SLOVENSKI STANDARD

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Kemična analiza železovih zlitin - Določevanje svinca v jeklih - Plamenska atomska absorpcijska spektrometrična metoda

Chemical analysis of ferrous materials - Determination of lead in steels - Flame atomic absorption spectrometric method

Chemische Analyse von Eisen- und Stahlwerkstoffen - Bestimmung des Bleigehaltes von Stahl - Flammenatomabsorptionsspektrometrisches Verfahren

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

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EUROPEAN STANDARD
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of content, lead, atomic absorption spectrometry, flame photometry

English version

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European Committee for Standardization
Comité Européen de Normalisation
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Central Secretariat : Rue Bréderode 2, B-1000 Brussels

EN 10 181 Page 2

Brief History

This European Standard takes over the content of EURONORM 181-85 "Chemical analysis of ferrous materials - Determination of lead in steels - 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 has been 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.

It has been adopted and ratified by CEN BT on 1988-11-05.

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

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Note in clauses 1 and 9 EURONORM shall read EUROPEAN STANDARD.

Chemical analysis of ferrous materials
Determination of lead in steels
Flame atomic absorption spectrometric method

CONTENTS

1. SCOPE AND FIELD OF APPLICATION

This EURONORM specifies a method for the determination of lead in steels by means of flame atomic absorption spectrometry.

The method is applicable to non-alloy and low-alloy steels with lead contents of 0.003 to 0.5% (m/m) (see Annex).

2. REFERENCE

EURONORM 18 — Selection and preparation of samples and test pieces for steel and iron and steel products.

General guidelines for the application of flame atomic absorption spectrometric methods are in course of preparation.

3. PRINCIPLE

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

Spraying of the solution into an air-acetylene flame. Determination of the lead by means of the spectrometric measurement of the atomic absorption of the 283.3 nm line emitted by a lead radiation source.

The instrument is calibrated by addition of a lead standard solution to a similar matrix to that of the test solution.

4. REAGENTS

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

Carefully check the lead content of all reagents.

If possible, use only freshly prepared distilled or deionized water.

4.1 Iron, of high purity, with a lead content < 0.001% (m/m)

4.2 Hydrochloric acid, ρ 1.19 g/ml approximately, diluted 1 + 1 (V/V) (6 mol/l approximately)

4.3 Nitric acid, ρ 1.40 g/ml approximately, diluted 4 + 6 (V/V), (5.6 mol/l approximately)

4.4 Lead reference solution corresponding to 0.5 mg of lead per ml

Wash approximately 1 g of lead ($\geqslant$ 99.9% pure) in boiling hydrochloric acid (ρ 1.19 g/ml, diluted 1 + 7 (V/V)) and afterwards with water and acetone. Dry the lead in a vacuum desiccator.

Weigh, to the nearest 0.001 g, approximately 0.5 g of the purified lead. Transfer the weighed mass to a 400 ml beaker and dissolve in 25 ml nitric acid (ρ 1.40 g/ml approximately, diluted 1 + 4 (V/V)) by heating.

Boil the solution to remove oxides of nitrogen, cool and transfer the solution to a 1 000 ml volumetric flask, dilute to

the mark with water and mix. Calculate the concentration of lead, in mg/ml.

5. APPARATUS

Ordinary laboratory equipment and

5.1 Atomic absorption spectrometer; a lead radiation source; supplies of air and acetylene sufficiently pure to give a 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 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 0.50% and 0.05% lead in the sample, the precision called for (as two standard deviations) would be 0.015% and 0.005% lead 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.

(a) Characteristic concentration. — The characteristic concentration for lead in a matrix similar to the final test solution shall be better than $0.6 \mu g/ml$.

(b) Limit of detection. — The limit of detection of lead in a matrix similar to the final sample test solution shall be better than $0.3 \mu g/ml$.

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: $m = 2\,g \pm 5\%$.

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 30 ml of hydrochloric acid (4.2). Heat gently until the reaction ceases. Oxidize with 5 ml nitric acid (4.3). Boil the solution for 5 minutes to remove oxides of nitrogen. Cool, transfer the solution to a 100 ml volumetric flask, dilute to the mark with water and mix.

If a residue has been left in the solution, filter off a portion through a dry medium textured filter paper, discarding the first runnings.

7.3.2 Preparation of the calibration solutions

Place 10 g of iron (4.1) in an 800 ml beaker and add 100 ml of hydrochloric acid (4.2). Heat gently until the reaction ceases. Oxidize with 20 ml of nitric acid (4.3).

Boil the solution for 5 minutes to remove oxides of nitrogen. Transfer to a 250 ml volumetric flask, dilute to the mark with water and mix.

Transfer a series of 25.0 ml aliquots to 50 ml volumetric flasks. Add, by means of a burette or pipette, respectively 0; 1.0; 2.0; 3.0; 4.0; 5.0; 6.0; 8.0 and 10.0 ml of the lead reference solution (4.4), dilute to the mark with water and mix.

NOTE — 1 ml of lead reference solution (4.4) is equivalent to approximately 0.05% (m/m) in the sample.

7.3.3 Adjustment of atomic absorption spectrometer (5.1)

Type of lamp:	Lead radiation source.
Wavelength:	283.3 nm.
Flame:	Air-acetylene clear fuel-lean flame adjusted for maximum lead response.
Lamp current — 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:

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

(b) the need to shield the eyes of the operator from u.v. radiation by means of tinted glass;

(c) the need to keep the burner head clear of deposits. A badly clogged burner may cause a flash back.

(d) make sure that the liquid trap is filled with water.

7.3.4 Optimization of the atomic absorption spectrometer settings

Follow the manufacturer's instructions for preparing the instrument for use.

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 clear 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.

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 acceptable precision (see 5.1.1), thus showing that the instrument has achieved stability. Select two calibration solutions, one having an absorbance just lower than the test 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.

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 lead 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 lead contents expected.

Prepare a calibration graph by plotting the net mean absorbance values of the calibration solutions against micrograms of lead per millilitre, calculated according to 4.4. Refer the net absorbances of the two adjacent calibration solutions to the graph. If these two calibration readings do not deviate from the graph by more than the permitted precision criteria then the test solution readings are acceptable.

8. EXPRESSION OF RESULTS

The percentage by mass of lead (Pb) is given by the expression

$$Pb \% \, (m/m) = \frac{(b-c) \cdot 100}{10^6} \cdot \frac{100}{m} = \frac{b-c}{100 \, m}$$

Where:

b is the concentration of lead in the test solution, expressed in $\mu g/ml$.

c is the concentration of lead in the reagent blank solution, expressed in $\mu g/ml$.

m is the mass of the test portion, in g.

9. TEST REPORT

The test report shall contain the following particulars:

(a) the method of analysis used by reference to this EURO-NORM;

(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 operations not specified in this EURONORM 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.

ANNEX

Samples tested

The method has been tested by analysts of different laboratories on the following certified reference materials (CRM):

CRM	Alloy type	Lead %
BCS 271	Mild steel	0.0025
BCS 330	Mild steel	0.003
BCS 274	Mild steel	0.008
BCS 456	Mild steel	0.010
BCS 328	Mild steel	0.015
BCS 329	Mild steel	0.050
BCS 212/1	Leaded steel	0.22
NBS 130a	Leaded steel	0.228
EURONORM 039-2	Leaded steel (1.5% Mn; 0.2% S)	0.308

The method has also been applied for the analysis of the following highly-alloyed CRMs, with acceptable results:

BCS 336: Cr-Ni stainless steel   0.0007% Pb
NBS 160a: Cr-Ni-Mo stainless steel 0.001% Pb