

INTERNATIONAL STANDARD

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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*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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*.

Annexes A and B of this International Standard are for information only.

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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.

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 $[(CH_3COO)_2Zn \cdot 2H_2O]$ 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 ($FeCl_3 \cdot 6H_2O$) 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 $[NH_2C_6H_4N(CH_3)_2 \cdot 2HCl]$ 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.