INTERNATIONAL STANDARD

ISO 10701

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Steel and iron — Determination of sulfur content — Methylene blue spectrophotometric method

Aciers et fontes — Dosage du soufre — Méthode spectrophotométrique au bleu de méthylène

Reference number
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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 10701 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

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Steel and iron — Determination of sulfur content — Methylene blue spectrophotometric method

1 Scope

This International Standard specifies a methylene blue spectrophotometric method for the determination of sulfur in steel and iron.

The method is applicable to sulfur contents between 0,000 3 % (*m/m*) and 0,010 % (*m/m*). However, niobium, silicon, tantalum and titanium interfere in the determination of sulfur.

Depending on the concentration of the interfering elements, the application ranges and test portion masses given in table 1 apply.

Table 1

Maximum allowable content of the interfering elements % (*m/m*)				**Test portion**	**Application ranges Δw_S** % (*m/m*)
Nb	**Si**	**Ta**	**Ti**	g	
0,5	1,0	0,3	1,0	1,0	0,000 3 to 0,001 0
1,0	2,0	0,6	2,0	0,50	0,001 0 to 0,010

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 a mixture of hydrochloric and nitric acids.

Evaporation with perchloric acid until white fumes appear to remove hydrochloric and nitric acids.

Dissolution of the salts in hydrochloric acid. Evolution of hydrogen sulfide by reducing with a mixture of hydroiodic and hypophosphorous acids in a nitrogen atmosphere, distillation, and absorption into zinc acetate solution.

Formation of methylene blue by reacting with *N*,*N*-dimethyl-*p*-phenylenediamine and iron(III) solution.

Spectrophotometric measurement at a wavelength of about 665 nm.

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4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade with a very low sulfur content, and only freshly prepared grade 2 water as specified in ISO 3696.

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

4.2 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 15.

4.3 Perchloric acid, ρ about 1,54 g/ml.

4.4 Hydrobromic acid, ρ about 1,48 g/ml.

4.5 Mixture of hydrochloric and nitric acids.

Mix one volume of hydrochloric acid (4.1) and one volume of nitric acid, ρ about 1,40 g/ml.

Prepare immediately before use.

4.6 Reducing reagent solution.

Transfer 200 ml of hydriodic acid [about 57 % *(m/m)*] and 50 ml of hypophosphorous acid [about 50 % *(m/m)*] into the purifying apparatus (see figure 1). Purge with nitrogen (4.12) at a flowrate of 100 ml/min for 10 min, to mix the acids and expel air from the system. Switch on the electric heating mantle. Heat to boiling and boil gently for about 120 min at a temperature of about 115 °C in a current of nitrogen. When purification is completed (see 10.3), switch off the electric heating mantle. Then cool the solution and keep it in a brown bottle.

4.7 Absorbing solution.

Dissolve 5 g of zinc acetate dihydrate [$(CH_3COO)_2Zn.2H_2O$] in 400 ml of water. Add 200 ml of sodium hydroxide solution, 30 g/l, and 70 g of ammonium chloride, and then dilute to 1 000 ml with water.

4.8 Iron, 10 g/l solution.

Weigh, to the nearest 0,01 g, 1,00 g of pure iron which is free from sulfur as sulfate. Transfer to a 300 ml beaker, cover with a watch glass, dissolve by heating with an addition of 20 ml of hydrochloric acid (ρ about 1,19 g/ml, diluted 1 + 1) and boil gently for about 10 min. Then add 2 ml of nitric acid (ρ about 1,40 g/ml) drop by drop to oxidize iron. Remove the oxides of nitrogen by boiling, and cool to room temperature. Transfer to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

4.9 Iron(III) chloride, solution.

Dissolve 1 g of iron(III) chloride hexahydrate ($FeCl_3.6H_2O$) in about 40 ml of water. Add 10 ml of hydrochloric acid (4.1) and dilute to 100 ml with water.

4.10 *N,N*-dimethyl-*p*-phenylenediamine in hydrochloric acid medium.

Dissolve 0,5 g of *N,N*-dimethyl-*p*-phenylenediamine chloride [$NH_2C_6H_4N(CH_3)_2.2HCl$] in about 100 ml of water. Add 230 ml of hydrochloric acid (4.1) and dilute to 500 ml with water.

4.11 Sulfur, standard solution.

4.11.1 Stock solution, corresponding to 1 g of S per litre.

Weigh, to the nearest 0,000 1 g, 5,435 2 g of potassium sulfate [minimum assay: 99,5 % *(m/m)*], previously dried at 110 °C for 2 h and cooled to room temperature in a desiccator. Dissolve in water, transfer to a 1 000 ml one-mark volumetric flask quantitatively, dilute to the mark and mix.

1 ml of this stock solution contains 1 mg of S.

4.11.2 Standard solution A, corresponding to 10 mg of S per litre.

Transfer 10,0 ml of the stock solution (4.11.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

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

4.11.3 Standard solution B, corresponding to 1 mg of S per litre.

Transfer 10,0 ml of the standard solution (4.11.2) to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare the solution immediately before use.

1 ml of this standard solution contains 1 µg of S.

4.12 Nitrogen.

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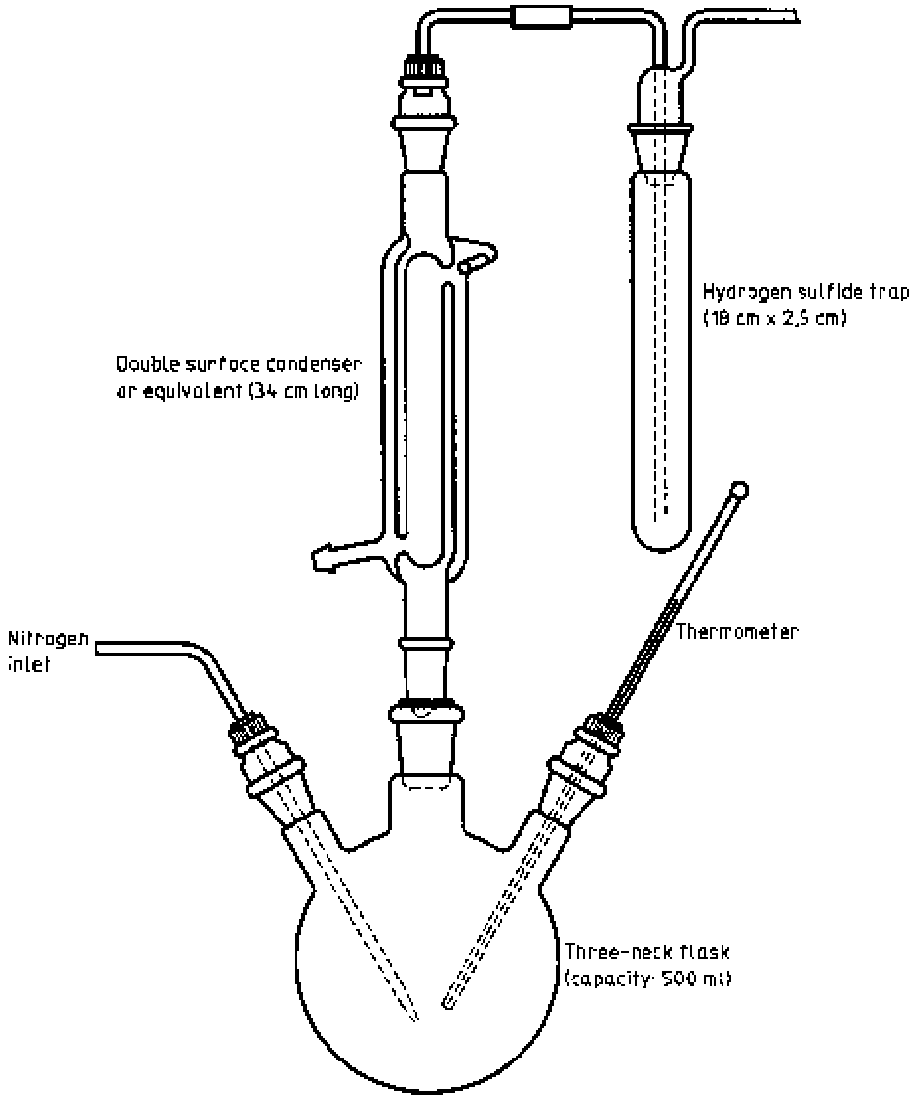

Figure 1 — Example of an apparatus for purification of the reducing mixture

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5 Apparatus

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

Ordinary laboratory apparatus, and

5.1 Apparatus for reduction and distillation

Assemble the apparatus for reduction and distillation as shown in figure 2. Close-fitting ground-glass joints shall be used.

When the apparatus is used for the first time, or after a long period of disuse, blank tests shall be carried out repetitively until stable low blank values are obtained.

5.1.1 Decomposition flask, about 300 ml in volume.

5.1.2 Reflux condenser, about 150 mm in length.

5.1.3 Gas washing bottle, about 150 ml in volume.

5.1.4 Absorption flask, one-mark volumetric flask of capacity 20 ml or 100 ml.

5.2 Spectrophotometer, equipped to measure absorbance at a wavelength of about 665 nm.

Dimensions in millimetres

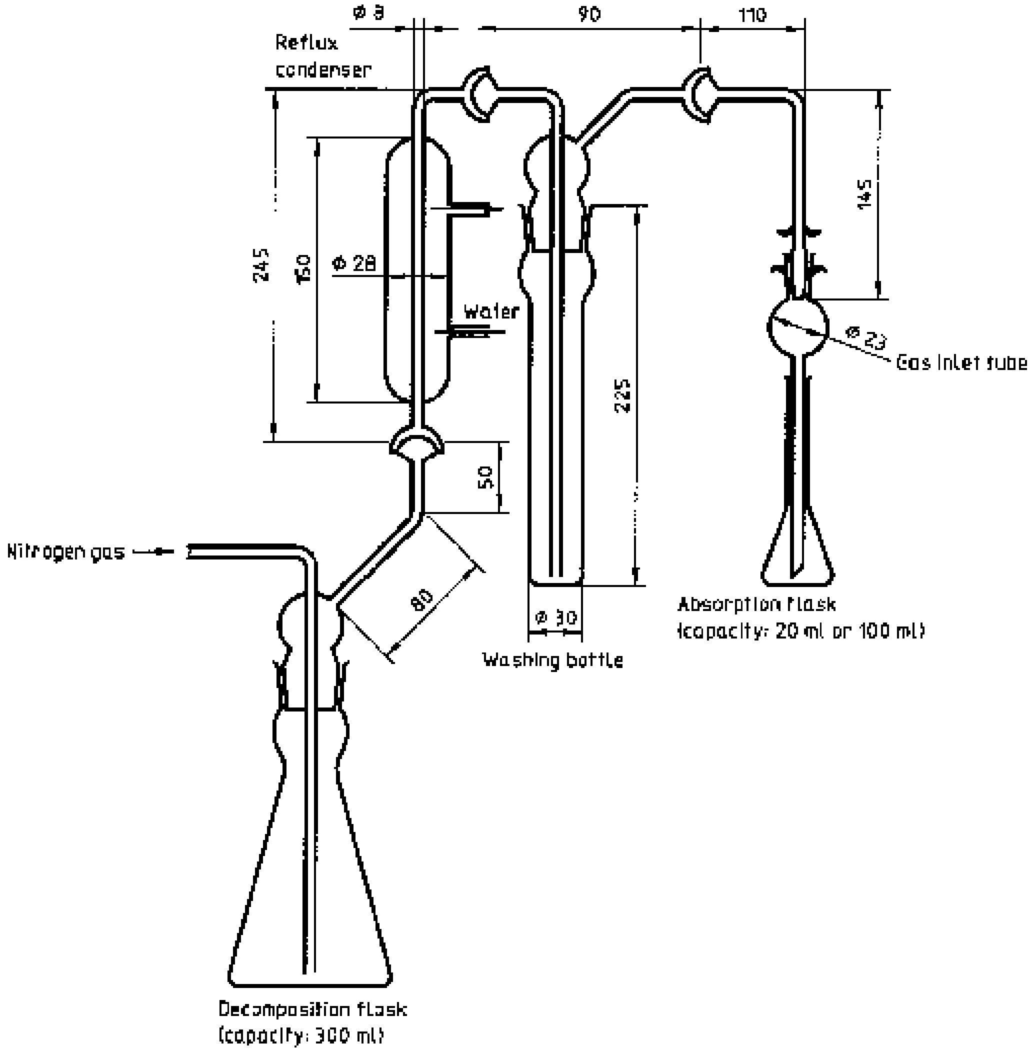

Figure 2 — Example of an apparatus for reduction and distillation

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6 Sampling

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

7 Procedure

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic matter in general.

7.1 Test portion

Weigh, to the nearest 1 mg, the mass given below as a function of the expected sulfur content:

a) sulfur contents from 0,000 3 % (*m/m*) to 0,001 0 % (*m/m*), mass of test portion about 1,00 g;

b) sulfur contents from 0,001 0 % (*m/m*) to 0,010 % (*m/m*), mass of test portion about 0,50 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. It is recommended that the blank value does not exceed 0,7 µg of sulfur for sulfur contents up to 0,001 % (*m/m*), or 1,5 µg of sulfur for sulfur contents from 0,001 % (*m/m*) to 0,010 % (*m/m*).

7.3 Determination

7.3.1 Preparation of the test solution

Place the test portion (7.1) in a decomposition flask (5.1.1). Add 15 ml of the mixture of hydrochloric and nitric acids (4.5). After standing at room temperature for about 30 min, heat gently until solvent action ceases.

Then, using a pipette, add 5,0 ml of perchloric acid (4.3) and 1,0 ml of iron solution (4.8), heat and evaporate until white fumes appear. After cooling, add 5 ml of hydrochloric acid (4.1). (See clause 9 for a possible modification of this procedure.) Heat again and evaporate to fuming on a hotplate at a temperature of about 300 °C. Then continue to evaporate until free from white fumes of perchloric acid and dry.

After cooling, add 10 ml of hydrochloric acid (4.1), heat to dissolve the salts and cool to room temperature.

7.3.2 Reduction and distillation

Add 20 ml of the reducing reagent solution (4.6) to the decomposition flask (5.1.1) and allow to stand for 10 min.

Pour 30 ml of water into the gas washing bottle (5.1.3). Place the appropriate volume of absorbing solution (4.7) in an absorption vessel (5.1.4), according to the expected sulfur content to be determined, as follows:

a) for sulfur contents less than 0,001 0 % (*m/m*), introduce 10 ml of the absorbing solution (4.7) into a 20 ml absorption flask;

b) for sulfur contents from 0,001 0 % (*m/m*) to 0,010 % (*m/m*), introduce 50 ml of the absorbing solution (4.7) into a 100 ml absorption flask.

With water flowing through the reflux condenser (5.1.2), connect the decomposition flask (5.1.1) containing the test solution. Pass nitrogen (4.12) through the apparatus at a flowrate of 100 ml/min, as shown in figure 2. Heat the test solution to a temperature of 114 °C to 118 °C for 30 min. This will normally be achieved by setting the hotplate temperature to about 250 °C (see 10.2). Evolved gases are conveyed by the nitrogen carrier gas through the gas washing bottle (5.1.3) to the absorption vessel.

7.3.3 Colour development

7.3.3.1 For sulfur contents up to 0,001 0 % (*m/m*)

Disconnect the 20 ml absorption flask (5.1.4) and the gas inlet tube from the apparatus. Keeping the tip of the tube in the absorbing solution, add 1,0 ml of hydrochloric acid (4.2) from the upper end of the tube by using a micropipette to wash the inside surface of the tube, followed by rinsing with 1 ml of water.

Remove the gas inlet tube, gently swirl the 20 ml absorption flask, and allow to stand for 20 min at 25 °C in a thermostat. Then add 2,0 ml of *N,N*-dimethyl-*p*-phenylenediamine solution (4.10) to the absorption flask (5.1.4) and shake gently. Immediately add 0,4 ml of iron(III) chloride solution (4.9), and shake vigorously for 1 min. Dilute to the mark with water and mix. Allow to stand for 15 min.

7.3.3.2 For sulfur contents from 0,001 0 % (*m/m*) to 0,010 % (*m/m*)

Disconnect the 100 ml absorption flask (5.1.4) and the gas inlet tube from the apparatus. Keeping the tip of the tube in the absorbing solution, add 1,0 ml of hydrochloric acid (4.2) from the upper end of the tube

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by using a micropipette to wash the inside surface of the tube, followed by rinsing with 1 ml or 2 ml of water.

Remove the gas inlet tube, gently swirl the 100 ml absorption flask, and allow to stand for 20 min at 25 °C in a thermostat. Then add 10,0 ml of *N*,*N*-dimethyl-*p*-phenylenediamine solution (4.10) to the absorption flask (5.1.4) and shake gently. Immediately add 2,0 ml of iron(III) chloride solution (4.9), and shake vigorously for 1 min. Dilute to the mark with water and mix. Allow to stand for 15 min.

7.3.4 Spectrophotometric measurement

7.3.4.1 For sulfur contents up to 0,001 0 % (*m/m*)

Carry out the spectrophotometric measurements with a cell of 1 cm optical path length at a wavelength of about 665 nm, after adjusting the spectrophotometer (5.2) to zero against water.

7.3.4.2 For sulfur contents from 0,001 0 % (*m/m*) to 0,010 % (*m/m*)

Carry out the spectrophotometric measurements with a cell of 1 cm optical path length at a wavelength of about 665 nm, after adjusting the spectrophotometer (5.2) to zero against water.

7.4 Establishing the calibration graph

7.4.1 Preparation of calibration solutions

Introduce 1,0 ml of iron solution (4.8) into a series of six decomposition flasks (5.1.1) and add the respective volumes of sulfur standard solution (4.11) which are given in table 2. Then add 15 ml of the mixture of hydrochloric and nitric acids (4.5), and 5,0 ml of perchloric acid (4.3), heat and evaporate to fuming. Proceed as described in the second paragraph of 7.3.1 from "After cooling, add ..." to the end of 7.3.3.

Table 2

Sulfur content % (*m/m*)	**Volume of sulfur standard solution A** (4.11.2) ml	**Volume of sulfur standard solution B** (4.11.3) ml	**Corresponding mass of sulfur** µg
up to 0,001 0	—	0[1]	0
	—	1,0	1,0
	—	3,0	3,0
	—	5,0	5,0
	—	7,5	7,5
	—	10,0	10,0
from 0,001 0 to 0,010	0[1]	—	0
	0,5	—	5
	1,0	—	10
	2,0	—	20
	3,0	—	30
	5,0	—	50

[1] Zero member.

7.4.2 Spectrophotometric measurement

7.4.2.1 For sulfur contents up to 0,001 0 % (*m/m*)

Carry out the spectrophotometric measurements with a cell of 1 cm optical path length at a wavelength of about 665 nm, after adjusting the spectrophotometer (5.2) to zero against the zero member (see table 2).

7.4.2.2 For sulfur contents from 0,001 0 % (*m/m*) to 0,010 % (*m/m*)

Carry out the spectrophotometric measurements with a cell of 1 cm optical path length at a wavelength of about 665 nm, after adjusting the spectrophotometer (5.2) to zero against the zero member (see table 2).

7.4.3 Plotting the calibration graph

Prepare the calibration graph by plotting the absorbance against the sulfur concentrations, expressed in micrograms per 20 ml (see 7.3.3.1) or per 100 ml of the colour-developed solution (see 7.3.3.2).

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8 Expression of results

8.1 Method of calculation

Convert the absorbance measured in 7.3.4 into the corresponding mass, expressed in micrograms of sulfur, in the colour-developed test solution (see 7.3.3.1 or 7.3.3.2) by using the calibration graph (see 7.4.3).

The sulfur content, w_S, expressed as a percentage by mass, is given by the equation

$$w_S = (m_{S,1} - m_{S,0}) \times \frac{1}{10^6} \times \frac{100}{m}$$

$$= (m_{S,1} - m_{S,0}) \times \frac{1}{10^4 m}$$

where

$m_{S,0}$ is the mass, expressed in micrograms, of sulfur in the blank test solution;

$m_{S,1}$ is the mass, expressed in micrograms, of sulfur in the test solution;

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

8.2 Precision

A planned trial of this method was carried out by 11 or 13 laboratories, at 16 levels of sulfur, each laboratory making three determinations (see notes 1 and 2) of sulfur content at each level.

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

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

The data obtained showed a logarithmic relationship between sulfur content and repeatability (r) or reproducibility (R and R_w) of the test results (see note 3) as summarized in table 3. The graphical representation of the figures is shown in figure B.1.

Table 3

Sulfur content % (m/m)	Repeatability r	Reproducibility R	 R_w
0,000 3	0,000 08	0,000 17	0,000 11
0,000 5	0,000 11	0,000 23	0,000 14
0,001 0	0,000 17	0,000 37	0,000 21
0,002 0	0,000 27	0,000 60	0,000 31
0,005 0	0,000 47	0,001 11	0,000 51
0,010 0	0,000 72	0,001 71	0,000 74

NOTES

1 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.

2 The third determination was carried out at a different time (on a different day) by the same operator as in note 1 above, using the same apparatus with a new calibration.

3 From the result 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 case

For a test portion containing selenium, the 3rd to 5th lines of the 2nd paragraph of 7.3.1 should be changed to "After cooling, add 5 ml of hydrochloric acid (4.1) and 5 ml of hydrobromic acid (4.4). Heat again and evaporate until white fumes appear and until absolutely dry, on a hotplate at a temperature of about 300 °C."

10 Notes on procedure

10.1 Due to the extraordinary sensitivity of the method, it is important to eliminate sulfur as a contamination source. In practice, it may be useful to provide a room for this type of analysis. If the analyst chooses to run two reagent blanks, the lower one usually is the correct one. Spurious contamination by sulfur has been observed when new flasks, scrupulously cleaned with aqua regia, were used.

It is essential that all sample treatments be carried out in a scrupulously clean laboratory atmosphere, i.e. free from sulfuric acid fumes and any vapours or dusts containing sulfur or sulfur compounds.

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To keep the sulfur blank values low and constant (e.g. $x = 0{,}50$ µg and $\sigma = 0{,}15$ µg of sulfur), special care should be given to the selection of all acids used, but it does not necessitate a purification procedure by distillation.

10.2 Chemically, the reduction of sulfate to hydrogen sulfide is a difficult reaction. To ensure a complete recovery of sulfur, the reaction conditions shall be closely controlled. The optimum reducing temperature is 114 °C to 118 °C. If the reducing solution is excessively diluted by the sample solution, the boiling point is decreased and the reduction kinetics are slowed appreciably. At temperatures above 120 °C, the acid mixture shows signs of decomposition of hypophosphorous acid and an increased formation of phosphine.

The required temperature of the hotplate shall be established by a preliminary blank test with a thermometer dipped in the heated solution.

10.3 To check the effectiveness of the purification, connect the hydrogen sulfide trap (see figure 1) containing 10 ml of the absorbing solution (4.7) and continue heating for 30 min.

Disconnect the trap. Develop the colour according to the 2nd to 10th lines of 7.3.3.1, using a close-fitting ground-glass stopper as needed. Dilute to about 20 ml by adding 8 ml of water and mix. Allow to stand for 15 min.

Carry out the spectrophotometric measurement with a cell of 1 cm optical path length at a wavelength of about 665 nm, after adjusting the spectrometer (5.2) to zero against water.

The purification is completed when the obtained absorbance is below 0,055 (corresponding to 1 µg of S).

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.

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Annex A
(informative)

Additional information on the international cooperative tests

Table 3 was derived from the results of international analytical trials carried out in 1989 and 1991 on 12 steel samples and four iron samples in nine countries involving 11 or 13 laboratories.

The results of the trials were reported in document ISO/TC 17/SC 1 N 839, March 1990 and document ISO/TC 17/SC 1 N 915, February 1992. The graphical representation of the precision data is given in annex B.

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

NOTE 4 These four samples were omitted from the regression calculation for the following reasons.

Sample 1) (pure iron): sulfur content is less than the lower limit of this method.

Sample 7) (stainless steel): titanium content 2,1 % (*m/m*).

Sample 10) (high-silicon steel): silicon content 3,7 % (*m/m*).

Sample 12) (nodular iron): silicon content 2,3 % (*m/m*).

Table A.1

Sample	Sulfur content, % (*m/m*)			Precision		
	Certified	Found		Repeatability	Reproducibility	
		$\overline{w}_{S,1}$	$\overline{w}_{S,2}$	r	R	R_w
1) JSS 002-2 (pure iron)	0,000 08	0,000 08	0,000 09	0,000 06	0,000 10	0,000 09
2) JSS 003-2 (pure iron)	0,000 4	0,000 42	0,000 42	0,000 10	0,000 16	0,000 16
3) ECRM 096-1 (unalloyed steel)	0,000 9	0,000 84	0,000 84	0,000 17	0,000 32	0,000 23
4) JSS 244-4 (unalloyed steel)	0,001 5	0,001 54	0,001 55	0,000 24	0,000 77	0,000 40
5) JSS 240-8 (unalloyed steel)	0,006 0	0,005 73	0,005 69	0,000 82	0,001 12	0,000 78
6) ECRM 480-1 (cast iron)	0,008 6	0,008 17	0,008 19	0,001 07	0,002 03	0,000 95
7) NBS 348a (stainless steel)	0,000 7	0,000 53	0,000 52	0,000 11	0,000 30	0,000 09
8) JSS 654-10 (stainless steel)	0,000 7	0,000 64	0,000 62	0,000 13	0,000 29	0,000 10
9) JSS 611-8 (high-speed steel)	0,001 3	0,001 33	0,001 34	0,000 16	0,000 53	0,000 23
10) ECRM 191-1 (high-silicon steel)	0,001 7	0,001 93	0,001 89	0,000 41	0,001 44	0,000 72
11) ECRM 285-1 (high-alloy steel)	0,002 4	0,002 14	0,002 11	0,000 41	0,000 76	0,000 28
12) ECRM 481-1 (nodular iron)	0,004	0,003 24	0,003 20	0,000 53	0,001 69	0,000 41
13) JSS 650-9 (stainless steel)	0,005 3	0,005 38	0,005 40	0,000 62	0,000 87	0,000 39
14) ECRM 235-1 (high-alloy steel)	0,007 2	0,006 81	0,006 80	0,000 16	0,000 63	0,000 33
15) JSS 654-7 (stainless steel)	0,009 3	0,009 12	0,009 06	0,000 42	0,001 58	0,000 57
16) NBS 339 (Se-stainless steel)	0,013$^{1)}$	0,012 5	0,012 4	0,001 25	0,003 13	0,001 08

$\overline{w}_{S,1}$: general mean within a day

$\overline{w}_{S,2}$: general mean between days

1) Test portion = 0,25 g

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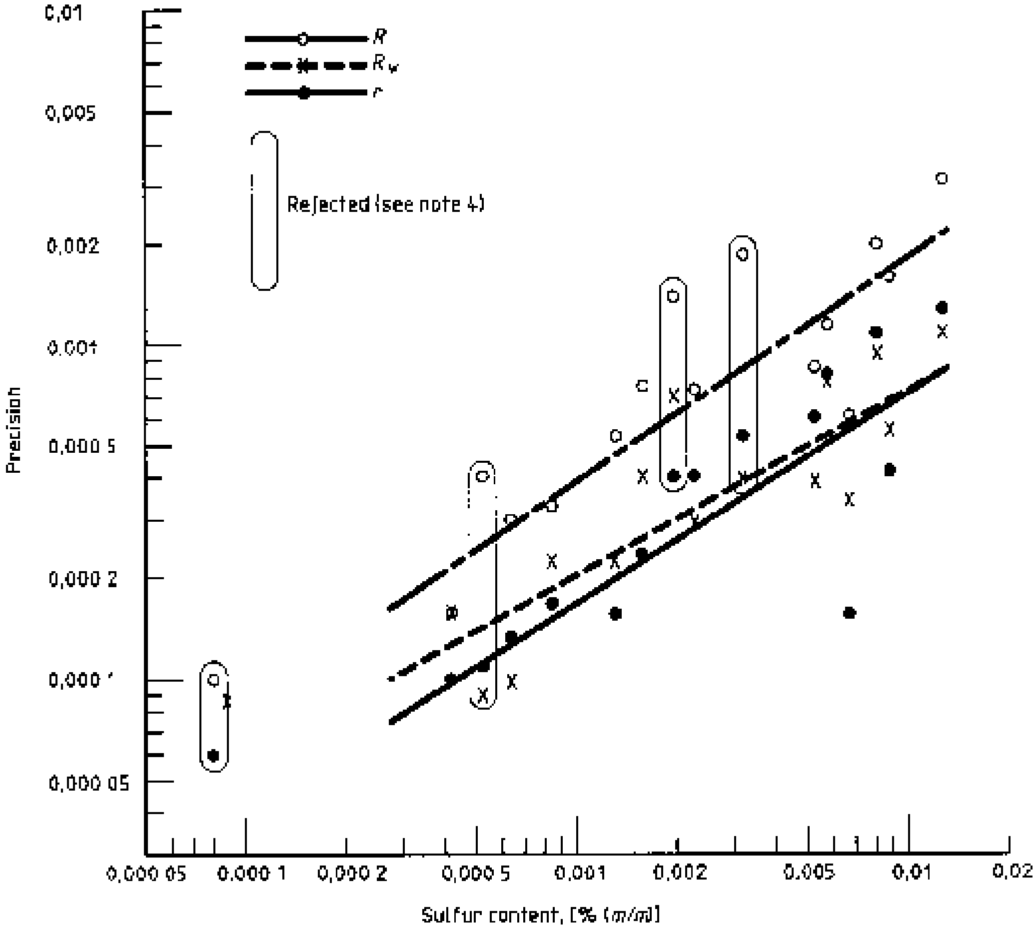
Annex B

(informative)

Graphical representation of precision data



$\lg r = 0{,}622\,4 \lg \overline{w}_{S,1} - 1{,}895\,5$

$\lg R = 0{,}675\,6 \lg \overline{w}_{S,1} - 1{,}400\,5$

$\lg R_w = 0{,}546\,7 \lg \overline{w}_{S,2} - 2{,}036\,8$

where

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

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

Figure B.1 — Logarithmic relationship between sulfur content (w_S) and repeatability (r) or reproducibility (R and R_w)

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