



SLOVENSKI STANDARD
SIST EN 10188:1997

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**Kemična analiza železovih zlitin - Določevanje kroma v jeklih in železovih litinah -
Plamenska atomska absorpcijska spektrometrična metoda**

Chemical analysis of ferrous materials - Determination of chromium in steels and irons -
Flame atomic absorption spectrometric method

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

Analyse chimique des matériaux sidérurgiques - Dosage du chrome dans les aciers et
les fontes - Méthode par spectrométrie d'absorption atomique dans la flamme

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English version

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CEN

European Committee for Standardization
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Brief History

This European Standard takes over the contents of EURONORM 188-87 "Chemical analysis of ferrous materials - Determination of chromium in steels and irons - Flame atomic absorption spectrometric method" prepared by ECISS/TC 20 "Methods of chemical analysis" the secretariat of which is allocated to the Dansk Standardiseringsrad (DS).

It was submitted to the CEN Formal Vote following the decision of the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization on 1987-11-24/25.

According to the Common CEN/CENELEC Rules, following countries are bound to implement this European Standard:

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Chemical analysis of ferrous materials
Determination of chromium in steels and irons
Flame atomic absorption spectrometric method

CONTENTS

	Page
1 SCOPE AND FIELD OF APPLICATION	2
2 REFERENCES	2
3 PRINCIPLE	2
4 REAGENTS	2
5 APPARATUS	2
6 SAMPLING	3
7 PROCEDURE	3
7.1 Test portion	3
7.2 Blank test	3
7.3 Determination	3
7.4 Establishment of the calibration graph	4
8 EXPRESSION OF RESULTS	5
9 TEST REPORT	5
ANNEX: Precision data	6

1 SCOPE AND FIELD OF APPLICATION

This European Standard specifies a method for the determination of chromium in steels and irons by means of flame atomic absorption spectrometry.

The method is applicable to non-alloy and low-alloy steels and irons with chromium contents of 0.002 to 2.0 % (m/m) (see Annex).

2 REFERENCES

Euronorm 18 – Selection and preparation of samples and test pieces for steel and iron and steel products.

Information Circular No 8 – Recommendations for the drafting of standard methods of analysis employing flame atomic absorption spectrometry for the chemical analysis of iron and steel.

Information Circular No 9 – Operational guidelines for the application of flame atomic absorption spectrometry in standard methods for the chemical analysis of iron and steel.

3 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. Extraction of the melt in acid and addition of the extract to the reserved filtrate.

Determination of the chromium by means of the spectrometric measurement of the atomic absorption of the 357.87 nm line emitted by a chromium hollow cathode lamp when the solution is nebulized into a nitrous oxide acetylene flame.

4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent quality and having a very low chromium content, and only distilled water or water of equivalent purity.

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

4.2 Potassium hydrogen sulphate (KHSO₄).

4.3 Hydrochloric acid, ρ 1.19 g/ml approximately, (12 mol/l approximately).

4.4 Nitric acid, ρ 1.40 g/ml approximately, (14 mol/l approximately).

4.5 Sulphuric acid, ρ 1.84 g/ml approximately, (18 mol/l approximately).

4.6 Hydrofluoric acid, ρ 1.15 g/ml approximately, (20 mol/l approximately).

4.7 Sulphuric acid, ρ 1.84 g/ml approximately, diluted 1+3 (V/V), (4.5 mol/l approximately).

4.8 Background solution

Weigh 10 g of iron (4.1) into a 1 litre beaker. Add 10 g of potassium hydrogen sulphate (4.2) and 150 ml of hydrochloric acid (4.3). Heat gently until dissolved, then oxidize with 30 ml of nitric acid (4.4), added in small portions. Allow to cool, transfer to a 250 ml volumetric flask, dilute to the mark with water and mix.

4.9 Chromium reference solution, corresponding to 1 mg of chromium per ml.

Weigh 1.000 g of high purity chromium metal (≥ 99.7% (m/m) pure). Transfer to a 250 ml beaker and dissolve in 40 ml of hydrochloric acid (4.3). Cool, transfer to a 1 000 ml volumetric flask, dilute to the mark with water and mix.

Note: ρ equals the density.

SIST EN 10188:1997 APPARATUS

Ordinary laboratory equipment and

5.1 Atomic absorption spectrometer: a chromium hollow cathode lamp; supplies of 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 7.3.4, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the following performance requirements.

5.1.1 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.

The standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero calibration solution) 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 % and 0.02 % chromium in the sample, the

precision called for (as two standard deviations) would be 0.003 % and 0.001 % chromium respectively, assuming curve linearity.

5.1.2 Additional performance requirements

It is also desirable that the instrument should conform to the following additional performance requirements (see Information Circular No 8, Section 2):

- (a) Characteristic concentration — The characteristic concentration for chromium in a matrix similar to the final sample test solution shall be better than 0.25 µg/ml.
- (b) Limit of detection — The limit of detection of chromium in a matrix similar to the final sample test solution shall be better than 0.05 µg/ml.
- (c) Curve linearity — The slope of the calibration curve covering the top 20 % concentration range (expressed as a change in absorbance) shall not be less than 0.7 of the value of slope for the bottom 20 % concentration range determined in the same way.

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.

5.2 Ancillary equipment

A strip chart recorder and/or digital readout device is recommended to evaluate the criteria in 5.1 and for all subsequent measurements.

6 SAMPLING

Sampling shall be carried out in accordance with Euronorm 18.

7 PROCEDURE

7.1 Test portion

Weigh the mass (m) indicated below to the nearest 0.001 g:

$$m = 1 \text{ g} \pm 1 \%$$

7.2 Blank test

With each analytical run, carry out an analysis on a reagent blank in parallel with the test portion analysis, using identical reagents, conditions, analytical procedures and dilutions throughout.

7.3 Determination

7.3.1 Preparation of the test solution

Place the test portion (7.1) in a 250 ml beaker. Add 15 ml of hydrochloric acid (4.3), cover the beaker and heat gently

until solvent action ceases. Oxidize with 3 ml of nitric acid (4.4) added dropwise and boil for 1 minute to remove oxides of nitrogen. Cool, dilute with about 15 ml of water and filter through a pulp pad prepared from macerated filter paper, collecting the filtrate in a 100 ml volumetric flask. Wash the filter with water, collecting the washings in the same 100 ml volumetric flask, but taking care to leave sufficient room for the fusion extract of the acid insoluble residue, about 20 ml, to be added later. Reserve the filtrate. Transfer the filter and residue to a small platinum capsule or crucible, dry and ignite at as low a temperature as possible until all carbonaceous matter is removed and finally at about 800 °C for at least 15 minutes. Cool, add 2 drops of sulphuric acid solution (4.7) and about 2 ml of hydrofluoric acid (4.6). Evaporate to dryness, heat to about 800 °C and cool. Add 1 g of potassium hydrogen sulphate (4.2) and fuse carefully until a clear melt is obtained.

Note: For residues which contain chromium carbide, prolonged heating may 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 (4.5) and repeating the fusion until the residue has dissolved.

Cool and transfer the crucible and solidified melt to a small beaker. Add 10 ml of water and 2 ml of hydrochloric acid (4.3) and heat gently until the solid has dissolved. Cool, add the extract to the reserved filtrate in the 100 ml volumetric flask, dilute to the mark with water and mix. This is the sample solution S.

Set up the instrument as described in 7.3.3. Test the solution S at various approximate dilutions to obtain the dilution factor D, that brings the solution near the midway of the optimum working range of the instrument, e.g. 0.25 to 0.45 absorbance units. Accurately dilute the sample solution S with water by the dilution factor D to obtain the test solution N. Dilute the blank test solution (7.2) in the same way.

Note: Volumes of sample solution S less than 5 ml shall not be taken for dilution.

7.3.2 Preparation of the calibration solution

If the test solution N gives an absorbance above 0.25, accurately dilute the standard chromium solution (4.9) to give a stock solution approximately 600 times the characteristic concentration (5.1.2). Using a burette, transfer 0, 5, 10, 15, 20 ml of the above chromium stock solution to 100 ml volumetric flasks. Add $25 \times \frac{1}{D}$ ml of background solution (4.8) to each and dilute to the mark with water (D is the dilution of the sample solution).

If the test solution N gives an absorbance below 0.25, prepare two solutions each containing 25 ml of background solution (4.8) per 100 ml, one containing 5 µg/ml of chromium, the other no added chromium. Using these, a rough estimate of the chromium in the test solution can be made. Accurately dilute the standard chromium solution (4.9) to give a stock solution of concentration 10 times the chromium level in the test solution, as estimated above. Using a burette, transfer 0, 5, 10, 15, 20 ml of the chromium stock solution to 100 ml volumetric flasks. Add 25 ml of background solution (4.8) to each and dilute to the mark with water.

The top standard shall contain at least sufficient chromium to give full scale deflection when using maximum scale expansion.

7.3.3 Adjustment of atomic absorption spectrophotometer (5.1)

Type of lamp: chromium hollow cathode.
Wavelength: 357.87 nm.
Flame: nitrous oxide/acetylene.
Lamp current: follow manufacturer's recommendations.
Slit width: follow manufacturer's recommendations.

Note – The manufacturer's recommendations should be closely followed and particular attention is drawn to the following safety points:

- the explosive nature of acetylene, and regulations concerning its use;
- the need to shield the eyes of the operator from UV radiation by means of tinted glass;
- the need to keep the burner head clear of deposits. A badly clogged burner may cause a flash back;
- the need to ensure that the liquid trap is filled with water.

See also Information Circular No 9 – Appendix C.

7.3.4 Optimization of the atomic absorption spectrometer settings

Follow the manufacturer's instructions for preparing the instrument for use. (See also Information Circular No 9 – Appendix A).

When the current to the lamp, the wavelength and the flow of gas have been adjusted and the burner lit, spray water until the indication has stabilized. Set the absorbance value to zero. Choose a damping setting or integration time to give a signal steady enough to fulfil the precision requirements (5.1.1).

Adjust the flame to be fuel-lean and the burner height to about 1 cm below the light path. Spraying, alternately, the high and zero calibration solutions, adjust the gas flow and burner position (horizontally, vertically and rotationally) until the difference in absorbance between the calibration solutions is at a maximum. Check that the spectrometer is set on the required wavelength.

Evaluate the criteria of 5.1.1 to ensure that the instrument is suitable for the determination.

7.3.5 Atomic absorption measurements

Set the scale expansion so that the top calibration solution gives nearly full scale deflection. Aspirate the calibration solutions in ascending order repetitively until each gives the specified precision (5.1.1) thus showing that the instrument has achieved stability. Select two calibration solutions, one having an absorbance just lower than the test portion solution and one just higher. Aspirate these first in ascending order, then in descending order, with the test solution as the

middle solution in each case, measuring the absorbance in relation to water. Aspirate the complete range of calibration solutions again.

It is recognized that these procedures cannot be followed with automatic instruments which accept two calibration solutions only. In this case, it is suggested that the two 'sandwiching' solutions should not be used for the primary calibration but should be analysed alternately with the test solution.

Spray a calibration solution at frequent intervals during the measurement of a batch of determinations. Clean the burner if the results show loss of precision caused by clogging.

Obtain the net mean absorbance of each calibration solution by subtracting the mean absorbance of the zero calibration solution.

Obtain the mean absorbance of the test solution and the mean absorbance of the reagent blank solution.

Convert the mean absorbances of the test solution and of the reagent blank solution to micrograms of chromium per millilitre by means of the calibration graph (7.4).

7.4 Establishment of the calibration graph

It is necessary to draw up a new calibration graph for each series of determinations, and for the range of chromium contents expected.

If pure metals and reagents have been used, the blank test and zero calibration standard should give equal and negligibly small absorbances. In this case, prepare a calibration graph by plotting the mean absorbance values of the calibration solutions against micrograms of chromium per millilitre. Refer the mean test solution absorbances and the absorbances of the two adjacent calibration solutions to the graph.

If, however, the zero calibration solution has a significant absorbance, a more complicated procedure is required. (See Information Circular No 8, Section 5.) In this case, the concentration of chromium in the zero calibration solution can be calculated using the formula:

$$C_z = C_1 \times \frac{A_z}{A_1 - A_z}$$

where

C_z is the concentration of chromium in the zero calibration solution.

C_1 is the concentration of chromium added to the first calibration solution.

A_z is the absorbance of the zero calibration solution.

A_1 is the absorbance of the first calibration solution.

The derived value C_z is then added to each of the nominal calibration concentrations in order to obtain a mean calibration curve passing through the origin. Refer the absorbances of the reagent blank, the test solution and the two adjacent calibration solutions to this graph. Subtract the concentration of the reagent blank from the other concentrations.

If the results of the two calibration readings do not deviate from the graph by more than the permitted precision criteria then the test solution readings are acceptable.

- c is the concentration of chromium in the reagent blank solution expressed in µg/ml.
- D is the dilution factor applied in 7.3.1.
- m is the mass, in g, of the test portion.

8 EXPRESSION OF RESULTS

The percentage by mass of chromium (Cr) is given by the expression

$$\text{Cr \% (m/m)} = \frac{(b - c) \times D \times 100}{10^6} \times \frac{100}{m} = \frac{(b - c) \times D}{100 m}$$

where

- b is the concentration of chromium in the test solution expressed in µg/ml.

9 TEST REPORT

The test report shall contain the following particulars:

- (a) the method of analysis used by reference to this European Standard;
- (b) the results obtained, as well as the form in which they are expressed;
- (c) any particular details which may have been noted during the determination;
- (d) any optional not specified in this European Standard or any optional operations which could have had an influence on the result;
- (e) all indications necessary for the identification of the sample;
- (f) the laboratory and the date of analysis.

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