SLOVENSKI STANDARD
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Kemijska analiza železovih zlitin - Določevanje selena v jeklih - Elektrotermična atomska absorpcijska spektrometrija

Chemical analysis of ferrous materials - Determination of selenium in steels - Electrothermal atomic absorption spectrometric method

Chemische Analyse der Eisen und Stahlwerkstoffe - Bestimmung des Selengehaltes in Stahl

Analyse chimique des produits ferreux - Détermination du sélénium dans les aciers - Méthode par spéctrométrie d'absorption atomique éléctrothermique

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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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TECHNICAL REPORT

RAPPORT TECHNIQUE

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Chemical analysis of ferrous materials - Determination of selenium in steels - Electrothermal atomic absorption spectrometric method

Analyse chimique des produits ferreux - Détermination du sélénium dans les aciers - Méthode par spéctrométrie d'absorption atomique éléctrothermique

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Selen in Stahl - Spektrometrisches Verfahren mit elektrothermischer Atomadsorption

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Foreword

This document (FprCEN/TR 10362:2014) 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 Technical Committee Approval.

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1 Scope

This Technical Report specifies an electrothermal atomic absorption spectrometric method for the determination of selenium in steels.

The method is applicable to selenium contents between 0,000 4 % (m/m) and 0,02 % (m/m).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 14284, *Steel and iron - Sampling and preparation of samples for the determination of chemical composition (ISO 14284)*

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids and dilution of the solution to a known volume.

Introduction of a known volume of the solution into the electrothermal atomizer of an atomic absorption spectrometer.

Measurement of the atomic absorption of the 196,0 nm spectral line energy emitted by a selenium hollow-cathode lamp, using background correction by Zeeman.

Calibration by the standard addition technique.

4 Reagents

During the analysis, use only reagents of recognised analytical grade and only grade 3 water, as specified in EN ISO 3696.

4.1 **Nitric acid,** HNO_3 (ρ_{20} = 1,40 g/ml)

4.2 **Hydrochloric acid,** HCl (ρ_{20} = 1,19 g/ml)

4.3 Matrix modifiers

The matrix modifiers described in 4.3.1 and 4.3.2 are recommended. Each laboratory has to investigate on its own equipment which of them is the most suitable, regarding sensitivity and recovery.

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4.3.1 Palladium-nickel modifier

Prepare a palladium solution (1 mg/ml Pd) by dissolving 167 mg of $PdCl_2$ in 100 ml of hot water and 1 ml of nitric acid (4.1).

Prepare a nickel solution (1 mg/ml Ni) by dissolving 1 gram of nickel metal (Ni > 99,999 %) in 20 ml of water, 20 ml of nitric acid (4.1) and 5 ml of hydrochloric acid (4.1). Heat until the metal is dissolved. After cooling, transfer the solution into a 1 l one-mark volumetric flask, dilute to the mark with water and mix well.

Into a 50 ml volumetric flask, mix 35 ml of the 1 mg/ml palladium solution with 15 ml of the 1 mg/ml nickel solution. This solution contains 700 µg/ml Pd and 300 µg/ml Ni.

4.3.2 Palladium-magnesium modifier

Prepare a $PdCl_2$ solution by dissolving 500 mg of $PdCl_2$ in 100 ml of hot water and 1 ml of nitric acid (4.1).

Prepare a $Mg(NO_3)_2$ solution by dissolving 350 mg of $Mg(NO_3)_2$. $6H_2O$ in 100 ml of water and 1 ml of nitric acid (4.1).

Mix equal volumes of the $PdCl_2$ solution and the $Mg(NO_3)_2$ solution.

4.4 Selenium, 1 g/l standard solution

Weigh (1 ± 0,000 1) g of selenium (99,9 % purity), transfer into a 100 ml beaker and cover with a watch glass.

Dissolve it in 35 ml of nitric acid (4.1). Heat to complete dissolution at a temperature just below the boiling point (approximately 150 °C) during at least 30 minutes. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of selenium.

4.5 Selenium, 0,01 g/l standard solution

Transfer 10,0 ml of selenium standard solution (4.4) into a 1 000 ml one-mark volumetric flask. Add 120 ml of hydrochloric acid (4.2) and 40 ml of nitric acid (4.1). Dilute to the mark with water and mix well.

1 ml of this standard solution contains 0,01 mg of selenium.

4.6 **Pure iron,** containing less than 0,000 1 % (mass fraction) of selenium

5 Apparatus

All volumetric glassware shall be Class A and calibrated, in accordance with ISO 648 or ISO 1042 as appropriate.

Before use, all glassware shall be cleaned by boiling with hydrochloric acid to remove any chemical contamination.

5.1 Auto sampler equipped with micropipettes of capacity 10 µl to 50 µl

5.2 Atomic absorption spectrometer and electrothermal atomizer

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This shall be equipped with a selenium hollow-cathode lamp (HCL) or an electrodeless discharge lamp (EDL) and supplied with pure argon.

The instrument shall be equipped with a Zeeman background correction.

An electrothermal atomizer equipped with a L'vov platform, mounted in a pyrolytically coated graphite tube, supplied with argon as purge gas, is recommended.

The characteristic mass for selenium shall be less than 30 pg of Se.

6 Sampling

Sampling shall be carried out in accordance with EN ISO 14284 or with an appropriate national standard for steels.

7 Procedure

7.1 Test portion

According to the expected selenium content (x) of the test sample, weigh to the nearest 0,1 mg, a test portion as indicated in Table 1.

Table 1 — Mass of the test portion

Expected selenium content (x) of the test sample (µg/g)	**Mass of the test portion** (mg)
0 < x < 15	2 000
15 ≤ x < 35	1 000
35 ≤ x < 75	400
75 ≤ x < 150	200
150 ≤ x ≤ 200	100

7.2 Blank test

Carry out a blank test simultaneously with the determination (see 7.3), following the same procedure and using the same quantities of all reagents as used for the determination, but substituting pure iron (4.6) for the test portion.

7.3 Preparation of the test solution

Transfer the test portion (7.1) into a 250 ml beaker tall form. Cover the beaker with a watch glass and add 10 ml of water, 10 ml of nitric acid (4.1) and 30 ml of hydrochloric acid (4.2).

Heat the sample at a temperature just below the boiling point (approximately 150 °C) during at least two hours. Allow the solution to cool. If necessary, filter through a medium texture filter paper and collect the filtrate in a 250 ml one-mark volumetric flask. Wash the filter paper several times with hot water and collect the washings in the same 250 ml volumetric flask.

Allow the solution to cool, dilute to the mark with water and mix.

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7.4 Preparation of the standard addition solutions for the test solution

Into a series of four 50 ml one-mark volumetric flasks introduce 25 ml aliquots of the test solution (7.3). Using a burette or a pipette, add the volumes of selenium standard solution (4.5) shown in Table 2. Dilute to the mark with water and mix.

These solutions are labelled S1, S2, S3 and S4 respectively.

Table 2 — Standard addition solutions for the test solution

Label of the solution	Selenium standard solution volume µl	Concentration of selenium in the test addition solutions µg/ml
S1	0	0
S2	200	0,04
S3	400	0,08
S4	600	0,12

7.5 Preparation of the standard addition solutions for the blank test

Into a series of four 50 ml one-mark volumetric flasks introduce 25 ml aliquots of the blank solution (7.2). Using a burette or a pipette, add the volumes of selenium standard solution (4.5) shown in Table 3. Dilute to the mark with water and mix.

These solutions are labelled B1, B2, B3 and B4 respectively.

Table 3 — Standard addition solutions for the blank test

Label of the solution	Selenium standard solution volume µl	Concentration of selenium in the test addition solutions µg/ml
B1	0	0
B2	200	0,04
B3	400	0,08
B4	600	0,12

8 Measurements

Set the required instrument parameters and align the electrothermal atomiser according to the manufacturer's instructions (see NOTE 1 and 2).

Adjust the wavelength in the region of 196,0 nm to minimum absorbance.

NOTE 1 The operating parameters for electrothermal atomic absorption spectrometry vary considerably from an instrument to another and much more than for flame atomic absorption spectrometry. Typical operating parameters are given in Annex A. Annex B details the instruments and the instrumental conditions used by the laboratories having participated to the interlaboratory test programme (see Clause10).

Fill the auto sampler with the blank standard addition solutions (7.4), the test standard addition solutions (7.3) and the matrix-modifier (4.3.1 or 4.3.2).