-104.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 6. Apparatus

- 6.1 *Beakers*, 250-mL.
- 6.2 *Jones Reductor Column*, 30-cm long by 2 cm in outer diameter, with a 100-mL reservoir.<sup>3</sup> Attach a piece of vinyl tubing to the end of the delivery tube long enough to extend to the bottom of the filtering flask (see Test Method E507).
- 6.3 *Vacuum Filtering Flask*, heavy-walled, 1000-mL.
- 6.4 *Buret*, 50-mL, Class A (0.1-mL divisions).
- 6.5 *Stirring Motor*, ~~magnetic, and TFE-fluorocarbon-covered stirring bars, 7.11 by 14.2 mm (5/16 by 19/16 in.)~~, ~~magnetic, and TFE-fluorocarbon-covered stirring bars, 7.11 by 14.2 mm.~~

## 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>4</sup> Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

7.3 *Ceric Sulfate*, 0.10 *N* standardized using sodium molybdate following the procedure given in Annex A2.

NOTE 1—Refer to Section 8 on Hazards in Practice D7442 for hazards associated with handling of acids.

7.4 *Ferric Ammonium Sulfate Solution*—[FeNH<sub>4</sub>(SO<sub>4</sub>)<sub>2</sub>·12 H<sub>2</sub>O]—Dissolve 200 g of [FeNH<sub>4</sub>(SO<sub>4</sub>)<sub>2</sub>·12 H<sub>2</sub>O] in 1000 mL of water containing 20 mL of concentrated sulfuric acid. Add potassium permanganate solution dropwise until a pink color persists for 1 min.

7.5 *Ferriin Indicator*, 0.025 *M* solution of 1, 10-phenanthroline ferrous sulfate complex.

7.6 ~~*Hydrochloric Acid (HCl)*~~Hydrochloric Acid (HCl), concentrated.

7.7 *Jones Reductor Amalgam*.

7.8 *Mercuric Chloride (HgCl<sub>2</sub>) Solution*, approximately 2 %. Dissolve 10 g of mercuric chloride (HgCl<sub>2</sub>) in water. Transfer to a 500-mL volumetric flask, dilute to volume, and mix.

7.9 *Molybdenum Standard Solution*—Dissolve 1.0 g, weighed to the nearest 0.1 mg, of sodium molybdate dihydrate (Na<sub>2</sub>MoO<sub>4</sub>·2H<sub>2</sub>O) in water containing 10 mL of H<sub>2</sub>SO<sub>4</sub>, transfer to a 1-L volumetric flask, dilute to volume, and mix (1 mL = 0.5948 mg MoO<sub>3</sub>).

7.10 *Phosphoric Acid (H<sub>3</sub>PO<sub>4</sub>), concentrated*—Mix 130 mL of concentrated H<sub>2</sub>SO<sub>4</sub> with 250 mL of water. Add the acid slowly with constant stirring. Add 140 mL of concentrated H<sub>3</sub>PO<sub>4</sub> and 15 mL of a saturated solution of MnSO<sub>4</sub> (approximately 60 g/100 mL). Dilute to 1000 mL with water.

7.11 *Potassium Permanganate (KMnO<sub>4</sub>)*, approximately 0.1 *M* solution.

7.12 *Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>)*, concentrated, 1 + 1 and 1 + 19, dilutions with water, corresponding to 18 and 1.8 *N* solutions, respectively. Cautiously add the concentrated sulfuric acid to the water, mix well, and allow to cool.

7.13 *Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>)*, dilute, 1 + 99, warm, dilutions with water, corresponding to 0.18 *N* solution. Cautiously add the concentrated sulfuric acid to the water and mix well.

7.14 *Zinc Metal*, 20 mesh, low iron content.

## 8. Sampling

8.1 The selection of a representative analytical sample from the bulk material is outside the scope of the present method. It is presumed that parties using this test method for comparison purposes will have agreed on the selection of an analytical sample which is appropriate to their needs.

8.2 Pulverize the analytical sample to pass a 150- $\mu$ m (No. 100) sieve. Ignite the pulverized sample for 30 min at 550°C in a muffle furnace. Allow to cool in a desiccator.

## 9. Procedure

9.1 Transfer a 0.75-g specimen, weighed to the nearest 1 mg, into a 250-mL beaker. Add 20 mL of 1 + 1 H<sub>2</sub>SO<sub>4</sub> solution and heat to fumes on a hot plate. Cool, rinse down with 10 mL of water and take to fumes again. Cool, add 100 mL of water and heat to dissolve. Cool to room temperature.

<sup>3</sup> Withdrawn.

<sup>3</sup> The sole source of supply of the apparatus known to the committee at this time is Part No. 8035-J10 from Thomas Scientific, 99 High Hill Rd. at 295, Swedesboro, NJ 08085. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.