SLOVENSKI STANDARD
oSIST prEN 10181:2018

01-september-2018

Jeklo - Določevanje svinca - Plamenska atomska absorpcijska spektrometrična metoda (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)

Ta slovenski standard je istoveten z: **prEN 10181**

ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.080.20	Jekla	Steels

oSIST prEN 10181:2018 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 10181

June 2018

ICS 77.040.30

Will supersede 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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee ECISS/TC 102.

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.

This draft European Standard was established by CEN 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.

CEN members are the national standards bodies of 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 United Kingdom.

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.

Warning : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.

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

Page

European foreword

This document (prEN 10181:2018) has been prepared by Technical Committee ECISS/TC 102 “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 10181:1989.

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 % and 0,5 %.

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)*

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

Weigh (0,500 ± 0,001) g of lead (Pb ≥ 99,99 %) and transfer into a heat-resistant glassware of suitable size. Add 25 ml of nitric acid (ρ_{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 beaker with a watch glass and heat gently until the reaction ceases. Oxidise 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.

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

It is also desirable that the instrument should 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 0,06 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 of lead.

7 Sampling

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

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.

Table 2 — Calibration for lead contents between 0,05 and 0,5 %

Lead standard solution volume (5.4) 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	0,5	0,01	0,05
2,0	1,0	0,02	0,10
3,0	1,5	0,03	0,15
4,0	2,0	0,04	0,20
5,0	2,5	0,05	0,25
6,0	3,0	0,06	0,30
8,0	4,0	0,08	0,40
10,0	5,0	0,10	0,50

8.3.3 Adjustment of the atomic absorption spectrometer

Fit the lead hollow-cathode lamp (see 6.2) to the atomic absorption spectrometer (6.2), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 283,3 nm to minimum absorbance, if possible. Following manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

NOTE Other suitable radiation sources can also be used.

Evaluate the criteria given in 6.2.1 to ensure that the instrument is suitable for the determination.

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

a) the explosive nature of acetylene, and regulations concerning its use;

b) the need to shield the eyes of the operator from ultraviolet radiation by means of tinted glass;

c) the need to keep the burner head clear of deposits because a badly clogged burner may cause a flashback;

d) the need to ensure that the liquid trap is filled with water;

e) the need to always spray water between the test solutions, blank solution and/or calibration solutions.

8.3.4 Spectrometric measurements

NOTE If pure metals and reagents have been used, the blank test and zero member should give very small absorbance readings with a negligibly small difference.

8.3.4.1 Spectrometric measurement of the calibration solutions

Aspirate the relevant series of calibration solutions (8.3.2.2 or 8.3.2.3) depending on the expected lead content in succession into the flame and measure the absorbance for each solution. Take care to keep the aspiration rate constant throughout the preparation of the calibration curve. Spray water through the burner after each measurement (see note).

NOTE For some instruments it is preferable to match the acid concentration of the washing solution with that of the test solution.

Establish the calibration curve using the measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or an off-line computer for regression calculations or prepare a graphical representation.

8.3.4.2 Spectrometric measurement of the test solutions

8.3.4.2.1 Preliminary spectrometric measurement

Carry out a preliminary measurement of the test solution (8.3.1) at the same time as the spectrometric measurements are carried out on the calibration solutions (see 8.3.2). Estimate the preliminary analyte amount by using the calibration curve (8.3.4.2.2.1).

8.3.4.2.2 Spectrometric measurement

8.3.4.2.2.1 Use of the calibration curve

Measure the absorbance of the blank test solution and repeat the measurements of the test solution; derive the corresponding concentrations directly from the calibration curve.