



Designation: D 1977 – 03

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 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 the standard. The values given in parentheses are for information only.

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 Specification 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 Pipets³

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

3. Terminology

3.1 *Definitions*—See Terminology D 3766.

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 Al₂O₃(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.

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 *TFE Fluorocarbon Beaker*, 250 mL.

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