



# SLOVENSKI STANDARD

## SIST-TP CEN/TR 10353:2012

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### Kemična analiza železovih zlitin - Analiza ferosilicija - Določevanje Al, Ti in P z direktno spektrometrijo optične emisije z induktivno sklopljeno plazmo

Chemical analysis of ferrous materials - Analysis of Ferro-silicon - Determination of Al, Ti and P by inductively coupled plasma optical emission spectrometry

Chemische Analyse von Ferrolegierungen - Analyse von Ferrosilicium - Bestimmung von Al, Ti und P durch induktiv gekoppeltes Plasma und optische Emissionsspektrometrie

Analyse chimique des matériaux ferreux - Analyse du ferro-silicium - Détermination de Al, Ti et P par spectrométrie d'émission optique avec source à plasma induit

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TECHNICAL REPORT  
RAPPORT TECHNIQUE  
TECHNISCHER BERICHT

**CEN/TR 10353**

November 2011

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ICS 77.080.10

English Version

**Chemical analysis of ferrous materials - Analysis of ferro-silicon  
- Determination of Al, Ti and P by inductively coupled plasma  
optical emission spectrometry**

Analyse chimique des matériaux ferreux - Analyse du ferro-silicium - Détermination de Al, Ti et P par spectrométrie d'émission optique avec source à plasma induit

Chemische Analyse von Ferrolegierungen - Analyse von Ferrosilizium - Bestimmung von Al, Ti und P durch induktiv gekoppeltes Plasma und optische Emissionsspektrometrie

This Technical Report was approved by CEN on 24 April 2011. It has been drawn up by the Technical Committee ECISS/TC 102.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (CEN/TR 10353:2011) 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 10353:2011 (E)

## 1 Scope

This Technical Report describes an inductively coupled plasma optical emission spectrometric method for the determination of Al, Ti and P contents in ferro-silicon materials.

The method is applicable to:

- Al contents between 0,2 and 2 %;
- Ti contents between 0,02 and 0,25 %;
- P contents between 0,005 and 0,05 %.

The procedure is valid for the analytical lines given in Table 1. This table also gives, for each line, the spectral interferences, which shall be corrected.

NOTE The interferences extent as well as other possible interferences depend on the temperature in the plasma and on the optical resolution of the spectrometer used.

**Table 1 — Spectral lines recommended together with the interferences which shall be corrected**

Element	Wavelength (nm)	Interferences
Al	308,22	V
Ti	337,28	V, Ni
P	178,29	Mo

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## 2 Normative references

The following referenced documents are indispensable for the application 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 648:2008)*

EN ISO 1042, *Laboratory glassware — One-mark volumetric flasks (ISO 1042:1998)*

## 3 Principle

Dissolution of a test portion with nitric, hydrofluoric and perchloric acids. Addition of hydrochloric acid.

Filtration and ignition of the acid insoluble residue.

Fusion of the residue with sodium hydrogen sulphate, dissolution of the melt and addition of this solution to the reserved filtrate.

After suitable dilution and, if necessary, addition of an internal reference element, the solution is filtered and nebulised into an inductively coupled plasma optical emission spectrometer.

The intensity of the emitted light from each element is then measured (simultaneously with that emitted from the internal reference element, where relevant).

## 4 Reagents

During the analysis, use only reagents of recognised analytical grade and only distilled water or water of equivalent purity.

The same reagents should be used for the preparation of calibration solutions and of sample solutions.

### 4.1 Hydrofluoric acid, HF ( $\rho_{20} = 1,13$ g/ml)

**WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.**

### 4.2 Hydrochloric acid, HCl ( $\rho_{20} = 1,19$ g/ml)

### 4.3 Hydrochloric acid, solution 1 + 1

Add 500 ml of hydrochloric acid (4.2) to 500 ml of water.

### 4.4 Hydrochloric acid, solution 1 + 9

Add 50 ml of hydrochloric acid (4.2) to 450 ml of water.

### 4.5 Nitric acid, HNO<sub>3</sub> ( $\rho_{20} = 1,40$ g/ml)

### 4.6 Perchloric acid, HClO<sub>4</sub> ( $\rho_{20} = 1,68$ g/ml)

### 4.7 Sodium hydrogen sulphate

### 4.8 Pure iron

The purity of the iron selected shall be at least ten times better than the low limit of the scope of each element included in the present method.

### 4.9 Aluminium, 0,5 g/l standard solution

Weigh  $(0,5 \pm 0,001)$  g of aluminium (99,98 % purity) and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid solution (4.3) and heat gently until aluminium is completely dissolved. 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 0,5 mg of Al.

### 4.10 Titanium, 0,5 g/l standard solution

Weigh  $(0,5 \pm 0,001)$  g of titanium (99,98 % purity) and transfer into a 400 ml beaker. Add 25 ml hydrochloric acid solution (4.3) and 5 to 10 drops of hydrofluoric acid (4.1). Heat gently until titanium is completely dissolved. 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 0,5 mg of Ti.

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**CEN/TR 10353:2011 (E)****4.11 Titanium, 0,05 g/l standard solution [freshly prepared]**

Transfer 20 ml of titanium standard solution (4.10) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 0,05 mg of Ti.

**4.12 Phosphorus, 0,1 g/l standard solution [freshly prepared]**

Weigh  $(0,4393 \pm 0,001)$  g of dried potassium dihydrogen phosphate and transfer into a 250 ml beaker. Dissolve it with water. 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 0,1 mg of P.

**4.13 Phosphorus, 0,01 g/l standard solution [freshly prepared]**

Transfer 20,0 ml of phosphorus standard solution (4.12) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this solution contains 0,01 mg of P.

**4.14 Scandium, 1 g/l solution**

Weight  $(1,534 \pm 0,001)$  g of scandium oxide,  $\text{Sc}_2\text{O}_3$ , (99,98 % purity) and transfer into a 600 ml beaker. Dissolve in 40 ml nitric acid (4.5). Transfer into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

**5 Apparatus**

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

**5.1 Platinum crucibles and covers****5.2 Polytetrafluoroethylene (PTFE) beakers****5.3 Optical emission spectrometer, equipped with inductively coupled plasma**

This shall be equipped with a nebulisation system. The instrument used will be satisfactory if, after optimising in accordance with the manufacturer's instructions, it meets the performance criteria given in Annex A.

The spectrometer can be either of simultaneous or of sequential type. A sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal reference element emitted light. In this case the sequential spectrometer can be used for the measurement method using the internal reference element. If the sequential spectrometer is not equipped with this arrangement, the internal reference element shall not be used.

**6 Sampling**

Sampling shall be carried out in accordance with appropriate international or national standards for ferro alloys.



## 7 Procedure

### 7.1 Preparation of the test solution

#### 7.1.1 Test portion

Weigh, to the nearest 0,001 g, 0,5 g of the test sample.

#### 7.1.2 Dissolution of the test portion

Transfer the test portion (7.1.1) into a platinum crucible (5.1) having a suitable capacity or into a polytetrafluoroethylene (PTFE) beaker (5.2).

Add 10 ml of nitric acid (4.5) and, in small portions, 10 ml of hydrofluoric acid (4.1). After each addition swirl the crucible or the beaker and allow the reaction to subside. Then heat to complete the dissolution.

**NOTE** If the internal reference element is not used, care must be taken in order to avoid uncontrolled evaporation of the acids, since differences in acid concentrations in the different solutions will give interference. A way to homogenise the residual acid concentration is the evaporation of the sample solution to dryness, followed by the dissolution of the salts.

Add 2,5 ml of perchloric acid (4.6) and heat until copious white fumes appear. Cool, rinse the sides of the crucible or that of the beaker with water and swirl to dissolve the salts. Repeat the heating to copious fumes.

Cool and add 15 ml of hydrochloric acid (4.4). Heat gently, in order to dissolve all salts. Filter the solution through a close texture filter paper, collect the filtrate into a 250 ml beaker and rinse the crucible or the PTFE beaker and the filter with hot water. Reserve the filtrate.

Transfer the filter into a platinum crucible (5.1), dry at about 105 °C and then heat at about 600 °C to remove organic matter. Ignite at 1 000 °C, to remove volatile oxides, for 15 min.

Cool, add 0,5 g of sodium hydrogen sulphate (4.7) and melt. Cool and dissolve the fusion products with the minimum quantity of water. Add this solution quantitatively to the filtrate in the 250 ml beaker.

Transfer the solution quantitatively into a 100 ml volumetric flask. If the internal reference element is used add, with a calibrated pipette, 0,5 ml of the scandium solution (4.14).

Make up to the mark with water and mix well.

### 7.2 Preparation of the calibration solutions

#### 7.2.1 Aluminium: calibration solutions

Transfer 0,15 g of pure iron (4.8) in each of a series of six 400 ml beakers and dissolve with 10 ml of nitric acid (4.5).

**NOTE** The iron amount (0,15 g) corresponds to a test portion of a ferro-silicon having a 30-70 % average composition.

After dissolution add 2,5 ml of perchloric acid (4.6) and evaporate to copious fumes. After cooling at room temperature, add 15 ml of hydrochloric acid (4.4) and heat gently, in order to dissolve the salts. Add 0,5 g of sodium hydrogen sulphate (4.7) and swirl to dissolve this reagent.

With calibrated pipettes, add the volumes of the aluminium standard solution (4.9) shown in Table 2.

Transfer each solution quantitatively into a series of 100 ml volumetric flasks. If the internal reference element is used add, with a calibrated pipette, 0,5 ml of the scandium solution (4.14).

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Dilute to the mark with water and mix well.

**Table 2 — Composition of the calibration solutions for aluminium**

Calibration solution label	Aluminium standard solution volumes (4.9) (ml)	Corresponding aluminium mass (mg)	Corresponding aluminium content in the sample (%)
0	0	0	0
1	2	1,0	0,2
2	4	2,0	0,4
3	7	3,5	0,7
4	10	5,0	1,0
5	20	10,0	2,0

### 7.2.2 Titanium: calibration solutions

Proceed as specified in 7.2.1 from "Transfer 0,15 g of pure iron..." until "...and swirl to dissolve the salts."

With calibrated pipettes, add the volumes of the titanium standard solutions (4.10 or 4.11) shown in Table 3.

Transfer each solution quantitatively into a series of 100 ml volumetric flasks. If the internal reference element is used add, with a calibrated pipette, 0,5 ml of the scandium solution (4.14).

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Dilute to the mark with water and mix well. [iteh.ai/catalog/standards/sist/cce7338b-3e4f-4844-b7b7-9e04437bebe7/sist-tp-cen-tr-10353-2012](https://standards.iteh.ai/catalog/standards/sist/cce7338b-3e4f-4844-b7b7-9e04437bebe7/sist-tp-cen-tr-10353-2012)

Table 3 — Composition of the calibration solutions for titanium

Calibration solution label	Titanium standard solutions volumes (4.10 or 4.11) (ml)	Corresponding titanium mass (mg)	Corresponding titanium content in the sample (%)
0	0	0	0
1	2,0 (4.11)	0,10	0,02
2	5,0 (4.11)	0,25	0,05
3	10,0 (4.11)	0,50	0,10
4	2,0 (4.10)	1,00	0,20
5	2,5 (4.10)	1,25	0,25

### 7.2.3 Phosphorus: calibration solutions

Proceed as specified in 7.2.1 from "Transfer 0,15 g of pure iron..." until "...and swirl to dissolve the salts."

With calibrated pipettes, add the volumes of the phosphorus standard solutions (4.12 or 4.13) shown in Table 4.

Transfer each solution quantitatively into a series of 100 ml volumetric flasks. If the internal reference element is used add, with a calibrated pipette, 0,5 ml of the scandium solution (4.14).

Dilute to the mark with water and mix well.

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Table 4 — Composition of the calibration solutions for phosphorus

Calibration solution label	Phosphorus standard solutions volumes (4.12 or 4.13) (ml)	Corresponding phosphorus mass (mg)	Corresponding phosphorus content in the sample (%)
0	0	0	0
1	2,5 (4.13)	0,025	0,005
2	5,0 (4.13)	0,050	0,010
3	10,0 (4.13)	0,100	0,020
4	15,0 (4.13)	0,150	0,030
5	2,5 (4.12)	0,250	0,050

### 7.2.4 Multi-elemental calibration solutions (Al, Ti and P)

Proceed as specified in 7.2.1 from "Transfer 0,15 g of pure iron..." until "...and swirl to dissolve the salts."

With calibrated pipettes, add the volumes of the aluminium, titanium and phosphorus standard solutions (4.9, 4.10 or 4.11 and 4.12 or 4.13) shown in Table 5.

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Transfer each solution quantitatively into a series of 100 ml volumetric flasks. If the internal reference element is used add, with a calibrated pipette, 0,5 ml of the scandium solution (4.14).

Dilute to the mark with water and mix well.

NOTE In this way any possible matrix effect will be minimized.

**Table 5 — Composition of the multi-elemental calibration solutions (Al, Ti and P)**

Calibration solution label	Aluminium standard solution volumes (4.9) (ml)	Titanium standard solution volumes (4.10) (ml)	Titanium standard solution volumes (4.11) (ml)	Phosphorus standard solution volumes (4.12) (ml)	Phosphorus standard solution volumes (4.13) (ml)
0	0	0	0	0	0
1	20 [2,0 %]	---	2,0 [0,02 %]	---	15,0 [0,030 %]
2	10 [1,0 %]	---	5,0 [0,05 %]	2,5 [0,05 %]	---
3	7 [0,7 %]	---	10,0 [0,10 %]	---	5,0 [0,010 %]
4	4 [0,4 %]	2,0 [0,20 %]	---	---	2,5 [0,005 %]
5	2 [0,2 %]	2,5 [0,25 %]	---	---	10,0 [0,020 %]

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### 7.3 Spectrometric measurements (standards.iteh.ai)

#### 7.3.1 Adjustment of the apparatus

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Start the inductively coupled plasma optical emission spectrometer (5.3) and let it stabilise in accordance with the manufacturer's instructions before taking any measurements.

At the wavelengths of the analytical lines specified in Table 1, adjust all relevant instrumental parameters, as well as the pre-spraying and the integrating times, according to the instrument manufacturer's instructions while aspirating (for each element) the highest concentration calibration solution.

NOTE Depending on the instrument configuration these parameters may include the outer, intermediate or central gas flow-rates, the torch position, the entrance slits, the exit slits and the photomultiplier tubes voltage.

Prepare the software for measurements of the intensity, and for the calculation of the mean value and relative standard deviation corresponding to each analytical line.

If the internal reference element (scandium in this case) is used, prepare the software to calculate the ratio between the intensity of each analyte and the scandium intensity.

The intensity of the internal reference element shall be measured simultaneously with that of the analyte intensity.

#### 7.3.2 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the calibration solutions (7.2.1, 7.2.2, 7.2.3 or 7.2.4). It is recommended to use a simultaneous spectrometer for measurements using "internal reference element" (ratio mode) (see 5.3).

For each calibration solution, carry out three to five integrations and calculate the corresponding mean intensity or mean ratioed intensity.