



SLOVENSKI STANDARD

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Jeklo in lito železo - Določevanje kroma - Plamenska atomska absorpcijska spektrometrična metoda (FAAS)

Steels and cast irons - Determination of chromium content - Flame atomic absorption spectrometric method (FAAS)

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Chrom in Stahl und Eisen - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Aciers et fontes - Détermination de la teneur en chrome - Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

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English Version

Steels and cast irons - Determination of chromium content - Flame atomic absorption spectrometric method (FAAS)

Aciers et fontes - Détermination de la teneur en chrome - Méthode par spectrométrie d'absorption atomique dans la flamme (SAAF)

Stahl und Gusseisen - Bestimmung des Chromanteils - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 459/SC 2.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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prEN 10188:2023 (E)**European foreword**

This document (prEN 10188:2023) has been prepared by Technical Committee CEN/TC 459/SC 2 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 10188:1989.

In comparison with the previous edition, the following technical modifications have been made:

- Normative references: updated;
- Clause 3 Terms and definitions: added;
- Subclauses 5.10 and 5.11: added;
- Sub Clauses 6.1 and 6.2: added;
- In 6.2.3: definition of limit of detection added;
- Sub Clause 6.3: reworded;
- Clause 7: updated;
- Subclause 8.3.1: partially aligned with ISO 10138:1991;
- Subclause 8.3.2: aligned with ISO 10138:1991;
- Subclause 8.3.4: aligned with EN 10136:2019;
- Bibliography: added.

1 Scope

This document specifies a flame atomic absorption spectrometric method (FAAS) for the determination of chromium content in steels and cast irons.

The method is applicable to non-alloy and low-alloy steels and cast irons with chromium contents between 0,002 % (by mass) to 2,0 % (by mass).

The method can be adapted to lower or higher chromium contents by changing the test portion or the dilution process, provided the criteria in 6.3.2 and 6.3.3 are still met.

2 Normative references

The following documents are referred to in the text in 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.

EN ISO 648, *Laboratory glassware - Single-volume pipettes (ISO 648)*

EN ISO 1042, *Laboratory glassware - One-mark volumetric flasks (ISO 1042)*

EN ISO 14284, *Steel and iron - Sampling and preparation of samples for the determination of chemical composition (ISO 14284)*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— IEC Electropedia: available at <https://www.electropedia.org/>

— ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

Dissolution of a test portion with hydrochloric acid followed by oxidation with nitric acid. Filtration and ignition of the acid insoluble residue. Removal of silica with hydrofluoric acid. Fusion of the residue with potassium hydrogen sulphate, dissolution of the melt in acid and addition of the solution obtained to the reserved filtrate.

Nebulisation of the test solution into a nitrous oxide/acetylene flame of an atomic absorption spectrometer.

Spectrometric measurement of the atomic absorption of the 357,87 nm spectral line emitted by a chromium hollow-cathode lamp.

NOTE Other suitable radiation sources can also be used, provided the criteria in 6.3.2 and 6.3.3 are still met.

5 Reagents

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

5.1 Iron of high purity, with a chromium content < 1 µg/g

5.2 Potassium hydrogen sulphate (KHSO₄)

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5.3 Hydrochloric acid, ρ_{20} 1,19 g/ml approximately

5.4 Nitric acid, ρ_{20} 1,40 g/ml approximately

5.5 Sulphuric acid, ρ_{20} 1,84 g/ml approximately

5.6 Hydrofluoric acid, ρ_{20} 1,15 g/ml approximately

5.7 Sulphuric acid, solution 1 + 3

Carefully add 50 ml of sulphuric acid (5.5) to 150 ml of water.

Allow to cool and mix.

5.8 Iron base solution, 40 g/l

Weigh 10 g of iron (5.1) into a 1 l beaker. Add 10 g of potassium hydrogen sulphate (5.2) and 150 ml of hydrochloric acid (5.3). Cover with a watch glass and heat gently until the iron is dissolved, then oxidize with 30 ml of nitric acid (5.4), added in small portions.

Allow to cool, transfer the solution into a 250 ml volumetric flask, dilute to the mark with water and mix.

5.9 Chromium standard solution, 1 g/l

Weigh $(1,000 \pm 0,001)$ g of chromium (Cr $\geq 99,7$ %). Transfer into a 250 ml beaker and dissolve in 40 ml of hydrochloric acid (5.3).

Allow to cool and transfer the solution quantitatively into a 1 000 ml volumetric flask, dilute to the mark with water and mix.

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

5.10 Chromium standard solution, 0,4 g/l

Transfer 40 ml of chromium standard solution (5.9) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,4 mg of chromium.

5.11 Chromium standard solution, 0,1 g/l

Transfer 10 ml of chromium standard solution (5.9) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,1 mg of chromium.

6 Apparatus

6.1 Ordinary laboratory equipment

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

6.2 Platinum crucible, of capacity 30 ml

6.3 Atomic absorption spectrometer

6.3.1 General

The spectrometer shall be equipped with a chromium hollow-cathode lamp or other suitable radiation source and supplied with nitrous oxide and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from chromium.

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 performance criteria given in 6.3.2 and 6.3.3.

6.3.2 Minimum precision

The standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution shall not exceed 1,5 % of the mean absorbance of this solution.

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

For example, if the top and bottom calibration solutions represent 0,10 % (by mass) and 0,02 % (by mass) chromium in the sample, the precision called for (as two standard deviations) would be 0,003 % and 0,001 % chromium respectively, assuming the curve linear.

6.3.3 Additional performance requirements

6.3.3.1 General

The instrument should also conform to the additional performance requirements given in 6.3.3.2 and 6.3.3.3.

6.3.3.2 Characteristic concentration

The characteristic concentration for chromium in a matrix similar to the final test solution shall be lower than 0,25 µg/ml.

6.3.3.3 Limit of detection

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 chromium in a matrix similar to the final test solution shall be less than 0,05 µg/ml.

6.3.3.4 Calibration linearity

The slope of the calibration line 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 determined in the same way.

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For instruments with automatic standardization using only two calibration solutions, one being twice the concentration of the other, the acceptable curve linearity is limited to such an extent that the absorption of the lower calibration solution does not exceed 0,55 of the absorption of the higher calibration solution.

6.4 Ancillary equipment

Scale expansion can be used until the noise observed is greater than the readout 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 signals obtained.

7 Sampling

Sampling shall be carried out in accordance with EN ISO 14284 or appropriate national standards for steels and cast irons.

8 Procedure**8.1 Test portion**

Weigh to the nearest 1 mg, approximately 1,0 g of the test sample.

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 reagents as used for the determination.

8.3 Determination**8.3.1 Preparation of the test solution**

Transfer the test portion (8.1) into a 250 ml beaker. Add 15 ml of hydrochloric acid (5.3), cover the beaker with a watch glass and heat gently until acid action ceases. Oxidize with 3 ml of nitric acid (5.4) added dropwise and boil for 1 min to remove nitrogen oxides.

Allow to cool, add about 15 ml of water, filter through a close texture filter paper and collect the filtrate in a 250 ml beaker. Wash the filter several times with hot water, collecting the washings in the same beaker. Reserve the filtrate as main solution.

Transfer the filter and its content into a platinum crucible, dry and ignite at a temperature as low as possible until all carbonaceous matter is removed and finally at about 800 °C for at least 15 min.

Allow to cool, add 2 drops of sulphuric acid solution (5.7) and about 2 ml of hydrofluoric acid (5.6). Evaporate to dryness, heat to about 800 °C and allow to cool.

Add 1 g of potassium hydrogen sulphate (5.2) and fuse carefully until a clear melt is obtained.

Note For residues which contain chromium carbides, prolonged heating can be necessary for complete fusion. The potassium hydrogen sulphate can be regenerated by allowing the melt to cool, adding 2 drops of sulphuric acid solution (5.5) and repeating the fusion until all the residues has been decomposed.

Allow to cool and transfer the crucible and the solidified melt into a small beaker. Add 10 ml of water and 2 ml of hydrochloric acid (5.3) and heat gently until the melt product is dissolved.

Allow to cool and add the solution obtained to the reserved filtrate (main solution).

According to the expected chromium content in the sample, transfer the test solution:

- a) into a 100 ml one-mark volumetric flask, for chromium contents up to 0,1 % (by mass), dilute to the mark with water and mix;

- b) into a 500 ml one mark volumetric flask, for chromium contents between 0,1 % (by mass) and 2,0 % (by mass), dilute to the mark with water and mix.

8.3.2 Preparation of the calibration solutions

8.3.2.1 General

Table 1 and Table 2 contain typical sets of calibration solutions. The range of concentrations in a set of solutions may be changed in order to better bracket the expected sample solution concentration.

8.3.2.2 Chromium contents up to 0,10 % (by mass)

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of chromium standard solution (5.11) shown in Table 1 and 25 ml of the iron base solution (5.8). Dilute to the mark with water and mix.

Table 1 — Calibration for chromium contents up to 0,1 %

Chromium standard solution volume (5.11) ml	Corresponding chromium mass mg	Corresponding chromium concentration after final dilution mg/ml	Corresponding chromium content in the sample %
0 ^a	0	0	0
1,0	0,1	0,001	0,01
2,0	0,2	0,002	0,02
4,0	0,4	0,004	0,04
6,0	0,6	0,006	0,06
8,0	0,8	0,008	0,08
10,0	1,0	0,010	0,10
^a Zero member 1,00 0,010 0,10			

8.3.2.3 Chromium contents between 0,10 (by mass) and 0,5 % (by mass)

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of chromium standard solution (5.11) shown in Table 2 and 5 ml of the iron base solution (5.8). Dilute to the mark with water and mix.