SLOVENSKI STANDARD

SIST EN 10177:2019

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Nadomešča:

SIST EN 10177:1998

Jeklo - Določevanje kalcija - Metoda s plamensko atomsko absorpcijsko spektrometrijo (FAAS)

Steels - Determination of calcium content - Flame atomic absorption spectrometric method (FAAS)

Stahl - Bestimmung des Calciumgehalt - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

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

Ta slovenski standard je istoveten z: **EN 10177:2019**

ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.080.20	Jekla	Steels

SIST EN 10177:2019 **en,fr,de**

EUROPEAN STANDARD

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EUROPÄISCHE NORM

EN 10177

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ICS 77.040.30

Supersedes EN 10177:1989

English Version

Steels - Determination of calcium content - Flame atomic absorption spectrometric method (FAAS)

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

Stahl - Bestimmung des Calciumgehalt - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

This European Standard was approved by CEN on 22 April 2019.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents

European foreword

This document (EN 10177:2019) has been prepared by Technical Committee CEN/TC 459 "ECISS - European Committee for Iron and Steel Standardization"[1], the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by December 2019, and conflicting national standards shall be withdrawn at the latest by December 2019.

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

This document supersedes EN 10177:1989.

In comparison with EN 10177:1989, the following significant technical changes were made:

- Clause 1: limits of the scope changed;
- Normative references: revised;
- Clause 4: possibility for using aqua regia for the dissolution and other suitable radiation sources added;
- Clause 5: preparation of iron base solution added;
- 8.3: details regarding the preparation of the test solution added
- 8.3.3: calibration solutions expanded;
- 8.3.5 and Clause 9: bracketing method for the spectrometric measurements added;
- Clause 10: editorially updated;
- Bibliography: added.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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[1] Through its subcommittee SC 2 "Methods of chemical analysis for iron and steel" (secretariat: SIS)

1 Scope

This document specifies a flame atomic absorption spectrometric method (FAAS) for the determination of calcium content in non-alloy and low alloy steels.

The method is applicable to calcium contents between 4 µg/g and 120 µg/g.

The method can be adapted to higher calcium contents by changing the test portion or the dilution process, provided the criteria in 6.2.2 and 6.2.3 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 http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

Dissolution of a test portion in hydrochloric acid followed by oxidation with nitric acid.

NOTE 1 Aqua regia can be used for simultaneous dissolution and oxidation of the test portion.

Addition of a solution of potassium chloride and nebulization of the test solution into an acetylene/nitrous oxide flame of an atomic absorption spectrometer.

NOTE 2 Potassium chloride is added to suppress ionization of calcium.

Spectrometric measurement of the atomic absorption of the 422,7 nm spectral line emitted by a calcium hollow-cathode lamp.

NOTE 3 Other suitable radiation sources can also be used, provided the criteria in 6.2.2 and 6.2.3 are still met.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

The following concentrations and amounts can be modified, provided the changes are taken into account in 8.3 and Clause 9.

5.1 Pure iron.

5.2 Hydrochloric acid solution, 1 + 1.

Add 500 ml of hydrochloric acid (ρ_{20} = 1,19 g/ml, approximately) to 500 ml of water and mix.

5.3 Nitric acid solution, 4 + 6.

Add 400 ml of nitric acid (ρ_{20} = 1,40 g/ml, approximately) to 600 ml of water and mix.

5.4 Potassium chloride solution, 190 g/l.

5.5 Calcium standard solution, 1 g/l.

Weigh (2,500 ± 0,001) g of pure calcium carbonate, dried at 105 °C and transfer into glassware of suitable size. Add 20 ml of water and then, slowly, 20 ml of hydrochloric acid (5.2).

When dissolved, dilute to 100 ml approximately with water and 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 solution contains 1 mg of calcium.

5.6 Calcium standard solution, 0,02 g/l.

Transfer 5,0 ml of calcium standard solution (5.5) into a 250 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 0,02 mg of calcium.

5.7 Calcium standard solution, 0,002 g/l.

Transfer 20,0 ml of calcium standard solution (5.6) into a 200 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 0,002 mg of calcium.

5.8 Iron base solution, 50 g/l.

Weigh, to the nearest 0,01 g, 10,00 g of pure iron (5.1) and transfer into a heat-resistant glassware of suitable size. Add 100 ml of hydrochloric acid (5.2), cover the glassware with a watch glass and heat gently until the reaction ceases. Oxidize with 25 ml of nitric acid (5.3) and then boil the solution for 5 min to remove nitrogen oxides.

Allow to cool and transfer the solution quantitatively into a 200 ml one-mark volumetric flask.

Add 2,5 ml of potassium chloride (5.4), dilute to the mark with water and mix.

6 Apparatus

6.1 Ordinary laboratory apparatus

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

All glassware shall be washed in hydrochloric acid (5.2) and then in water. The quantity of calcium present in the glassware and the volumetric flasks can be checked by measuring the absorption of distilled water introduced in the glassware after the acid washing.

6.2 Atomic absorption spectrometer

6.2.1 General

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

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 6.2.2 and 6.2.3.

6.2.2 Minimum precision

The standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution shall not exceed 3 % 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 1 % of the mean absorbance of the most concentrated calibration solution.

6.2.3 Additional performance requirements

6.2.3.1 General

The instrument should also conform to the additional performance requirements given in 6.2.3.2 and 6.2.3.3.

6.2.3.2 Characteristic concentration

The characteristic concentration for calcium in a matrix similar to the final test solution shall be lower than 0,05 µg/ml.

6.2.3.3 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 calcium in a matrix similar to the final test solution shall be less than 0,025 µg/ml.

7 Sampling

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

Degrease the chips with any appropriate solvent (acetone, ethyl ether, etc.), avoiding contamination of the sample when handling.

Calcium is liable to non-uniform distribution throughout the steel and it is recommended that the laboratory sample should contain not less than 25 g of chips, from which the test portion (8.1) should be taken.

8 Procedure

NOTE The following concentrations, amounts and glassware volumes can be modified, provided the changes are taken into account in all appropriate sub clauses of Clauses 8 and 9 and technically validated.

8.1 Test portion

Weigh to the nearest 1 mg, approximately 2,0 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 400 ml beaker. Add 20 ml of hydrochloric acid (5.2), cover the beaker with a watch glass and heat gently until the acid action ceases.

Oxidize with 5 ml of nitric acid (5.3) and then boil the solution for 5 min to remove nitrogen oxides.

8.3.2 Treatment of the test solution

8.3.2.1 Calcium contents up to 0,006 %

Allow to cool and transfer the test solution quantitatively into a 100 ml one-mark volumetric flask.

WARNING — Do not filter, unless absolutely necessary. If unavoidable, use only filters known to be calcium free.

Add 0,5 ml of potassium chloride (5.4), dilute to the mark with water and mix.

For test solutions giving a calcium absorbance greater than about 0,6, the test solution should be diluted and compared with calibration solutions diluted in the same way. These more diluted solutions should also be used if problems with burner blockage are encountered.

8.3.2.2 Calcium contents between 0,004 % and 0,012 %

Allow to cool and transfer the test solution quantitatively into a 200 ml one-mark volumetric flask.

WARNING — Do not filter, unless absolutely necessary. If unavoidable, use only filters known to be calcium free.

Add 0,5 ml of potassium chloride (5.4), dilute to the mark with water and mix.

For test solutions giving a calcium absorbance greater than about 0,6, the test solution should be diluted and compared with calibration solutions diluted in the same way. These more diluted solutions should also be used if problems with burner blockage are encountered.

8.3.3 Preparation of the calibration solutions

8.3.3.1 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.

8.3.3.2 Calcium contents up to 0,006 % (mass fraction)

Into each of a series of 50 ml one-mark volumetric flasks, introduce the volumes of calcium standard solution (5.7) shown in Table 1 and 20 ml of the iron base solution (5.8). Dilute to the mark with water and mix.