SLOVENSKI STANDARD
# SIST EN 10351:2011

01-november-2011

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**Kemična analiza železovih zlitin - Analiza z direktno spektrometrijo optične emisije z induktivno sklopljeno plazmo nelegiranih in malolegiranih jekel - Določevanje Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (celotni) in Sn [rutinska metoda]**

Chemical analysis of ferrous materials - Inductively coupled plasma optical emission spectrometric analysis of unalloyed and low alloyed steels - Determination of Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) and Sn [Routine method]

Chemische Analyse von Eisenwerkstoffen - Analyse von unlegierten und niedrig legierten Stählen mittels optischer Emissionsspektrometrie mit induktiv gekoppeltem Plasma - Bestimmung von Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (gesamt) und Sn [Routineverfahren]

Analyse chimique des matériaux ferreux - Analyse des aciers non alliés et faiblement alliés par spectrométrie d'émission optique avec source à plasma induit - Détermination de Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) et Sn [Méthode de routine]

**Ta slovenski standard je istoveten z:** **EN 10351:2011**

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**ICS:**

| | | |
|---|---|---|
| 77.040.30 | Kemijska analiza kovin | Chemical analysis of metals |

**SIST EN 10351:2011** **en,fr,de**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

# EN 10351

March 2011

ICS 77.140.10; 77.140.45

English Version

# Chemical analysis of ferrous materials - Inductively coupled plasma optical emission spectrometric analysis of unalloyed and low alloyed steels - Determination of Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) and Sn [Routine method]

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 10351:2011: E

# Contents

# Foreword

This document (EN 10351:2011) 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.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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## 1 Scope

This European Standard specifies an inductively coupled plasma optical emission spectrometry routine method for the analysis of unalloyed and low alloyed steels, whose iron content shall be at least 95 %.

This method is applicable to the elements listed in Table 1 within the ranges shown.

**Table 1 — Application ranges**

| Element | Mass fraction % | |
|---|---|---|
| | min. | max. |
| Mn | 0,005 | 2,00 |
| P | 0,005 | 0,05 |
| Cu | 0,005 | 0,80 |
| Ni | 0,010 | 2,00 |
| Cr | 0,010 | 1,60 |
| Mo | 0,005 | 0,80 |
| V | 0,002 | 0,40 |
| Co | 0,002 | 0,10 |
| Al (total) | 0,020 | 0,30 |
| Sn | 0,001 | 0,10 |

In all cases, the ranges specified can be extended or adapted (after validation) for the determination of other mass fractions, provided that the iron content in the samples under concern is above 95 %.

Other elements may be included. However such elements and their mass fractions should be carefully checked, taking into account the possible interferences, the sensitivity, the resolution and the linearity criteria of each instrument and each wavelength.

Depending also on the sensitivity of each instrument, suitable dilutions of the calibration and the test sample solutions may be necessary.

Moreover, even if the method described is "multi elemental", it is not absolutely necessary to carry out the determination of all the elements of its scope simultaneously: the measurement conditions have to be optimised by each laboratory, depending on the performances of each apparatus available.

NOTE 1 The accuracy of the method is unsatisfactory for phosphorus contents from 0,05 to 0,1 %.

NOTE 2 The trueness of the method couldn't be checked for vanadium contents below 0,05 %.

NOTE 3 The precision of the method is unsatisfactory for aluminium (total) contents below 0,02 %.

## 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.

CEN/TR 10345:2008, *Guideline for statistical data treatment of inter laboratory tests for validation of analytical methods*

EN ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition (ISO 14284:1996)*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

## 3 Principle

Dissolution of a test portion with nitric and hydrochloric acids. Filtration and ignition of the acid insoluble residue. Removal of silica with hydrofluoric acid. Fusion of the residue with a mixture of orthoboric acid and potassium carbonate, dissolution of the melt with acid and addition of this solution to the reserved filtrate.

After suitable dilution and, if necessary, addition of an internal reference element, nebulisation of the solution into an inductively coupled plasma optical emission spectrometer and measurement of the intensity of the emitted light from each element (including, where relevant, the intensity of the internal reference element).

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

### 4.2 Hydrochloric acid, solution 1 + 1

Add 500 ml of hydrochloric acid (4.1) to 500 ml of water.

### 4.3 Nitric acid, $HNO_3$ ($\rho_{20}$ = 1,33 g/ml)

### 4.4 Nitric acid, solution 1 + 1

Add 500 ml of nitric acid (4.3) to 500 ml of water.

### 4.5 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.**

### 4.6 Sulphuric acid, $H_2SO_4$ ($\rho_{20}$ = 1,84 g/ml)

### 4.7 Sulphuric acid, solution 1 + 1

Add 25 ml of sulphuric acid (4.6) to 25 ml of water and allow it to cool.

### 4.8 Fusion reagent

#### 4.8.1 Fusion mixture

Mix one part by mass of orthoboric acid, $H_3BO_3$ and one part of potassium carbonate anhydrous, $K_2CO_3$.

#### 4.8.2 Fusion mixture, 100 g/l solution

In a suitable beaker, dissolve 25 g of the fusion mixture (4.8.1). Heat if necessary. After cooling, transfer the solution quantitatively into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix well.

### 4.9 Aluminium, 1 g/l standard solution

Weigh (0,5 ± 0,001) g of aluminium (99,99 % purity) and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid solution (4.2) and heat gently until aluminium is completely dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Al.

### 4.10 Chromium 1 g/l standard solution

Weigh (0,5 ± 0,001) g of chromium (99,99 % purity) and transfer into a 250 ml beaker. Add 40 ml of hydrochloric acid (4.1) and heat gently until chromium is completely dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cr.

### 4.11 Cobalt 1 g/l standard solution

Weigh (0,5 ± 0,001) g of cobalt (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 5 ml of hydrochloric acid (4.1) and 5 ml of nitric acid (4.3). Heat gently until the metal is dissolved and then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Co.

### 4.12 Copper 1 g/l standard solution

Weigh (0,5 ± 0,001) g of copper (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cu.

### 4.13 Manganese 1 g/l standard solution

The manganese used to prepare the solution is released from superficial oxide possibly present by introducing a few grams of metal in a 250 ml beaker containing 150 to 160 ml of water and 15 to 20 ml of sulphuric acid (4.6). Shake and after a few seconds, allow the solution to settle and add water. Repeat the water cleaning several times. Remove the metallic manganese and rinse with acetone. Dry the metal in an oven at 100 °C for 2 minutes or with a hair dryer. Cool in a desiccator.

Weigh (0,5 ± 0,001) g of this purified manganese and transfer into a 250 ml beaker. Dissolve it in 5 ml of hydrochloric acid (4.1) and 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mn.

### 4.14 Molybdenum 1 g/l standard solution

Weigh (0,5 ± 0,001) g of molybdenum (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 10 ml of hydrochloric acid (4.1) and 10 ml of nitric acid (4.3). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mo.

### 4.15 Nickel 1 g/l standard solution

Weigh (0,5 ± 0,001) g of nickel (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Ni.

### 4.16 Phosphorus 1 g/l standard solution

Weigh (2,197 ± 0,001) g of dried potassium dihydrogen phosphate, transfer into a 250 ml beaker and dissolve it with water. Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of P.

### 4.17 Tin 1 g/l standard solution [freshly prepared]

Weigh (0,5 ± 0,001) g of tin (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 50 ml of hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Sn.

### 4.18 Vanadium 1 g/l standard solution

Weigh (0,5 ± 0,001) g of vanadium (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 30 ml of hydrochloric acid (4.1) and 10 ml of nitric acid (4.3). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of V.

NOTE Standard solutions whose preparations are described under items 4.9 to 4.18 can be replaced by commercial available standard solutions, provided that they are supplied by a recognised producer and their traceability fully documented.

### 4.19 Internal reference element solution, 1 g/l

Choose a suitable element to be added as internal reference and prepare a 1 g/l solution.

NOTE Elements as Cd, Fe, Sc and Y were used for this purpose during the validation precision test of this method.

### 4.20 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.

## 5 Apparatus

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

### 5.1 Close texture filter paper and/or 0,45 µm cellulose nitrate filter

### 5.2 Filter funnel suitable for 0,45 µm filters

Two-pieces acid-resistant filter funnel with a support screen between the funnel body and stem, designated for the vacuum filtration of liquids. The stem of the filter is fitted with a ground glass cap stopper or a rubber stopper for insertion into an opening of the vacuum vessel.

### 5.3 Vacuum vessel

With an opening to allow the insertion of the stopper of the filter funnel stem.

### 5.4 Platinum crucibles and covers

### 5.5 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.

## 6 Sampling

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

## 7 Sample solution preparation

### 7.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample.

### 7.2 Preparation of the test solution

#### 7.2.1 Dissolution of the test portion

Transfer the test portion (7.1) into a 250 ml or 400 ml beaker.

Add 5 ml of nitric acid (4.3) and 20 ml of hydrochloric acid solution (4.2). Cover and heat gently until the attack reaction ceases. Heat at a temperature of about 95 °C, for about 5 minutes. Allow to cool.

Rinse the beaker cover and the walls of the beaker with water.

### 7.2.2 Filtration of the test solution

#### 7.2.2.1 Including total aluminium determination

Place a 0,45 µm cellulose nitrate filter (5.1) on the support screen of a filter funnel (5.2). Moisten the filter with water and join the body and stem of the funnel. Insert the stopper of the filter funnel stem into a vacuum vessel (5.3). Apply vacuum gently to the vacuum vessel and filter the solution. Wash the funnel sides and residue with warm water several times.

Stop the vacuum gently.

Wipe the funnel sides with a close texture filter paper, then place the cellulose nitrate filter in this filter paper and fold them.

Transfer the folded filters into a platinum crucible (5.4) and cover with a suitable platinum cover.

Transfer the filtrate quantitatively into a 200 ml one-mark volumetric flask.

#### 7.2.2.2 Excluding total aluminium determination

Filter the solution through a close texture filter paper and collect the filtrate into a 200 ml one-mark volumetric flask.

Wash the filter paper and residue with warm water several times and collect the washings in the volumetric flask.

Transfer the filter into a platinum crucible (5.4) and cover with a suitable platinum cover.

### 7.2.3 Treatment of the insoluble residue

Introduce the crucible in a COLD furnace, then switch it on in order to reach a temperature of about 900 °C. Allow to stand at 900 °C for about 15 min. Cool the crucible. Add into the crucible 0,5 to 1,0 ml of sulphuric acid solution (4.7) and 2 ml of hydrofluoric acid (4.5). Evaporate to dryness and cool.

Add 1,0 g of the fusion mixture (4.8.1). Fuse the content of the crucible in a muffle furnace at 1 000 °C for 15 min to 20 min. Cool the crucible and add 20 ml of hydrochloric acid solution (4.2) to the solidified melt. Heat gently, in order to dissolve the fusion products. Allow the crucible to cool and add this solution quantitatively to the filtrate in the 200 ml volumetric flask.

If an internal reference element is used add, with a calibrated pipette, 10 ml of the internal reference element solution (4.19).

NOTE 1 Depending on the instrument performances, the volume and/or the concentration of the internal reference element solution may be different.

Dilute to the mark with water and mix.

NOTE 2 Depending on the instrument performances, suitable dilutions of the sample solution and of the calibration solutions may be necessary.

## 8 Calibration process

### 8.1 Steps

The calibration process is subdivided into calibration and drift compensation by re-calibration.

### 8.2 Preparation of the multi-elemental calibration solutions

Weigh the quantities of pure iron (4.20) shown in Table 2 and introduce them into each of a series of 250 ml or 400 ml beakers.

Add 20 ml of the hydrochloric acid solution (4.2) to each beaker. Cover with a watch glass and heat gently. When the attack reaction ceases, add 5 ml of nitric acid (4.3). Heat until nitrous fumes have been expelled.

Rinse the cover and the walls of the beaker with water and heat to boiling.

Allow to cool and transfer each calibration solution into a 200 ml one-mark volumetric flask.

With calibrated pipettes, add the quantities (volumes) of the mono-elemental standard solutions (4.9 to 4.18) or of commercial available mono-elemental standard solutions of each element shown in Table 2.

Add to each volumetric flask 10 ml of the fusion mixture solution (4.8.2) and 10 ml of hydrochloric acid (4.1).

If an internal reference element is used add, with a calibrated pipette, 10 ml of the internal reference element solution (4.19).

NOTE 1 Refer to NOTE 1 under 7.2.3.

Dilute to the mark with water and mix.

NOTE 2 Refer to NOTE 2 under 7.2.3.

Following this same procedure, also prepare a pure iron solution (labelled F0 in Table 2), to be used to control if the lowest content for each element is enough high to be considered as "different from the zero point".

Strong care should be taken in order to ensure that the quantity of acids remains about the same in the calibration solutions (**namely when commercially available standard solutions are used**), in the sample solutions and in the Certified Reference Materials solutions prepared to check the trueness of the calibration.

The calibration curves obtained may be stored within a computer, connected to the inductively coupled plasma optical emission spectrometer.

NOTE 3 The trueness of these calibration functions should be checked by measuring at least two suitable Certified Reference Materials or Internal Reference Materials sample solutions (just above the low point and just below the high point of each calibration range).

**Table 2 — Composition of the multi-elemental calibration solutions**

| Element | Label | | | | |
|---|---|---|---|---|---|
| | F0 | F1 | F2 | F3 | F4 |
| Fe | *1 000,0 mg*<br>*100,00 %* | *958,3 mg*<br>*95,83 %* | *979,7 mg*<br>*97,97 %* | *971,2 mg*<br>*97,12 %* | *989,7 mg*<br>*98,97 %* |
| Mn<br>(4.13) | ------ | 20 mg [2,0 %]<br>20 ml | 2 mg [0,2 %]<br>2 ml | 0,02 mg [0,002 %]<br>[a] | 0,2 mg [0,02 %]<br>[b] |
| P<br>(4.16) | ------ | 0,05 mg [0,005 %]<br>[c] | 0,1 mg [0,01 %]<br>[d] | 0,5 mg [0,05 %]<br>[e] | 1 mg [0,1 %]<br>1 ml |
| Cu<br>(4.12) | ------ | 0,5 mg [0,05 %]<br>[f] | 8 mg [0,8 %]<br>8 ml | 0,05 mg [0,005 %]<br>[g] | 1 mg [0,1 %]<br>1 ml |
| Ni<br>(4.15) | ------ | 20 mg [2,0 %]<br>20 ml | 5 mg [0,5 %]<br>5 ml | 0,05 mg [0,005 %]<br>[h] | 0,5 mg [0,05 %]<br>[i] |
| Cr<br>(4.10) | ------ | 0,05 mg [0,005 %]<br>[j] | 0,5 mg [0,05 %]<br>[k] | 16 mg [1,6 %]<br>16 ml | 5 mg [0,5 %]<br>5 ml |
| Mo<br>(4.14) | ------ | 0,05 mg [0,005 %]<br>[l] | 0,5 mg [0,05 %]<br>[m] | 8 mg [0,8 %]<br>8 ml | 1 mg [0,1 %]<br>1 ml |
| V<br>(4.18) | ------ | 0,02 mg [0,002 %]<br>[n] | 4 mg [0,4 %]<br>4 ml | 0,2 mg [0,02 %]<br>[o] | 1 mg [0,1 %]<br>1 ml |
| Co<br>(4.11) | ------ | 0,02 mg [0,002 %]<br>[p] | 0,1 mg [0,01 %]<br>[q] | 1 mg [0,1 %]<br>1 ml | 0,05 mg [0,005 %]<br>[r] |
| Al<br>(4.9) | ------ | 0,03 mg [0,003 %]<br>[s] | 0,1 mg [0,01 %]<br>[t] | 3 mg [0,3 %]<br>3 ml | 0,5 mg [0,05 %]<br>[u] |
| Sn<br>(4.17) | ------ | 1 mg [0,1 %]<br>1 ml | 0,05 mg [0,005 %]<br>[v] | 0,01 mg [0,001 %]<br>[w] | 0,1 mg [0,01 %]<br>[x] |

[a] 2 ml of a 0,01 g/l manganese standard solution

[b] 2 ml of a 0,1 g/l manganese standard solution

[c] 5 ml of a 0,01 g/l phosphorus standard solution

[d] 1 ml of a 0,1 g/l phosphorus standard solution

[e] 5 ml of a 0,1 g/l phosphorus standard solution

[f] 5 ml of a 0,1 g/l copper standard solution

[g] 5 ml of a 0,01 g/l copper standard solution

[h] 5 ml of a 0,01 g/l nickel standard solution

[i] 5 ml of a 0,1 g/l nickel standard solution

[j] 5 ml of a 0,01 g/l chromium standard solution

[k] 5 ml of a 0,1 g/l chromium standard solution

[l] 5 ml of a 0,01 g/l molybdenum standard solution

[m] 5 ml of a 0,1 g/l molybdenum standard solution

[n] 2 ml of a 0,01 g/l vanadium standard solution

[o] 2 ml of a 0,1 g/l vanadium standard solution

[p] 2 ml of a 0,01 g/l cobalt standard solution

[q] 1 ml of a 0,1 g/l cobalt standard solution

[r] 5 ml of a 0,01 g/l cobalt standard solution

[s] 3 ml of a 0,01 g/l aluminium standard solution

[t] 1 ml of a 0,1 g/l aluminium standard solution

[u] 5 ml of a 0,1 g/l aluminium standard solution

[v] 5 ml of a 0,01 g/l tin standard solution

[w] 1 ml of a 0,01 g/l tin standard solution

[x] 1 ml of a 0,1 g/l tin standard solution