



Designation: D8207 – 18

# Standard Test Method for Determination of Metals in Purified Terephthalic Acid (PAT) by Atomic Absorption (AA) Spectrometry<sup>1</sup>

This standard is issued under the fixed designation D8207; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of aluminum, chromium, cobalt, titanium, iron, manganese, molybdenum, sodium and nickel in purified terephthalic acid (PAT) by graphite furnace atomic absorption (GFAA) spectrometry. It is applicable to PAT samples containing sodium, chromium, cobalt, titanium, manganese, molybdenum and nickel over 0.004 mg/kg, and iron and aluminum over 0.054 mg/kg.

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. Some specific hazards statements are given in 6.3, 6.10, 6.11, Section , and 9.1.*

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:*<sup>2</sup>

D1193 Specification for Reagent Water

<sup>1</sup> 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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<sup>2</sup> 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.

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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

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

E300 Practice for Sampling Industrial Chemicals

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

E1184 Practice for Determination of Elements by Graphite Furnace Atomic Absorption Spectrometry

2.2 *Other Document:*<sup>3</sup>

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

## 3. Summary of Test Method

3.1 PAT sample in a platinum crucible is ignited and burned, then ashed at 750°C approximately for 45 min in a muffle furnace, and finally dissolved with sulfuric acid solution. The sample solution is analyzed by graphite furnace atomic absorption spectrophotometer. The external standard calibration is used for quantification.

## 4. Significance and Use

4.1 The presence of metals in PAT 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.

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

## 5. Apparatus

5.1 *Atomic Absorption Spectrometer with Graphite Furnace*, having background correction capable of removing nonspecific absorbance.

<sup>3</sup> Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

5.2 *Hollow Cathode Lamps.*

5.3 *Autosampler or Manual Pipettor.*

5.4 *Analytical Balance,* readable to  $\pm 0.0001$  g.

5.5 *Muffle Furnace,* with temperature control as (750  $\pm$  25°C).

5.6 *Platinum Crucible,* with capacity of 60 to 100 mL and weight no more than 22 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 of the analytical reagents of the American Chemical Society, where such specifications are available.<sup>4</sup> 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.

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

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**, Type II. Other reagent water types may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without lessening the bias and precision of the determination.

6.3 *Sulfuric Acid*—Ultrapure, over 96 % (w/w). It may be purchased. Metal impurities should be no more than 1  $\mu\text{g}/\text{kg}$ . (**Warning**—Poison, danger, corrosive. Causes severe burns. Harmful or fatal if swallowed or inhaled.)

6.4 *Sulfuric Acid Solution*—A 1:1 (v/v) mixture of sulfuric acid (6.3) and water.

6.5 *Single Element Standard Solution (100 mg/L)*—Standards either purchased or prepared from high purity grade chemicals or metals in **Table 1**.

6.6 *Multi-element Standard Stock Solution (1.0 mg/L)*—Pipette 1.0 mL of each single element standard solution (6.5) into a 100 mL volumetric flask, mix and dilute to mark with water (6.2).

6.7 *Mixed Working Standards*—Freshly prepared by transferring into twelve 100-mL volumetric flasks 0.00, 0.20, 0.40, 0.60, 0.80, 1.00, 2.00, 4.00, 6.00, 8.00, 10.00, 12.00 mL of the multi-element standard stock solution (6.6) and dilute with water (6.2) to the mark to give 0.0, 2.0, 4.0, 6.0, 8.0, 10.0, 20.0, 40.0, 60.0, 80.0, 100.0 and 120.0  $\mu\text{g}/\text{L}$  working standards.

NOTE 2—The ranges of the calibration curves for both iron and nickel are suggested to be between 0 and 20  $\mu\text{g}/\text{L}$  for being linear. And for other elements, the ranges are suggested to be between 0 and 120  $\mu\text{g}/\text{L}$ .

6.8 *Sodium Standard Solution (0.1 mg/L)*—Pipette 0.1 mL of sodium single element sodium standard solution (6.5) into a 100 mL volumetric flask, mix and dilute to mark with water (6.2).

6.9 *Sodium Working Standards*—Freshly prepared by transferring into six 100-mL volumetric flasks 0.00, 1.00, 2.00, 3.00, 4.00, 5.00 mL of sodium standard solution (6.8). Dilute with water (6.2) to the mark to give 0.0, 1.0, 2.0, 3.0, 4.0 and 5.0  $\mu\text{g}/\text{L}$  sodium working standards. For best results, prepare these working standards daily and store in plastic bottles, or as required.

6.10 *Argon Gas*—99.99 % minimum purity, (**Warning**—Argon is a compressed gas under high pressure.) for graphite furnace gas flow system.

6.11 *Ethanol*—Over 99.8 % (w/w). Metal impurities should be no more than 1  $\mu\text{g}/\text{kg}$ . (**Warning**—Ethanol is highly flammable.)

6.12 *Filter Paper*—Quantitative grade.

## 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, Test Specimens, and Test Units

8.1 Use only representative samples obtained as described in Practice **E300**, unless otherwise specified.

## 9. Preparation of Apparatus

9.1 Consult the manufacturer's instructions for the operation of the graphite furnace atomic absorption spectrometer. The present test method assumes that good operating procedures are followed. Because of the differences in equipment, all instrumental operating instructions cannot be provided. Instead, the analyst shall refer to the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, linear dynamic range, interference effects, and appropriate background correction shall be investigated and established for each individual analyte on that particular instrument. (**Warning**—Beware of high voltage and high temperature conditions when using this instrument.)

9.2 Recommended graphite furnace atomic absorption operating conditions are listed in **Table 1**.

**TABLE 1 Recommended Atomic Absorption Spectrometer Working Conditions**

Element	Wavelength nm	Slit Width nm	Pyrolysis Temperature °C	Atomization Temperature °C
Al	309.3	0.7	900	2600
Cr	357.9	0.2	800	2300
Co	240.7	0.2	400	2500
Ti	364.3	0.2	900	2600
Fe	248.3	0.2	800	2500
Mn	279.5	0.2	1000	2200
Mo	313.3	0.7	1000	2600
Na	589.0	0.2	500	2000
Ni	232.0	0.2	800	2500

<sup>4</sup> *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 *Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.