



Standard Test Method for Determination of Low Levels of Antimony in Carbon and Low-Alloy Steel by Electrothermal Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E 1852; 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 antimony in carbon and low-alloy steel in the 0.0005 through 0.010 % range.

1.2 If this test method is used to test materials having contents less than 0.001 % antimony, users of different laboratories will experience more than the usual 5 % risk that their results will differ by more than 50 % relative error.

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.*

2. Referenced Documents

2.1 ASTM Standards:

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials²

E 1184 Practice for Electrothermal (Graphite Furnace) Atomic Absorption Analysis²

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analyses²

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method²

E 1770 Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment³

2.2 ISO Standards:

ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility for a Standard Test Method by Inter-Laboratory Tests⁴

ISO 10698 Steel—Determination of Antimony Content—Electrothermal Atomic Absorption Spectrometric Method⁴

3. Summary of Test Method

3.1 The sample is dissolved in hydrochloric and nitric acids and diluted to volume. An appropriate aliquot is injected into the electrothermal atomizer of an atomic absorption spectrometer, which is equipped with a background correction. The sample is dried, pyrolyzed, and atomized. The absorbance of the radiation from the external light source is measured and compared to the absorbance of samples to which known amounts of the sought element were added.

NOTE 1—In general, the deuterium correction system should be able to correct for the broad-band background absorbance up to 0.5 to 0.6 absorbance units. Zeeman systems should compensate for background levels as high as 1.0 to 1.5 absorbance units.

4. Significance and Use

4.1 This test method is to be used for the determination of trace levels of antimony in carbon and low-alloy steel. 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 background corrector and appropriate signal output device, such as video display screen, digital computer, printer or strip chart recorder, and autosampler. It is recommended that the instrument meet the following performance requirements after adjusting the instrument and optimizing the furnace heating program as described in Practice E 1770.

5.1.1 The characteristic mass determined in accordance with Practice E 1770 for antimony shall be less than 25 pg or within the manufacturer's tolerance.

5.1.2 The minimum precision of the most concentrated blank addition solution shall not exceed 10 % of the mean absorbance of the same solution. The minimum precision of the least concentrated blank addition solution (excluding Solution B₀) shall not exceed 4 % of the mean absorbance of the most concentrated blank addition solution when determined in accordance with Practice E 1770.

5.1.3 The limit of detection of antimony as described in

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 E01.01 on Iron, Steel, and Ferroalloys.

Current edition approved Dec. 10, 1996. Published February 1997.

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 West 42nd Street, 13th Floor, New York, NY 10036.