



Designation: D1977-98 Designation: D 1977 – 03 (Reapproved 2008)

Standard Test Method for Nickel and Vanadium in FCC Equilibrium Catalysts by Hydrofluoric/Sulfuric Acid Decomposition and Atomic Spectroscopic Analysis¹

This standard is issued under the fixed designation D 1977; 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 is intended for the determination of nickel and vanadium in equilibrium catalysts where the vanadium and nickel concentrations are greater than 50 and 25 mg/kg, respectively.

1.2

1.1 This test method covers the determination of nickel and vanadium in equilibrium catalysts where the vanadium and nickel concentrations are greater than 50 and 25 mg/kg, respectively.

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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

D 1193 Specifications for Reagent Water

D 3766 Terminology Relating to Catalysts and Catalysis

E 105 Practice for Probability Sampling of Materials

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 288 Specification for Laboratory Glass Volumetric Flasks

E 456 Terminology Relating to Quality and Statistics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 U.S. Federal Specification

Federal Spec NNN-P-395C Tolerance for Class A Pipets⁴ U.S. Federal Specification:³

Federal Specification NNN-P-395C Tolerance for Class A Pipets

3. Summary of Test Method

3.1 The test specimen (as received) is decomposed with hydrofluoric and sulfuric acids. After complete volatilization of the hydrofluoric acid and cooling, the sulfate salts are diluted to the appropriate concentration range for analysis by flame atomic absorption, direct current plasma emission, or inductively coupled plasma emission spectroscopies. The instrument is calibrated with matrix-matched standards. Solutions of the test specimen are analyzed. Terminology

3.1 Definitions—See Terminology D 3766.

4. Significance and Use

4.1 This test method is a procedure by which catalyst samples may be compared on an inter- or intra-laboratory basis. Catalyst producers and user should find this test method to be of value. Summary of Test Method

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¹ This test method is under the jurisdiction of ASTM Committee D-32 on Catalyst and is the direct responsibility of Subcommittee D-32.03 on Chemical Composition. Current edition approved March 10, 1998. Published October 1998. Originally published as D1977-91. Last previous edition D1977-91.

² 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. Current edition approved April 1, 2008. Published April 2008. Originally approved in 1991. Last previous edition approved in 2003 as D 1977-03.

³ 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 Vol 11.01, volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

absorption, direct current plasma emission, or inductively coupled plasma emission spectroscopies. The instrument is calibrated with matrix-matched standards. Solutions of the test specimen are analyzed.

5. Significance and Use

5.1 This test method is a procedure by which catalyst samples may be compared on an inter- or intra-laboratory basis. Catalyst producers and user should find this test method to be of value.

6. Interferences

6.1 The enhancement of alumina in the samples are overcome by using matrix-matched standards. Any dilutions needed to achieve the working ranges for vanadium and nickel must contain the same Al_2O_3 (7800 ppm) concentration as the standards.

6.2 If using optical emission, consult tables showing interfering line near analyte lines; if significant overlap occurs, one must apply inter-element correction or choose an alternate emission line.

6.7. Apparatus

6.7.1 *Analytical balance*, capable of weighing to nearest 0.1 mg.

6.7.2 *Hot plate*, capable of maintaining $250^\circ\text{C} \pm 10^\circ\text{C}$ at surface.

6.7.3 *TFE fluorocarbon beaker*

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

7.2 *Hot Plate*, capable of maintaining $250 \pm 10^\circ\text{C}$ at surface.

7.3 *TFE Fluorocarbon Beaker*, 250 mL.

6.7.4 *Volumetric flasks*

7.4 *Volumetric Flasks*, borosilicate glass, 50, 100, 250, 500, and 1000-mL capacity conforming to Specification E 288.

6.5

7.5 *Pipettes*, borosilicate glass, 5, 10, and 25 mL, conforming to Federal Specification NNN-P-395C.

6.6

7.6 *Bottles*, polyethylene, 100 and 1000 mL.

7.

8. Reagents

7.1

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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 sufficient purity to permit its use without lessening the accuracy of the determination.

7.2

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean type IV reagent water, as defined in Specification D 1193.

7.3

8.3 *Required Reagents:*

7.3.1 *Hydrofluoric acid*

8.3.1 *Hydrofluoric Acid* (HF), concentrated, 48 %.

7.3.2 *Sulfuric acid*

8.3.2 *Sulfuric Acid*, (H_2SO_4), concentrated, 98 %.

7.3.3 *Sulfuric acid*

8.3.3 *Sulfuric Acid*, 49 volume %, add slowly, while stirring, one part of concentrated H_2SO_4 (98 %) to one part of water, then cool.

7.3.4 *Hydrochloric acid*

7.3.5 *Hydrochloric acid*

8.3.5 *Hydrochloric Acid*, 19 volume %, add slowly, while stirring, one part of concentrated HCl (38 %) to one part of water, then cool.

7.3.6

8.3.6 *Nitric Acid* (HNO_3), concentrated, 70 %.

7.3.7

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

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.