



SLOVENSKI STANDARD
oSIST prEN ISO 4943:2022
01-januar-2022

Jeklo in lito železo - Določevanje bakra - Plamenska atomska absorpcijska spektrometrična metoda (ISO/DIS 4943:2021)

Steel and cast iron - Determination of copper content - Flame atomic absorption spectrometric method (ISO/DIS 4943:2021)

Stahl und Gusseisen - Bestimmung des Kupfergehalts -
Flammenatomabsorptionsspektrometrisches Verfahren (ISO/DIS 4943:2021)

Aciers et fontes - Détermination du cuivre - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO/DIS 4943:2021)

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Steel and cast iron — Determination of copper content — Flame atomic absorption spectrometric method

Aciers et fontes — Dosage du cuivre — Méthode par spectrométrie d'absorption atomique dans la flamme

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 4943:1985), which has been technically revised. The main changes compared to the previous edition are:

- extension of the determination range;
- re-organization of a precision test;
- re-assessment of the precision data.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Steel and cast iron — Determination of copper content — Flame atomic absorption spectrometric method

1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of copper in steel and cast iron.

The method is applicable to copper contents in the range of 0,003 % (mass fraction) to 3,0 % (mass fraction).

2 Normative references

The following documents are referred to in the text such a way that some or all of their content constitutes requirements 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 648, *Laboratory glassware — Single-volume pipettes*

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

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

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

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3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

Dissolution of a test portion in a mixture of hydrochloric, nitric and perchloric acids.

Nebulisation of the test solution into an air/acetylene flame of an atomic absorption spectrometer. Spectrometric measurement of the atomic absorption of the 324,7 nm or 327,4 nm spectral line emitted by a copper hollow-cathode lamp.

NOTE Other suitable radiation sources can also be used.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

5.1 Pure iron, containing less than 0,000 5 % (mass fraction) of copper or of low and known copper content.

ISO/DIS 4943:2021(E)**5.2 Hydrochloric acid-nitric acid mixture.**

Mix three parts by volume of hydrochloric acid (ρ about 1,19 g/ml), one part by volume of nitric acid (ρ about 1,40 g/ml), and two parts by volume of water.

Prepare this mixture immediately before use.

5.3 Hydrochloric acid-nitric acid-perchloric acid mixture.

Mix 20 ml of hydrochloric acid (ρ about 1,19 g/ml) with 55 ml of nitric acid (ρ about 1,40 g/ml) and 75 ml of perchloric acid (ρ about 1,54 g/ml).

NOTE Perchloric acid (ρ about 1,67 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1,54 g/ml) are equivalent to 79 ml of perchloric acid (ρ about 1,67 g/ml).

5.4 Copper standard solution, 1,0 g/l.

Weigh (1,000 \pm 0,001) g of copper [purity > 99,95 % (mass fraction)]. Transfer into a 400 ml beaker, add 25 ml of nitric acid (ρ about 1,40 g/ml, diluted 1 + 4) and cover with a watch-glass. Heat gently to complete dissolution. Allow to cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of Cu.

5.5 Copper standard solution, 20 mg/l.

Transfer 20,0 ml of the standard solution (5.4) into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 20 μ g of Cu.

Prepare this standard solution immediately before use.

6 Apparatus

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

All glassware shall first be washed in hydrochloric acid (ρ about 1,19 g/ml, diluted 1 + 1), and then in water. The quantity of copper present in the beakers and flasks can be checked by measuring the absorption of distilled water introduced in the glassware after the acid wash.

Ordinary laboratory apparatus and the following shall be used.

6.1 Atomic absorption spectrometer

The spectrometer shall be equipped with a copper hollow-cathode lamp, and supplied with air and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from copper.

NOTE Other suitable radiation sources can also be used.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 8.3.4, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the precision criteria given in 6.1.1 to 6.1.3.

The instrument should also conform to the additional performance requirement given in 6.1.4.

6.1.1 Minimum precision (see Annex A)

Calculate the standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution. This standard deviation shall not exceed 1,0 % of the mean absorbance.

Calculate the standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero member). This standard deviation shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

6.1.2 Limit of detection (see Annex A)

The limit of detection is a number, expressed in units of concentration (or amount) that describes the lowest concentration level (or amount) of an element that can be determined to be statistically different from an analytical blank.

The limit of detection of copper in a matrix similar to the final test portion solution shall be better than 0,05 µg/ml.

6.1.3 Calibration linearity (see Annex A)

The slope of the calibration curve covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 times the value of the slope for the bottom 20 % of the concentration range (expressed as a change in absorbance) determined in the same way.

For instruments with automatic calibration using two or more standards, it shall be established prior to the analysis, by obtaining absorbance readings, that the above requirements for graph linearity are fulfilled.

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6.1.4 Characteristic concentration (see Annex A)

The characteristic concentration for copper in a matrix similar to the final test portion solution shall be better than 0,08 µg/ml for 324,7 nm.

6.2 Ancillary equipment.

Scale expansion can be used until the noise observed is greater than the read-out error and is always recommended for absorbances below 0,1. If scale expansion has to be used and the instrument does not have the means to read the value of the scale expansion factor, the value can be calculated by measuring a suitable solution with and without scale expansion and then dividing the signal obtained.

7 Sampling

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

8 Procedure

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic matter in general. All evaporations shall be carried out in fume cupboards suitable for use with perchloric acid.

Ensure that the spray system and drainage system are washed free from perchloric acid after use.

8.1 Test Portion

Weigh, to the nearest 0,001 g, approximately 0,50 g of the test sample.

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8.2 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, including pure iron (5.1) instead of the test portion.

8.3 Determination

8.3.1 Preparation of the test solution

8.3.1.1 Dissolution of the test portion

Place the test portion (8.1) in a 250 ml beaker. Add, in small portions, 20 ml of the acid mixture (5.3) (see Note), cover the beaker with a watch-glass and heat gently until the acid action ceases. Evaporate until dense white fumes of perchloric acid appear. Continue fuming at a suitable temperature to maintain a steady reflux of white perchloric acid fumes on the walls of the beaker until salts crystallization.

NOTE For samples not readily soluble in the acid mixture (5.3), first dissolve in 10 ml of the acid mixture (5.2) before adding 20 ml of the acid mixture (5.3).

Allow to cool, add 25 ml of water and heat gently to dissolve the salts. Cool again and transfer the test solution quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Filter by decantation through a dry medium texture filter paper to remove any residue or precipitate, for example graphite, silica or tungstic acid, and collect the filtrate in a dry beaker after discarding the first portions.

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8.3.1.2 Dilution of the test solution (standards.iteh.ai)

Depending on the copper content expected in the test portion, prepare the test solution in accordance with a), b), or c), as follows:

- a) If the expected copper content is up to 0,1 % (mass fraction), use the filtrate (see 8.3.1.1) undiluted.
- b) If the expected copper content is between 0,1 % (mass fraction) and 1,0 % (mass fraction), transfer 10,0 ml of the test solution into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.
- c) If the expected copper content is between 1,0 % (mass fraction) and 3,0 % (mass fraction), transfer 5,0 ml of the test solution into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

In the cases where the test solution has to be diluted, the blank test (8.2) shall be diluted in the same way.

8.3.2 Preparation of the calibration solutions

Place (5,00 ± 0,01) g of the pure iron (5.1) in a 500 ml beaker. Add in small portions 200 ml of the acid mixture (5.3) and heat gently until the dissolution is achieved.

Evaporate until dense white fumes of perchloric acid appear. Continue fuming at a suitable temperature to maintain a steady reflux of white perchloric acid fumes on the walls of the beaker until salts crystallization.

Allow to cool, add 100 ml of water and heat gently to dissolve the salts. Cool again and transfer the solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

8.3.2.1 Copper contents up to 0,1 % (mass fraction)

Transfer into a series of 100 ml one-mark volumetric flasks 50,0 ml of the iron solution (8.3.2). Using a burette or pipette, add the volumes of the copper standard solution (5.5) given in Table 1. Dilute to the mark with water and mix.

Table 1 — Calibration solutions for copper contents up to 0,1 %

Volume of copper standard solution (5.5) ml	Mass of copper mg	Corresponding content of copper in the test sample %
0 ¹⁾	0	0
0,5	0,01	0,002
2,5	0,05	0,010
5,0	0,10	0,020
10,0	0,20	0,040
15,0	0,30	0,060
20,0	0,40	0,080
25,0	0,50	0,100

1) Zero member

8.3.2.2 Copper contents between 0,1 % (mass fraction) and 1,0 % (mass fraction)

Transfer into a series of 100 ml one-mark volumetric flasks 5,0 ml of the iron solution (8.3.2). Using a burette or pipette, add the volumes of the copper standard solution (5.5) given in Table 2. Dilute to the mark with water and mix.

Table 2 — Calibration solutions for copper contents between 0,1 % and 1,0 %

Volume of copper standard solution (5.5) ml	Mass of copper mg	Corresponding content of copper in the test sample %
0 ¹⁾	0	0
2,5	0,05	0,10
5,0	0,10	0,20
10,0	0,20	0,40
15,0	0,30	0,60
20,0	0,40	0,80
25,0	0,50	1,00

1) Zero member

8.3.2.3 Copper contents between 1,0 % (mass fraction) and 3,0 % (mass fraction)

Transfer into a series of 100 ml one-mark volumetric flasks 1,0 ml of the iron solution (8.3.2). Using a burette or pipette, add the volumes of the copper standard solution (5.5) given in Table 3. Dilute to the mark with water and mix.