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**Steel and iron — Determination of arsenic  
content — Spectrophotometric method**

*Aciers et fontes — Dosage de l'arsenic — Méthode  
spectrophotométrique*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17058 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 arsenic content — Spectrophotometric method

## 1 Scope

This International Standard specifies a method for the determination of the arsenic content in steel and iron using a spectrophotometric method after distillation.

The method is applicable to an arsenic content between 0,000 5 % (mass fraction) and 0,10 % (mass fraction).

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

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

ISO 14284:1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

## 3 Principle

Dissolution of a test portion in a hydrochloric acid and nitric acid mixture.

Prolonged heating with sulfuric acid until white fumes are given off.

Reduction of arsenic in the presence of hydrazine sulfate and potassium bromide, then separation by distillation as the trivalent chloride, absorbed in the nitric acid solution, in which the As(III) is oxidized back to the pentavalent state As(V).

Formation of the molybdenum blue complex between ammonium molybdate and arsenic which is reduced by hydrazine sulfate.

Spectrophotometric measurement at a wavelength of approximately 840 nm.

## 4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 3 water specified in ISO 3696:1987.

**4.1 Hydrochloric acid**,  $\rho$  about 1,19 g/ml

**4.2 Nitric acid**,  $\rho$  about 1,40 g/ml

**4.3 Nitric acid**,  $\rho$  about 1,40 g/ml, diluted 3 + 1

**4.4 Acid mixture**

Add one volume of nitric acid (4.2) to four volumes of hydrochloric acid (4.1).

**4.5 Sulfuric acid**,  $\rho$  about 1,84 g/ml

**4.6 Sulfuric acid**,  $\rho$  about 1,84 g/ml, diluted 1 + 1

While cooling under water and swirling, add, in small portions, a volume of sulfuric acid (4.5) to an equal volume of water.

**4.7 Sulfuric acid**,  $\rho$  about 1,84 g/ml, diluted 1 + 6

While cooling under water and swirling, add, in small portions, a volume of sulfuric acid (4.5) to six volumes of water.

**4.8 Mixture of reductant powder**

Weigh, in proportion, 2,5 g of hydrazine sulfate and 10,0 g of potassium bromide, transfer to a mortar and, using a pestle, grind finely and mix well for use.

**4.9 Ammonium molybdate**,  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ , 10 g/l

**4.10 Hydrazine sulphate**,  $\text{N}_2\text{H}_6\text{SO}_4$  0,6 g/l

**4.11 Arsenic**, standard solution

**4.11.1 Stock solution**, corresponding to 0,20 g of arsenic per litre

Weigh, to the nearest 0,000 1 g, 0,132 0 g of arsenic trioxide ( $\text{As}_2\text{O}_3$ ).

Place in a 100 ml beaker, carefully add 10 ml of nitric acid (4.2), cover with a watch glass, heat gently until dissolution is complete. Add 2 ml of sulphuric acid (4.6), heat cautiously to expel oxides of nitrogen and then evaporate to dense, white fumes.

Remove the beaker from the hot plate, cool somewhat, and wash down the cover and sides of the beaker with a little water.

Again evaporate to copious white fumes.

Remove, cool and add about 10 ml of water, warm gently until salts are dissolved and the solution becomes clear. Cool to room temperature, transfer the solution quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this stock solution contains 0,2 mg of arsenic.

**4.11.2 Standard solution A**, corresponding to 0,01 g of arsenic per litre

Transfer 25,0 ml of the stock solution (4.11.1) to a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately before use.

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

**4.11.3 Standard solution B**, corresponding to 0,002 g of arsenic per litre.

Transfer 50,0 ml of the standard solution (4.11.2) to a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 2 µg of arsenic.

**4.12 Potassium Bromide****5 Apparatus**

All volumetric glassware shall be class A, in accordance with ISO 385-1:1984, ISO 648:1977 and ISO 1042:1998.

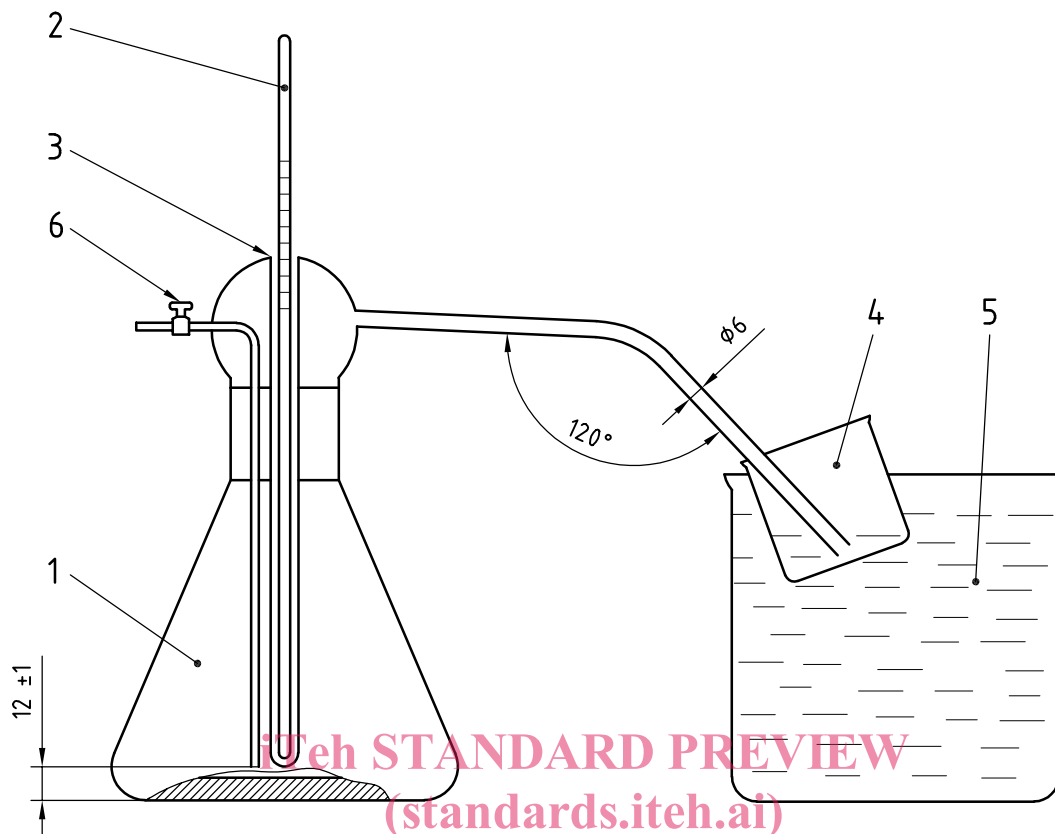
Ordinary laboratory apparatus and the following shall be used.

**5.1 Spectrophotometer**, suitable for measuring the absorbance of the solution at a wavelength of 840 nm with 40 mm (or 10 mm) optical cells

**5.2 Distillation apparatus**, see attached diagram (Figure 1)

**6 Sampling**

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel.



**Key**

- 1 conical flask (distillation flask), 250 ml
- 2 thermometer
- 3 thermometer well
- 4 receiving beaker, 100 ml
- 5 cooling beaker, 1 000 ml
- 6 valve

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**Figure 1 — Distillation apparatus for arsenic determination**

**7 Procedure**

**7.1 Test portion sampling**

Weigh, to the nearest 0,1 mg, a test portion of the sample in accordance with Table 1.

**Table 1 — Test portion sampling**

Expected arsenic content % (mass fraction)	Test portion g
0,000 5 to 0,001	1,0
0,001 to 0,006	0,50
0,006 to 0,100	0,10



## 7.2 Blank test

In parallel with the determination and the same procedure, carry out a blank test using the same quantities of all the reagents.

## 7.3 Determination

### 7.3.1 Preparation of the test solution

Place the test portion (see 7.1) in a 150 ml beaker. Add 10 ml to 20 ml of the acid mixture (4.4). Cover the beaker with a watch-glass and heat gently to complete dissolution.

Cool the solution, remove the watch-glass and add 20 ml of sulphuric acid (4.6). Replace the watch-glass leaving small opening to allow for the expulsion fumes and evaporate to dense white fumes.

### 7.3.2 Distillation

Transfer the test solution (see 7.3.1) quantitatively to the distillation flask, rinse down the cover and the sides of the beaker with approximately 10 ml of water. If any undissolved sulfate salts remain, add a small amount of water and warm until the salts are in solution.

Cool in water, add 10 ml of hydrochloric acid (4.1), 2,0 g of mixed reductant powder (4.8), and mix.

Add 10 ml of nitric acid (4.3) to the receiving beaker, and place the beaker in the cooling beaker containing cold water.

Assemble the apparatus for distillation (5.2). Heat the distillation flask at a low temperature, distil until the temperature rises to 125 °C, keeping the entire distillation procedure within 6 min to 10 min. Open the valve to avoid the solution's flowing back from the receiving beaker, and remove the distillation flask from the heat source.

Rinse down the tip of the outlet with a small amount of water. Collect the washings in the receiving beaker. Add 3,5 ml of sulfuric acid (4.7) and mix.

Replace the watch-glass leaving a small opening to allow for the expulsion of fumes and evaporate to dense white sulfuric acid fumes. During this procedure no bubbles should appear.

Remove and cool somewhat, rinse down the cover and the sides of the beaker, and again evaporate to white fumes to expel the oxides of nitrogen.

Remove the beaker from the heat source and allow it to cool. Rinse down the cover and transfer the solution quantitatively to a 50 ml one-mark volumetric flask.

### 7.3.3 Formation of the coloured complex

Add 2,5 ml of ammonium molybdate solution (4.9) and 2,5 ml of hydrazine sulfate solution (4.10) to the flask, dilute to the mark with water and mix. Warm the flask in a boiling water bath for 10 min. Remove the flask and cool to room temperature.

### 7.3.4 Spectrophotometric measurements

Carry out spectrophotometric measurements of each solution at a wavelength of about 840 nm after having adjusted the spectrophotometer to zero absorbance by reference to water using 40 mm (or 10 mm) cells.