



SLOVENSKI STANDARD

SIST EN 10181:2019

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Nadomešča:
SIST EN 10181:1997

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

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

Stahl - Bestimmung des Bleianteils - Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

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

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EUROPEAN STANDARD

EN 10181

NORME EUROPÉENNE

EUROPÄISCHE NORM

June 2019

ICS 77.040.30

Supersedes EN 10181:1989

English Version

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

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

Stahl - Bestimmung des Bleianteils -
Flammenatomabsorptionsspektrometrisches
Verfahren (FAAS)

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

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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

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Contents	Page
European foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions.....	4
4 Principle.....	4
5 Reagents.....	4
6 Apparatus.....	5
7 Sampling.....	6
8 Procedure.....	6
8.1 Test portion.....	6
8.2 Blank test.....	6
8.3 Determination.....	7
8.3.1 Preparation of the test solution.....	7
8.3.2 Preparation of the calibration solutions.....	7
8.3.3 Adjustment of the atomic absorption spectrometer.....	8
8.3.4 Spectrometric measurements.....	8
9 Expression of results.....	9
9.1 Use of the calibration curve.....	9
9.2 Use of bracketing method.....	10
10 Test report.....	10
Annex A (informative) Samples tested.....	11
Bibliography.....	12

European foreword

This document (EN 10181:2019) 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 10181:1989.

In comparison with EN 10181:1989, the following significant technical changes were made:

- Clause 1: lower limit 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 lead standard solution, 0,05 g/l and iron base solution added;
- 8.3.2: calibration solutions expanded;
- 8.3.3: background correction specified;
- 8.3.4 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.

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

EN 10181:2019 (E)**1 Scope**

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

The method is applicable to lead contents between 0,005 % (weight percent) and 0,5 % (weight percent).

The method can be adapted to lower or higher lead 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.

Nebulization of the test solution into an air/acetylene flame of an atomic absorption spectrometer.

Spectrometric measurement of the atomic absorption of the 283,3 nm spectral line emitted by a lead hollow-cathode lamp.

NOTE 2 Other suitable radiation sources can also be used and measurements can also be carried out at 217,0 nm, 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, with lead content < 0,001 %.

5.2 Hydrochloric acid solution, 1 + 1.

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

5.3 Nitric acid solution, 4 + 6.

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

5.4 Lead standard solution, 0,5 g/l.

Weigh $(0,500 \pm 0,001)$ g of lead ($\text{Pb} \geq 99,99\%$) and transfer into a heat-resistant glassware of suitable size. Add 25 ml of nitric acid ($\rho_{20} = 1,40$ g/ml, approximately), diluted 1 + 4.

Cover with a watch glass, and, if necessary, heat gently to assist dissolution. When dissolution is complete boil to remove nitrogen oxides.

Allow to cool to room temperature 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 0,5 mg of lead.

5.5 Lead standard solution, 0,05 g/l.

Transfer 10,0 ml of lead standard solution (5.4) 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 0,05 mg of lead.

5.6 Iron base solution, 40 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 20 ml of nitric acid (5.3), then boil the solution for 5 min to remove nitrogen oxides.

Allow to cool and transfer the solution quantitatively into a 250 ml one-mark volumetric flask.

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.

6.2 Atomic absorption spectrometer**6.2.1 General**

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

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 8.3.3, 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.

EN 10181:2019 (E)

6.2.2 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.

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 lead in a matrix similar to the final test solution shall be lower than 0,6 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 lead in a matrix similar to the final test solution shall be less than 0,3 µg/ml.

7 Sampling

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

8 Procedure

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

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 250 ml beaker. Add 30 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.

NOTE Aqua regia can be also used for dissolution.

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

Dilute to the mark with water and mix.

If some residue has been left in the solution, filter the solution through a dry, medium-texture filter paper, discarding the first portions.

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 Lead contents up to 0,050 %

Into each of a series of 50 ml one-mark volumetric flasks, introduce the volumes of lead standard solution (5.5) shown in Table 1 and 25 ml of the iron base solution (5.6). Dilute to the mark with water and mix.

Table 1 — Calibration for lead contents up to 0,050 %

Lead standard solution volume (5.5) ml	Corresponding lead mass mg	Corresponding lead concentration after final dilution mg/ml	Corresponding lead content in the sample %
0	0	0	0
1,0	0,05	0,001	0,005
2,0	0,10	0,002	0,010
4,0	0,20	0,004	0,020
6,0	0,30	0,006	0,030
8,0	0,40	0,008	0,040
10,0	0,50	0,010	0,050

8.3.2.3 Lead contents between 0,05 % and 0,5 %

Into each of a series of 50 ml one-mark volumetric flasks, introduce the volumes of lead standard solution (5.4) shown in Table 2 and 25 ml of the iron base solution (5.6). Dilute to the mark with water and mix.