



Designation: D1977 – 22

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 D1977; 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 nickel and vanadium in equilibrium catalysts where the vanadium and nickel concentrations are greater than 50 and 25 mg/kg, respectively.

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](#)

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

[D4481 Test Method for Total Nickel in Fresh Alumina-Base Catalysts](#)

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

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

[E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)

[E288 Specification for Laboratory Glass Volumetric Flasks](#)

[E456 Terminology Relating to Quality and Statistics](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

2.2 *U.S. Federal Specification:*³

[Federal Specification NNN-P-395C Tolerance for Class A Pipets](#)

3. Terminology

3.1 *Definitions*—See Terminology [D3766](#).

4. Summary of Test Method

4.1 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.

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.

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.

³ Available from DLA Document Services, Building 4/D, 700 Robbins Ave., Philadelphia, PA 19111-5094, <http://quicksearch.dla.mil>.

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

7. Apparatus

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

7.2 *Hot Plate*, capable of maintaining 250 ± 10 °C at surface.

7.3 *PTFE Fluorocarbon Beaker*, 250 mL and PTFE watch glass.

7.4 *Volumetric Flasks*, borosilicate glass, 50, 100, 250, 500, and 1000 mL capacity conforming to Specification E288.

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

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

7.7 *Laboratory Fume Hood* suitable for use with hydrofluoric acid.

8. Reagents

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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean type IV reagent water or better, as defined in Specification D1193.

8.3 Required Reagents:

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

8.3.2 *Sulfuric Acid, (H₂SO₄)*, concentrated, 98 %.

8.3.3 *Sulfuric Acid*, 49 volume %, add slowly, while stirring, one part of concentrated H₂SO₄ (98 %) to one part of water, then cool.

8.3.4 *Hydrochloric Acid*, concentrated, 38 %.

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

8.3.6 *Nitric Acid (HNO₃)*, concentrated, 70 %.

8.3.7 *Reference Standard Solution*—1000 mg/L nickel (see Note 1).

NOTE 1—If emission spectrometry is to be used, standards must contain no interfering element(s) in concentration(s) great enough to yield an interference of more than 0.1% of the analytical response.

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. (USP), Rockville, MD.

8.3.8 *Reference Standard Solution*—1000 mg/L vanadium (see Note 1).

8.3.9 *Aluminum Chloride*, reagent grade, AlCl₃·6H₂O

8.3.10 *Aluminum Stock Solution*—39 000 mg/L Al₂O₃, dissolve 184.5 g of AlCl₃·6H₂O in water and dilute to 1 L and store in a polyethylene bottle.

8.3.11 *Hydrogen Peroxide*—3 % solution.

9. Sampling

9.1 The selection of a representative analytical sample from the bulk material is outside the scope of this test method. Parties using this test method for comparison purposes will have agreed on the selection of an analytical sample. If a sampling procedure is desired, Practice E105 is recommended.

10. Preparation of Standards

10.1 *Nickel*—Prepare standard solutions containing 0, 5.0, 20.0, and 50.0 mg/L Ni in a matrix of 7800 mg/L Al₂O₃ and 10 % hydrochloric acid by transferring 0, 5.0, 20.0, and 50.0 mL of the 1000-mg/L solution to 1000 mL volumetric flasks containing 200 mL of the 39 000 mg/L Al₂O₃ solution and 100 mL concentrated hydrochloric acid. Dilute solutions to volume with distilled water and store in polyethylene bottles.

10.2 *Vanadium*—Prepare standard solutions containing 0, 10.0, 25.0, 50.0 and 100.0 mg/L V in a matrix of 7800 mg/L Al₂O₃ and 10 % hydrochloric acid by transferring 0, 10.0, 25.0, 50.0, and 100.0 mL of the 1000-mg/L solution to 1000 mL volumetric flasks containing 200 mL of the 39 000 mg/L Al₂O₃ solution and 100 mL concentrated hydrochloric acid. Dilute solutions to volume with distilled water and store in polyethylene bottles.

11. Procedure

11.1 **Warning**—This test method requires the use of an extremely hazardous substance, hydrofluoric acid. Additional information for the safe handling of this chemical, HF, is included as Appendix X1 and Appendix X2. Read Appendix X1 and Appendix X2 and assemble the necessary safety equipment before proceeding with this test method.

Warning—All heating of acid solutions on a hot plate or with a gas burner should be conducted in a fume hood. Fusions on a gas burner should be conducted in a fume hood.

11.2 Weigh three test specimens of approximately 2.0 g each to the nearest 0.1 mg and transfer to 250 mL PTFE-fluorocarbon beakers. A reagent blank should be carried along with each set of samples.

11.3 First, add 10 mL 48 % sulfuric acid, then 10 mL concentrated nitric acid, and 10 mL concentrated hydrofluoric acid at last.

11.4 Transfer beaker and contents to a hot plate (no hotter than 250 °C to avoid melting the beaker) and evaporate to near dryness.

11.5 Remove beaker from hot plate and cool to ambient temperature.

11.6 Add 20 mL 19 % hydrochloric acid and 30 mL 3% hydrogen peroxide, cover with a PTFE watch glass and return beaker to hot plate.

11.7 Heat solution to boiling and continue to boil until all the salts are dissolved.

11.8 After dissolution is complete, remove beaker from hot plate and cool to ambient temperature.

11.9 Wash watch glass, catching washings in the beaker, and transfer solution quantitatively to a polymethylpentene (PMP) plastic 100-mL volumetric flask. Dilute to volume with water and mix well.

11.10 Using direct current argon plasma spectrometry (DCP), inductively coupled argon plasma spectrometry (ICP), or atomic absorption spectrophotometry (AAS), determine concentration of analytes in solutions of test specimens. If apparent concentration is greater than that of the most concentrated standard, perform an additional dilution so that result is bracketed by two or more standards. If internal standard is required, calibration standard must match preparation sample matrix.

12. Calculations

12.1 Calculate the concentration of nickel and vanadium as follows:

$$\text{mg/L metal} = \frac{C \times D}{S} \quad (1)$$

where:

- C = concentration of metal in solution, mg/kg,
- D = volume equivalent of dilution used for analysis; if solution 10.8 is used directly, $D = 100$ mL. If a further dilution is needed, $D = 100 \text{ mL} \times V_2/V_1$, where V_1 is the aliquot of solution 10.8 and V_2 is the volume to which it was diluted, and
- S = Sample mass.

13. Precision and Bias

13.1 *Test Program*—An interlaboratory study was conducted in which the named property was measured in three separate test materials in eight separate laboratories. Practice E691, modified for non-uniform data sets, was followed for the data reduction period.

13.2 *Precision*—Pairs of test results obtained by a procedure similar to that described in the study are expected to differ in absolute value by less than $2.772 S$, where $2.772 S$ is the 95 % probability interval limit on the difference between two test results, and S is the appropriate estimate of standard deviation. Definitions and usage are given in Terminology E456 and Practice E177, respectively.

Test Results for Ni (Consensus Mean)	95 % Repeatability Interval (Within Laboratory)	95 % Reproducibility Interval (Between Laboratories)
208 mg/kg	8 mg/kg (3.9 % of mean)	14 mg/kg (6.7 % of mean)
152 mg/kg	5 mg/kg (3.3 % of mean)	24 mg/kg (15.8 % of mean)
437 mg/kg	9 mg/kg (2.1 % of mean)	31 mg/kg (7.1 % of mean)

Test Results for V (Consensus Mean)	95 % Repeatability Interval (Within Laboratory)	95 % Reproducibility Interval (Between Laboratories)
1191 mg/kg	43 mg/kg (3.6 % of mean)	201 mg/kg (16.9 % of mean)
1898 mg/kg	72 mg/kg (3.8 % of mean)	303 mg/kg (16.0 % of mean)
3346 mg/kg	82 mg/kg (2.4 % of mean)	531 mg/kg (15.9 % of mean)

13.3 *Bias*—This test method described is without known bias since there is by definition no absolute standard for comparison.

14. Keywords

14.1 acid decomposition; atomic spectroscopy; ECAT; FCC equilibrium catalysts; nickel; vanadium

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