



Designation: D5863 – 22

Standard Test Methods for Determination of Nickel, Vanadium, Iron, and Sodium in Crude Oils and Residual Fuels by Flame Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation D5863; 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 These test methods cover the determination of nickel, vanadium, iron, and sodium in crude oils and residual fuels by flame atomic absorption spectrometry (AAS). Two different test methods are presented.

1.2 *Procedure A, Sections 8–14*—Flame AAS is used to analyze a sample that is decomposed with acid for the determination of total Ni, V, and Fe.

1.3 *Procedure B, Sections 15–20*—Flame AAS is used to analyze a sample diluted with an organic solvent for the determination of Ni, V, and Na. This test method uses oil-soluble metals for calibration to determine dissolved metals and does not purport to quantitatively determine nor detect insoluble particulates. Hence, this test method may underestimate the metal content, especially sodium, present as inorganic sodium salts.

1.4 The concentration ranges covered by these test methods are determined by the sensitivity of the instruments, the amount of sample taken for analysis, and the dilution volume. A specific statement is given in [Note 1](#).

1.5 For each element, each test method has its own unique precision. The user can select the appropriate test method based on the precision required for the specific analysis.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard, unless specifically stated. Other units that appear in this standard are included for information purposes only or because they are in embedded pictures that cannot be edited.

1.7 *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 deter-*

mine the applicability of regulatory limitations prior to use. Specific warning statements are given in [8.1](#), [9.2](#), [9.5](#), [11.2](#), [11.4](#), and [16.1](#).

1.8 *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

[D4057](#) Practice for Manual Sampling of Petroleum and Petroleum Products

[D4175](#) Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

[D4177](#) Practice for Automatic Sampling of Petroleum and Petroleum Products

[D6299](#) Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these test methods, refer to Terminology [D4175](#).

4. Summary of Test Method

4.1 *Procedure A*—One to twenty grams of sample are weighed into a beaker and decomposed with concentrated sulfuric acid by heating to dryness. The residual carbon is burned off by heating at 525 °C in a muffle furnace. The inorganic residue is digested in dilute nitric acid, evaporated to incipient dryness, dissolved in dilute nitric and made up to volume with dilute nitric acid. Interference suppressant is

¹ These test methods are under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products, Liquid Fuels, and Lubricants and are the direct responsibility of Subcommittee [D02.03](#) on Elemental Analysis.

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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.

*A Summary of Changes section appears at the end of this standard

added to the dilute nitric acid solution. The solution is nebulized into the flame of an atomic absorption spectrometer. A nitrous oxide/acetylene flame is used for vanadium and an air/acetylene flame is used for nickel and iron. The instrument is calibrated with matrix-matched standard solutions. The measured absorption intensities are related to concentrations by the appropriate use of calibration data.

4.2 Procedure B—Sample is diluted with an organic solvent to give a test solution containing either 5 % (m/m) or 20 % (m/m) sample. The recommended sample concentration is dependent on the concentrations of the analytes in the sample. For the determination of vanadium, interference suppressant is added to the test solution. The test solution is nebulized into the flame of an atomic absorption spectrometer. A nitrous oxide/acetylene flame is used for vanadium and an air/acetylene flame is used for nickel and sodium. The measured absorption intensities are related to concentrations by the appropriate use of calibration data.

5. Significance and Use

5.1 When fuels are combusted, metals present in the fuels can form low melting compounds that are corrosive to metal parts. Metals present at trace levels in petroleum can deactivate catalysts during processing. These test methods provide a means of quantitatively determining the concentrations of vanadium, nickel, iron, and sodium. Thus, these test methods can be used to aid in determining the quality and value of the crude oil and residual oil.

6. Purity of Reagents

6.1 Reagent grade chemicals shall be used for 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.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 When determining metals at concentrations less than 1 mg/kg, use ultra-pure grade reagents.

6.3 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification **D1193**.

7. Sampling and Sample Handling

7.1 The objective of sampling is to obtain a sample for testing purposes that is representative of the entire quantity. Only representative samples obtained as specified in Practices **D4057** and **D4177** shall be used. Do not fill the sample container more than two-thirds full

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.

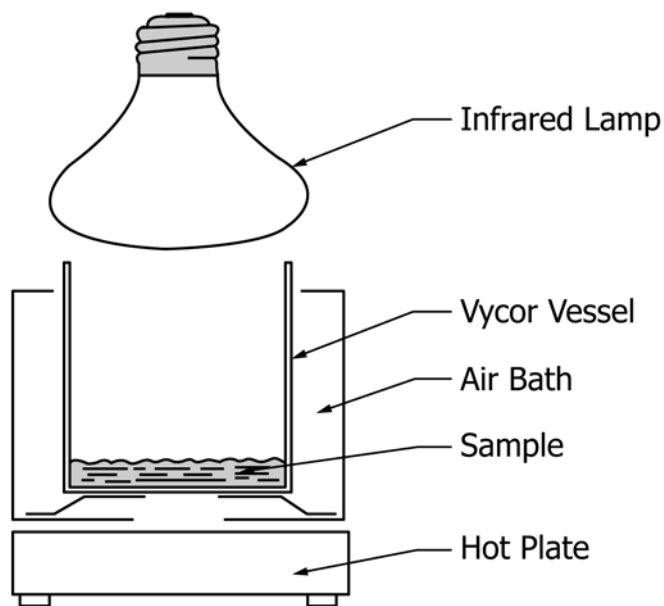


FIG. 1 Decomposition Apparatus

7.2 Prior to weighing, stir the sample and then shake the sample in its container. If the sample does not readily flow at room temperature, heat the sample to a sufficiently high and safe temperature to ensure adequate fluidity.

PROCEDURE A—FLAME ATOMIC ABSORPTION AFTER ACID DECOMPOSITION OF THE SAMPLE

8. Apparatus

8.1 Atomic Absorption Spectrometer, complete instrument with hollow cathode lamps and burners with gas supplies to support air-acetylene and nitrous oxide-acetylene flames. (**Warning**—Hazardous. Potentially toxic and explosive. Refer to the manufacturer's instrument manual for associated safety hazards.)

8.2 Sample Decomposition Apparatus (optional)—This apparatus is described in **Fig. 1**. It consists of a borosilicate glass 400 mL beaker for the test solution, an air bath (**Fig. 2**) that rests on a hot plate and a 250 W infrared lamp supported 2.5 cm above the air bath. A variable transformer controls the voltage applied to the lamp.

8.3 Glassware—Borosilicate glass 400 mL beakers, volumetric flasks of various capacities and pipettes of various capacities. When determining concentrations below 1 mg/kg, all glassware must be thoroughly cleaned (or soaked overnight) with 5 % HNO_3 and rinsed five times with water.

8.4 Electric Muffle Furnace, capable of maintaining $525^\circ\text{C} \pm 25^\circ\text{C}$ and sufficiently large to accommodate 400 mL beakers. The capability of an oxygen bleed is advantageous and optional.

8.5 Steam Bath.

8.6 Temperature Controlled Hot Plate, (optional).

8.7 Drying Oven, (optional), explosion-proof, if used to heat crude oils to obtain fluidity.