



Standard Test Method for Determination of Lead in Nickel Alloys by Electrothermal Atomic Absorption Spectrometric Method¹

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 atomic absorption spectrometry.

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.

1.2 *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 see Practices E 50.

2. Referenced Documents

2.1 ASTM Standards:

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²

E 1184 Practice for Electrothermal (Graphite Furnace) Atomic Absorption Analysis³

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and Spectroscopic Atomic Analysis³

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method³

E 1770 Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment³

2.2 ISO Standards:

ISO Standard 11437—Nickel Alloys—Determination of Trace-Element Content—Electrothermal Atomic Absorption Spectrometric Method—Part 2: Determination of Lead Content⁴

ISO Guide 5725—Accuracy, Trueness, and Precision of Measurements, Methods and Results⁴

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.

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.

5. Apparatus

5.1 *Atomic Absorption Spectrometer*, with electrothermal atomizer, 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.

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

5.3 *Pyrolytic Graphite Platforms*, L'vov design, fitted to the tubes specified in 5.2.

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

6. Instrument Parameters and Criteria

6.1 The atomic absorption spectrometer and electrothermal 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 6.4-6.7.

6.2 Calibration solutions S₀ through S₃ prepared in accordance with 7.9 and summarized in Table 1 shall be used for testing criteria.

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

6.3.1 The injection volume is 20 µL.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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 Oct. 10, 1996. Published December 1996.

² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.