Designation: D3605 – 22

Standard Test Method for Trace Metals in Gas Turbine Fuels by Atomic Absorption and Flame Emission Spectroscopy[1]

This standard is issued under the fixed designation D3605; 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 sodium, lead, calcium, and vanadium in Specification D2880 Grade Nos. 0-GT through 4-GT fuels at 0.5 mg⁄kg level for each of the elements. This test method is intended for the determination of oil-soluble metals and not waterborne contaminants in oil-water mixtures.

1.1.1 Test Method D6728 is suggested as an alternative test method for the determination of these elements in Specification D2880.

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 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]

D2880 Specification for Gas Turbine Fuel Oils

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

D6728 Test Method for Determination of Contaminants in Gas Turbine and Diesel Engine Fuel by Rotating Disc Electrode Atomic Emission Spectrometry

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 The samples are prepared to conform with the requirements of the method of standard additions, which is selected to obviate problems encountered with the direct analysis of typical gas turbine fuels that exhibit significant variations in physical properties. Different, but known, amounts of analyte are added to two portions of sample. These, together with the unaltered sample, are burned in the flame of an atomic absorption instrument that measures light absorption by the atomized metals. The analysis of the sample portions with added analyte provides the calibration information necessary to calculate the analyte content of the unaltered sample.

4.2 Lead is determined by atomic absorption in a premixed air-acetylene flame, and sodium is determined by atomic absorption or atomic emission in a premixed air-acetylene flame. Calcium and vanadium are determined by atomic absorption or atomic emission in a premixed nitrous oxide-acetylene flame.

4.3 Most experience with this test method has been in the atomic absorption mode, although flame emission has been used successfully. Details in the subsequent sections are written for the atomic absorption mode. If the flame emission mode is used, minor details in the subsequent sections must be altered to conform to standard practice for flame emission spectroscopy. The precision statement applies only to the atomic absorption mode.

NOTE 1—Some GT fuel users may wish to determine potassium in addition to other metals included in this method. Potassium can be determined in a manner similar to that for sodium using a potassium hollow cathode lamp, (unless flame emission mode is used) a wavelength of 766.4 mm, and an appropriate organo-potassium standard. Precision

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

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

data for potassium have not been determined.

5. Significance and Use

5.1 Knowledge of the presence of trace metals in gas turbine fuels enables the user to predict performance and, when necessary, to take appropriate action to prevent corrosion.

6. Apparatus

6.1 *Atomic Absorption Spectrometer,* capable of measuring radiation over the wavelength range from 280 nm to 600 nm. The instrument must be capable of measuring low-level signals (approximately 1 % absorption or 0.004 absorbance unit per milligram per litre vanadium). The instrument should also be equipped as follows.

6.1.1 *Burner,* with variable nebulizer and auxiliary oxidant supply to reduce non-atomic absorption from unburned hydrocarbons which cause interferences.

6.1.1.1 *Burner Head,* capable of supporting a nitrous oxide-acetylene flame.

6.1.1.2 *Burner Head,* single- or multiple-slot, capable of supporting an air-acetylene flame.

6.1.2 *Electronic Detection System,* capable of reading to the nearest 0.1 % absorption or 0.0004 absorbance.

6.1.2.1 The text describes the measurement of absorption signals that is, either percent absorption or absorbance. For instruments reading in percent absorption, absorption signals of 0.1 % absorption must be measurable. For instruments reading in absorbance, signals of 0.0004 absorbance must be measurable.

6.1.3 *Hollow Cathode Lamp Power Supply,* regulated to minimize drift.

6.1.4 *Monochromator,* capable of resolving the 318.34 nm – 318.40 nm vanadium doublet from the 318.54 nm vanadium line.

6.1.5 *Hollow Cathode Lamps,* one each for calcium, sodium, lead, and vanadium.

NOTE 2—Electrodeless-discharge lamps can be an acceptable alternative, but the precision of this method was determined with hollow cathode lamps.

6.1.6 When the instrument has flame-emission capability, the emission technique can be used for the analyses of sodium, calcium, and vanadium.

6.2 *Volumetric Flasks,* 25 mL.

6.3 *Glass Vials,* 40 mL, screw-cap type, polyethylene-lined caps.

6.4 *Syringe,* 100 µL, Hamilton type or equivalent.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in 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 ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *1,2,3,4-tetrahydronaphthalene,*[4] practical grade, analyte-sterile.

NOTE 3—Analyte-sterile 1,2,3,4-tetrahydronaphthalene can be prepared by extracting a portion of tetralin with an equal amount of hydrochloric acid in a covered screw-cap vial. Heat the vial on a steam bath for 1 h and shake the vial for 1 h. If the acid extracted 1,2,3,4-tetrahydronaphthalene and unextracted 1,2,3,4-tetrahydronaphthalene give indistinguishable absorption signals for each of the analytes under optimal experimental conditions, the unextracted 1,2,3,4-tetrahydronaphthalene can be used throughout this method.

7.3 *Organometallic Standards*—Oil-soluble salts of sodium, lead, calcium, and vanadium of known concentration.[5]

7.4 *Mixed Standard*—Prepare a mixed standard containing 250 mg/L each of sodium, lead, calcium, and vanadium by dissolving the appropriate amounts of organometallic standards in 1,2,3,4-tetrahydronaphthalene and making the required dilutions. Prepare fresh daily, as needed.

7.5 *Quality Control Samples,* preferably are portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section 9.

8. Sampling

8.1 Samples shall be taken in accordance with the instructions in Practice D4057 or D4177.

9. Procedure

9.1 Fill two clean 25 mL volumetric flasks to the line with sample. With the microlitre syringe add 50 µL of mixed standard to one flask and 100 µL to the other. Touch the needle of the syringe to the inner wall of the flask to ensure quantitative transfer of the standard. Invert and mix the contents. (The two flasks are now spiked with 0.5 mg/L and 1.0 mg/L of sodium, lead, calcium, and vanadium). Alternatively, weigh 25.0 g of sample into each of two clean disposable glass vials and add the standard in the same manner.

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[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.

[4] Tetralin (1,2,3,4-tetrahydronaphthalene), manufactured by E. I. duPont de Nemours and Co., has been found satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] Conostan standards, available from Conostan Division, Continental Oil Co., Ponca City, OK 74601, were used in determining the precision quoted in this method. Other standards are available from the Office of Standard Reference Materials, Room B314, Chemistry Bldg., National Institute of Standards and Technology, Washington, DC 20234, and from Angstrom, Inc., P. O. Box 252, Belleville, MI 48111, but the precision statement may or may not apply to results obtained with these standards.