



Designation: E 1852 – 96^{ε1}

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.

^{ε1} NOTE—An Editorial correction was made in June 1997.

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 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 for Spectroscopic Atomic Analyses³

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 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—

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 E01.01 on Iron, Steel, and Ferroalloys.

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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.

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 μg 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 Practice E 1770 shall be less than 20 pg.

5.1.4 Unless the instrument is provided with automatic curve correction circuitry, the graph linearity shall not be less than 0.95 when determined in accordance with Practice E 1770.

5.2 *Graphite Tubes*, with pyrolytic coating and grooves for graphite platform, suitable for use with the electrothermal atomizer used.

5.3 *Graphite Platform*, pyrolytic graphite, L'vov design, to fit graphite tubes specified in 5.2.

5.4 *Labware*—To prevent contamination of the sample(s), all beakers, lids, volumetric flasks, and funnels must be cleaned with hot HNO₃ (1 + 1) before use.

6. Reagents

6.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents shall conform to Practices E 50. It is important that antimony shall not exceed 0.01 µg/mL in each of the reagents and 0.001 µg/mL in the water.

6.2 *Antimony Stock Solution (1 mL = 1 mg Sb)*—Dissolve 0.100 ± 0.0001 g high-purity antimony (minimum 99.9 % Sb) in 30 mL HCl + 5 mL HNO₃ in a 100-mL beaker. Boil gently to expel oxides of nitrogen. Cool and transfer the solution into a 100-mL volumetric flask. Dilute to mark with HNO₃(1 + 1) and mix. Store in polypropylene or high density polyethylene bottle.

6.3 *Antimony Standard Solution (1 mL = 10 µg Sb)*—Transfer 1.0 mL of the antimony stock solution to a 100-mL volumetric flask, dilute to the mark with HNO₃(1 + 1), and mix. Prepare this solution immediately before use.

7. Sampling and Sample Preparation

7.1 Sampling and sample preparation is to be performed by procedures agreed on between buyer and seller.

7.2 The sampling procedures shall not involve any steps or operations that can result in the loss of antimony in the sample.

NOTE 2—Arc melting of the sample or induction melting of the sample under vacuum may result in significant loss of several elements that have a low vapor pressure. Arc melting of the sample should be avoided and induction melting should be performed only in an at least partial inert atmosphere.

7.3 The laboratory sample is normally in the form of turnings, millings, or drillings. No further mechanical preparation of the sample is necessary.

7.4 The laboratory sample shall be cleaned by first washing in acetone and air drying.

7.5 If brazed alloy tools are used in the preparation of the sample, the sample shall be further cleaned by pickling in diluted nitric acid for a few minutes. The sample shall then be washed several times with water, then several times with acetone and air dried.

8. Procedure

8.1 *Sample Size*—For samples containing between 0.0005 and 0.0050 % antimony, the sample size shall be ≈1.00 g, weighed to the nearest 0.1 mg. For samples containing between 0.0050 and 0.010 % antimony, the sample shall be ≈0.25 g weighed to the nearest 0.1 mg.

8.2 *Blank*—Simultaneously with the sample, a blank test using the same quantities of all reagents shall be carried along. The antimony contents of the blank should be no greater than 10 ppb.

8.3 *Test Solution*—Transfer the test portion in accordance with 8.1 into a 250-mL beaker. Add 5 mL HCl and 50 mL HNO₃. Cover the beaker with a watch glass, heat gently until the reaction ceases, and boil for 1 min to remove the oxides of nitrogen.

8.3.1 If sample contains tungsten or niobium, or both, transfer test portion (see 9.1) to a 100-mL beaker and add 1 mL orthophosphoric acid, 15 mL hydrochloric acid, and 5 mL nitric acid. Cover beaker with watch glass, and heat gently until reaction ceases. Evaporate the solution to 2 to 3 mL; then add 25 mL nitric acid. Boil for 1 min to remove nitrous oxides. Carry along a separate blank test corresponding to this procedure.

8.3.2 Allow the solution, which may contain carbides, to cool. Add about 15 mL water, filter through medium texture filter paper, and collect the filtrate in a 200-mL volumetric flask. Wash the filter paper several times with warm water and collect the washings in the flask. Dilute to the mark with water and mix.

8.4 *Test Addition Solutions*—Transfer separate 20.0-mL aliquot of the test solution into a series of five 100-mL volumetric flasks. Using a micropipette, inject the respective volumes of antimony standard solution indicated in Table 1. Dilute to the mark with water and mix. These solutions are referred to as S₀, S₁, S₂, S₃, and S₄, respectively.

8.5 *Blank Addition Solutions*—Transfer separate 20.0-mL aliquot of the blank solution into a series of five 100-mL volumetric flasks. Using a micropipette, add the respective volumes of antimony standard solution indicated in Table 2. Dilute to the mark with water and mix. These solutions are referred to as B₀, B₁, B₂, B₃, and B₄, respectively.

8.6 Optical parameters used for the determination of antimony are given in Table 3.

8.7 The atomic absorption spectrometer and the electrothermal atomizer should be adjusted and optimized as described in Practice E 1770.

NOTE 3—The volume injected into the atomizer should be between 10

TABLE 1 Test Addition Solutions

Name of Solution	Volume of Antimony Standard Solution Added, µL	Concentration of Antimony Added in Test Addition Solutions, ng/mL	Corresponding Mass of Antimony Added, ng	
			Volume Injected, 10 µL	Volume Injected, 50 µL
S ₀	0	0	0.0	0.0
S ₁	100	10	0.1	0.5
S ₂	200	20	0.2	1.0
S ₃	400	40	0.4	2.0
S ₄	500	50	0.5	2.5