



Designation: D 5153 – 91 (Reapproved 1997)

## Standard Test Method for Palladium in Molecular Sieve Catalyst by Atomic Absorption<sup>1</sup>

This standard is issued under the fixed designation D 5153; 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 the determination of palladium in molecular sieve-containing fresh catalysts with about 0.5 weight % of palladium.

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 Water<sup>2</sup>

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>3</sup>

E 456 Terminology Relating to Quality and Statistics<sup>3</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>3</sup>

2.2 *U.S. Federal Specification:*

Federal Spec. NNN-P-395C Tolerance for Class A Pipets<sup>4</sup>

### 3. Summary of Test Method

3.1 The test sample is treated with a mixture of sulfuric and hydrofluoric acids. Upon dissolution, the excess hydrofluoric acid is expelled. Aqua regia and lanthanum chloride are added and the solution is diluted to a specific volume; and palladium concentration is determined by atomic absorption spectrophotometry. Absorbance of the samples is bracketed using a set of narrow range, matrix matched standards. A second sample taken at the same time is used to determine loss on ignition.

### 4. Significance and Use

4.1 This test method provides a means of determining the palladium content in fresh catalysts containing molecular sieves.

4.2 This test method is not intended to cover samples containing precious metals other than palladium.

### 5. Apparatus

5.1 *Analytical Balance*, capable of weighing to nearest 0.1 mg.

5.2 *Atomic absorption spectrophotometer*.

5.3 *Beakers*, TFE-fluorocarbon, 100-mL.

5.4 *Crucibles*, porcelain, 10-mL.

5.5 *Crucible Cover*, porcelain, for 10-mL crucible.

5.6 *Desiccator*.

5.7 *Flasks*, Erlenmeyer, 2000-mL.

5.8 *Graduated Cylinders*, glass, 5-mL, 10-mL, 25-mL, 50-mL, 250-mL, 500-mL, 1000-mL.

5.9 *Graduated Cylinder*, plastic, 10-mL.

5.10 *Hot plate*.

5.11 *Muffle furnace*, electrically heated, capable of 1000°C.

5.12 *Pipets*, 4-mL, 6-mL, 8-mL, 10-mL.

5.13 *Volumetric Flasks*, 100-mL, 500-mL.

5.14 *Watch glasses*, TFE-fluorocarbon, for 100-mL beaker.

5.15 *Weighing Papers*.

### 6. Reagents and Materials

6.1 *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>5</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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Specification D 1193.

6.3 *Hydrochloric acid*, concentrated, 36.5–38.0 wt. % or 12 M, sp gr 1.18.

6.4 *Hydrofluoric acid*, concentrated, 48.0–51.0 wt. % or 28.9 M, sp gr 1.17.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-32 on Catalysts and is the direct responsibility of Subcommittee D32.03 on Chemical Composition.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, ATTN: NPODS.

<sup>5</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 "Analytical Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."