



Designation: ~~E1834-96 (Reapproved 2002)~~ Designation: E 1834 - 09

Standard Test Method for Determination of Lead in Nickel Alloys by Electrothermal Graphite Furnace Atomic Absorption Spectrometric Method Spectrometry¹

This standard is issued under the fixed designation E 1834; 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 lead in nickel ~~and nickel~~ alloys in the concentration range of 0.00005 % to 0.001 % by ~~electrothermal-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 will experience more than the usual 5% risk that their results will differ by more than 50% relative error.

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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 ~~practice~~ test method, see Practices E 50.

2. Referenced Documents

2.1 ASTM Standards:²

E 50 ~~Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals, Ores, and Related Materials~~

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

E 135 ~~Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials~~

E 882 ~~Guide for Accountability and Quality Control in the Chemical Analysis Laboratory~~

E 1184 ~~Practice for Electrothermal (Graphite Furnace) Atomic Absorption Analysis~~

E1452 ~~Practice for Preparation of Calibration Solutions for Spectrophotometric and Spectroscopic Atomic Analysis~~

E 1601 ~~Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method~~

E 1770 ~~Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment~~ <http://www.astm.org/standards/e1834-09>

2.2 ISO Standards: ~~ISO Standard 11437—Nickel Alloys—Determination of Trace-Element Content—Electrothermal Atomic Absorption Spectrometric Method—Part 2: Determination of Lead Content~~³

ISO Guide 5725—Accuracy, Trueness, and Precision of Measurements, Methods and Results³—ISO Guide 5725:1986 Precision of

Test Methods — Determination of Repeatability and Reproducibility for a Standard Test Method by Inter-Laboratory Tests

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

3. Summary of Test Method

~~3.1~~ The sample is dissolved in a mixture of nitric acid, hydrofluoric acid, and water. The sample and a nickel-ammonium phosphate matrix modifier are injected into the electrothermal atomizer of an atomic absorption spectrometer, which is provided with a background corrector. Measurement of the absorbance is made at a wavelength of 283.3 nm. The lead concentration is determined from a calibration curve established with nickel solutions containing a known amount of lead. Terminology

3.1 Definitions—For definitions of terms used in this test method, refer to Terminology E 135.

¹ 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.

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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. Significance and Use

4.1 This test method is intended to be used for the determination of trace levels of lead in nickel and nickel alloys. 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 be followed. Summary of Test Method

4.1 The sample is dissolved in a mixture of HNO₃, 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 be followed. Appropriate quality control practices must be followed such as those described in Guide E 882.

6. Apparatus

6.1

6.1 Atomic Absorption Spectrometer, with electrothermal-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

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

6.4

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

7.

7. Instrument Parameters and Criteria

7.1 The atomic absorption spectrometer and electrothermal-graphite furnace atomizer are satisfactory if, after adjustment of the instruments and optimization of the furnace heating program as described in Practice E 1770, they meet the criteria given in 7.4-7.7.

7.2 Calibration solutions S₀

7.2 Calibration solutions S₀ through S₅ prepared in accordance with 7.9-8.9 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 µL.

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 E 1770 shall be within 20 % of that given in the manufacturer's literature.

7.5

7.5 Precision—The variability obtained from calibration solution S₃ shall not exceed 10 % of the mean absorbance of the same solution, and the variability of calibration solution S₁ shall not exceed 4 % of the mean absorbance of solution S₃ when determined in accordance with Practice E 1770.

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 ^A
	µg/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

^A Based 0.500 g sample/100 mL sample solution (910.1).

TABLE 2 Nominal Composition of Test Samples, %

Sample	Pb	Co	Cr	Mo	Ta	Ti	Al	Hf	W	V	Ni
1	0.0001	15	15	5	...	2.5	2.5	Balance
2	0.001	15	15	5	...	2.5	2.5	Balance
3	<0.0001	10	8	...	2.5	1.5	5	1.5	10	...	Balance
4	0.0004	10	8	...	2.5	1.5	5	1.5	10	...	Balance
5	0.001	10	8	...	2.5	1.5	5	1.5	10	...	Balance
6	<0.00005	14	10	3	...	4.5	6	1	Balance

TABLE 3 Results of the Statistical Analysis, Lead in Nickel Alloys

Test Material	Average Pb Found, ppm $\mu\text{g/g}$	Repeatability (s_r , ISO 5725) ^A	Reproducibility (S_M , ISO 5725 Practice E 1601) ^B	Reproducibility Index (Practice E 1601)
6	0.04	0.27	0.26	650
3	0.14	0.33	0.42	300
1	1.6	0.26	0.45	28
4	4.0	0.59	0.86	21
2	9.6	1.31	2.13	22
5	10.7	1.42	2.06	19

^AEquivalent to $2.8 \times S_M$ where S_M is minimum standard deviation (see Practice E 1601).

^BEquivalent to R , reproducibility index (see Practice E 1601).

6.6

7.6 Limit of Detection—The limit of detection of lead as described in Practice E 1770 shall be less than 20 pg (equivalent to 1.0 $\mu\text{g/L}$ lead in the test solution or 0.2 ppm $\mu\text{g/g}$ in the original sample).

7.7.7 Linearity—The linearity of the calibration as determined in Practice E 1770 shall not be not less than 0.7.

7.8. Reagents

7.1

8.1 Purity and Concentration of Reagents—The purity and concentration of common chemical reagents and water shall conform to Practices E 50. The reagents should be free of₂ or contain minimal amounts (< 0.01 $\mu\text{g/g}$) of₂ lead.

7.28.2 Dissolution Acid Mixture—To 150 mL water carefully add 150 mL nitric acid HNO_3 and 150 mL hydrofluoric acid. HF. Mix and store in plastic bottle.

7.38.3 Pure Nickel Metal—99.9 % minimum purity nickel, and shall contain less than 0.1 ppm $\mu\text{g/g}$ lead.

7.48.4 Nickel, Stock Solution (50 g/L)— Dissolve 25 g \pm 0.1 g of the pure nickel in 200 mL of nitric acid (1:1) 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.

7.5

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 with water and mix.

7.68.6 Nickel Nitrate-Ammonium Phosphate Matrix Modifier Solution—Dissolve 6.0 g \pm 0.1 g high purity ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$) in 50 mL water. Transfer to 100 mL volumetric flask and add 20 mL of nickel solution (7.58.5). Dilute with water and mix. This solution shall be freshly prepared.

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

7.88.8 Lead Standard Solution (1.0 mg/L)— Pipette 10.0 mL of the lead reference solution (7.78.7) into a 1-L volumetric flask. Add 20 mL nitric acid and HNO_3 , dilute to volume with water volume, and mix. This solution shall be freshly prepared.

7.98.9 Calibration Solutions—Transfer 10.0 mL of the nickel solution (50 g/L) to each of six 100-mL volumetric flasks. Add respectively 0, (0, 1.0, 2.0, 3.0, 4.0, and 6.0) mL of lead standard solution (7.8). Dilute with water solution, dilute, and mix. These calibration solutions contain (0, 10.0, 20.0, 30.0, 40.0, and 60.0 $\mu\text{g-Pb/L}$; 60.0) $\mu\text{g/L}$ lead. These calibration solutions shall be identified as calibration solutions S_0 through S_5 , respectively (see Table 1).

8.

9. Sampling and Sample Preparation

8.1 Sampling and sample preparation are to be performed by procedures agreed upon between buyer and seller.