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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**

*Acier et fer — Dosage de neuf éléments par spectrométrie de masse  
avec plasma induit par haute fréquence —*

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



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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*

# 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.

## 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.

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>

| 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.

**10.1.2.1 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) 1,0 ml is taken and added to a 100 ml volumetric flask containing about 50 ml of ultra-pure water (4.5). The multi-element solution is made up to volume with ultra-pure water (4.5). The standard solution is mixed and stored in the volumetric flask.

**Table 3 — Multi-standard solution<sub>10</sub>**

| Volume of each standard stock solution – Standard <sub>1 000</sub><br>ml | Mass<br>µg | Volume of volumetric flask<br>ml | Concentration of each element in volumetric flask after dilution<br>mg/l |
|--|------------|----------------------------------|--|
| 1,0  | 1 000      | 100                              | 10   |

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

Dispense 1,0 ml of multi-standard<sub>10</sub> into a 100 ml volumetric flask, make the solution up to volume with ultra-pure water (4.5) and mix well.

**Table 4 — Multi-standard solution<sub>0,1</sub>**

| Volume of multi-standard <sub>10</sub><br>ml | Mass<br>µg | Volume of volumetric flask<br>ml | Concentration. of each element in the volumetric flask after dilution<br>mg/l |
|--|------------|----------------------------------|---|
| 1,0  | 10         | 100                              | 0,10  |

**10.2 Standard solutions of the element Ce**

The element Ce must be determined separately, since there is a risk of precipitation of CeF<sub>3</sub> if hydrofluoric acid is used. The preparation of the standard solution Ce-Standard<sub>10</sub> should be a straightforward procedure following the dilution scheme starting with 10.1.1.1 and 10.1.2.1, respectively. Start with the cerium standard stock solution (4.16.3).

**11 Preparation of internal standard solutions (“internal standards”) — Y, Rh and Lu****11.1 Preparation in polystyrene test tubes**

It is mandatory to use internal standards when analysing several samples, since there is always instrumental drift due to the heavy matrix in wet-digested steel samples.

Internal standards of the elements Rh, Y and Lu are prepared in three separate polystyrene test tubes, respectively. About 3 ml of ultra-pure water (4.5) is dispensed into each of three 10 ml polystyrene test tubes, and to the first test tube is added 100 µl of the standard stock solution of Rh (4.16.6), to the second test tube is added 100 µl of the standard stock solution of Y (4.16.7) and to the third test tube is added 100 µl of the standard stock solution of Lu (4.16.8). All three test tubes are made up to volume with ultra-pure water (4.5) by weighing, and the test tubes are then sealed with parafilm and mixed. The concentration of the internal standard in each of the polystyrene tubes is 10 mg/l. Further dilution will be necessary, following the procedure for standard solutions (for Rh and Y 10.1.1.1 and 10.1.1.2, and for Lu 10.2.) and for calibration solutions (for Rh and Y 12.1.1 and 12.2.1, and for Lu 12.1.2 and 12.2.2).

The concentration of the internal standard should be approximately the same as the element concentration to be determined. This can be a problem when multi-element determinations are carried out. The deviation in concentrations between internal standard and the element to be determined shall not exceed two orders of magnitude. In such cases, different concentrations can be used for Y and Rh. If this is not possible, the elements to be determined shall be split into two or more groups before analysis.

Since many of the concentrations of the elements are not known prior to analysis, a pre-analysis should be done to determine the concentration level of the elements of concern. In several steel materials, the concentrations will be too low to be determined. Then it can be suitable to choose a concentration of the internal standard of 1 µg/l.

## 11.2 Preparation in volumetric flasks

It is mandatory to use internal standards when analysing several samples since there is always an instrumental drift due to the heavy matrix in the wet-digested steel samples.

The internal standards of the element Y, Rh and Lu are prepared in three separate 100 ml volumetric flasks, respectively. About 50 ml ultra-pure water (4.5) is dispensed into each of three volumetric flasks, and to the first volumetric flask is added 1,0 ml of the standard stock solution of Rh (4.16.6), to the second volumetric flask is added 1,0 ml of the standard stock solution of Y (4.16.7) and to the third volumetric flask is added 1,0 ml of the standard stock solution of Lu (4.16.8). All three volumetric flasks are made up to volume with ultra-pure water (4.5) and mixed. The concentration of the internal standard in each of the volumetric flasks is 10 mg/l. Further dilution will be necessary. Then the dilution manner follows the scheme for standard solutions (for Y and Rh 10.1.2.1 to 10.1.2.2 and for Lu 10.2) and for calibration solutions (for Y and Rh 12.1.1 and 12.2.1 and for Lu 12.1.2 and 12.2.2).

The concentration of the internal standard should approximately be the same as the element concentration to be determined. This can be a problem when multi-element determinations are carried out. However, common sense must be used, and the deviation in concentration between internal standard and the element to be determined may not exceed two orders of magnitude. In such cases different concentrations can be used for Y and Rh. If this is not possible, the elements to be determined must be split up in two or more groups before analysis.

Since many of the concentrations of the elements are not known prior to analysis, a pre-analysis should be done in order to find out the concentration level of the elements of concern. In several steel materials it will be found that the concentrations are too low to be determined. Then it can be suitable to choose an internal standard concentration of 1 µg/l.

## 12 Calibration blank solution and calibration solutions

Calibration blank and calibration solutions for the elements Sn, Sb and Pb are prepared covering the concentration range 0,4 µg/l to 200 µg/l according to the procedures in 12.1 and 12.2. Calibration solutions for the element Ce shall cover the concentration range 5 µg/l to 1 000 µg/l, and for the element Bi the calibration range shall cover the concentration range 0,3 µg/l to 40 µg/l. A calibration blank and at least five calibration solutions shall be prepared to establish a calibration graph, and the calibration solutions shall cover the concentration range of the unknown samples. All calibration solutions shall be matched with the element Fe using the 10 000 mg/l solution (see 4.17), and acids used for wet digestion giving the same concentrations as can be expected in the steel samples. Finally internal standards are added: Y and Rh for the multi-element calibration blank (for the elements Sn, Sb, Pb and Bi) as well as for the five multi-element calibration solutions (for the elements Sn, Sb, Pb and Bi), and Lu for the Ce-calibration blank as well as for the five Ce-calibration solutions.

### 12.1 Preparation in volumetric flasks

At least six 100 ml volumetric flasks are prepared, which are then used for preparation of a calibration blank solution and calibration solutions for establishment of a calibration graph. The concentrations of the calibration solutions shall be chosen in the same concentration range as for the samples, covering the range evenly.

Preparation of calibration solutions in volumetric flasks can be done according to the procedures in 12.1.1 and 12.1.2. All calibration solutions are made up to volume with ultra-pure water (4.5).

### 12.1.1 Preparation of multi-element calibration blank solution and multi-element calibration solutions — Sn, Sb, Pb and Bi

To establish a calibration graph, a calibration blank solution and at least five calibration solutions are needed.

Thus dispense about 50 ml ultra-pure water (4.5) into six 100 ml volumetric flasks, and then prepare the solutions according to the scheme below by adding Fe-matrix solution (see 4.17), mineral acids and multi-standard solutions. Internal standard solutions (Y and Rh) shall be added corresponding to the expected concentrations of the multi-elements in the test solution. After that, make the solutions up to volume (see Table 5).

### 12.1.2 Preparation of Ce-calibration blank solution and Ce-calibration solutions

To establish a calibration graph, a calibration blank and at least five calibration solutions are needed.

Thus dispense about 50 ml ultra-pure water (4.5) into six 100 ml volumetric flasks, and then prepare the solutions according to the scheme below by adding Fe-matrix solution (see 4.17), mineral acids and Ce-standard solutions. Internal standard solution (Lu) shall also be added corresponding to the expected concentration of Ce in the test solution. After that, make the solutions up to volume (see Table 6).

NOTE HF shall not be added.

## 12.2 Preparation in polystyrene test tubes

At least six 10 ml polystyrene test tubes are prepared, which are then used for preparation of a calibration blank solution and calibration solutions for establishment of a calibration graph. The concentrations of the calibration solutions have to be chosen in the same concentration range as for the samples.

Preparation of calibration solutions in polystyrene test tubes can be done according to 12.2.1 and 12.2.2. All calibration solutions are made up to volume with ultra-pure water (4.5) by weighing. The test tubes are sealed with parafilm and then mixed.

### 12.2.1 Preparation of multi-element calibration blank and multi-element calibration solutions using Sn, Sb, Pb and Bi

To establish a calibration graph, a calibration blank solution and at least five calibration solutions are needed.

Dispense into six polystyrene test tubes about 3 ml ultra-pure water (4.5), and then prepare the solutions according to the procedure below by adding Fe-matrix solution (see 4.17), mineral acids and multi-standard solutions. Internal standard solutions (Y and Rh) shall be added to give concentrations corresponding to multi-element concentrations in the test solution. Then make the solutions up to volume with ultra-pure water (4.5) by weighing, seal the test tubes with parafilm and mix (see Table 7).

### 12.2.2 Preparation of Ce-calibration blank solution and Ce-calibration solutions

To establish a calibration graph, a calibration blank solution and at least five calibration solutions are needed.

Dispense into six polystyrene test tubes about 3 ml ultra-pure water (4.5), and then prepare the solutions according to the procedure below by adding Fe-matrix solution (see 4.17), mineral acids and Ce-standard solutions. Internal standard solution (Lu) shall be added corresponding to the concentration Ce in the test solution. Then make the solutions up to volume with ultra-pure water (4.5) by weighing, seal the test tubes with parafilm and mix (see Table 8).

HF shall not be added.

Table 5 — Preparation of multi-element calibration blank solution and multi-element calibration solutions – Sn, Sb, Pb and Bi

| Addition of Fe matrix solution <sup>b</sup><br>10 000 mg/l<br>ml | Addition of HCl<br>(4.1)<br>ml | Addition of HNO <sub>3</sub><br>(4.2)<br>ml | Addition of HF<br>(4.3)<br>ml | Volume of standard stock solutions <sup>a</sup><br>1 000 mg/l<br>µl | Volume of multi-standard <sub>10</sub><br>µl | Volume of multi-standard <sub>0,1</sub><br>µl | Concentration of multi-elements in calibration solution<br>µg/l | Mass of multi-elements<br>µg |
|--|--------------------------------|---|-------------------------------|---|--|---|---|------------------------------|
| 10   | 3                              | 1   | 0,5                           | 0   | 0  | 0   | 0   | 0                            |
| 10   | 3                              | 1   | 0,5                           | 50  | 0  | 0   | 500   | 50                           |
| 10   | 3                              | 1   | 0,5                           | 20  | 0  | 0   | 200   | 20                           |
| 10   | 3                              | 1   | 0,5                           | 0   | 1 000  | 0   | 100   | 10                           |
| 10   | 3                              | 1   | 0,5                           | 0   | 500  | 0   | 50  | 5                            |
| 10   | 3                              | 1   | 0,5                           | 0   | 200  | 0   | 20  | 2                            |
| 10   | 3                              | 1   | 0,5                           | 0   | 100  | 0   | 10  | 1                            |
| 10   | 3                              | 1   | 0,5                           | 0   | 50   | 0   | 5   | 0,5                          |
| 10   | 3                              | 1   | 0,5                           | 0   | 20   | 0   | 2   | 0,2                          |
| 10   | 3                              | 1   | 0,5                           | 0   | 0  | 1 000   | 1   | 0,1                          |
| 10   | 3                              | 1   | 0,5                           | 0   | 0  | 500   | 0,5   | 0,05                         |
| 10   | 3                              | 1   | 0,5                           | 0   | 0  | 200   | 0,2   | 0,02                         |

<sup>a</sup> There are four separate standard stock solutions for the elements Sn (4.16.1), Sb (4.16.2), Pb (4.16.4) and Bi (4.16.5).

<sup>b</sup> See 4.17.

Table 6 — Preparation of Ce-calibration blank solution and Ce-calibration solutions

| Addition of Fe matrix solution <sup>a</sup><br>10 000 mg/l<br>ml | Addition of HCl<br>(4.1)<br>ml | Addition of HNO <sub>3</sub><br>(4.2)<br>ml | Volume of standard stock solution Ce<br>(4.16.3)<br>µl | Volume of Ce-standard <sub>10</sub><br>µl | Concentration of Ce in calibration solution<br>µg/l | Mass of Ce<br>µg |
|--|--------------------------------|---|--|---|---|------------------|
| 10   | 3                              | 1   | 0  | 0   | 0   | 0                |
| 10   | 3                              | 1   | 200  | 0   | 2 000   | 200              |
| 10   | 3                              | 1   | 100  | 0   | 1 000   | 100              |
| 10   | 3                              | 1   | 50   | 0   | 500   | 50               |
| 10   | 3                              | 1   | 20   | 0   | 200   | 20               |
| 10   | 3                              | 1   | 0  | 1 000                                     | 100   | 10               |
| 10   | 3                              | 1   | 0  | 500                                       | 50  | 5                |
| 10   | 3                              | 1   | 0  | 200                                       | 20  | 2                |
| 10   | 3                              | 1   | 0  | 100                                       | 10  | 1                |
| 10   | 3                              | 1   | 0  | 50  | 5   | 0,5              |
| 10   | 3                              | 1   | 0  | 20  | 2   | 0,2              |

<sup>a</sup> See 4.17.



**Table 7 — Preparation of multi-element calibration blank and multi-element calibration solutions — Sn, Sb, Pb and Bi**

| Addition of Fe matrix solution <sup>a</sup><br>10 000 mg/l | Addition of HCl<br>(4.1) | Addition of HNO <sub>3</sub><br>(4.2) | Addition of HF<br>(4.3) | Volume of Multi-standard <sub>10</sub> | Volume of Multi-standard <sub>0,1</sub> | Concentration of multi-elements in calibration solution | Mass of multi-elements |
|--|--------------------------|---------------------------------------|-------------------------|--|---|---|------------------------|
| ml   | μl                       | μl                                    | μl                      | μl                                     | μl                                      | μg/l  | μg                     |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 0                                       | 0   | 0                      |
| 1,0  | 300                      | 100                                   | 50                      | 500                                    | 0                                       | 500   | 5,0                    |
| 1,0  | 300                      | 100                                   | 50                      | 200                                    | 0                                       | 200   | 2,0                    |
| 1,0  | 300                      | 100                                   | 50                      | 100                                    | 0                                       | 100   | 1,0                    |
| 1,0  | 300                      | 100                                   | 50                      | 50                                     | 0                                       | 50  | 0,5                    |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 2 000                                   | 20  | 0,2                    |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 1 000                                   | 10  | 0,1                    |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 500                                     | 5   | 0,05                   |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 200                                     | 2   | 0,02                   |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 100                                     | 1   | 0,01                   |
| 1,0  | 300                      | 100                                   | 50                      | 0                                      | 50                                      | 0,5   | 0,005                  |

<sup>a</sup> See 4.17.

**Table 8 — Preparation of Ce-calibration blank solution and Ce-calibration solutions**

| Addition of Fe matrix solution <sup>a</sup><br>10 000 mg/l | Addition of HCl<br>(4.1) | Addition of HNO <sub>3</sub><br>(4.2) | Volume of Ce-standard <sub>10</sub> | Volume of Ce-standard <sub>0,1</sub> | Concentration of Ce in calibration solution | Mass of Ce |
|--|--------------------------|---------------------------------------|-------------------------------------|--------------------------------------|---|------------|
| ml   | μl                       | μl                                    | μl                                  | μl                                   | μg/l  | μg         |
| 1,0  | 300                      | 100                                   | 0                                   | 0                                    | 0   | 0          |
| 1,0  | 300                      | 100                                   | 2 000                               | 0                                    | 2 000                                       | 20,0       |
| 1,0  | 300                      | 100                                   | 1 000                               | 0                                    | 1 000                                       | 10,0       |
| 1,0  | 300                      | 100                                   | 500                                 | 0                                    | 500   | 5,0        |
| 1,0  | 300                      | 100                                   | 200                                 | 0                                    | 200   | 2,0        |
| 1,0  | 300                      | 100                                   | 100                                 | 0                                    | 100   | 1,0        |
| 1,0  | 300                      | 100                                   | 50                                  | 0                                    | 50  | 0,5        |
| 1,0  | 300                      | 100                                   | 0                                   | 2 000                                | 20  | 0,2        |
| 1,0  | 300                      | 100                                   | 0                                   | 1 000                                | 10  | 0,1        |
| 1,0  | 300                      | 100                                   | 0                                   | 500                                  | 5   | 0,05       |
| 1,0  | 300                      | 100                                   | 0                                   | 200                                  | 2   | 0,02       |

<sup>a</sup> See 4.17.

### 13 Conditioning of the ICP-MS instrument

There are several types of commercial ICP-MS instrument, with their own instruction manuals, which are usually also available via the instrument software. These should be studied carefully before beginning.

Mass calibration of the instrument by running a mass calibration solution (see 4.18) is crucial for good performance. In order to optimise the detector signal, run a 100 µg/l calibration solution of the element Sb, while adjusting the operational parameters of the instrument. These two procedures shall be carried out on a daily basis.

Prior to start of the ICP-MS measurements, the tubing and glassware of the instrument should be rinsed by pumping a wash solution (4.6) through the system for 5 min.

### 14 ICP-MS measurements

Begin the measurements with a calibration blank solution and then continue with five calibration solutions, starting with the lowest element concentration and ending with the highest. Next analyse a blank test solution (see 9.2) in order to check the blank test solution value and also to determine if there is any carry-over effect from the highest calibration solution. If so, a longer wash time is needed between samples. After the blank test solution, analyse ten test samples followed by a calibration standard solution ("control sample"). Repeat this procedure again with ten test samples and a calibration standard solution, and so on. Thus every tenth sample shall be a calibration solution ("control sample"), and the last sample shall be a calibration solution even if the number of test samples analysed is less than ten.

The concentration of the calibration solutions shall cover the element concentrations of the test samples.

NOTE The calibration standard solution ("control sample") is analysed as a test sample, e.g. a 100 µg/l concentration should give the same intensity as obtained in the calibration curve. A CRM-sample can also be used as a control sample.

### 15 Plotting of calibration graphs

It is necessary to prepare a new calibration curve for each series of determinations. If pure metals and reagents have been used, the blank test should not give any significant contribution to the mass spectrum signal.

A calibration graph (calibration curve) shall be prepared by plotting the mass spectral intensities (usually in counts per second, cps) of the calibration solutions against the mass concentrations of calibration solutions (µg/l). Blank subtraction shall be used. Furthermore, calibration curves should be calculated using one or two internal standards, and the mass concentrations of those should be in the same concentration range as for the test sample.

Plotting of the calibration graph and calculations of the test sample concentrations are done automatically by the software of the ICP-MS instrument. The linearity of the calibration graph should be checked by calculating the regression coefficient, and a value better than 0,999 should be obtained.

Mass spectral intensities of test samples are then measured and, using blank subtraction, the concentrations of the test samples are obtained from the calibration curve.

If the blank intensity is the same or higher than that of the calibration solutions and the sample solutions, precautions shall be taken. In such a case, it is important that the blank intensity be very stable in order to do a blank subtraction. High blank intensity can be due to interference or several interferences, which can be reduced or even eliminated if another isotope is chosen. However, in the case of mono-isotopic elements there are no such possibilities, and better control of the background signal is needed.

## 16 Expression of results

### 16.1 Method of calculation

The concentration of each element of concern refers to the intensities (counts per second or mV) of the test solution and the blank solution, respectively. In the calibration graph, all results are obtained by blank subtraction (see Clause 15).

The mass fraction,  $w$ , of each element, expressed as ( $\mu\text{g/g}$ ) is given by the equation:

$$w = \frac{M}{m}$$

where

$M$  is the mass of the element of concern (in  $\mu\text{g}$ ) in the test solution, derived from the calibration curve;

$m$  is the mass (in g) of the test portion.

### 16.2 Precision

The precision test of this method was carried out by ten laboratories, at 10 to 12 levels (depending on the element) of each element, with each laboratory making three determinations (see Note 1 and Note 2) of each element.

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1, ISO 5725-2 and ISO 5725-3, i.e. one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day) by the same operator as in Note 1 above, using the same apparatus with a new calibration.

From the three results obtained in Note 1 and Note 2, the repeatability ( $r$ ), the within-laboratory reproducibility ( $R_w$ ) and the reproducibility ( $R$ ) were calculated in accordance with the procedure described in C.1 (Three-factor staggered-nested experiment) of Annex C in ISO 5725-3.

The test samples used are listed in Table A.1 for Sn, Tables A.2 and A.3 for Sb, Tables A.4 and A.5 for Ce, Tables A.6 and Table A.7 for Pb and Tables A.8 and A.9 for Bi. The tables are presented together with logarithmic graphs of the five elements investigated (see Annex A).

The results obtained were treated statistically in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3.

The data obtained showed a logarithmic relationship between the mass fraction of the five elements, respectively; repeatability ( $r$ ), reproducibility ( $R$ ) and reproducibility within-laboratories ( $R_w$ ) of test results are summarised in Table 9 for Sn, Table 10 for Sb, Table 11 for Ce, Table 12 for Pb and Table 13 for Bi.

Table 9 — Sn — Repeatability and reproducibility

| Mass fraction of tin<br>$\mu\text{g/g}$ | Repeatability<br>$r$ | Reproducibility<br>within laboratory<br>$R_w$ | Reproducibility<br>$R$ |
|---|----------------------|---|------------------------|
| 5                                       | 0,77                 | 1,96  | 3,18                   |
| 10                                      | 1,33                 | 3,16  | 5,74                   |
| 30                                      | 3,13                 | 6,72  | 14,63                  |
| 50                                      | 4,66                 | 9,55  | 22,60                  |
| 100                                     | 8,00                 | 15,39   | 40,78                  |
| 150                                     | 10,97                | 20,33   | 57,59                  |
| 200                                     | 13,73                | 24,78   | 73,57                  |

Table 10 — Sb — Repeatability and reproducibility

| Mass fraction of<br>antimony<br>$\mu\text{g/g}$ | Repeatability<br>$r$ | Reproducibility<br>within laboratory<br>$R_w$ | Reproducibility<br>$R$ |
|---|----------------------|---|------------------------|
| 1   | 0,14                 | 0,32  | 0,57                   |
| 5   | 0,56                 | 1,35  | 2,42                   |
| 10  | 1,01                 | 2,52  | 4,50                   |
| 20  | 1,82                 | 4,72  | 8,36                   |
| 40  | 3,28                 | 8,82  | 15,56                  |
| 70  | 5,27                 | 14,61   | 25,68                  |
| 100   | 7,15                 | 20,15   | 35,34                  |
| 150   | 10,09                | 29,06   | 50,80                  |
| 200   | 12,89                | 37,66   | 65,73                  |

Table 11 — Ce — Repeatability and reproducibility

| Mass fraction of<br>cerium<br>$\mu\text{g/g}$ | Repeatability<br>$r$ | Reproducibility<br>within laboratory<br>$R_w$ | Reproducibility<br>$R$ |
|---|----------------------|---|------------------------|
| 10  | 0,92                 | 1,07  | 4,19                   |
| 40  | 2,65                 | 3,72  | 13,97                  |
| 70  | 4,05                 | 6,15  | 22,71                  |
| 100   | 5,32                 | 8,47  | 30,96                  |
| 200   | 9,02                 | 15,78   | 56,52                  |
| 300   | 12,29                | 22,70   | 80,38                  |
| 500   | 18,14                | 35,91   | 125,56                 |
| 700   | 23,44                | 48,56   | 167,78                 |
| 1 000   | 30,76                | 66,89   | 228,69                 |

Table 12 — Pb — Repeatability and reproducibility

| Mass fraction of lead<br>$\mu\text{g/g}$ | Repeatability<br>$r$ | Reproducibility<br>within laboratory<br>$R_w$ | Reproducibility<br>$R$ |
|--|----------------------|---|------------------------|
| 0,5                                      | 0,12                 | 0,32  | 0,42                   |
| 1  | 0,20                 | 0,52  | 0,78                   |
| 2  | 0,35                 | 0,85  | 1,44                   |
| 4  | 0,60                 | 1,38  | 2,65                   |
| 7  | 0,94                 | 2,03  | 4,35                   |
| 10                                       | 1,24                 | 2,61  | 5,95                   |
| 20                                       | 2,15                 | 4,23  | 10,97                  |
| 50                                       | 4,43                 | 8,00  | 24,60                  |
| 100                                      | 7,67                 | 12,98   | 45,32                  |

Table 13 — Bi — Repeatability and reproducibility

| Mass fraction of<br>bismuth<br>$\mu\text{g/g}$ | Repeatability<br>$r$ | Reproducibility<br>within laboratory<br>$R_w$ | Reproducibility<br>$R$ |
|--|----------------------|---|------------------------|
| 0,3  | 0,05                 | 0,07  | 0,12                   |
| 0,6  | 0,10                 | 0,14  | 0,25                   |
| 1  | 0,14                 | 0,23  | 0,42                   |
| 2  | 0,26                 | 0,44  | 0,88                   |
| 6  | 0,63                 | 1,24  | 2,81                   |
| 10   | 0,96                 | 2,00  | 4,83                   |
| 20   | 1,70                 | 3,84  | 10,05                  |
| 30   | 2,37                 | 5,62  | 15,43                  |

## 17 Test report

The test report shall include the following information:

- all information necessary for identification of the sample, the laboratory and the date of analysis;
- the method used, by reference to this part of ISO 16918;
- the results, and the form in which they are expressed;
- any unusual features noted during the determination;
- any operation not specified in this part of ISO 16918 or any optional operation which may have influenced the results.

## Annex A (informative)

### Additional information on the international co-operative tests

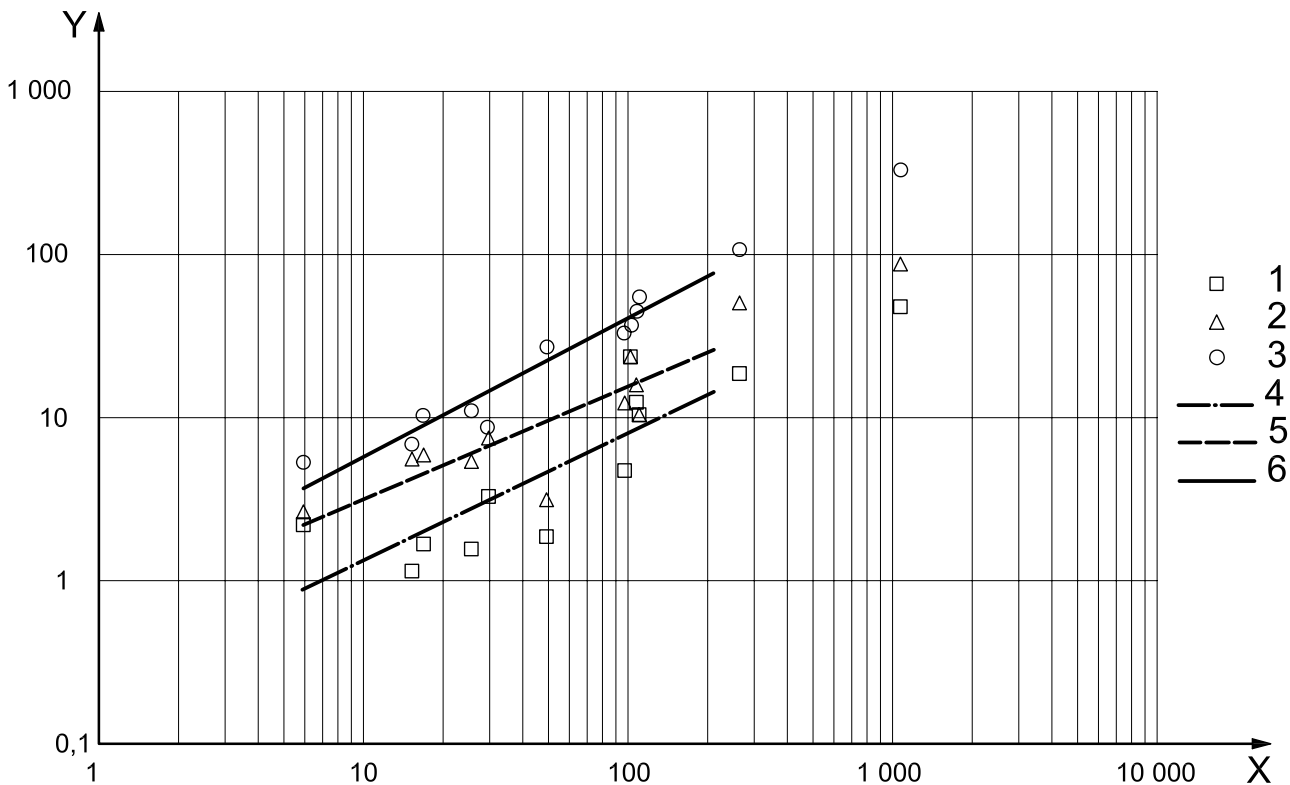
Graphical representations of the precision data are given in Figure A.1 for Sn, Figure A.2 for Sb, Figure A.3 for Ce, Figure A.4 for Pb and Figure A.5 for Bi in connection with Table A.1 (Sn), Tables A.2 and A.3 (Sb), Tables A.4 and A.5 (Ce), Tables A.6 and A.7 (Pb) and Tables A.8 and A.9 (Bi), respectively.

The investigations were carried out over the entire concentration range of 1 µg/g to 1 000 µg/g, but the applicable concentration ranges for determination of the five elements, respectively, are given in the Scope (Clause 1) in this part of ISO 16918.

Not all CRMs are used for every element, and moreover when there is no certified value or approximate value given for a CRM, it is marked with a question mark “?”.

The results in Tables A.1 to A.9 were derived from international trials carried out in 2001 to 2005 on twelve steel samples plus five spiked steel samples in six countries involving ten laboratories.

The samples used are listed in the tables below.



**Key**

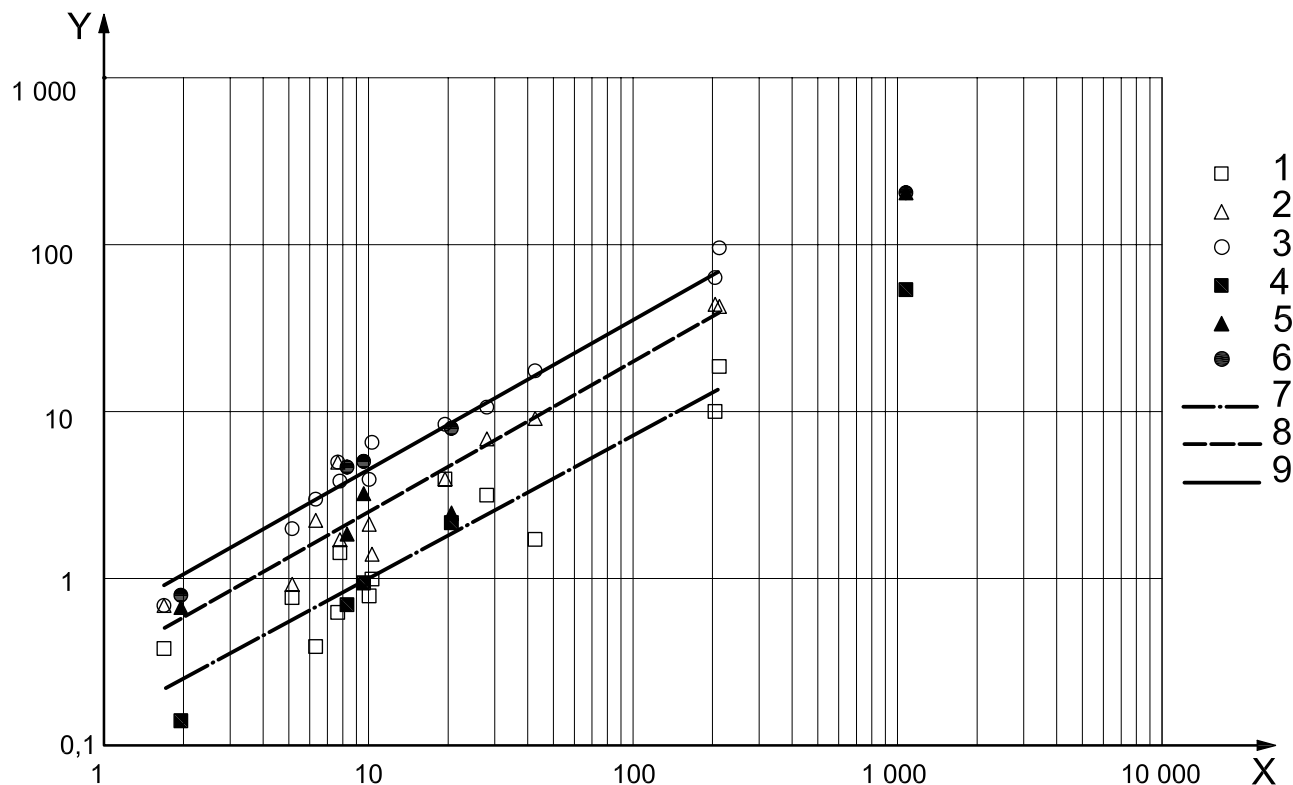
|   |                                 |                        |
|---|---------------------------------|------------------------|
| X | Mass fraction of Sn, µg/g       |                        |
| Y | Precision, µg/g                 |                        |
| 1 | authentic- <i>r</i>             | 4 <i>r</i>             |
| 2 | authentic- <i>R<sub>W</sub></i> | 5 <i>R<sub>W</sub></i> |
| 3 | authentic- <i>R</i>             | 6 <i>R</i>             |

**Figure A.1 — Logarithmic relationship between mass fraction of tin and repeatability or reproducibility**

Table A.1 — Mass fraction of Sn — Authentic samples

Concentrations in µg/g

| Sample Identification   | Certified | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|-----------|--------------|---------------|----------------------|-----------------|
|   |           |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 090-1, BAS<br>1 % Carbon steel (WG53-1)                            | 44        | 49,2         | 1,86          | 3,14                 | 26,95           |
| ECRM 295-1, BAS<br>High alloy steel (WG53-2)                            | 25        | 29,7         | 3,28          | 7,50                 | 8,83            |
| ECRM 179-2, BAM<br>Tool steel (WG53-3)                                  | ?         | 102          | 23,60         | 23,60                | 36,57           |
| ECRM 188-1, IRSID<br>Low alloy steel (WG53-4)                           | ?         | 15,2         | 1,14          | 5,57                 | 6,98            |
| JK1C, JK/SIMR<br>Low impurity iron (WG53-5)                             | 24        | 25,6         | 1,56          | 5,40                 | 11,05           |
| JK37, JK/SIMR<br>High alloy steel (WG53-6)                              | 20        | 16,9         | 1,67          | 5,91                 | 10,19           |
| NIST 361<br>Low alloy steel (AISI4340)<br>(WG53-7)                      | 100       | 97,2         | 4,73          | 12,33                | 32,85           |
| ECRM 187-1, BAM<br>Low alloy steel (WG53-8)                             | 110       | 110          | 10,43         | 10,43                | 54,49           |
| GBW01222, SBQTS<br>Carbon steel (WG53-9)                                | 260       | 264          | 18,62         | 50,53                | 106,04          |
| GBW01619, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-14) | 103       | 107          | 12,38         | 15,92                | 44,47           |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15) | 1 040     | 1 072        | 47,78         | 87,37                | 328,81          |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)                            | ?         | 5,92         | 2,20          | 2,65                 | 5,34            |



**Key**

X Mass fraction of Sb, µg/g

Y Precision, µg/g

- 1 authentic-*r*
- 2 authentic-*R<sub>W</sub>*
- 3 authentic-*R*
- 4 spike-*r*
- 5 spike-*R<sub>W</sub>*
- 6 spike-*R*
- 7 *r*
- 8 *R<sub>W</sub>*
- 9 *R*

**Figure A.2 — Logarithmic relationship between mass fraction of antimony and repeatability or reproducibility**



Table A.2 — Mass fraction of Sb — Authentic samples

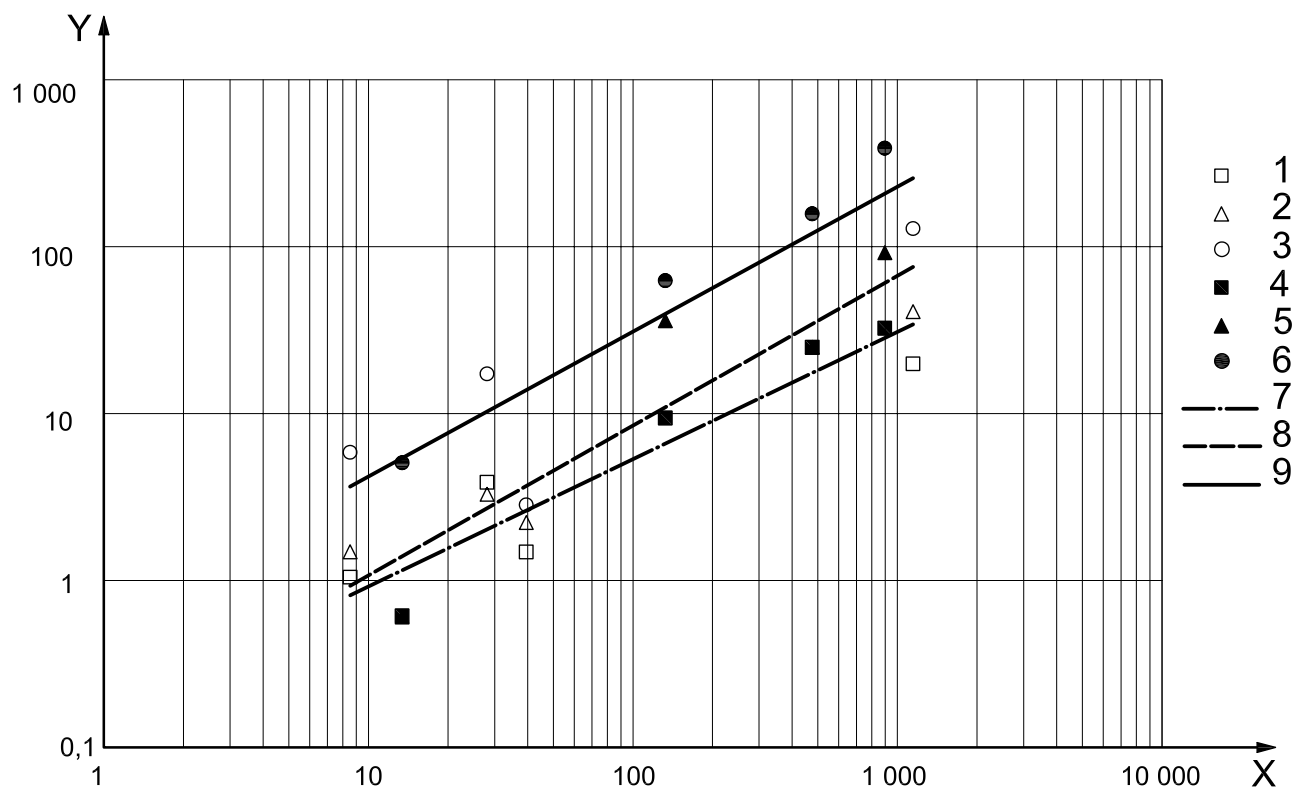
Concentrations in µg/g

| Sample Identification   | Certified | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|-----------|--------------|---------------|----------------------|-----------------|
|   |           |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 090-1, BAS<br>1 % Carbon steel (WG53-1)                            | 9,0       | 10,3         | 0,99          | 1,40                 | 6,52            |
| ECRM 295-1, BAS<br>High alloy steel (WG53-2)                            | 7         | 7,8          | 1,43          | 1,71                 | 3,83            |
| ECRM 179-2, BAM<br>Tool steel (WG53-3)                                  | 17,5      | 19,5         | 3,94          | 3,94                 | 8,39            |
| ECRM 188-1, IRSID<br>Low alloy steel (WG53-4)                           | 4,8       | 5,15         | 0,77          | 0,92                 | 1,98            |
| JK1C, JK/SIMR<br>Low impurity iron (WG53-5)                             | 1,7       | 1,69         | 0,38          | 0,69                 | 0,69            |
| JK37, JK/SIMR<br>High alloy steel (WG53-6)                              | 6,2       | 6,32         | 0,39          | 2,23                 | 2,98            |
| NIST 361<br>Low alloy steel (AISI4340)<br>(WG53-7)                      | 42        | 42,7         | 1,71          | 9,10                 | 17,51           |
| ECRM 187-1, BAM<br>Low alloy steel (WG53-8)                             | ?         | 28,1         | 3,15          | 6,87                 | 10,62           |
| GBW01222, SBQTS<br>Carbon steel (WG53-9)                                | ?         | 10,1         | 0,78          | 2,11                 | 3,91            |
| GBW01619, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-14) | 204       | 212,         | 18,57         | 42,57                | 95,39           |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15) | 7,4       | 7,66         | 0,63          | 4,97                 | 4,97            |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)                            | 190       | 204          | 10,00         | 43,83                | 63,40           |

Table A.3 — Mass fraction of Sb — Spiked samples

Concentrations in µg/g

| Sample Identification   | Certified      | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|----------------|--------------|---------------|----------------------|-----------------|
|   |                |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 295-1, BAS<br>High alloy steel (WG 53-2)<br>Spike 1b                           | 7              | 9,61         | 0,94          | 3,23                 | 5,02            |
| ECRM 179-2, BAM<br>Tool steel (WG 53-3)<br>Spike 2b                                 | 17,5           | 20,6         | 2,15          | 2,47                 | 7,93            |
| JK1C, JK/SIMR<br>Low impurity iron (WG 53-5)<br>Spike 3b                            | 1,7<br>+ 1 000 | 1 080        | 53,56         | 204,12               | 204,12          |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15)<br>Spike 4b | 7,4            | 8,3          | 0,69          | 1,85                 | 4,65            |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)<br>Spike 5b                            | 1,7            | 1,95         | 0,14          | 0,67                 | 40,79           |



**Key**

X Mass fraction of Ce, µg/g

Y Precision, µg/g

- 1 authentic-*r*
- 2 authentic- $R_W$
- 3 authentic- $R$
- 4 spike-*r*
- 5 spike- $R_W$
- 6 spike- $R$
- 7 *r*
- 8  $R_W$
- 9  $R$

**Figure A.3 — Logarithmic relationship between mass fraction of cerium and repeatability or reproducibility**

Table A.4 — Mass fraction of Ce — Authentic samples

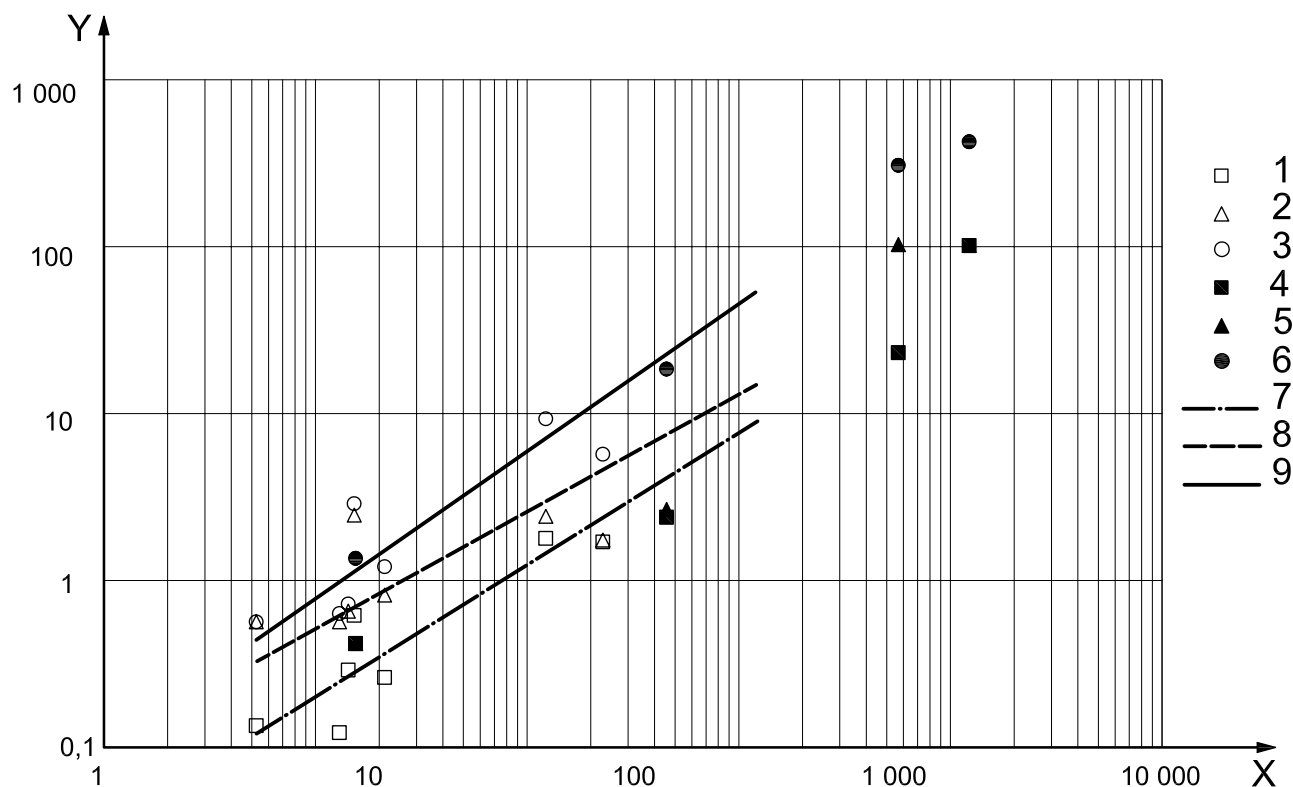
Concentrations in µg/g

| Sample Identification   | Certified | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|-----------|--------------|---------------|----------------------|-----------------|
|   |           |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 090-1, BAS<br>1% Carbon steel (WG53-1)                             | < 0,045   |              |               |                      |                 |
| ECRM 295-1, BAS<br>High alloy steel (WG53-2)                            | ?         |              |               |                      |                 |
| ECRM 179-2, BAM<br>Tool steel (WG53-3)                                  | ?         |              |               |                      |                 |
| ECRM 188-1, IRSID<br>Low alloy steel (WG53-4)                           | ?         |              |               |                      |                 |
| JK1C, JK/SIMR<br>Low impurity iron (WG53-5)                             | < 0,2     |              |               |                      |                 |
| JK37, JK/SIMR<br>High alloy steel (WG53-6)                              | 1 230     | 1 150        | 19,83         | 40,87                | 128,15          |
| NIST 361<br>Low alloy steel (AISI4340)<br>(WG53-7)                      | 40        | 39,52        | 1,48          | 2,22                 | 2,83            |
| ECRM 187-1, BAM<br>Low alloy steel (WG53-8)                             | ?         |              |               |                      |                 |
| GBW01222, SBQTS<br>Carbon steel (WG53-9)                                | 8,5       | 7,33         | 1,11          | 1,20                 | 7,86            |
| GBW01619, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-14) | 83        | 64,3         | 56,00         | 56,00                | 61,98           |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15) | 0,36      |              |               |                      |                 |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)                            | ?         |              |               |                      |                 |

Table A.5 — Mass fraction of Ce — Spiked samples

Concentrations in µg/g

| Sample Identification   | Certified      | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|----------------|--------------|---------------|----------------------|-----------------|
|   |                |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 295-1, BAS<br>High alloy steel (WG 53-2)<br>Spike 1b                           | ?<br>+ 110     | 133          | 9,39          | 36,05                | 62,62           |
| ECRM 179-2, BAM<br>Tool steel (WG 53-3)<br>Spike 2b                                 | < 0,3<br>+ 11  | 12,6         | 0,59          | 2,25                 | 8,31            |
| JK1C, JK/SIMR<br>Low impurity iron (WG 53-5)<br>Spike 3b                            | < 0,2<br>+ 0,1 |              |               |                      |                 |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15)<br>Spike 4b | ?<br>+ 400     | 477          | 24,99         | 24,99                | 157,25          |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)<br>Spike 5b                            | < 0,2<br>+ 800 | 896          | 32,50         | 92,00                | 389,06          |



**Key**

X Mass fraction of Pb,  $\mu\text{g/g}$

Y Precision,  $\mu\text{g/g}$

- 1 authentic-*r*
- 2 authentic- $R_W$
- 3 authentic- $R$
- 4 spike-*r*
- 5 spike- $R_W$
- 6 spike- $R$
- 7  $r$
- 8  $R_W$
- 9  $R$

**Figure A.4 — Logarithmic relationship between mass fraction of lead and repeatability or reproducibility**

Table A.6 — Mass fraction of Pb — Authentic samples

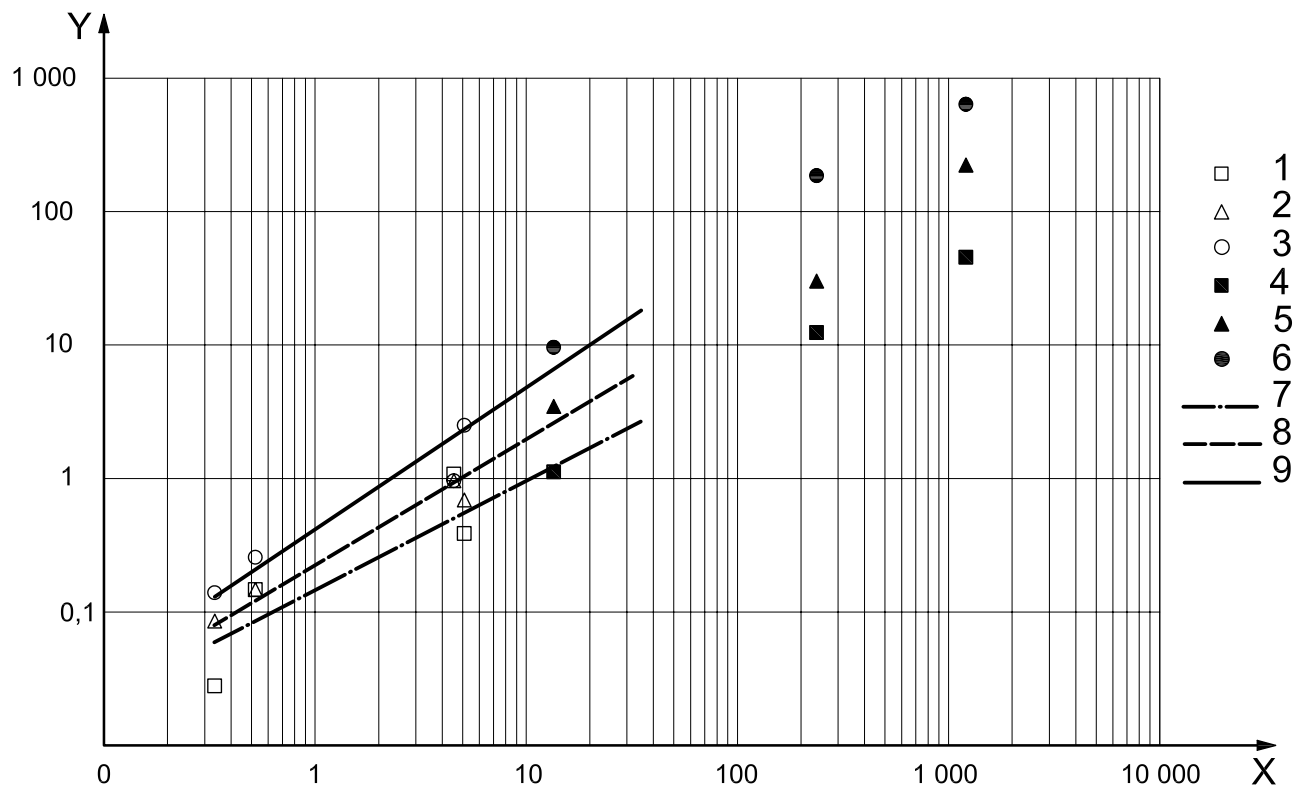
Concentrations in µg/g

| Sample Identification   | Certified | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|-----------|--------------|---------------|----------------------|-----------------|
|   |           |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 090-1, BAS<br>1 % Carbon steel (WG53-1)                            | 23,9      | 22,8         | 1,70          | 1,75                 | 5,71            |
| ECRM 295-1, BAS<br>High alloy steel (WG53-2)                            | ?         |              |               |                      |                 |
| ECRM 179-2, BAM<br>Tool steel (WG53-3)                                  | 1,3       | 1,43         | 0,29          | 0,65                 | 0,72            |
| ECRM 188-1, IRSID<br>Low alloy steel (WG53-4)                           | < 1       |              |               |                      |                 |
| JK1C, JK/SIMR<br>Low impurity iron (WG53-5)                             | < 0,3     |              |               |                      |                 |
| JK37, JK/SIMR<br>High alloy steel (WG53-6)                              | 1,29      | 1,30         | 0,12          | 0,56                 | 0,63            |
| NIST 361<br>Low alloy steel (AISI4340)<br>(WG53-7)                      | 0,25      |              |               |                      |                 |
| ECRM 187-1, BAM<br>Low alloy steel (WG53-8)                             | ?         | 0,52         | 0,13          | 0,57                 | 0,57            |
| GBW01222, SBQTS<br>Carbon steel (WG53-9)                                | ?         |              |               |                      |                 |
| GBW01619, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-14) | 12        | 12,3         | 1,79          | 2,43                 | 9,32            |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15) | 2,2       | 1,52         | 0,62          | 2,46                 | 2,89            |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)                            | ?         | 2,12         | 0,26          | 0,82                 | 1,21            |

Table A.7 — Mass fraction of Pb — Spiked samples

Concentrations in µg/g

| Sample Identification   | Certified        | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|------------------|--------------|---------------|----------------------|-----------------|
|   |                  |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 295-1, BAS<br>High alloy steel (WG 53-2)<br>Spike 1b                           | ?<br>+ 40        | 45,7         | 2,39          | 2,67                 | 18,48           |
| ECRM 179-2, BAM<br>Tool steel (WG 53-3)<br>Spike 2b                                 | 1,3              | 1,55         | 0,42          | 0,42                 | 1,36            |
| JK1C, JK/SIMR<br>Low impurity iron (WG 53-5)<br>Spike 3b                            | < 0,3<br>+ 0,1   |              |               |                      |                 |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15)<br>Spike 4b | 2,2<br>+ 540     | 567          | 23,17         | 102,86               | 307,30          |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)<br>Spike 5b                            | < 0,3<br>+ 1 100 | 1 230        | 101,76        | 101,76               | 424,71          |



**Key**

X Mass fraction of Bi, µg/g

Y Precision, µg/g

- 1 authentic-*r*
- 2 authentic-*R<sub>W</sub>*
- 3 authentic-*R*
- 4 spike-*r*
- 5 spike-*R<sub>W</sub>*
- 6 spike-*R*
- 7 *r*
- 8 *R<sub>W</sub>*
- 9 *R*

**Figure A.5 — Logarithmic relationship between mass fraction of bismuth and repeatability or reproducibility**

Table A.8 — Mass fraction of Bi — Authentic samples

Concentrations in µg/g

| Sample Identification   | Certified | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|-----------|--------------|---------------|----------------------|-----------------|
|   |           |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 090-1, BAS<br>1 % Carbon steel (WG 53-1)                           | < 0,2     |              |               |                      |                 |
| ECRM 295-1, BAS<br>High alloy steel (WG 53-2)                           | ?         |              |               |                      |                 |
| ECRM 179-2, BAM<br>Tool steel (WG 53-3)                                 | < 0,3     |              |               |                      |                 |
| ECRM 188-1, IRSID<br>Low alloy steel (WG 53-4)                          | < 0,2     |              |               |                      |                 |
| JK1C, JK/SIMR<br>Low impurity iron (WG 53-5)                            | < 0,1     |              |               |                      |                 |
| JK37, JK/SIMR<br>High alloy steel (WG 53-6)                             | 0,32      | 0,33         | 0,03          | 0,09                 | 0,14            |
| NIST 361 (AISI4340)<br>Low alloy steel (WG 53-7)                        | 4         | 5,10         | 0,39          | 0,69                 | 2,50            |
| ECRM 187-1, BAM<br>Low alloy steel (WG 53-8)                            | ?         |              |               |                      |                 |
| GBW01222, SBQTS<br>Carbon steel (WG 53-9)                               | ?         |              |               |                      |                 |
| GBW01619, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-14) | 4,2       | 4,54         | 1,08          | 0,96                 | 0,96            |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy (WG53-15) | 0,5       | 0,52         | 0,15          | 0,15                 | 0,26            |
| JSS 175-5, ISIJ<br>Low alloy steel (WG53-17)                            | ?         | 2,12         | 0,26          | 0,82                 | 1,21            |

Table A.9 — Mass fraction of Bi — Spiked samples

Concentrations in µg/g

| Sample Identification   | Certified        | Overall mean | Repeatability | Reproducibility      | Reproducibility |
|---|------------------|--------------|---------------|----------------------|-----------------|
|   |                  |              | <i>r</i>      | <i>R<sub>w</sub></i> | <i>R</i>        |
| ECRM 295-1, BAS<br>High alloy steel<br>Spike 1b (WG 53-2)                           | ?<br>+ 200       | 237          | 12,40         | 30,20                | 185,65          |
| ECRM 179-2, BAM<br>Tool steel<br>Spike 2b (WG 53-3)                                 | < 0,3<br>+ 10    | 13,5         | 1,12          | 3,48                 | 9,62            |
| JK1C, JK/SIMR<br>Low impurity iron<br>Spike 3b (WG 53-5)                            | < 0,1<br>+ 0,1   |              |               |                      |                 |
| GBW01622, SBQTS<br>Iron/Nickel base<br>High temperature alloy<br>Spike 4b (WG53-15) | ?<br>0,5         |              |               |                      |                 |
| JSS 175-5, ISIJ<br>Low alloy steel<br>Spike 5b (WG53-17)                            | < 0,1<br>+ 1 000 | 1 210        | 45,45         | 223,75               | 635,71          |

## Annex B (informative)

### Interferences in the determination of elements Sn, Sb, Ce, Pb and Bi using ICP- MS

| Isotope | Interferences  |
|---------|--|
| 116Sn   | 116Cd, 100MoO <sup>+</sup> , 232Th <sup>2+</sup>                               |
| 117Sn   | 100MoOH <sup>+</sup> , 238U <sup>2+</sup> , 40Ar <sup>77</sup> Se <sup>+</sup> |
| 118Sn   | 102RuO <sup>+</sup> , 102PdO <sup>+</sup> , 40Ar <sup>78</sup> Se <sup>+</sup> |
| 119Sn   | 103RhO <sup>+</sup>  |
| 120Sn   | 104PdO <sup>+</sup> , 104RuO <sup>+</sup> , (120Te <sup>+</sup> )              |
| 122Sn   | 124Te <sup>+</sup> , 106PdO <sup>+</sup>                                       |
| 124Sn   | 124Te <sup>+</sup> , 108PdO <sup>+</sup> , 124Xe <sup>+</sup>                  |
| 121Sb   | 105PdO <sup>+</sup>  |
| 123Sb   | 123Te, 107AgO <sup>+</sup>   |
| 140Ce   |  |
| 142Ce   |  |
| 204Pb   | 204Hg <sup>+</sup>   |
| 206Pb   |  |
| 207Pb   | 191IrO <sup>+</sup>  |
| 208Pb   |  |
| 209Bi   | 193IrO <sup>+</sup>  |



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