



Designation: E1834 – 09

# Standard Test Method for Determination of Lead in Nickel Alloys by Graphite Furnace Atomic Absorption Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E1834; 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 lead in nickel alloys in the concentration range 0.00005 % to 0.001 % by graphite furnace atomic absorption spectrometry (GF-AAS).

NOTE 1—If this test method is used to test materials having contents less than 0.0001 % lead, users in different laboratories may experience more than the usual 5 % risk that their results will differ by more than 50 % relative error.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazards associated with the use of this test method, see Practices E50.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1184 Practice for Electrothermal (Graphite Furnace) Atomic Absorption Analysis

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.08 on Ni and Co and High Temperature Alloys.

Current edition approved July 15, 2009. Published August 2009. Originally approved in 1996. Last previous edition approved in 2002 as E1834 – 96 (2002). DOI: 10.1520/E1834-09.

<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.

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1770 Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment

2.2 *ISO Standards:*<sup>3</sup>

ISO 5725:1986 Precision of Test Methods — Determination of Repeatability and Reproducibility for a Standard Test Method by Inter-Laboratory Tests

ISO 11437:1994 Nickel Alloys — Determination of Trace-Element Content — Electrothermal Atomic Absorption Spectrometric Method—Part 2: Determination of Lead Content

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

## 4. Summary of Test Method

4.1 The sample is dissolved in a mixture of HNO<sub>3</sub>, HF, and water and diluted to a known volume. A nickel-ammonium phosphate matrix modifier is added to an appropriate aliquot and a portion is injected into the graphite furnace atomizer of an atomic absorption spectrometer, which is provided with a background corrector. The sample is dried, pyrolyzed, and atomized. The absorbance of the resonance spectral line of lead is measured at 283.3 nm and compared with that from matrix-matched calibration solutions.

## 5. Significance and Use

5.1 This test method is used for the determination of trace levels of lead in nickel alloys by GF-AAS to check compliance with compositional specifications. It is assumed that the procedure will be performed by trained analysts capable of performing common laboratory practices skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory and proper waste disposal procedures will

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

be followed. Appropriate quality control practices must be followed such as those described in Guide E882.

## 6. Apparatus

6.1 *Atomic Absorption Spectrometer*, with graphite furnace atomizer, and equipped with an appropriate background corrector, a signal output device, such as a video display screen, a digital computer, a printer or strip chart recorder, and an autosampler.

6.2 *Pyrolytically Coated Graphite Tubes*, grooved, conforming to the instrument manufacturer's specifications.

6.3 *Pyrolytic Graphite Platforms*, L'vov design, fitted to the tubes specified in 6.2.

6.4 *Radiation Source*, lead hollow cathode lamp, or electrodeless discharge lamp with an appropriate power supply.

## 7. Instrument Parameters and Criteria

7.1 The atomic absorption spectrometer and graphite furnace atomizer are satisfactory if, after adjustment of the instrument and optimization of the furnace heating program as described in Practice E1770, they meet the criteria given in 7.4-7.7. For general discussion of the theory and instrumental requirements of GF-AAS, see Practice E1184.

7.2 Calibration solutions  $S_0$  through  $S_5$  prepared in accordance with 8.9 and summarized in Table 1 shall be used for testing criteria.

7.3 The parameters for the determination of lead and the establishing of the instrument criteria are as follows:

7.3.1 The injection volume is 20  $\mu$ L.

7.3.2 The peak area absorbance integration measurements shall be at a wavelength of 283.3 nm.

7.4 *Characteristic Mass*—The characteristic mass determined in accordance with Practice E1770 shall be within 20 % of that given in the manufacturer's literature.

7.5 *Precision*—The variability obtained from calibration solution  $S_3$  shall not exceed 10 % of the mean absorbance of the same solution, and the variability of calibration solution  $S_1$  shall not exceed 4 % of the mean absorbance of solution  $S_3$  when determined in accordance with Practice E1770.

7.6 *Limit of Detection*—The limit of detection of lead as described in Practice E1770 shall be less than 20 pg (equivalent to 1.0  $\mu$ g/L lead in the test solution or 0.2  $\mu$ g/g in the original sample).

7.7 *Linearity*—The linearity of the calibration as determined in Practice E1770 shall be not less than 0.7.

**TABLE 1 Lead Concentration of Calibration Solutions**

Calibration Solution	Concentration of Pb in Calibration Solution		Pb Concentration in Calibration Solution Corresponds to Concentration of Pb in Sample <sup>A</sup>
	$\mu$ g/L	ng/mL	
$S_0$	0	0	0
$S_1$	10	10	2
$S_2$	20	20	4
$S_3$	30	30	6
$S_4$	40	40	8
$S_5$	60	60	12

<sup>A</sup> Based 0.500 g sample/100 mL sample solution (10.1).

## 8. Reagents

8.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents and water shall conform to Practices E50. The reagents should be free of, or contain minimal amounts (< 0.01  $\mu$ g/g) of, lead.

8.2 *Dissolution Acid Mixture*—To 150 mL water carefully add 150 mL  $HNO_3$  and 150 mL HF. Mix and store in plastic bottle.

8.3 *Pure Nickel Metal*—99.9 % minimum purity nickel, and shall contain less than 0.1  $\mu$ g/g lead.

8.4 *Nickel, Stock Solution (50 g/L)*—Dissolve 25 g  $\pm$  0.1 g of the pure nickel in 200 mL of  $HNO_3$  (1 + 1) in a 600-mL beaker. Cool the solution and transfer to a 500-mL volumetric flask. Dilute to the mark and mix.

NOTE 2—If large nickel turnings or chunks are used, gentle heating may be required to complete the dissolution. When using carbonyl nickel powder, the solution must be filtered to remove undissolved carbon.

8.5 *Nickel, Solution (5.0 g/L)*—Transfer 10.0 mL of the Nickel Stock Solution to a 100-mL volumetric flask. Dilute to the mark and mix.

8.6 *Nickel Nitrate-Ammonium Phosphate Matrix Modifier Solution*—Dissolve 6.0 g  $\pm$  0.1 g high purity ammonium dihydrogen phosphate ( $NH_4H_2PO_4$ ) in 50 mL water. Transfer to 100 mL volumetric flask and add 20 mL of nickel solution (8.5). Dilute and mix. This solution shall be freshly prepared.

8.7 *Lead Reference Solution (100 mg/L)*—Dissolve 0.100 g  $\pm$  0.001 g of lead metal (99 % minimum purity) in 20 mL  $HNO_3$  (1 + 1) in a 250-mL beaker. Heat to assist dissolution. Cool and transfer to 1-L volumetric flask, dilute to mark, and mix. Store in polypropylene or high density polyethylene bottle.

8.8 *Lead Standard Solution (1.0 mg/L)*—Pipette 10.0 mL of the lead reference solution (8.7) into a 1-L volumetric flask. Add 20 mL  $HNO_3$ , dilute to volume, and mix. This solution shall be freshly prepared.

8.9 *Calibration Solutions*—Transfer 10.0 mL of the nickel solution (50 g/L) to each of six 100-mL volumetric flasks. Add (0, 1.0, 2.0, 3.0, 4.0, and 6.0) mL of lead standard solution, dilute, and mix. These calibration solutions contain (0, 10.0, 20.0, 30.0, 40.0, and 60.0)  $\mu$ g/L lead. These calibration solutions shall be identified as calibration solutions  $S_0$  through  $S_5$ , respectively (see Table 1).

## 9. Sampling and Sample Preparation

9.1 Sampling and sample preparation are to be performed by normal procedures agreed upon between the parties, or in the event of a dispute, in accordance with the relevant standard, if one is available.

9.2 The sampling procedure shall not involve any steps or procedures that can result in the loss or pick up of lead in the sample.

NOTE 3—Sometimes a heterogeneous scrap composite is homogenized by melting, then milling a laboratory sample from the solid. Arc melting of the sample or induction melting of the sample under vacuum can result in significant loss of several elements, including lead, that have a low vapor pressure. Arc melting of the sample should be avoided and induction melting should be performed only in a partially inert atmosphere.