



Designation: D4782 – 24

Standard Test Method for Palladium in Molecular Sieve Catalyst by Wet Chemistry¹

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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 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

[B215 Practices for Sampling Metal Powders](#)

[D1193 Specification for Reagent Water](#)

[D3766 Terminology Relating to Catalysts and Catalysis](#)

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

[E105 Guide for Probability Sampling of Materials](#)

[E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process](#)

[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](#)

3. Summary of Test Method

3.1 The test sample is treated with hydrofluoric acid and evaporated to dryness to remove silica; the residue is fused with potassium pyrosulfate, and the cooled melt is dissolved in 4 % H_2SO_4 . A measured excess of 0.01 N potassium iodide (KI) is added to precipitate palladium as palladium iodide (PdI_2). The precipitate is filtered and washed, and a small amount of sodium sulfite is added to the filtrate to reduce any free iodine to iodide. The excess iodide is titrated with 0.01 N AgNO_3 potentiometrically, using a silver electrode versus a calomel electrode. A second sample taken at the same time is used to determine loss on ignition. **Warning**—This test method requires the use of an extremely hazardous substance, hydrofluoric acid. Refer to [Appendix X1](#). Assemble the necessary equipment and materials before proceeding with this test method.

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 metals other than palladium.

5. Apparatus

5.1 *pH Meter with Millivolt Scale* or automatic recording titrator.

5.2 *Silver-Sulfide Electrode.*

5.3 *Silver-Silver Chloride Double Junction* reference electrode with 10 % KNO_3 in the outer chamber.

5.4 *Fisher Burner.*

5.5 *Low-Temperature Muffle Furnace, 450 °C.*

5.6 *High-Temperature Muffle Furnace, 1000 °C.*

5.7 *Hot-Plate, with sand bath as a preferred option.*

5.8 *Magnetic Stirrer and TFE-Fluorocarbon-Coated Stirring Bars.*

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 www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

5.9 *Burets*, 25-mL, with 0.1-mL graduations or an equivalent.

5.10 *Volumetric Flasks*, 1000-mL.

5.11 *High-Silica Beakers*, 400-mL, with disposable borosilicate beakers as an option. Alternatively, a petri dish can be used for the fusion step, with polypropylene beakers being used thereafter.

5.12 *Watch Glasses*, preferably ribbed, 87 mm.

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

5.14 *Weighing Papers*.

5.15 *Porcelain Crucibles*, 10-mL.

5.16 *Graduated Cylinders*, 5-mL, 10-mL, 25-mL, 100-mL.

5.17 *Filter Paper*, Whatman No. 40, 11 cm.

5.18 *Funnel*, filter.

5.19 *Desiccator*.

5.20 *Crucible Cover*, porcelain.

5.21 Fume hood suitable for the materials in use.

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.³ 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 *Desiccant Molecular Sieve*, type 4A.

6.4 *Filter Paper Pulp*.

NOTE 1—Refer to Appendix on Hazards in Practice D7442 for hazards associated with handling of acids. Appendix X1 on safe handling of hydrofluoric acid is included with this test method.

6.5 *Hydrofluoric Acid (HF)*, 48 %.

6.6 *Potassium Iodide Solution*, 0.01 N—Dissolve 1.66 g of potassium iodide (KI) in distilled water, dilute to 1000 mL, and mix well.

6.7 *Potassium Pyrosulfate (K₂S₂O₈)*.

6.8 *Silver Nitrate Solution*, 0.01000 N—Dissolve 1.6989 g of silver nitrate (AgNO₃) in distilled water, dilute to 1000 mL, and mix well.

NOTE 2—The preparation of the AgNO₃ standard solution is a critical step. If any traces of chloride, organic matter or reducing agents are

present in the water, more AgNO₃ will be used in the back-titration and the final result will be low. Standardization is recommended.

6.9 *Sodium Sulfite Solution*, 10 % Dissolve 10 g of anhydrous sodium sulfite (Na₂SO₃) in 100 mL of distilled water.

6.10 *Sulfuric Acid (H₂SO₄)* concentrated, sp gr 1.84.

6.11 *Sulfuric Acid*, 48 % or 9 M—Add slowly, stir one part concentrated H₂SO₄ (96 %) to one part water, then cool.

6.12 *Sulfuric Acid*, 4 % or 0.72 M—Dilute 80 mL of 48 % sulfuric acid to 1000 mL.

7. Procedure

7.1 *Weighing*—Prepare a carefully riffled, finely ground sample of ambient-equilibrated catalyst. For example, the sample could be thinly spread on filter paper and exposed to room conditions for 16 h. **Warning**—All catalyst acid digestions and fusions should be carried out in a laboratory fume hood suitable for the materials in use.

7.1.1 Catalytic materials are, by nature, non-homogeneous in that they have a particle size distribution. In addition, the chemical composition as well as the physical properties of the catalytic materials can vary from particle to particle. Sampling a nonhomogeneous material in order to obtain a representative specimen for analysis can be difficult. It is recommended that the analyst develop a sampling plan suitable for the material being tested. For example, the test specimen should be obtained from larger composites by riffing or splitting in accordance with subsection 5.12 of STP447A6 with the aim of obtaining a test specimen that mirrors all of the properties of the larger composite. Guide E105 provides guidance on constructing a sampling plan and Practice E122 assists the analyst with determining the representative sample size. The analyst is also encouraged to consult STP B215-20 which contains excellent sampling guidance.

7.1.2 For determination of percent loss on ignition at 1000 °C, ignite a porcelain crucible at 1000 °C for at least 30 min, place in desiccator to cool, and weigh to nearest 0.1 mg. Transfer approximately 2.0 g of sample to the crucible and weigh to the nearest 0.1 mg.

7.1.3 For determination of palladium, transfer approximately 1.6 g of sample, weighed to the nearest 0.1 mg, into a 400-mL high-silica beaker.

7.2 *Loss on Ignition*:

7.2.1 Place the porcelain crucible containing the test sample in a muffle furnace maintained at 450 °C and heat for 30 min.

7.2.2 Transfer the crucible to a muffle furnace maintained at 1000 °C and heat for at least 1.5 h to constant weight.

7.2.3 Remove the crucible from the furnace, place in desiccator to cool, and weigh to nearest 0.1 mg.

7.2.4 Calculate weight percent loss on ignition at 1000 °C as follows:

$$\text{Weight \% LOI} = \frac{(I - F)}{(I)} \times 100 \quad (1)$$

where:

I = initial sample weight, and

F = final sample weight.

7.3 *Preparation of Test Sample for Titration*

³ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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.