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**Kemična analiza železovih zlitin - Analiza nelegiranih in malolegiranih jekel z optično emisijsko spektrometrijo z induktivno sklopljeno plazmo - Določevanje Si, Mn, P, Cu, Ni, Cr, Mo, Sn in topnega Al [rutinska metoda]**

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

**iTeh STANDARD PREVIEW**

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

[SIST EN 10355:2014](https://standards.itih.ai/catalog/standards/sist/0db635ef-a77b-4c1e-9762-10355-2014)

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

**Ta slovenski standard je istoveten z: EN 10355:2013**

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EUROPEAN STANDARD

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Chemical analysis of ferrous materials - Inductively coupled  
plasma optical emission spectrometric analysis of unalloyed and  
low alloyed steels - Determination of Si, Mn, P, Cu, Ni, Cr, Mo  
and Sn, following dissolution with nitric and sulphuric acids  
[Routine method]

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 Si, Mn, P, Cu, Ni, Cr, Mo et Sn, après  
mise en solution par les acides nitrique et sulfurique  
[Méthode de routine]

Chemische Analyse von Eisenwerkstoffen - Analyse von  
unlegierten und niedrig legierten Stählen mittels optischer  
Emissionsspektrometrie mit induktiv gekoppeltem Plasma -  
Bestimmung von Si, Mn, P, Cu, Ni, Cr, Mo und Sn nach  
Lösen in Salpeter- und Schwefelsäure [Routineverfahren]

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Management Centre: Avenue Marnix 17, B-1000 Brussels

| <b>Contents</b>   | <b>Page</b> |
|---|-------------|
| Foreword.....   | 3           |
| 1 Scope.....  | 4           |
| 2 Normative references.....   | 4           |
| 3 Principle.....  | 5           |
| 4 Reagents.....   | 5           |
| 5 Apparatus .....   | 7           |
| 6 Sampling.....   | 7           |
| 7 Sample solution preparation .....   | 7           |
| 8 Calibration process.....  | 9           |
| 9 Determination.....  | 11          |
| 10 Expression of results.....   | 14          |
| 11 Precision.....   | 14          |
| 12 Test report .....  | 17          |
| <b>Annex A</b> (informative) <b>Plasma optical emission spectrometer – Suggested performance criteria to be checked</b> ..... | <b>18</b>   |
| <b>Annex B</b> (normative) <b>Synoptic of the operations related to Clause 9</b> .....  | <b>22</b>   |
| <b>Annex C</b> (informative) <b>Composition of the test samples used for the validation precision test</b> .....              | <b>23</b>   |
| <b>Annex D</b> (informative) <b>Detailed results obtained from the validation precision test</b> .....                        | <b>24</b>   |
| <b>Annex E</b> (informative) <b>Graphical representation of the precision data</b> .....                                      | <b>32</b>   |
| <b>Bibliography</b> .....   | <b>39</b>   |

## Foreword

This document (EN 10355:2013) 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 February 2014, and conflicting national standards shall be withdrawn at the latest by February 2014.

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## EN 10355:2013 (E)

## 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 standard differs from the similar standard EN 10351:2011 in that it is optimised for the determination of silicon.

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

The sample preparation described may not completely dissolve samples having a combination of high chromium and substantial carbon. Incomplete dissolution may also affect the determination of manganese and molybdenum in these samples. For this reason, the scope of the method is limited to chromium contents  $\leq 0,9$  %, whereas the scope of EN 10351 covers a range of up to 1,6 % chromium.

**Table 1 — Application ranges**

| Element | Mass fraction % |      |
|---------|-----------------|------|
|         | min.            | max. |
| Si      | 0,020           | 0,45 |
| Mn      | 0,005           | 1,40 |
| P       | 0,005           | 0,10 |
| Cu      | 0,005           | 0,60 |
| Ni      | 0,010           | 2,00 |
| Cr      | 0,010           | 0,90 |
| Mo      | 0,005           | 0,60 |
| Sn      | 0,010           | 0,10 |

SIST EN 10355:2014

NOTE For tin, see NOTE 2 under Clause 11.  
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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.

## 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)*

### 3 Principle

Dissolution of a test portion with a nitric-sulphuric acids mixture. Addition of an ammonium peroxodisulphate solution and boiling. After cooling, addition of a hydrogen peroxide solution.

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<sub>3</sub> ( $\rho_{20} = 1,33$  g/ml).

**4.4 Nitric acid**, solution 1 + 1.

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

**4.5 Sulphuric acid**, H<sub>2</sub>SO<sub>4</sub> ( $\rho_{20} = 1,84$  g/ml).

**4.6 Hydrogen peroxide**, H<sub>2</sub>O<sub>2</sub> 30 % (mass fraction) solution.

**4.7 Hydrogen peroxide**, H<sub>2</sub>O<sub>2</sub> 3 % (mass fraction) solution.

Dilute 50 ml of hydrogen peroxide (4.6) with water to 500 ml.

**4.8 Nitric-sulphuric acids mixture.**

Add to approximately 500 ml of water 50 ml of nitric acid (4.3) and 50 ml of sulphuric acid (4.5) and mix. After cooling, dilute to 1 l with water and mix.

**4.9 Ammonium peroxodisulphate solution**, 20 g/l.

Dissolve 10 g of ammonium peroxodisulphate in 500 ml water.

**4.10 Chromium 1 g/l standard solution.**

Weigh ( $0,5 \pm 0,001$ ) 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.

**EN 10355:2013 (E)****4.11 Copper 1 g/l standard solution.**

Weigh  $(0,5 \pm 0,001)$  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 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 Cu.

**4.12 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 (4.5). 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 min or with a hair dryer. Cool in a desiccator.

Weigh  $(0,5 \pm 0,001)$  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.

**4.13 Molybdenum 1 g/l standard solution.**

Weigh  $(0,5 \pm 0,001)$  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.

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1 ml of this solution contains 1 mg of Mo.

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**4.14 Nickel 1 g/l standard solution.**

Weigh  $(0,5 \pm 0,001)$  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, 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 Ni.

**4.15 Phosphorus 1 g/l standard solution.**

Weigh  $(2,197 \pm 0,001)$  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.

**4.16 Silicon 1 g/l standard solution.**

Using a suitable plastic beaker, dissolve  $(3,171 \pm 0,001)$  g of dried ammonium hexafluorosilicate  $[(\text{NH}_4)_2\text{SiF}_6]$  in hot water ( $\sim 80$  °C).

Allow to cool and transfer the solution quantitatively into a polyethylene 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.



1 ml of this solution contains 1 mg of Si.

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

Weigh  $(0,5 \pm 0,001)$  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.

NOTE Standard solutions whose preparations are described in 4.10 to 4.17 can be replaced by commercially available standard solutions, provided that they are supplied by a recognised producer and their traceability fully documented.

#### 4.18 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 such as Sc and Y are often used for this purpose.

#### 4.19 Pure iron.

The iron selected shall have a content of less than one tenth of the lower limit of each element as given in the scope.

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### 5 Apparatus

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

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#### 5.1 Microwave system.

#### 5.2 Gravimetric or volumetric dispensers.

#### 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.

### 6 Sampling

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

### 7 Sample solution preparation

#### 7.1 General

The method is optimised for use with automated systems, **open** microwave digestion units (5.1) and gravimetric dispensers (5.2).

NOTE This method was also tested with closed microwave units, after a suitable optimisation of the relevant operating conditions: temperatures and times.

**EN 10355:2013 (E)**

The method can also be used in a classical way with hot plates and manual additions.

**7.2 Test portion**

Weigh, to the nearest 1 mg, 250 mg of the test sample.

**7.3 Preparation of the test solution****7.3.1 Automated system, OPEN units**

Transfer the test portion (7.2) into a PTFE tube.

Add 10 g of the nitric-sulphuric acid mixture (4.8) and cover with a suitable cover (PTFE or some other microwave transparent material, perfectly clean and free from contamination. Screw caps shall be avoided with open microwave units). Use the microwave to heat the sample at about 120 °C for 7 min and then allow to cool for about 10 min.

Add 5 g of the ammonium peroxodisulphate solution (4.9), and cover. Heat at about 120 °C for 3 min by means of the microwave system, and then allow to cool for about 10 min.

Add 5 g of the hydrogen peroxide solution (4.7) and mix well. Cool for about 10 min.

The cooling time may be increased to ensure that gas evolution has ceased before analysing the sample.

The addition of 2 g HCl (4.1) may help to prevent the hydrolysis of silicon and tin.

If an internal reference element is used, add a suitable quantity of the internal reference solution (4.18), dilute the sample solution to 50 g, weighted to the nearest mg and mix well.

If necessary, filter the solution through a dry close-texture filter paper. Reject the first portions of the filtrate.

It is recommended to carry out the measurements on freshly prepared solutions, in order to avoid silicon hydrolysis.

**7.3.2 Manual procedure**

Transfer the test portion (7.2) into a 100 ml quartz or glassware conical flask (or in a 100 ml PTFE beaker).

**NOTE** It has been shown that perfectly clean quartz or ordinary glass conical flasks are suitable even for the determination of silicon.

Other plastic materials may be used, provided that they are heat resistant and free from contamination.

Add 10 ml of the nitric-sulphuric acid mixture (4.8). If beakers are used, cover with a PTFE<sup>1)</sup> watch glass and heat until gently boiling (the hotplate temperature shall be adjusted in order to allow a gentle boiling of the sample solution). Maintain the boiling for about 7 min to 10 min and then allow to cool for about 10 min.

Add 5 ml of the ammonium peroxodisulphate solution (4.9), cover and heat until gently boiling. Maintain the boiling for about 3 min to 5 min and then allow to cool for about 10 min.

Add 5 ml of the hydrogen peroxide solution (4.7) and mix well. Cool for about 10 min.

The cooling time may be increased to ensure that gas evolution has ceased before analysing the sample.

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1) Covers made from other materials can be suitable, provided that these materials are free from contamination.

Transfer the sample solution into a 50 ml one-mark volumetric flask and then, if an internal reference element is used, add, with a calibrated pipette, a suitable volume of the internal reference element solution (4.18).

The addition of 2 ml HCl (4.1) may help to prevent the hydrolysis of silicon and tin.

Dilute to the mark with water and mix well.

If necessary, filter the solution through a dry close-texture filter paper. Reject the first portions of the filtrate.

It is recommended to carry out the measurements on freshly prepared solutions, in order to avoid silicon hydrolysis.

## 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

Weight 2,500 g of pure iron (4.19) into a 500 ml beaker and add 100 ml of the nitric-sulphuric acids mixture (4.8). Heat at about 120 °C, for 7 min.

Add 50 ml of the ammonium peroxodisulphate solution (4.9), cover and heat at about 120 °C for 3 min. Boil gently and allow to cool.

Add 50 ml of the hydrogen peroxide solution (4.7) and mix well. Transfer into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix well.

Into each of a series of four 100 ml one-mark volumetric flasks transfer 50,0 ml of this iron solution and, with calibrated pipettes, add the volumes of the mono-elemental standard solutions (4.10 to 4.17) or of commercial available mono-elemental standard solutions shown in Table 2.

If an internal reference element is used add, with a calibrated pipette a suitable volume of the internal reference element solution (4.18); this volume shall be double of that added to the sample solution (7.3).

The addition of 4 ml HCl (4.1) may help to prevent the hydrolysis of silicon and tin.

Dilute to the mark and mix well.

It is recommended to carry out the inductively coupled plasma optical emission spectrometric measurements on freshly prepared solutions, in order to avoid silicon hydrolysis.

In a fifth 100 ml one-mark volumetric flasks transfer 50,0 ml of the above 10 g/l iron solution. Dilute to the mark and mix well.

Use this solution to test whether or not the lowest concentration of each element is high enough to be considered as "different from the zero point".

Strong care should be taken in order to ensure that the quantity of acids in the calibration solutions (**particularly 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 remain the same.

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

## EN 10355:2013 (E)

The trueness of these calibration functions should be verified 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   |                               |                                 |   |
|--------------|---|-------------------------------|---------------------------------|---|
|              | F1  | F2                            | F3                              | F4  |
| Si<br>(4.16) | 0,1 mg [0,02 %] <sup>a</sup>                      | 0,25 mg [0,05 %] <sup>b</sup> | 2,25 mg [0,45 %] <sup>c</sup>   | 0,5 mg [0,1 %] <sup>d</sup>                       |
| Mn<br>(4.12) | 7 mg [1,4 %]<br>7 ml                              | 1 mg [0,2 %]<br>1 ml          | 0,025 mg [0,005 %] <sup>e</sup> | 0,1 mg [0,02 %] <sup>f</sup>                      |
| P<br>(4.15)  | 0,025 mg [0,005 %] <sup>g</sup>                   | 0,05 mg [0,01 %] <sup>h</sup> | 0,25 mg [0,05 %] <sup>i</sup>   | 0,5 mg [0,1 %] <sup>j</sup>                       |
| Cu<br>(4.11) | 0,25 mg [0,05 %] <sup>k</sup>                     | 3,0 mg [0,6 %]<br>3 ml        | 0,025 mg [0,005 %] <sup>l</sup> | 0,5 mg [0,1 %] <sup>m</sup>                       |
| Ni<br>(4.14) | 10 mg [2,0 %]<br>10 ml                            | 2,5 mg [0,5 %]<br>2,5 ml      | 0,05 mg [0,01 %] <sup>n</sup>   | 0,25 mg [0,05 %] <sup>o</sup>                     |
| Cr<br>(4.10) | 0,05 mg [0,01 %] <sup>p</sup>                     | 0,25 mg [0,05 %] <sup>q</sup> | 4,5 mg [0,9 %]<br>4,5 ml        | 2,5 mg [0,5 %]<br>2,5 ml                          |
| Mo<br>(4.13) | 0,05 mg [0,01 %] <sup>r</sup>                     | 0,25 mg [0,05 %] <sup>s</sup> | 3 mg [0,6 %]<br>3 ml            | 0,5 mg [0,1 %] <sup>t</sup>                       |
| Sn<br>(4.17) | 0,5 mg [0,1 %] <sup>u</sup>                       | 0,05 mg [0,01 %] <sup>v</sup> | 0,25 mg [0,05 %] <sup>w</sup>   | 0,1 mg [0,02 %] <sup>x</sup>                      |
| <sup>a</sup> | 1 ml of a 0,1 g/l silicon standard solution       |                               | <sup>m</sup>                    | 5,0 ml of a 0,1 g/l copper standard solution      |
| <sup>b</sup> | 2,5 ml of a 0,1 g/l silicon standard solution     |                               | <sup>n</sup>                    | 5,0 ml of a 0,01 g/l nickel standard solution     |
| <sup>c</sup> | 22,5 ml of a 0,1 g/l silicon standard solution    |                               | <sup>o</sup>                    | 2,5 ml of a 0,1 g/l nickel standard solution      |
| <sup>d</sup> | 5,0 ml of a 0,1 g/l silicon standard solution     |                               | <sup>p</sup>                    | 2,5 ml of a 0,01 g/l chromium standard solution   |
| <sup>e</sup> | 2,5 ml of a 0,01 g/l manganese standard solution  |                               | <sup>q</sup>                    | 2,5 ml of a 0,1 g/l chromium standard solution    |
| <sup>f</sup> | 10 ml of a 0,01 g/l manganese standard solution   |                               | <sup>r</sup>                    | 5,0 ml of a 0,01 g/l molybdenum standard solution |
| <sup>g</sup> | 2,5 ml of a 0,01 g/l phosphorus standard solution |                               | <sup>s</sup>                    | 2,5 ml of a 0,1 g/l molybdenum standard solution  |
| <sup>h</sup> | 5,0 ml of a 0,01 g/l phosphorus standard solution |                               | <sup>t</sup>                    | 5,0 ml of a 0,1 g/l molybdenum standard solution  |
| <sup>i</sup> | 2,5 ml of a 0,1 g/l phosphorus standard solution  |                               | <sup>u</sup>                    | 5,0 ml of a 0,1 g/l tin standard solution         |
| <sup>j</sup> | 5,0 ml of a 0,1 g/l phosphorus standard solution  |                               | <sup>v</sup>                    | 5,0 ml of a 0,01 g/l tin standard solution        |
| <sup>k</sup> | 2,5 ml of a 0,1 g/l copper standard solution      |                               | <sup>w</sup>                    | 2,5 ml of a 0,1 g/l tin standard solution         |
| <sup>l</sup> | 2,5 ml of a 0,01 g/l copper standard solution     |                               | <sup>x</sup>                    | 10,0 ml of a 0,01 g/l tin standard solution       |

## 8.3 Re-calibration

Drifts in the spectrometer readings shall be corrected by using the re-calibration procedure as described in the manufacturer's instruction manual.

For that purpose the synthetic multi-elemental solutions (8.2), used for the calibration or suitable Certified Reference Materials or Internal Reference Materials solutions may be used.

It is strongly recommended to carry out a re-calibration of the instrument before the analysis of each set of unknown samples.

## 9 Determination

### 9.1 Synoptic

The synoptic of the operations described in this paragraph is given in Annex B.

### 9.2 Adjustment of the spectrometer

Start the inductively coupled plasma optical emission spectrometer 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 3, 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.

**Table 3 — Wavelengths for determinations related with unalloyed and low alloyed steels**

| Element | Wavelength (nm)                  |
|---------|----------------------------------|
| Si      | 185,0 or 251,6 or 288,2          |
| Mn      | 257,6 or 293,1 or 403,08         |
| P       | 178,2 or 178,3                   |
| Cu      | 217,9 or 324,7 or 327,4          |
| Ni      | 222,5 or 230,3 or 231,6          |
| Cr      | 205,6 or 206,6 or 267,7 or 285,0 |
| Mo      | 202,0 or 204,05 or 281,6         |
| Sn      | 189,9 or 190,0                   |
| Sc      | 361,4                            |
| Y       | 371,0                            |

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.

Other wavelengths may be used, provided that interferences, sensitivity, resolution and linearity criteria have been carefully investigated.

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.

Each time the internal reference element is used, prepare the software to calculate the ratio between the intensity of each analyte and the intensity of the internal reference element.

### 9.3 Spectrometric measurements of the calibration solutions

Carry out the spectrometric measurements of the calibration solutions (8.2). A simultaneous spectrometer shall be available for measurements using the internal reference element (ratio mode).

For each calibration solution, three to five integrations are necessary in order to calculate the mean intensities or the mean ratioed intensities.