Designation: D8130 – 17

Standard Test Method for Determination of Metals in Purified Terephthalic Acid (PTA) by Inductively Coupled Plasma Atomic Emission Spectrometric Method[1]

This standard is issued under the fixed designation D8130; 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 metal elements in purified terephthalic acid (PTA) by inductively coupled plasma atomic emission spectrometry (ICP-AES).This method is applicable to PTA samples containing sodium, chromium, cobalt, aluminum, titanium, potassium, magnesium, manganese, iron, nickel, molybdenum and calcium over 0.055 mg/kg, respectively.

1.2 In determining the conformance of the test results using this method to applicable specification, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

D7111 Test Method for Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Document:*[3]

EN ISO 8213 Chemical products for industrial use—Sampling techniques—Solid chemical products in the form of particles varying from powders to coarse lumps

2.3 *Other Document:*[4]

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Summary of Test Method

3.1 A PTA sample is dissolved in ammonium hydroxide solution. Calibration standards are prepared by mixing metallic standard materials in ammonium hydroxide solution. An internal standard material is added to the calibration standards and the sample solution to compensate for variations of test specimen introduction efficiency. The calibration standards and the sample solution are aspirated into the ICP-AES instrument. The concentrations of the elements in the sample solution are calculated by comparing emission intensity ratios of the sample solution and calibration standards to the internal standard.

4. Significance and Use

4.1 The presence of metals in PTA used for the production of polyester is undesirable because they may speed up or slow down the reaction and be impurities in the final product.

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

TABLE 1 Element and Recommended Wavelengths[A]

Element	Wavelength (nm)
Cobalt	228.616
Manganese	257.610
Iron	238.204
Chromium	267.716
Nickel	221.648
Sodium	589.592
Titanium	334.940
Molybdenum	202.033
Magnesium	285.231
Potassium	766.490
Calcium	317.933
Aluminum	396.153
Cadmium[B]	228.802
Yttrium[B]	371.029

[A] These wavelengths are only suggested and do not represent all possible choices. Other appropriate interference free wavelengths may also be used. Consult instrument manufacture's instructions or other ICP-AES references.
[B] Cadmium and Yttrium are used as internal standards.

4.2 Determination of the metals in PTA is often required. This test method is suitable for setting specifications and for use as an internal quality control where these products are produced or used.

4.3 This test method covers the determination of sodium, chromium, cobalt, aluminum, titanium, potassium, magnesium, manganese, iron, nickel, molybdenum and calcium.

5. Apparatus

5.1 *Inductively-Coupled Plasma Atomic Emission Spectrometer*—Any commercial sequential or simultaneous ICP-AES instrument capable of measuring emission intensities of the elements of interest (listed in Table 1).

5.2 *Analytical Balance,* readable to ±0.0001 g.

6. Reagents and Materials

6.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the reagent grade specification for analytical reagents of the American Chemical Society,[5] where such specifications are available. 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 performance or accuracy of the determination. Reagent chemicals shall be used for all tests.

NOTE 1—Calibration and detection limits of this test method can be biased by the purity of the reagents.

6.2 *Argon Gas*—99.99% minimum purity. (**Warning**—Argon is under high pressure.)

6.3 *Nitrogen Gas*—99.99% minimum purity. (**Warning**—Nitrogen is under high pressure.)

6.4 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type I reagent water conforming or exceeding Specification D1193. Freshly drawn water should be used for preparation of all stock and working standards, and sample solutions.

6.5 *Ammonium Hydroxide*—25 to 28 %(wt).

NOTE 2—The metal impurities in ammonia hydroxide should be no more than 10 µg/kg, and the non-metallic impurities should be no more than 100 mg/kg. The reagent may be purchased.

6.6 *Ammonium Hydroxide Solution*—Pipette 13 mL of ammonia hydroxide (6.5) to a 100 mL volumetric flask, fill the volumetric flask to the mark with water.

6.7 *Internal Standard Stock Solution*—1000 mg/L. The single element chosen for the internal standard should not be a component of the sample or calibration standard. Cadmium and yttrium were found to perform well as an internal standard for this test method and are recommended. The standard may be purchased.

6.8 *Internal Standard Solution*—100 mg/L. Pipette 10 mL of Internal Standard stock solution (6.7) to a 100 mL volumetric flask, fill the volumetric flask to the mark with water.

6.9 *Single Element Standard Stock Solution*—1000 mg/L. The test elements are listed in Table 1. The standards may be purchased.

6.10 *Single Element Standard Solution*—100 mg/L. Pipette 10 mL of the single element standard stock solution (6.9) to a 100 mL volumetric flask, fill the volumetric flask to the mark with water.

6.11 *Multi-element Mixed Standard Solution*—1.0 mg/L. Pipette 1 mL of each single element standard solution (6.10) to a 100 mL volumetric flask, fill the volumetric flask to the volume mark with water. Seal the volumetric flask and mix well. Prepare fresh multi-element mixed standard solution daily when samples are to be analyzed.

6.12 *Working Standards*—The working standards are prepared as follows: pipette 0, 0.5, 1.0, 2.0, 3.0 and 5.0 mL of the multi-element mixed standard solution (6.11) into six separate 100 mL volumetric flasks. And then pipette 0.2 mL of the internal standard (6.8) into each volumetric flask. Dilute to the mark with the ammonium hydroxide solution (6.6). Seal the volumetric flask and mix well. The working standards will be 0.0, 5.0, 10.0, 20.0, 30.0, 50.0 µg/L of each element of interest, and 200.0 µg/L of internal standard. Working standards are to be prepared daily when samples are to be analyzed.

7. Hazards

7.1 Consult current federal regulations, supplier's Safety Data Sheets, and local regulations for all materials used in this test method.

8. Sampling and Test Specimens

8.1 Use only representative samples obtained as described in EN ISO 8213, unless otherwise specified.

9. Preparation of Apparatus

9.1 Prepare the ICP spectrometer according to the manufacturer's instructions and parameter settings for the elements of

[5] *Reagent Chemicals, American Chemical Society Specifications*, 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.