



Designation: D1318 – 23

Standard Test Method for Sodium in Residual Fuel Oil (Flame Photometric Method)¹

This standard is issued under the fixed designation D1318; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method covers the determination of sodium in residual fuel oil by means of a flame photometer. Its precision in low ranges limits its application to samples containing more than 15 mg/kg sodium. Other elements commonly found in residual fuel oil do not interfere.

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 problems 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.* For specific hazard statements see 7.3, 7.5, 7.7, 9.2, 7.8, 7.9, and Note 3.

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

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

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is 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.

Measurement System Performance

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

4. Summary of Test Method

4.1 A weighed sample is reduced to a carbonaceous ash under controlled conditions. The residual carbon is removed by heating in a muffle furnace at 550 °C. The ash is dissolved, diluted to volume, and the sodium determined by means of a flame photometer.

5. Significance and Use

5.1 Excessive amounts of sodium can indicate the presence of materials that cause high wear of burner pumps and valves, and contribute to deposits of boiler heating surfaces.

6. Apparatus

6.1 *Flame Photometer*, capable of isolating the sodium doublet at 589 nm and stable enough to give repeatable measurements that do not vary more than 5 % of their mean in the 2 mg/kg to 20 mg/kg range of sodium.

6.2 *Platinum Dish*, 100 mL capacity, approximately 35 mm in depth.

6.3 *Electric Muffle Furnace*, capable of operating over a variable range from 200 °C to 600 °C and of maintaining a temperature of 550 °C $\pm$ 50 °C.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades can be used, provided it is first ascertained that the reagent is of

³ *ACS 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.

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

sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or III of Specification **D1193**.

7.3 Hydrochloric Acid (sp gr 1.19)—Concentrated hydrochloric acid (HCl). (**Warning**—Poison. Causes severe burns. Harmful or fatal if swallowed or inhaled.)

7.4 Hydrochloric Acid (1+9)—Mix 1 volume of HCl (sp gr 1.19) with 9 volumes of water.

7.5 Hydrofluoric Acid (48 %)—Concentrated hydrofluoric acid (HF). (**Warning**—Poison. Causes severe burns. Harmful or fatal if swallowed or inhaled.)

7.6 Sodium Solution, Standard (1000 mg Na/L)—Dissolve $3.088 \text{ g} \pm 0.005 \text{ g}$ of dried sodium sulfate (Na_2SO_4) in water and dilute to 1 L in a volumetric flask. Store in a polyethylene bottle.

7.7 Sulfuric Acid (1+1)—Carefully add, while stirring, 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) to 1 volume of water. (**Warning**—Poison. Causes severe burns. Harmful or fatal if swallowed or inhaled.)

7.8 Ethyl Alcohol, $\text{C}_2\text{H}_6\text{O}$, 95 % reagent grade. (**Warning**—Flammable.)

7.9 2-Propanol, $\text{C}_3\text{H}_8\text{O}$, 99 % reagent grade (**Warning**—Flammable.)

8. Sampling

8.1 Sampling shall be done in accordance with Practices **D4057** or Test Method **D4177**.

8.2 Use a clear, clean glass pint bottle, previously rinsed twice with HCl (1+9) and once with water and allowed to dry, for sampling the bulk material or plant streams. Obtain a representative sample but do not fill the bottle more than about two-thirds full. Warm viscous samples until they can be mixed readily. Stir up any material that has settled out and shake the sample for 3 min just prior to weighing it out.

8.3 Optimum sample size for most instruments is that which contains from 0.5 mg to 1.0 mg of sodium. Estimate the sample size as follows (see **Note 1**):

Sample size, g = $750/\text{estimated sodium content, mg/kg}$ (1)

NOTE 1—An estimate of the maximum amount of sodium in a sample can be obtained from its ash value. For example, an ash of 0.01 % would undoubtedly have less than 0.005 % sodium (50 ppm). If there is no estimate as to the probable sodium range in a sample, it is more expedient to weigh out a large amount, for example 60 g, because the test method provides for dilution of sample solutions that are more concentrated than the standards.

9. Calibration

9.1 Dilute the sodium solution (1000 mg Na/L) so as to obtain solutions containing 2 mg/L, 4 mg/L, 6 mg/L, 8 mg/L, 10 mg/L, 12 mg/L, 15 mg/L, 18 mg/L, and 20 mg/L (approximately equivalent to mg/kg). Store all dilute sodium solutions in polyethylene bottles.

9.2 Prepare the flame photometer for use as described in the manufacturer's instruction manual. Carefully adjust the pres-

sure of the gases (**Warning**—Dangerous.) used for flame combustion in the order described by the manufacturer until optimum control is achieved. Select a standard approximately in the middle of the optimum range of the instrument being used. While atomizing this medium-range standard, adjust the wavelength selector to the greatest response for the sodium doublet at about 589 nm and adjust all controls of the instrument to optimum performance. Finally, adjust the sensitivity control to give a proper scale reading.

9.3 Atomize each of the standard solutions and record the scale response for each. Run repeat checks on the medium-range standard selected in **7.2** after each of these standards to determine whether the flame photometer is functioning properly. Make the indicated adjustments, if required, and rerun the standards.

9.4 Prepare a working curve by plotting the milligrams of sodium per litre against the scale readings.

10. Procedure

10.1 Preparation of Sample:

10.1.1 Weigh into a thoroughly clean, dry 100 mL platinum dish the appropriate size sample (**8.3**) with an accuracy of at least 1 part per 100 parts of sample (**Note 2**). Samples larger than 75 g require a second filling of the platinum dish; for such samples, obtain the sample weight from the difference between the initial and final weights of the sample bottle. Place the platinum dish containing the fuel oil on a silica triangle properly supported, and heat with a bunsen burner until the contents ignite and burn readily (**Note 3**). Continue heating with the burner in such a manner that the sample burns at a uniform and moderate rate and only ash and carbon remain after burning ceases. For samples larger than 75 g, cool the dish and fill it approximately two-thirds full with additional well-shaken sample, and burn as above.

NOTE 2—Handle the platinum dish only with platinum-tip tongs and do not touch it with the fingers during the test. Carefully dust the bottom of the dish with a clean camel-hair brush before each weighing.

NOTE 3—If sample contains an appreciable amount of water, as indicated by spattering when heated, add a few millilitres of ethyl-alcohol (95 %) (**Warning**—Flammable) or isopropyl alcohol (99 %) (**Warning**—Flammable) before heating. Include the alcohol in the blank determination.

10.1.2 Place the dish in a muffle furnace at no more than 200 °C (**Note 3**). Slowly raise the temperature to $550 \text{ }^\circ\text{C} \pm 50 \text{ }^\circ\text{C}$. Leave the muffle door slightly ajar until only a little carbon remains in the dish; then close the door and continue the ignition until no carbon is visible.

NOTE 4—The platinum dishes should be placed on silica plates or silica triangles on the floor of the muffle furnace. Particular care must be exercised to avoid contamination of the sample with particles from the roof, walls, and the door of the furnace. During the initial ignition, the opening of the muffle door must be carefully adjusted so that the air flow into the muffle is not excessive. Too great an air flow causes high temperatures in the burning carbon and also possible loss of ash from the dish.

10.1.3 Cool the dish to room temperature, add 1 mL of H_2SO_4 (1+1) and about 1 mL of HF to the platinum dish (**Note 5**), evaporate to dryness under a hood, and replace the dish in the muffle furnace at $550 \text{ }^\circ\text{C} \pm 50 \text{ }^\circ\text{C}$ for 15 min.