

SLOVENSKI STANDARD

SIST EN 10212:1998

01-avgust-1998

**Kemična analiza železovih zlitin - Določanje arzena v jeklih in železovih litinah -
Spektrofotometrijska metoda**

Chemical analysis of ferrous materials - Determination of arsenic in steel and iron -
Spectrophotometric method

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Arsen in Stahl und Eisen
mittels Spektralphotometrie

Analyse chimique des matériaux sidérurgiques - Dosage de l'arsenic dans les aciers et
les fontes - Méthode spectrophotométrique

Ta slovenski standard je istoveten z: EN 10212:1995

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77.040.30

Kemijska analiza kovin

Chemical analysis of metals

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English version

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CEN

European Committee for Standardization
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Foreword

This European Standard was prepared by the Technical Committee ECISS/TC 20 "Methods of chemical analysis" of which the secretariat is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 1995, and conflicting national standards shall be withdrawn at the latest by September 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

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1 Scope

This European Standard specifies a spectrophotometric method for the determination of arsenic in steel and iron. The method is applicable to all types of steel and iron with arsenic contents from 0,001% to 0,08% (m/m).

2 Normative Reference

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EURONORM 18: 1979, Selection and preparation of samples and test pieces for steel and iron and steel products.

3 Principle

Dissolution of a test portion in a mixture of nitric and hydrochloric acids followed by evaporation to dryness and prolonged heating of the dried residue.

Extraction of the residue with acid, reduction of the arsenic (As V to As III) by addition of potassium iodide, ascorbic acid and tin (II) chloride. Conversion of the arsenic to arsenic hydride (arsine) with zinc.

Absorption of the evolved arsine in a solution of silver diethyldithiocarbamate and 1-ephedrin in trichloromethane.

Spectrophotometric measurement of the reddish-violet coloured colloid at a wavelength between 500 nm and 520 nm.

4 Reagents

During the analysis, use only reagents of recognised analytical quality and only distilled water of water of equivalent purity.

- 4.1 Iron, of high purity, free of arsenic or of known low arsenic content
- 4.2 Zinc, of high purity, free of arsenic or of known low arsenic content.

Zinc shot, 0,5 mm to 2 mm, and zinc pellets, approximately 5 mm in diameter.

4.3 Hydrochloric acid, approximately ρ 1,19 g/ml.

4.4 Sulphuric acid, approximately ρ 1,84 g/ml, diluted 1 + 1

4.5 Hydrochloric - nitric acid mixture

Add 65 ml nitric acid, approximately ρ 1,40 g/ml, to 100 ml water; add the resulting diluted acid to 60 ml hydrochloric acid, (4.3) and mix.

4.6 Absorption solution

Dissolve 1,25 g of silver diethyldithiocarbamate ($C_5H_{10}AgNS_2$) and 0,825 g of 1-ephedrin in approximately 400 ml of trichloromethane (chloroform) in a 500 ml volumetric flask. Dilute to the mark with trichloromethane and mix.

After preparation a small residue may develop. Filter the solution after at least 20 h and store the solution in an amber coloured bottle. The solution can be used after the filtration for at least 3 months.

NOTE: The peak wavelength of the silver colloid may vary with the quality of the reagent and should be determined with each new batch of reagent.

4.7 Ascorbic acid, 100 g/l solution

This solution shall be freshly prepared each day.

4.8 Potassium iodide, 150 g/l solution

This solution shall be freshly prepared each day.

4.9 Tin (II) chloride, 400 g/l in hydrochloric acid solution

Dissolve 20 g of tin (II) chloride in approximately 40 ml of hydrochloric acid (4.3). Transfer to a 50 ml volumetric flask, dilute to the mark with hydrochloric acid (4.3) and mix.

This solution shall be freshly prepared each day.

4.10 Lead acetate wool

Impregnate cotton wool with lead acetate trihydrate solution, (10 g/l), squeeze out the excess solution and dry the wool at 105°C for several hours.

- 4.11 Arsenic standard solution, corresponding to 5 μg of arsenic per ml

Dissolve 0,3301 g of arsenic trioxide, dried at 105°C, in 20 ml sodium hydroxide solution (8 g/l). Transfer to a 1000 ml volumetric flask, dilute to the mark with water and mix. Transfer 20,0 ml of this solution to a 1000 ml volumetric flask, dilute to the mark with water and mix.

5 Apparatus

Ordinary laboratory equipment and

- 5.1 a spectrophotometer, suitable for measuring the absorbance of the solution at a wavelength of 500 nm to 520 nm (see 4.6), together with cells of 40 mm and 10 mm optical path length.
- 5.2 apparatus for the evolution and absorption of arsine as shown in figure 1.

NOTE: Approximate dimensions are shown in the figure. Important parameters are an adequate depth of immersion and sufficient free space around the capillary to allow the bubbles to rise without agglomeration. If these conditions are satisfied, variations in the apparatus should be permissible. It is essential that the arsine absorption is complete. This may be verified initially for a given piece of apparatus by coupling a second absorption vessel in series with the first.

6 Sampling

Sampling shall be carried out in accordance with EURONORM 18.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,0001 g, a test portion of 0,1 g \pm 0,005 g.

NOTE: For coarse samples, where the weighing of 0,1 g may be inconvenient or where a more representative sample is required, a larger portion may be dissolved in the acid-mixture (4.5). After the prolonged heating period and the extraction of the residue in hydrochloric acid (4.3), an aliquot equivalent to 0,1 g shall be taken and treated with 10 ml of sulphuric acid (4.4) as specified in 7.3.1.

7.2 Blank test

With each analytical run, carry out the calibration procedure as specified in 7.4. The zero member of the calibration series provides automatic compensation for arsenic in the reagents - see 7.4.3 and the note.

7.3 Determination

7.3.1 Preparation of the test solution

Transfer the test portion (7.1) to a 100 ml conical beaker, add 10 ml of hydrochloric-nitric acid mixture (4.5), cover the beaker with a watch-glass and digest until solvent action ceases. Rinse and remove the watch-glass and heat carefully until all liquid has been evaporated. Continue to heat the dried residue at a temperature of 300°C to 350°C for a period of about 3 h. Cool, add 10 ml of hydrochloric acid (4.3) and heat to dissolve the residue.

Add 10 ml of sulphuric acid (4.4) and heat carefully to remove oxides of nitrogen. Continue heating until copious white fumes of sulphur trioxide are evolved.

Cool, dilute with water to 40 ml, warming if necessary to dissolve the precipitated sulphates.

7.3.2 Reduction of arsenic (V) to arsenic (III)

Cool and add in the following order, 5 ml of potassium iodide solution (4.8), 5 ml of ascorbic acid solution (4.7) and 3 ml of tin (II) chloride solution (4.9). Allow to stand for 5 min.

NOTE: In the case of an unalloyed steel, the solution at this stage should be virtually colourless. Colouration is indicative of incomplete removal of nitric acid. Optimum conditions for fuming are best decided in the individual laboratory, with due regard being taken of the temperature variations occurring across hot plate surfaces and to spurting if the temperature rises too fast. A final hot plate temperature of approximately 200°C to 240°C has been found to be satisfactory.

7.3.3 Evolution of arsine and development of the colour

transfer the solution to the evolution flask (5.2), rinsing with 10 ml to 20 ml of water. Dry the neck of the flask, add a mixture of 3 g of zinc shot and 3 g of zinc pellets (4.2) and immediately connect the flask to the delivery tube by means of the ground glass stopper. As evolution commences, immerse the delivery tube into 20 ml of the absorption solution (4.6) contained in an absorption tube with a 20,0 ml mark. (See the figure).

NOTE: 1 It is important to avoid crystallisation at the tip of the capillary with the danger of blockage and pressure