



BSI Standards Publication

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

National foreword

This Published Document is the UK implementation of CEN/TR 10362:2014.

The UK participation in its preparation was entrusted to Technical Committee ISE/102, Methods of Chemical Analysis for Iron and Steel.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 84816 2

ICS 77.040.30; 77.080.20

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This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 November 2014.

Amendments issued since publication

Date	Text affected
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ICS 77.040.30; 77.080.20

English Version

**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 spectrométrie
d'absorption atomique électrothermique

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

This Technical Report was approved by CEN on 10 May 2014. It has been drawn up by the Technical Committee ECISS/TC 102.

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Foreword

This document (CEN/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.

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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)*

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

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, HNO₃ ($\rho_{20} = 1,40$ g/ml)

4.2 Hydrochloric acid, HCl ($\rho_{20} = 1,19$ 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.

4.3.1 Palladium-nickel modifier

Prepare a palladium solution (1 mg/ml Pd) by dissolving 167 mg of PdCl₂ in 100 ml of hot water and 1 ml of nitric acid (4.1).

Prepare a nickel solution (1 mg/ml) by dissolving 1 g of nickel (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.