



Designation: D 3943 – 00

Standard Test Method for Total Molybdenum in Fresh Alumina-Base Catalysts¹

This standard is issued under the fixed designation D 3943; 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 approval. A superscript epsilon (ϵ) 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 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 *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:

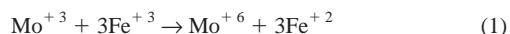
D 1193 Specification for Reagent Water²

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³

E 507 Test Method for Aluminum in Iron Ores by Atomic Absorption Spectroscopy³

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 (Mo^{+6} to Mo^{+3}) which in turn is reoxidized in the receiving flask according to the reaction:



3.2 The ferrous ion produced (three per 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 Fe^{+2} titrated in the reaction



is a direct measure of 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.

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.⁴ 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 E 507).

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, 7.9 by 14.2 mm ($5/16$ by $1/16$ in.).

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,

¹ This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D 32.03 on Chemical Composition.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.05.

⁴ Part No. 8035-J10. The sole source of supply of this apparatus known to the committee at this time is Arthur H. Thomas Co. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹ which you may attend.