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**Steel and iron — Determination of nine  
elements by the inductively coupled  
plasma mass spectrometric method —**

Part 1:

**Determination of tin, antimony, cerium,  
lead and bismuth**

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avec plasma induit par haute fréquence —*

*Partie 1: Dosage de l'étain, de l'antimoine, du cérium, du plomb et du  
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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 16918-1 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

ISO 16918 consists of the following parts, under the general title *Steel and iron — Determination of nine elements by the inductively coupled plasma mass spectrometric method*:

— Part 1: *Determination of tin, antimony, cerium, lead and bismuth*

— Part 2: *Determination of boron, silver, indium and thallium*

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# Steel and iron — Determination of nine elements by the inductively coupled plasma mass spectrometric method —

## Part 1: Determination of tin, antimony, cerium, lead and bismuth

### 1 Scope

This part of ISO 16918 specifies a method for analysing steel and iron for the trace element determinations of Sn, Sb, Ce, Pb and Bi using inductively coupled plasma mass spectrometry (ICP-MS). The method is applicable for trace elements in the mass fraction ranges ( $\mu\text{g/g}$ ) as follows:

Sn: 5  $\mu\text{g/g}$  to 200  $\mu\text{g/g}$ ; Sb: 1  $\mu\text{g/g}$  to 200  $\mu\text{g/g}$ ; Ce: 10  $\mu\text{g/g}$  to 1 000  $\mu\text{g/g}$ ; Pb: 0,5  $\mu\text{g/g}$  to 100  $\mu\text{g/g}$ ; Bi: from 0,3  $\mu\text{g/g}$  to 30  $\mu\text{g/g}$ .

Interferences in the determination of trace elements using ICP-MS are listed in Annex B.

### 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.

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

### 3 Principle

A test portion is dissolved in an acid-mixture of hydrochloric acid, nitric acid and hydrofluoric acid using either a microwave-assisted system or a traditional hot plate.

Diluted wet-digested samples are introduced into an inductively coupled plasma mass spectrometer (ICP-MS), via a peristaltic pump. Simultaneous measurements of the intensities of elements with atomic mass units of concern (mass spectra) are carried out using ICP-MS techniques.

Calibration blank and calibration solutions are matrix-matched with the major elements of steel, and mineral acids are used for wet-digestion.

Internal standards are used throughout in order to compensate for any instrument drift.

## 4 Reagents

During the analysis, unless other stated, use only reagents of high purity quality containing less than 0,000 1 % mass fraction of each element or equivalent purity. The % given below refers to % mass fraction.

- 4.1 **Hydrochloric acid**, 30 % HCl,  $\rho$  1,15 g/ml or 38 %,  $\rho$  1,19 g/ml.
- 4.2 **Nitric acid**, 70 % HNO<sub>3</sub>,  $\rho$  1,42 g/ml.
- 4.3 **Hydrofluoric acid**, 49 % HF,  $\rho$  1,16 g/ml.
- 4.4 **Nitric acid**, 65 % HNO<sub>3</sub>,  $\rho$  1,40 g/ml.
- 4.5 **Ultra-pure water**, produced by a water purification system giving a resistivity of 18 M $\Omega$ /cm or higher.
- 4.6 **Washing solution for ICP-MS.**

In a 500 ml plastic bottle (e.g. polyethylene) pour about 400 ml of ultra-pure water (4.5), then add 15 ml hydrochloric acid (4.1), 5 ml nitric acid (4.2) and 2,5 ml hydrofluoric acid (4.3) and make it up to volume with ultra-pure water (4.5).

The quality of the acids can be checked prior to use by a mass spectrum scan with the ICPMS instrument. It is recommended to use a solution of 300  $\mu$ l HCl (4.1) + 100  $\mu$ l HNO<sub>3</sub> (4.2) + 50  $\mu$ l HF (4.3) with about 3 ml ultra-pure water (4.5) and make it up to a volume of 10 ml. If peaks of elements of concern are present, a new flask of acid shall be used and a new check of the same elements shall be carried out.

- 4.7 **10 % nitric acid solution**, HNO<sub>3</sub> diluted 1+9.

In a 100 ml volumetric flask pour about 70 ml of ultra-pure water (4.5), then add 10 ml concentrated HNO<sub>3</sub> (4.2), and dilute to volume with ultra-pure water (4.5).

- 4.8 **NaOH solution**, 7,5 mol/l,  $\rho$  1,33 g/ml.

- 4.9 **NaOH solution**, 0,2 mol/l.

Dispense 2,7 ml of 7,5 mol/l NaOH (4.8) into a 100 ml volumetric flask, and dilute to volume with ultra-pure water (4.5).

The solution shall be stored in a polyethylene bottle or similar.

- 4.10 **Aqua regia (HCl+HNO<sub>3</sub> = 3+1).**

Prepare aqua regia in a 30 ml beaker (or similar) by dispensing 9 ml HCl (4.1) and 3 ml HNO<sub>3</sub> (4.2) into the beaker.

- 4.11 **Diluted aqua regia solution**, diluted 4+10.

Dispense 100 ml ultra-pure water (4.5) into a 150 ml flask. Then add 40 ml aqua regia (4.10) and mix. Do not make the solution up to volume.

- 4.12 **50 % nitric acid solution**, HNO<sub>3</sub> diluted 1+1.

In a 100 ml volumetric flask, pour about 30 ml of ultra-pure water (4.5), then add 50 ml concentrated HNO<sub>3</sub> (4.2) and dilute to volume with ultra-pure water (4.5).

- 4.13 **Perchloric acid**, 70 % HClO<sub>4</sub>,  $\rho$  1,68 g/ml.

**4.14 50 % hydrochloric acid solution**, HCl diluted 1+1.

In a 100 ml volumetric flask, pour about 30 ml of ultra-pure water (4.5), then add 50 ml concentrated HCl (4.1) and dilute to volume with ultra-pure water (4.5).

**4.15 Iron**, high purity quality containing less than 0,000 1 % mass fraction of each element.**4.16 Standard stock solutions**, corresponding to 1 000 mg element per litre.**4.16.1 Tin standard stock solution**

Dissolve 100,0 mg of high purity tin metal (99,9 % mass fraction, minimum) in 3 ml HCl (4.1) and 1 ml HNO<sub>3</sub> (4.2) in a 250 ml beaker. Heat gently to complete dissolution, cool, transfer into a 100 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the tin standard stock solution in a polyethylene bottle.

**4.16.2 Antimony standard stock solution**

Dissolve 100,0 mg of high purity antimony metal (99,9 % mass fraction, minimum) in 3 ml HCl (4.1) and 1 ml HNO<sub>3</sub> (4.2) in a 250 ml beaker. Heat gently to complete dissolution, cool, transfer into a 100 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the antimony standard stock solution in a polyethylene bottle.

**4.16.3 Cerium standard stock solution**

Dissolve 288,5 mg of pure cerium(IV) sulfate, Ce(SO<sub>4</sub>)<sub>2</sub> · 4H<sub>2</sub>O, in 50 ml of a solution of diluted aqua regia (4.11) in a 100 ml volumetric flask. After complete dissolution, make the solution up to volume with diluted aqua regia (4.11) and mix well.

Store the cerium standard stock solution in a polyethylene bottle.

**4.16.4 Lead standard stock solution**

Dissolve 100,0 mg of high purity lead metal (99,9 % mass fraction, minimum) in 20 ml of 50 % HNO<sub>3</sub> (4.12) in a 250 ml beaker. Heat gently to complete dissolution, cool, transfer into a 100 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the lead standard stock solution in a polyethylene bottle.

**4.16.5 Bismuth standard stock solution**

Dissolve 100,0 mg of high purity bismuth metal (99,9 % mass fraction, minimum) in 3 ml HCl (4.1) and 1 ml HNO<sub>3</sub> (4.2) in a 250 ml beaker. Heat gently to complete dissolution, cool, transfer into a 100 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the bismuth standard stock solution in a polyethylene bottle.

**4.16.6 Rhodium standard stock solution**

Dissolve 203,6 mg of pure rhodium(III) chloride, RhCl<sub>3</sub>, in 6 ml aqua regia (4.10), freshly prepared, in a 100 ml volumetric flask. After complete dissolution, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the rhodium standard stock solution in a polyethylene bottle.

#### 4.16.7 Yttrium standard stock solution

Dissolve 127,0 mg of pure yttrium trioxide,  $Y_2O_3$ , in 6 ml aqua regia (4.10), freshly prepared, in a 100 volumetric flask. After complete dissolution, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the yttrium standard stock solution in a polyethylene bottle.

#### 4.16.8 Lutetium standard stock solution

Dissolve 113,7 mg of pure lutetium trioxide,  $Lu_2O_3$ , in 6 ml aqua regia (4.10), freshly prepared, in a 100 ml volumetric flask. After complete dissolution, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the lutetium standard stock solution in a polyethylene bottle.

#### 4.16.9 Titanium standard stock solution

Dissolve 100,0 mg of pure titanium metal (99,9 % mass fraction, minimum) in 30 ml of 50 % HCl (4.14) and 0,2 ml of HF (4.3) in a 250 ml beaker. Heat gently to complete dissolution, cool, transfer into a 100 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the titanium standard stock solution in a polyethylene bottle.

#### 4.17 Iron matrix solution, 10 000 mg of Fe per litre

Weigh 0,5 g of the high purity iron (4.15) to the nearest 0,01 mg and transfer it to a 250 ml beaker. Add 20 ml ultra-pure water, and then 0,1 ml HCl (4.1) and finally 5 ml  $HNO_3$  (4.2). Heat gently to dissolve the iron chips. After complete dissolution, cool, transfer into a 50 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

Store the iron matrix solution in a polyethylene bottle.

#### 4.18 Mass calibration solution, 100 µg/l of each of the elements Ti, Y, Rh, Sb, Ce, Pb and Bi

Dispense about 50 ml ultra-pure water (4.5) into a 1 000 ml volumetric flask, and then add 100 µl of each of the standard stock solutions of Ti (4.16.9), Y (4.16.7), Rh (4.16.6), Sb (4.16.2), Ce (4.16.3), Pb (4.16.4) and Bi (4.16.5). Make the solution up to volume with ultra-pure water (4.5) and mix well.

## 5 Apparatus

**5.1 Laboratory glassware and plastic ware**, including volumetric flasks, watch-glasses, beakers, polyethylene bottles, polyethylene pipette tips, polystyrene tubes.

All volumetric glassware shall be Class A in accordance with ISO 648 and ISO 1042.

#### 5.2 Micro-pipettes

The following micro-pipettes are used: 5 µl to 40 µl, 50 µl to 200 µl, 100 µl to 1 000 µl and 1 ml to 5 ml.

**5.3 Microwave-assisted digestion system**, consisting of a laboratory microwave oven and a carousel or holder for polytetrafluoroethylene (PTFE) pressure vessels.

A time-step programme can be used, and during the wet-digestion procedure both pressure and temperature are registered and can be followed on a monitor.



## 5.4 ICP-MS instruments

### 5.4.1 Magnetic sector ICP-MS (high resolution ICP-MS)

### 5.4.2 Quadrupole ICP-MS (low resolution ICP-MS)

### 5.4.3 Time-of-flight ICP-MS (ICP-TOF-MS)

For optimum running of the ICP-MS instruments, the manual of each ICP-MS type shall be followed.

All three types of ICP-MS instrument are supplied with argon gas in order to supply an argon plasma. Prior to analysis, the argon plasma is switched on and shall remain on for 30 min to 60 min to stabilize the instrument. Meanwhile, ultra-pure water or washing solution should be pumped through the nebulizer and torch system. The warm-up period depends on the type of ICP-MS instrument used.

Mass calibration should be performed every morning before starting analysis; seven elements should be chosen in order to cover the periodic table of concern [Ti, Y, Rh, Sb, Ce, Pb and Bi (see 4.18)]. Other elements can be used in a mass calibration solution, but they shall cover the atomic mass unit range which will be used in the analysis.

Usually an auto-sampler device is connected to a peristaltic pump to automatically introduce samples into the plasma. Manual introduction can also be used.

The instruments are conditioned by optimising the sensitivity. It is then very important to set the operational parameters such as frequency, output power, plasma gas flow, auxiliary gas flow, nebulizer gas flow, sample uptake rate, detection mode, integration time/peak, number of points/peak, number of replicates and washing time. In practice, the sensitivity is optimised by introducing a calibration solution (e.g. a 100 µg/l rhodium calibration solution or any other suitable calibration solution) into the plasma and then adjusting the operational parameters.

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## 6 Measurement specifications

### 6.1 Minimum precision (RSD)

Calculate the standard deviation of 10 measurements of a 10 µg/l element concentration of each element, in a matrix-matched sample solution. The minimum precision (RSD) shall not exceed 5 %.

### 6.2 Limit of detection (LOD) and limit of quantification (LOQ)

Limit of detection (LOD) and limit of quantification (LOQ) are defined by the following equations respectively.

$$\text{LOD} = 3 \times \sigma \times \frac{C_s}{X_s - X_b}$$

$$\text{LOQ} = 10 \times \sigma \times \frac{C_s}{X_s - X_b}$$

where

$\sigma$  is the standard deviation of intensity for the blank solution at 10 measurements;

$X_s$  is the mean intensity for the standard solution at 10 measurements;

$X_b$  is the mean intensity for the blank solution at 10 measurements;

$C_s$  is the concentration of the standard solution, in µg/l.

## 7 Sampling

Sampling is carried out in accordance with ISO 14284 or appropriate national standards for steel.

## 8 Washing

All glassware and plastic materials are soaked in nitric acid (4.4) for at least 12 h and subsequently rinsed with ultra-pure water (4.5). The labware shall then be stored in a dust-free place.

## 9 Procedure

### 9.1 Test portion

Weigh 100 mg, to the nearest 0,01 mg, of a test portion (sample) to be analysed.

NOTE This International Standard specifies the procedure for which the mass of test portion is 100 mg, but less than 100 mg of the test portion, e.g. 10 mg, can be chosen.

### 9.2 Blank test solution [blank sample solution]

In parallel with the determination of unknown samples, a blank test solution shall be analysed. The blank test solution shall contain the same quantities of reagents as used for analysing unknown steel samples, plus the same mass of high purity iron (4.15) as the test portion.

### 9.3 Preparation of the test solution

#### 9.3.1 Test solution for the elements Sn, Sb, Pb and Bi

##### 9.3.1.1 Microwave-assisted digestion method

The test portion is quantitatively transferred to a PTFE pressure vessel (about 120 ml) and 3 ml HCl (4.1), 1 ml HNO<sub>3</sub> (4.2) and 0,5 ml HF (4.3) are added. The lid of the vessel is screwed tight. However, the acids can be added to the vessel and they can remain overnight in the vessel with the lid loosely tightened. This usually improves the wet-digestion procedure.

The wet digestion takes place in a microwave-assisted digestion system. The PTFE pressure vessels are placed in a carousel or a special holder, which is put in a laboratory microwave oven, and the wet-digestion is carried out by means of microwaves. The wet digestion is carried out according to a three-step procedure, i.e. starting at a low temperature of about 50 °C for 10 min, then raising the temperature to about 100 °C for 10 min, and finally raising the temperature to 150 °C to 200 °C for 10 min.

The three-step procedure can be carried out simply by regulating the power of the microwave oven. Thus the microwave-assisted digestion takes place for 30 min, and for a further 30 min the PTFE pressure vessels shall cool before being taken out of the microwave oven. The temperature in the PTFE pressure vessels shall be less than 50 °C before they are opened. Plastic gloves shall be worn when opening the PTFE pressure vessels.

**WARNING — Do not open the door of the microwave oven immediately after the end of the programme, since there is always a risk that the security membrane of the PTFE pressure vessels can rupture and blow out hot acids.**

After cooling, the contents of the PTFE pressure vessels are transferred quantitatively to a 100 ml polyethylene bottle or a 100 ml volumetric flask by carefully rinsing the PTFE pressure vessels with ultra-pure water (4.5), making the bottles or flasks up to volume with ultra-pure water (4.5) and mixing well.

### 9.3.1.2 Digestion using open vessels on a hot plate

Place the test portion in a 50 ml PTFE beaker or quartz beaker with graphite bottom. Add 3 ml HCl (4.1), cover the beaker with a watch-glass, and heat gently until solvent reaction ceases. Add 1 ml HNO<sub>3</sub> (4.2) and heat until fumes of nitrogen oxides have disappeared. Add 0,5 ml HF (4.3) and heat for 5 min. If necessary, cool and add 5 ml of HClO<sub>4</sub> (4.13) and heat strongly without a watch-glass until fuming commences.

Cover with a watch-glass and continue heating at a temperature at which a steady reflux of white perchloric acid fumes is maintained on the walls of the beaker. Continue heating until there are no perchloric acid fumes visible inside the beaker. Cool and transfer the solution quantitatively to a 100 ml volumetric flask by rinsing the beaker with ultra-pure water (4.5). Make the solution up to volume with ultra-pure water (4.5) and mix well.

**CAUTION — PTFE beakers with graphite bottoms can easily be destroyed by elevated temperatures, and consequently the temperature must be increased very slowly.**

### 9.3.2 Test solution for the element Ce

#### 9.3.2.1 Microwave-assisted digestion method

The test portion is quantitatively transferred to a PTFE pressure vessel (about 120 ml), and 3 ml HCl (4.1) and 1 ml HNO<sub>3</sub> (4.2) are added. The lid of the vessel is screwed tight. However, the acids can be added to the vessel and they can remain overnight in the vessel with the lid loosely tightened. This usually improves the wet-digestion procedure.

The wet digestion takes place in a microwave-assisted digestion system. The PTFE pressure vessels are placed in a carousel or a special holder, which is put in a laboratory microwave oven, and the wet-digestion is carried out by means of microwaves. The wet digestion is carried out according to a three-step procedure, i.e. starting at a low temperature of about 50 °C for 10 min, then raising the temperature to about 100 °C for 10 min, and finally raising the temperature to 150 °C to 200 °C for 10 min.

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The three-step procedure can be carried out simply by regulating the power of the microwave oven. Thus the microwave-assisted digestion takes place over 30 min, and for a further 30 min the PTFE pressure vessels shall cool before being taken out of the microwave oven. The temperature in the PTFE pressure vessels shall be less than 50 °C before they are opened. Plastic gloves shall be worn when opening the PTFE pressure vessels.

**WARNING — Do not open the door of the microwave oven immediately after the end of the programme, since there is always a risk that the security membrane of the PTFE pressure vessels can rupture and blow out hot acids.**

After cooling, the contents of the PTFE pressure vessels are transferred quantitatively to a 100 ml polyethylene bottle or a 100 ml volumetric flask by carefully rinsing the PTFE pressure vessels with ultra-pure water (4.5), making up to volume with ultra-pure water (4.5) and mixing well.

#### 9.3.2.2 Digestion using open vessels on a hot plate

Place the test portion in a 100 ml glass beaker or quartz beaker. Add 3 ml HCl (4.1), cover with a watch-glass and heat gently until solvent reaction ceases. Add 1 ml HNO<sub>3</sub> (4.2) and heat until fumes of nitrogen oxides have disappeared. If necessary, cool and add 5 ml of HClO<sub>4</sub> (4.13) and heat strongly without a watch-glass until fuming commences.

Cover with a watch-glass and continue heating at a temperature at which a steady reflux of white perchloric acid fumes is maintained on the walls of the beaker. Continue heating until there are no perchloric acid fumes visible inside the beaker. Cool and transfer the solution quantitatively to a 100 ml volumetric flask by rinsing the beaker with ultra-pure water (4.5), making the solution up to volume with ultra-pure water (4.5) and mixing well.

**10 Standard solutions**

Three standard solutions are prepared and used for further preparations of calibration solutions.

**10.1 Multi-element standard solutions of the elements Sn, Sb, Pb and Bi**

Multi-element standards of the four elements given above are prepared, starting with the standard stock solution of each element (4.16.1, 4.16.2, 4.16.4, 4.16.5).

**10.1.1 Preparation in polystyrene test tubes**

Preparation of standard solutions directly in 10 ml polystyrene test tubes is convenient and time-saving.

The solutions are made up to volume with ultra-pure water (4.5). The preparation of the two standard solutions is described in 10.1.1.1 and 10.1.1.2.

**10.1.1.1 Preparation of multi-standard solution — Multi-standard<sub>10</sub>: 10 mg/l**

From each of the four standard stock solutions (4.16.1, 4.16.2, 4.16.4, 4.16.5) take 100 µl and add to a 10 ml polystyrene test tube containing about 5 ml of ultra-pure water (4.5). Make up the multi-element solution to volume with ultra-pure water (4.5) by weighing<sup>1)</sup>. Seal the test tube with parafilm and mix the standard solution. See Table 1.

**Table 1 — Multi-standard solution<sub>10</sub>**  
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Volume of each standard stock solution – Standard <sub>1 000</sub>	Mass	Test tube volume	Concentration of each element in test tube after dilution
µl	µg	ml	mg/l
100	100	10	10

**10.1.1.2 Preparation of multi-standard solution — Multi-standard<sub>0,1</sub>: 0,1 mg/l**

Dispense 100 µl of multi-standard<sub>10</sub> into a 10 ml polystyrene test tube and make the solution up to volume with ultra-pure water (4.5) by weighing. Seal the test tube with parafilm and mix the standard solution. See Table 2.

**Table 2 — Multi-standard solution<sub>0,1</sub>**

Volume of multi-standard <sub>10</sub>	Mass	Test tube volume	Concentration of each element in test tube after dilution
µl	µg	ml	mg/l
100	1,0	10	0,10

**10.1.2 Preparation in volumetric flasks**

Preparation of standard solutions can be done in a 100 ml volumetric flask. All sample solutions are made up to volume with ultra-pure water (4.5). The multi-standard solutions are prepared according to 10.1.2.1 to 10.1.2.2.

1) Measuring net mass of solution in the test tube.