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Steel — Determination of antimony content — Electrothermal atomic absorption spectrometric method

*Aciers — Dosage de l'antimoine — Méthode par spectrométrie
d'absorption atomique à excitation électrothermique*



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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10698 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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International Organization for Standardization

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Steel — Determination of antimony content — Electrothermal atomic absorption spectrometric method

1 Scope

This International Standard specifies an electrothermal atomic absorption spectrometric method for the determination of the antimony content in steel.

The method is applicable to antimony contents between 0,000 5 % (*m/m*) and 0,010 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition*.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids and dilution of the solution to a known volume.

Introduction of a known volume of the solution into an electrothermal atomizer of an atomic absorption spectrometer.

Measurement of the atomic absorption of the 217,6 nm spectral resonance line energy emitted by an antimony lamp, using background correction.

Calibration by the standard additions technique.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 3 water as specified in ISO 3696.

4.1 Nitric acid, ρ about 1,40 g/ml.

4.2 Hydrochloric acid, ρ about 1,19 g/ml.

4.3 Orthophosphoric acid (H_3PO_4), ρ about 1,71 g/ml.

4.4 Nitric acid, ρ about 1,40 g/ml, diluted 1 + 1.

4.5 Antimony, standard solutions.

4.5.1 Stock solution, corresponding to 1,0 g of Sb per litre.

Weigh, to the nearest 0,1 mg, 0,100 g of the high

purity antimony metal [minimum purity 99,9 % (*m/m*)]. Transfer to a 100 ml beaker and dissolve in 30 ml of hydrochloric acid (4.2) and 5 ml of nitric acid (4.1). Heat until it is completely dissolved and boil gently to expel oxides of nitrogen. Cool and transfer the solution quantitatively to a 100 ml one-mark volumetric flask. Dilute to the mark with nitric acid (4.4) and mix. Store in a polyethylene bottle.

1 ml of this stock solution contains 1,0 mg of Sb.

4.5.2 Standard solution, corresponding to 0,010 g of Sb per litre.

Transfer 1,0 ml of the stock solution (4.5.1) to a 100 ml one-mark volumetric flask, dilute to the mark with nitric acid (4.4) and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 10 µg of Sb.

5 Apparatus

All volumetric glassware shall be grade A, in accordance with ISO 385-1, ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus, and

5.1 Micropipettes, of capacities 100 µl to 500 µl.

5.2 Autosampler, equipped with micropipettes of capacities 10 µl to 50 µl.

5.3 Atomic absorption spectrometer and electrothermal atomizer, equipped with a background corrector and a high-speed recorder or computerized readout.

The instrument shall be capable of using single-element hollow cathode or electrodeless discharge lamps operated at currents recommended by the manufacturer of the lamp and instrument.

The atomic absorption spectrometer and the electrothermal atomizer used will be satisfactory if, after optimization according to 7.3.4.2, they meet the precision criteria given in 5.3.1 to 5.3.3.

It is also desirable that the instrument should conform to the additional performance requirements given in 5.3.4.

5.3.1 Characteristic mass (see A.1)

The characteristic mass for antimony shall be less than 25 pg.

5.3.2 Minimum precision (see A.2)

The minimum precision of the most concentrated blank addition solution shall not exceed 10 % of the mean absorbance of the same solution and the minimum precision of the least concentrated blank addition solution (excluding solution B₁) shall not exceed 4 % of the mean absorbance of the most concentrated blank addition solution.

5.3.3 Limit of detection (see A.3)

The limit of detection of antimony shall be less than 20 pg.

5.3.4 Graph linearity (see A.4)

The graph linearity shall not be less than 0,95.

6 Sampling

Carry out sampling in accordance with ISO 377-2 or appropriate national standards for steel.

7 Procedure

7.1 Test portion

According to the expected antimony content, weigh, to the nearest 0,1 mg, the following mass (*m*) of the test portion:

- for antimony contents between 0,000 5 % (*m/m*) and 0,005 0 % (*m/m*): $m \approx 1,00$ g;
- for antimony contents between 0,005 0 % (*m/m*) and 0,010 % (*m/m*): $m \approx 0,25$ g.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents. The antimony content in the blank test solution should be not greater than 10 ppb.

7.3 Determination

7.3.1 Preparation of the test solution (see clause 10)

Place the test portion (7.1) in a 250 ml beaker. Add 5 ml of hydrochloric acid (4.2) and 5 ml of nitric acid (4.1). Cover the beaker with a watch glass, heat gently until the reaction ceases and boil for 1 min to remove oxides of nitrogen.

Allow the solution, which may contain carbides, to cool. Add about 15 ml of water, filter through a medium-texture filter paper and collect the filtrate in a 200 ml one-mark 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.

7.3.2 Preparation of the test addition solutions

Introduce separate 20,0 ml aliquots of the test solution (see 7.3.1) into a series of five 100 ml one-mark volumetric flasks. Then add, using a micropipette (5.1), the respective volumes of antimony standard

solution (4.5.2) indicated in table 1. Dilute to the mark with water and mix. These solutions are named S₁, S₂, S₃, S₄ and S₅, respectively.

7.3.3 Preparation of the blank addition solutions

Introduce separate 20,0 ml aliquots of the blank solution (see 7.2) into another series of five 100 ml one-mark volumetric flasks. Then add, using a micropipette (5.1), the respective volumes of antimony standard solution (4.5.2) indicated in table 2. Dilute to the mark with water and mix. These solutions are named B₁, B₂, B₃, B₄ and B₅, respectively.

Table 1

Name of the solution	Volume of antimony standard solution (4.5.2) added μl	Concentration of antimony added in the test addition solutions ng/ml	Corresponding mass of antimony added, ng	
			Volume injected	
			10 μl	50 μl
S ₁	0	0	0	0
S ₂	100	10	0,1	0,5
S ₃	200	20	0,2	1,0
S ₄	400	40	0,4	2,0
S ₅	500	50	0,5	2,5

Table 2

Name of the solution	Volume of antimony standard solution (4.5.2) added μl	Concentration of antimony added in the blank addition solutions ng/ml	Corresponding mass of antimony added, ng	
			Volume injected	
			10 μl	50 μl
B ₁	0	0	0	0
B ₂	100	10	0,1	0,5
B ₃	200	20	0,2	1,0
B ₄	400	40	0,4	2,0
B ₅	500	50	0,5	2,5