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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

Reference number
ISO 10698:1994(E)

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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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 0,0005 % (*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 B_1) 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:

a) for antimony contents between 0,000 5 % (*m/m*) and 0,005 0 % (*m/m*): $m \approx 1{,}00$ g;

b) 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 S_1, S_2, S_3, S_4 and S_5, 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 B_1, B_2, B_3, B_4 and B_5, 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/m	Corresponding mass of antimony added, ng Volume injected 10 µl	
			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

7.3.4 Measurement

7.3.4.1 Adjustment of atomic absorption spectrometer

See table 3.

Table 3

Element	Characteristic
Type of lamp	Antimony electrodeless discharge or cathode lamp
Wavelength	217,6 nm
Lamp current	Follow manufacturer's recommendations
Bandwidth	Follow manufacturer's recommendations
Background correction	Antimony line 217,6 nm is close to iron line 217,8 nm. If the zero member gives an absorbance comparable with the precision of the least concentrated calibration solution, background correction will be required

7.3.4.2 Optimizing the atomic absorption spectrometer settings and electrothermal atomizer

Set the required instrument parameters and align the electrothermal atomizer according to the manufacturer's instructions (see note 1).

NOTE 1 Optimum settings for the operating parameters vary from instrument to instrument. Scale expansion may have to be used to obtain the required readability.

Determine the optimum electrothermal atomizer parameters for the particular type of atomizer and sample volume, as recommended by the instrument manufacturer or by following normal laboratory practice.

Zero the instrument and set up the baseline on the recorder.

Check the zero stability and lack of spectral interference within the atomization system by running the pre-set heating programme for blank firing of the graphite atomizer. Repeat to ensure baseline stability.

Condition new graphite tubes prior to analytical use by firing at least twice.

Evaluate the criteria of 5.3.1 to 5.3.3 and the additional performance requirements of 5.3.4, to ensure that the instrument is suitable for the determination.

7.3.4.3 Spectrometric measurement

Using an autosampler (5.2), inject into the atomizer the predetermined volume (see note 2) of the test and blank addition solutions, in order of increasing instrument response.

NOTE 2 The volume injected into the atomizer should be between 10 µl and 50 µl, depending on sensitivity, matrix interferences and range of linearity.

Atomize each solution three times. Record the three readings by peak height measurements. Rank the values obtained in order of increasing numerical value ($x_1 < x_2 < x_3$), decide whether the smallest (x_1) or the largest (x_3) is suspected to be an outlier and apply Dixon's test:

$$(x_3 - x_2)/(x_3 - x_1)$$

or

$$(x_2 - x_1)/(x_3 - x_1)$$

If the calculated ratio is lower than 0,970, average the readings. If greater than 0,970, reject the outlier and average the remaining two readings.

Check the instrument for memory effects, especially at high analyte levels, by running the blank firing programme. Reset the baseline to zero if necessary. Record all the readings by peak height measurements for the determination.

7.3.5 Plotting of the additions graphs (see note 3)

Calculate the average of three instrument readings for each of the blank addition solutions (solutions B).

Plot the average instrument reading against the mass of antimony added, expressed in nanograms, in the blank addition solutions.

Calculate the average of three instrument readings for each of the test addition solutions (solutions S).

Plot the average instrument reading against the mass of antimony added, expressed in nanograms, in the test addition solutions.

NOTE 3 In this method, any non-specific absorption effect is eliminated by background correction of the instrument.

Antimony may be present in the reagents. As the blank test is incorporated in the additions graph, this may not pass through the origin.

The standard additions graphs for the blank and the sample should be parallel.

8 Expression of results

8.1 Method of calculation

Determine the masses of antimony in the test and blank addition solutions, $m_{Sb,1}$ and $m_{Sb,0}$, expressed in nanograms, as the two intercepts on the mass axis by extrapolating the resulting straight lines in the two additions graphs (see 7.3.5). The difference $(m_{Sb,1} - m_{Sb,0})$ gives the net mass of antimony, m_{Sb}, in the test addition solution (solution S_1).

The net mass of antimony, m_{Sb}, can also be calculated using the least-square-fit method applied to the two straight lines, the blank addition solutions (solutions B) and the test addition solutions (solutions S).

The equation of the straight line is

$$y = a + bm$$

where

a and b are two constants that correspond respectively to the intercept on the y-axis and the slope of the straight line. Calculate b and a using the least-square-fit method as follows:

$$b = \frac{n\sum m_i y_i - \sum m_i \sum y_i}{n\sum m_i^2 - \left(\sum m_i\right)^2}$$

$$a = \frac{1}{n}\left(\sum y_i - b\sum m_i\right)$$

The intercept on the x-axis with the straight line of slope b is $(- a/b)$, therefore

$$m_{Sb,1} = \frac{1}{nb_1}\left(\sum y_i - b_1\sum m_i\right)$$

$$m_{Sb,0} = \frac{1}{nb_0}\left(\sum y_i - b_0\sum m_i\right)$$

$$m_{Sb} = m_{Sb,1} - m_{Sb,0}$$

where

b is the coefficient of regression;

n is the number of the solutions analysed;

a is the intercept on the y-axis;

m_i is the mass, expressed in nanograms, of antimony added, in the test or blank addition solutions;

y_i is the absorbance corresponding to the test or blank addition solutions;

$m_{Sb,1}$ is the mass, expressed in nanograms, of antimony obtained, from the test addition solutions;

$m_{Sb,0}$ is the mass, expressed in nanograms, of antimony obtained, from the blank addition solutions.

The antimony content, w_{Sb}, expressed as a percentage by mass, is given by the equation

$$w_{Sb} = \frac{m_{Sb} \times \dfrac{V_2 \times 10^3}{V_1} \times \dfrac{V_4}{V_3}}{m \times 10^9} \times 100$$

$$= \frac{m_{Sb} \times \dfrac{10^5}{V_1} \times \dfrac{200}{20} \times 10^2}{m \times 10^9} = \frac{m_{Sb} \times 0,1}{m \times V_1}$$

where

m_{Sb} is the mass expressed in nanograms, of antimony, in the test addition solution (S_1);

V_1 is the volume injected, in microlitres, of a series of test addition solutions and of the blank addition solutions (see tables 1 and 2);

V_2 is the volume, in millilitres, of a series of test addition solutions and blank addition solutions (see 7.3.2 and 7.3.3);

V_3 is the volume, in millilitres, of the aliquot of the test and blank solutions (see 7.3.2 and 7.3.3);

V_4 is the volume, in millilitres, of the test and blank solutions (see 7.3.1);

m is the mass, in grams, of the test portion (see 7.1).

8.2 Precision

A planned trial of this method was carried out by 18 laboratories, using seven levels of antimony contents, each laboratory making three determinations of antimony at each level (see notes 4 and 5).

The test samples used are listed in table B.1.

The results obtained were treated statistically in accordance with ISO 5725.

The data obtained showed a logarithmic relationship between antimony content and repeatability (r) and reproducibility (R and R_w) of the test results (see note 6), as summarized in table 4. The graphical representation of the data is shown in figure C.1.

NOTES

4 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725, i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time.

5 The third determination was carried out at a different time (on the different day) by the same operator as in note 4, using the same apparatus with a new calibration.

6 From the results obtained on day 1, the repeatability (r) and reproducibility (R) were calculated using the procedure specified in ISO 5725. From the first result obtained on day 1 and the result obtained on day 2, the within-laboratory reproducibility (R_w) was calculated.

9 Special cases

Use the following procedure for test portions containing tungsten and/or niobium.

Introduce the test portion (7.1) into a 100 ml beaker. Add 1 ml of orthophosphoric acid (4.3), 15 ml of hydrochloric acid (4.2) and 5 ml of nitric acid (4.1). Cover the beaker with a watch glass and heat gently until the reaction ceases. Evaporate the solution till a volume of 2 ml to 3 ml is reached, then add 25 ml of nitric acid (4.4) and boil for 1 min to remove oxides of nitrogen.

Proceed as specified in the second paragraph of 7.3.1.

Carry out a separate blank test (7.2) corresponding to this procedure.

10 Notes on procedure

Because of the high sensitivity of electrothermal atomic absorption, stringent precautions shall be taken to clean all glassware and to avoid contamination of sample, standard and calibration solutions from foreign material and dust from the laboratory atmosphere.

11 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis;

b) the method used by reference to this International Standard;

c) the results, and the form in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard, or any optional operation which may have influenced the results.

Table 4

Antimony content	Repeatability	Reproducibility	
% (m/m)	r	R	R_w
0,000 5	0,000 20	0,000 35	0,000 18
0,001 0	0,000 28	0,000 54	0,000 27
0,002 0	0,000 41	0,000 83	0,000 41
0,005 0	0,000 65	0,001 5	0,000 71
0,010 0	0,000 92	0,002 2	0,001 1

Annex A
(normative)

Procedures for the determination of instrumental criteria

For the preparation of standard methods of analysis using electrothermal atomic absorption spectrometry, the values for instrumental criteria should be decided from interlaboratory test results, by the working group in charge.

A.1 Determination of characteristic mass, m_c

Prepare a solution with the same matrix concentration as the blank solution, but with the element of interest at the following known concentration:

ρ ng/ml to give an absorbance A of approximately 0,1 at a predetermined injection volume.

Inject into the atomizer a predetermined volume of this solution and then the same volume of the blank, without scale expansion, and measure the absorbances A and A_0.

The characteristic mass, m_c, is given by the equation

$$m_c = \frac{\rho \times 0{,}004\,4 \times V_1}{A - A_0}$$

where

V_1 is the injected volume expressed in microlitres, of the solution, with concentration ρ ng/ml;

A_0 is the absorbance of the blank solution.

A.2 Determination of minimum precision

Inject into the atomizer the predetermined volume of the most concentrated addition solution 10 times to obtain 10 individual absorbance readings A_{Ai} and calculate the mean value $\bar{A}_A$.

Inject into the atomizer the same volume of the least concentrated addition solution (excluding the zero member) 10 times to obtain 10 individual absorbance readings A_{Bi} and calculate the mean value $\bar{A}_B$.

The standard deviations s_A and s_B of the most and least concentrated addition solutions respectively are obtained from the equations

$$s_A = \sqrt{\frac{\sum (A_{Ai} - \bar{A}_A)^2}{9}}$$

$$s_B = \sqrt{\frac{\sum (A_{Bi} - \bar{A}_B)^2}{9}}$$

The minimum precisions of the most and least concentrated addition solutions are obtained from $s_A \times 100/\bar{A}_A$ and $s_B \times 100/\bar{A}_A$, respectively.

A.3 Determination of limit of detection, m_{min}

Prepare two solutions each containing the same matrix concentration as the blank solution, but with the element of interest at the following known concentrations:

— ρ' ng/ml to give an absorbance A of approximately 0,01 at a predetermined injection volume;

— matrix blank to give an absorbance A_0.

Inject into the atomizer the predetermined volume of the solution of concentration ρ' ng/ml and the blank solution, 10 times each, using sufficient scale expansion to make the fluctuations in signal clearly visible.

Obtain the mean absorbance readings $\bar{A}'$ and $\bar{A}_0$.

The standard deviation $s_{A'}$ is given by the equation

$$s_{A'} = \sqrt{\frac{\sum (A'_i - \bar{A}')^2}{9}}$$

where

A'_i is the individual measured absorbance reading;

$\bar{A}'$ is the mean value of A'_i.

The limit of detection m_{min}, in picograms, is given by the equation

$$m_{\text{min}} = \frac{\rho' \times V_1 \times s_{A'} \times k}{\bar{A}' - \bar{A}_0}$$

where

V_1 is the volume expressed in microlitres, of the solution injected;

k is normally taken as 2.

A.4 Criterion for graph linearity

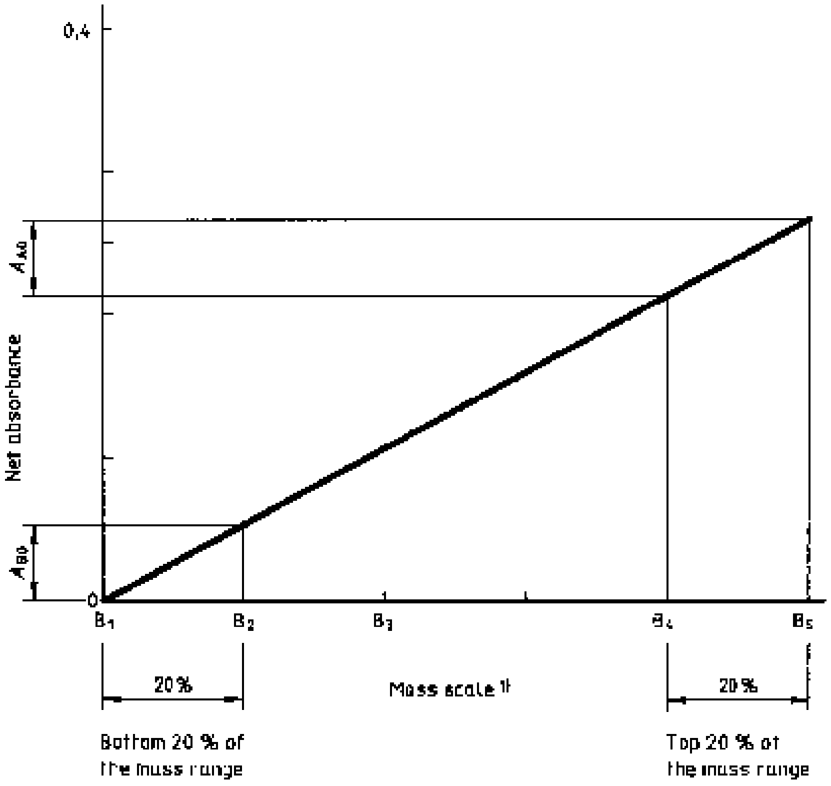
In the case of the standard additions method, the graph linearity (A_A/A_B) should be checked by following the procedure described in A.4.1 and A.4.2, and shall not be less than 0,95.

A.4.1 Linearity of the blank additions graph

Having established the blank additions graph, the value of the slope of the graph covering the top 20 % of the mass range, B_4 to B_5 (expressed as a change in absorbance), should not be less than 0,95 times the value of the slope of the bottom 20 % of the mass range, B_1 to B_2 (expressed as a change in absorbance) (see figure A.1).

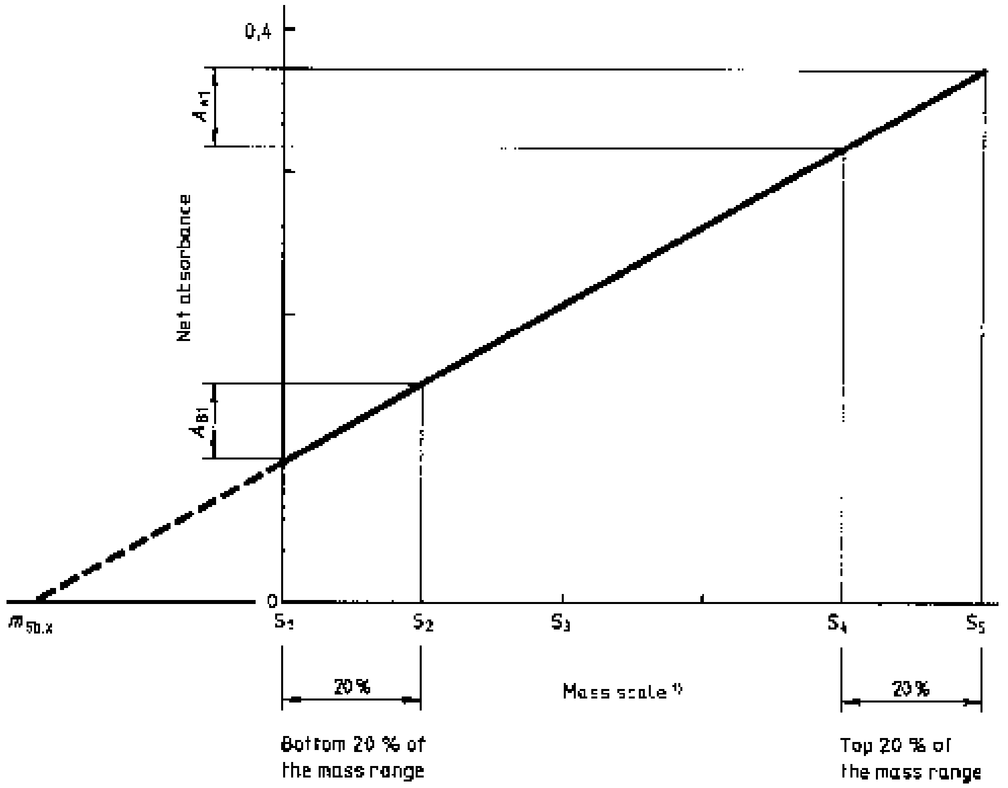
A.4.2 Linearity of the test additions graph

Having established the test additions graph, the value of the slope of the graph covering the top 20 % of the mass range, S_4 to S_5 (expressed as a change in absorbance), should not be less than 0,95 times the value of the slope of the bottom 20 % of the mass range, S_1 to S_2 (expressed as a change in absorbance) (see figure A.2).



1) Mass of antimony, expressed in nanograms, in the injected blank solution.

Figure A.1 — Blank additions graph



1) Mass of antimony, expressed in nanograms, in the injected test solution.

Figure A.2 — Test additions graph

A.4.3 Linearity of the graph throughout the entire mass range

A.4.3.1 Preparation of the solution for checking the graph linearity of the entire mass range

Obtain the predetermined mass of antimony, $m_{Sb,x}$, expressed in nanograms, in the test solution. In figure A.2, the intercept on the mass axis, obtained by extrapolating the resulting straight line, gives the mass of antimony.

Introduce separate 20,0 ml aliquots of the blank solution into four 100 ml one-mark volumetric flasks, then respectively add, using a micropipette, the following volumes, in microlitres, of antimony standard solutions.

Solution C$_1$: 0

Solution C$_2$: $\dfrac{[(m_{Sb,x} \times 10^5/V_1) + m_{Sb,y} \times 10^3] \times 1/5}{10}$

Solution C$_3$: $\dfrac{[(m_{Sb,x} \times 10^5/V_1) + m_{Sb,y} \times 10^3] \times 4/5}{10}$

Solution C$_4$: $\dfrac{[(m_{Sb,x} \times 10^5/V_1) + m_{Sb,y} \times 10^3]}{10}$

where

V_1 is the injected volume, in microlitres;

$m_{Sb,x}$ is the predetermined mass of antimony, in nanograms (this value is preferably the nearest multiple of 5);

$m_{Sb,y}$ is the mass of antimony, in micrograms, in 100 ml of the most concentrated solution (in tables 1 or 2, $m_{Sb,y} = 5$ µg).

Dilute to the mark with water and mix.

A.4.3.2 Plotting the additions graph

Atomize these solutions (C$_1$ to C$_4$) respectively as indicated in 7.3.4 and obtain the three instrument readings.

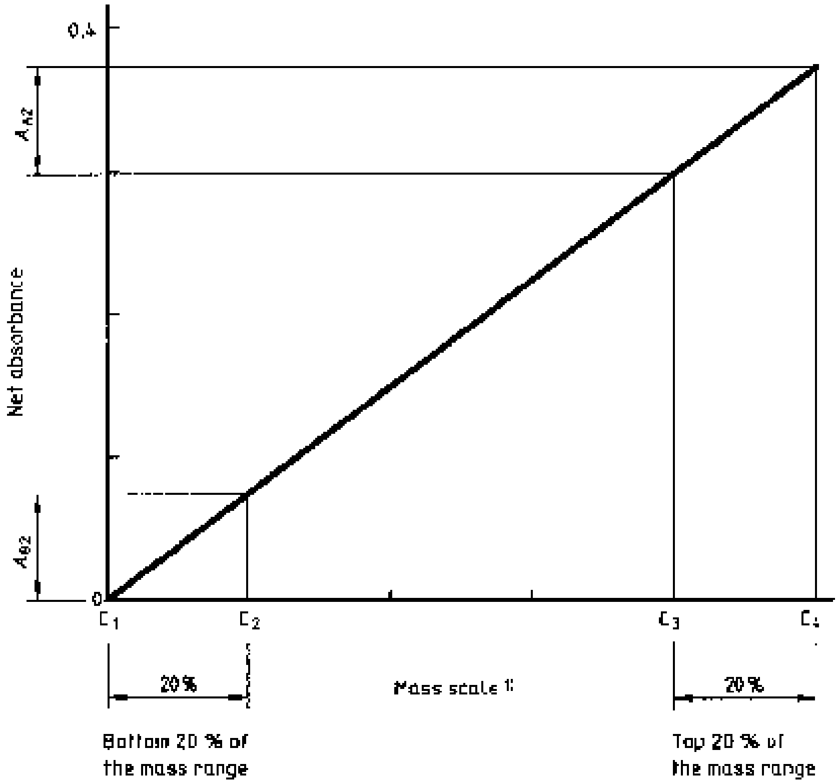
Calculate the average of these three instrument readings for four solutions.

Plot the average instrument reading against the mass of antimony, expressed in nanograms (see figure A.3).

A.4.3.3 Confirmation of the linearity of the graph

In the graph in figure A.3, throughout the entire mass range, the value of the slope of the graph covering the top 20 % of the mass range, C_3 to C_4 (expressed as a change in absorbance), should not be less than 0,95 times the value of the slope of the bottom 20 % of the mass range, C_1 to C_2 (expressed as a change in absorbance).

When the linearity is less than 0,95 times this slope, the volume injected into the atomizer should be controlled to fulfil the figure mentioned above. If the volume injected is as low as 10 µl, the volumes of the antimony standard solution added to the test and blank addition solutions (see table 1 or 2) should be decreased.



1) Mass of antimony, expressed in nanograms, in the injected blank solution and the test solution of predetermined mass of antimony.

Figure A.3 — Graph throughout the entire mass range

Annex B
(informative)

Additional information on the international co-operative tests

Table 4 was derived from the results of international analytical trials carried out in 1987 on seven steel samples in seven countries involving 16 laboratories.

The results of the trials were reported in document ISO/TC 17/SC 1 N 886, July 1991. The graphical representation of the precision data is given in annex C.

The test samples used are listed in table B.1.

Table B.1

Sample	Antimony content % (*m/m*)			Precision data		
	Certified	Found		Repeatability	Reproducibility	
		$\overline{w}_{Sb,1}$	$\overline{w}_{Sb,2}$	r	R	R_w
25Ni-20Cr-4,5Mo (stainless steel)	0,000 25 [1]	0,000 30	0,000 30	0,000 15	0,000 42	0,000 16
11Ni-17Cr-8Mo [2] (stainless steel)	0,000 5	0,000 53	0,000 52	0,000 20	0,000 33	0,000 19
13Ni-17Cr-3Mo (stainless steel)	0,001 4 [1]	0,001 31	0,001 31	0,000 29	0,000 44	0,000 35
30Ni (nickel steel)	0,002 4 [1]	0,002 25	0,002 22	0,000 28	0,000 72	0,000 27
ECRM 087-1 (carbon steel)	0,004 6	0,004 24	0,004 25	0,000 54	0,001 03	0,000 82
ECRM 085-1 (carbon steel)	0,007 3	0,006 91	0,006 84	0,000 80	0,001 89	0,000 66
BCS 456/1 (carbon steel)	0,012	0,012 4	0,012 5	0,001 32	0,003 90	0,001 62

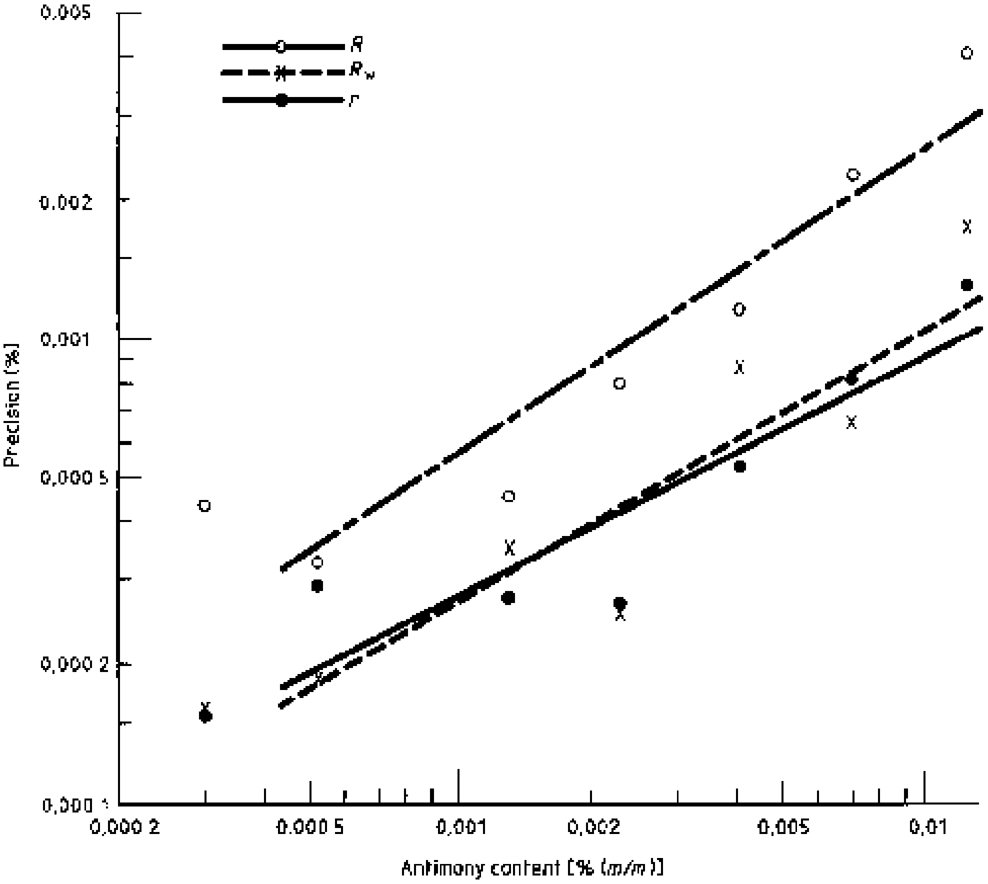
$\overline{w}_{Sb,1}$: general mean within a day
$\overline{w}_{Sb,2}$: general mean between days

1) Non-certified value

2) Sweden CRM: JK 8F

Annex C
(informative)

Graphical representation of precision data



$\lg r = 0{,}512\,7 \lg \overline{w}_{\mathrm{Sb},1} - 2{,}008\,7$
$\lg R_w = 0{,}592\,2 \lg \overline{w}_{\mathrm{Sb},2} - 1{,}784\,7$
$\lg R = 0{,}616\,1 \lg \overline{w}_{\mathrm{Sb},1} - 1{,}461\,8$

where

$\overline{w}_{\mathrm{Sb},1}$ is the average antimony content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{Sb},2}$ is the average antimony content, expressed as a percentage by mass, obtained between days.

Figure C.1 — Logarithmic relationship between antimony content (w_{Sb}) and repeatability (r) and reproducibility (R and R_w)

ICS 77.080.20

Descriptors: steels, chemical analysis, determination of content, antimony, atomic absorption spectrometric method.

Price based on 12 pages