



Designation: D5153 – 22

Standard Test Method for Palladium in Molecular Sieve Catalyst by Atomic Absorption Spectrophotometry¹

This standard is issued under the fixed designation D5153; 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 *Units*—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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

D7442 Practice for Sample Preparation of Fluid Catalytic Cracking Catalysts and Zeolites for Elemental Analysis by Inductively Coupled Plasma Optical Emission Spectroscopy

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

E1272 Specification for Laboratory Glass Graduated Cylinders

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 Composition.

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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.

2.2 *U.S. Federal Specification:*³

Federal Spec. NNN-P-395C Tolerance for Class A Pipets

3. Summary of Test Method

3.1 **Warning**—This test method requires the use of an extremely hazardous substance, hydrofluoric acid. Please refer to **Appendix X1** and **Appendix X2** for additional safety requirements.

3.2 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. 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 as the analysis sample, 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 (see Specification E1272).

³ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098.

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*.

5.16 *Fume Hood* suitable for use with hydrofluoric acid.

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.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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Specification D1193.

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.

NOTE 1—Refer to Section 8 of Practice D7442-08 for hazards associated with handling of acids.

6.5 *Nitric Acid*, concentrated, 69.0–71.0 wt. % or 15.7 M, sp gr 1.41.

6.6 *Sulfuric Acid*, concentrated, 95.0–98.0 wt. % or 18 M, sp gr 1.84.

6.7 *Aluminum Oxide* (Al_2O_3).

6.8 *Lanthanum Chloride* ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$).

6.9 *Palladium Wire*, 99.99 %.

6.10 *Aqua Regia*—Mix three parts by volume of concentrated hydrochloric acid (12 M) and one part by volume of concentrated nitric acid (15.7 M) immediately before use.

6.11 *Sulfuric Acid*, 15.8–16.3 wt. % or 3M. Cautiously add 250 mL of concentrated sulfuric acid (18 M) to 1250 mL of water—mix well and allow to cool.

6.12 *Palladium Standard Solution*, 500 mg/L. Dissolve 0.2500 ± 0.0001 g of palladium wire (99.99 %) in 25 mL of aqua regia. Evaporate the solution to dryness on a steam bath. Dissolve the remaining salts by addition of 25 mL of concen-

trated hydrochloric acid (12 M) and 25 mL of distilled water. Transfer the solution to a 500 mL volumetric flask and dilute to volume when cool.

NOTE 2—A commercially available atomic absorption palladium reference solution may be used if it is known to be reliable.

6.13 *Lanthanum Chloride Solution*—Dissolve 25.5 g of lanthanum chloride ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) in distilled water, dilute to 100 mL and mix well. This solution serves as an ionization suppressor in atomic absorption.

6.14 *Desiccant*, molecular sieve, type 4A.

7. Sampling

7.1 Obtain a representative sample of approximately 10 g of material from larger composites by riffing or splitting in accordance with subsection 5.12 of STP 447A⁵ or some other suitable means with the aim of obtaining a sample that represents the composition of the larger composite.

8. Procedure

8.1 *Preparation of Calibration Standards*:

8.1.1 If the concentration of aluminum oxide in the sample is unknown, digest a representative sample in acid and determine the aluminum concentration by atomic absorption spectrophotometry.

8.1.2 Transfer 30 mL of sulfuric acid (3M) to five TFE-fluorocarbon beakers.

8.1.3 To each beaker add the equivalent weight of aluminum oxide that would be present in 0.8000 g of sample as determined in 8.1.1.

NOTE 3—If it is available, 0.800 g of catalyst base material may be substituted for the aluminum oxide.

8.1.4 Transfer 0, 4, 6, 8 and 10 mL of the 500 mg/L palladium standard to the five TFE-fluorocarbon beakers.

8.1.5 Add 10 mL of concentrated hydrofluoric acid to each beaker.

8.1.6 Cover the beakers with TFE-fluorocarbon lids and digest on a hot plate at medium heat until all solid material is in solution (including any brown stains that may appear on the beaker walls) and light fumes are evolved.

NOTE 4—If brown stains do not redissolve, discard sample and prepare again.

8.1.7 Cool the solutions. Dilute to 70 mL with distilled water. Add 10 mL of aqua regia. Boil gently for a few minutes on a hot plate.

8.1.8 When the solution is cool, transfer quantitatively to a 500 mL volumetric flask.

8.1.9 Add 5 mL of the lanthanum chloride solution to each flask. Dilute to volume when the solution has reached room temperature.

8.1.10 The concentration of palladium will be 0, 4, 6, 8 and 10 mg/L.

NOTE 5—The standard solutions are stable for two months.

8.2 *Weighing*—Prepare a carefully riffled, finely ground sample of ambient-equilibrated catalyst. For example, the

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 *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.

⁵ *Manual on Test Sieving Methods*, ASTM STP 447A, ASTM International, 2005.