



Designation: D 3610 – 00

Standard Test Method for Total Cobalt In Alumina-Base Cobalt-Molybdenum Catalyst by Potentiometric Titration Method¹

This standard is issued under the fixed designation D 3610; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of cobalt (expressed as the oxide) in fresh cobalt-molybdenum catalyst, in the range of 0.5 to 10 % cobalt oxide.

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 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

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

3. Summary of Test Method

3.1 The sample is decomposed by adding water and sulfuric acid and then heating until completely dissolved. The cold solution is diluted with water and transferred to a 250-mL volumetric flask. An aliquot of this solution containing between 10 and 30 mg of cobalt is transferred to a 250-mL beaker containing measured volumes of potassium ferricyanide and ammonium citrate solutions, ammonia, and petroleum ether. The excess of ferricyanide is then back-titrated with a standard cobalt solution.

4. Significance and Use

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

5. Interferences

5.1 None of the elements normally found in fresh cobalt-molybdenum catalysts interferes with this method. (Elements such as nickel, phosphorus, silicon, aluminum, and molybdenum do not interfere; elements such as iron, chromium, vanadium, and manganese do interfere).

6. Apparatus

6.1 *Analytical Balance and Weights*—The balance used to weigh the sample shall have a precision of 0.1 mg. Analytical weights shall be of precision grade or calibrated against a set of certified standard weights.

6.2 *Buret*—The 50-mL buret used to deliver the standard potassium ferricyanide and standard cobalt solutions shall be of precision grade and shall be read to 0.01 mL by interpolation.

6.3 *Glassware*—Beakers used in the analysis of the sample shall be of chemical-resistant glass and free of etched surfaces. Before using, all glassware shall be cleaned in hot dilute hydrochloric acid and thoroughly rinsed with water.

6.4 *Potentiometric Titration Apparatus*—Apparatus No. 3B of Practices E 50, or equivalent.

6.5 *Hot Plate*—Capable of maintaining surface temperature of at least 300°C.

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.⁴ 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 D 1193, Type IV.

¹ This test method is under the jurisdiction of ASTM Committee D32 on Catalysts, and is the direct responsibility of Subcommittee D32.03 on Chemical Analysis.

Current edition approved April 10, 2000. Published May 2000. Originally published as D 3610 – 77. Last previous edition D 3610 – 88 (1993).

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

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

⁴ *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 *Analar 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.