



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