

Nederlandse praktijkrichtlijn

# **NPR-CEN/TR 10362**

(en)

Chemische analyse van ijzerhoudende materialen  
- Bepaling van selenium in staal - Elektrothermische  
atomaire absorptiespectrometrische methode

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

ICS 77.040.30; 77.080.20

januari 2015

Als Nederlandse praktijkrichtlijn is aanvaard:  
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Voor de in deze praktijkrichtlijn vermelde normatieve verwijzingen bestaan in Nederland de volgende equivalenten:

<u>vermelde norm</u>	<u>Nederlandse norm</u>	<u>titel</u>
EN ISO 648	NEN-EN-ISO 648	Laboratoriumglaswerk - Volume inhoud pipetten
EN ISO 1042	NEN-EN-ISO 1042	Laboratoriumglaswerk - Maatkolven met één streep
EN ISO 3696	NEN-EN-ISO 3696	Water voor analytische laboratoriumdoeleinden - Eisen en beproevingsmethoden

Voorbeeld  
Preview

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TECHNICAL REPORT

**CEN/TR 10362**

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

November 2014

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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**CEN/TR 10362:2014 (E)****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**, HNO<sub>3</sub> ( $\rho_{20} = 1,40$  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 PdCl<sub>2</sub> 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.



#### 4.3.2 Palladium-magnesium modifier

Prepare a  $\text{PdCl}_2$  solution by dissolving 500 mg of  $\text{PdCl}_2$  in 100 ml of hot water and 1 ml of nitric acid (4.1).

Prepare a  $\text{Mg}(\text{NO}_3)_2$  solution by dissolving 350 mg of  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in 100 ml of water and 1 ml of nitric acid (4.1).

Mix equal volumes of the  $\text{PdCl}_2$  solution and the  $\text{Mg}(\text{NO}_3)_2$  solution.

#### 4.4 Selenium standard solution, 1 g/l

Weigh, to the nearest 0,001 g, 1 g of high purity selenium [min 99,9 % (mass fraction)], 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 standard solution, 0,01 g/l

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 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 $\mu\text{l}$ to 50 $\mu\text{l}$

#### 5.2 Atomic absorption spectrometer and electrothermal atomizer

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

The instrument shall be fitted with Zeeman effect 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 selenium.

### 6 Sampling

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

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