BS ISO 7530-1:2015

BSI Standards Publication

# Nickel alloys — Flame atomic absorption spectrometric analysis

## Part 1: Determination of cobalt, chromium, copper, iron and manganese

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## National foreword

This British Standard is the UK implementation of ISO 7530-1:2015. It supersedes BS 7455-1:1991, BS 7455-2:1991, BS 7455-3:1991, BS 7455-4:1991, BS 7455-5:1991 and BS 7455-6:1991, which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee I/-, Miscellaneous standards materials and chemicals.

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# Nickel alloys — Flame atomic absorption spectrometric analysis —

## Part 1:
## Determination of cobalt, chromium, copper, iron and manganese

*Alliages de nickel — Analyse par spectrométrie d’absorption atomique dans la flamme —*

*Partie 1: Détermination du cobalt, du chrome, du cuivre, du fer et du manganèse*

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 155, *Nickel and nickel alloys*.

This second edition of ISO 7530-1 cancels and replaces ISO 7530-1:1990, ISO 7530-2:1990, ISO 7530-3:1990, ISO 7530-4:1990, ISO 7530-5:1990, and ISO 7530-6:1990, which have been technically revised.

ISO 7530 consists of the following parts, under the general title *Nickel alloys — Flame atomic absorption spectrometric analysis*:

- *Part 1: Determination of cobalt, chromium, copper, iron and manganese*
- *Part 7: Determination of aluminium content*
- *Part 8: Determination of silicon content*
- *Part 9: Determination of vanadium content*

# Introduction

This part of ISO 7530 describes five flame atomic absorption spectrometric methods for the determination of cobalt, chromium, copper, iron, and manganese in nickel alloys.

Although the methods are described independently, it is possible to determine more than one element on a single test solution by adjustment of the sample weight and initial and subsequent dilutions.

# Nickel alloys — Flame atomic absorption spectrometric analysis —

## Part 1:
## Determination of cobalt, chromium, copper, iron and manganese

## 1 Scope

This part of ISO 7530 describes flame atomic absorption spectrometric methods for the determination of cobalt, chromium, copper, iron, and manganese in nickel alloys which can be dissolved in the nitric-hydrochloric acids mixture specified.

For each element, the method is applicable to the content range between 0,01 % and 4 %.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

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

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

## 3 Principle

Dissolution of a test portion in a nitric–hydrochloric acids mixture, evaporation of acids excess, and dissolution of the salts.

Addition of an ionization suppressant if necessary and dilution of the solution to a known volume.

Nebulization of the test solution after suitable dilution, if necessary, into an air/acetylene or a nitrous oxide/acetylene flame of an atomic absorption spectrometer.

Measurement of the absorption of the energy of the resonance line from the spectrum of the element being determined and comparison with that of calibration solutions of the same element.

## 4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

### 4.1 Nitric acid, ($HNO_3$) $\rho_{20}$ = 1,41 g/ml.

### 4.2 Nitric acid solution, 1 + 1.

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

### 4.3 Hydrochloric acid, (HCl) $\rho_{20}$ = 1,18 g/ml.

### 4.4 Hydrochloric acid solution, 1 + 1.

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

### 4.5 Nitric-hydrochloric acids mixture.

**CAUTION — This acid mixture is highly corrosive and unstable. Noxious gas (chlorine) is liberated on standing. It shall be prepared and used in a fume cupboard and shall not be kept in a closed container.**

Carefully mix one part of nitric acid (4.2) and three parts of hydrochloric acid (4.3). This mixture is not stable and should be prepared only as needed.

### 4.6 Strontium chloride solution.

Transfer 113,5 g of strontium chloride hexahydrate ($SrCl_2 . 6H_2O$) into a 600 ml beaker and dissolve in 400 ml of hot water (50 °C to 60 °C). Cool, transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix.

### 4.7 Hydrogen peroxide, 300 g/l solution.

### 4.8 Cobalt standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of cobalt (Co ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid solution (4.4), cover with a watch glass, and heat gently until the cobalt is completely dissolved. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, add 35 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

1 ml of this solution contains 1,000 mg of cobalt.

### 4.9 Cobalt standard solution, 0,025 g/l.

Transfer 5,0 ml of cobalt standard solution (4.8) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,025 mg of cobalt.

### 4.10 Chromium standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of chromium (Cr ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid (4.4), cover with a watch glass, and heat to complete dissolution. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, add 35 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

1 ml of this solution contains 1,000 mg of chromium.

### 4.11 Chromium standard solution, 0,100 g/l.

Transfer 20,0 ml of chromium standard solution (4.10) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of chromium.

### 4.12 Copper standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of copper (Cu ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 50 ml of nitric acid (4.2), cover with a watch glass, and allow to stand until the reaction ceases. Heat to complete dissolution, boil to remove nitrogen oxides, and evaporate just to dryness. Cool, add 25 ml of hydrochloric acid (4.3), and evaporate just to dryness. Add again 25 ml of hydrochloric acid (4.3) and repeat the evaporation. Dissolve the salts in 50 ml of hydrochloric acid (4.4) and heat if necessary. Cool, transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

1 ml of this solution contains 1,000 mg of copper.

### 4.13 Copper standard solution, 0,100 g/l.

Transfer 10,0 ml of copper standard solution (4.12) into a 100 ml one-mark volumetric flask and add 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of copper.

### 4.14 Copper standard solution, 0,025 g/l.

Transfer 5,0 ml of copper standard solution (4.12) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,025 mg of copper.

### 4.15 Iron standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of iron (Fe ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid (4.4) and cover with a watch glass. Heat to initiate the reaction and complete dissolution. Cool to about 50 °C, cautiously add 1 ml of hydrogen peroxide (4.7), and bring to the boil to oxidize the iron. Cool, transfer into a 1 000 ml one-mark volumetric flask, and add 35 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

1 ml of this solution contains 1,000 mg of iron.

### 4.16 Iron standard solution, 0,050 g/l.

Transfer 10,0 ml of iron standard solution (4.15) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,050 mg of iron.

### 4.17 Manganese standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of manganese (Mn ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid (4.4) and 2 ml of nitric acid (4.1). Cover with a watch glass and heat to initiate the reaction and complete dissolution. Add 0,5 ml of hydrogen peroxide (4.7). Cool, transfer into a 1 000 ml one-mark volumetric flask, and add 50 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

1 ml of this solution contains 1,000 mg of manganese.

### 4.18 **Manganese standard solution**, 0,100 g/l.

Transfer 10,0 ml of manganese standard solution (4.17) into a 100 ml one-mark volumetric flask and add 2,5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of manganese.

### 4.19 **Manganese standard solution**, 0,010 g/l.

Transfer 5,0 ml of manganese standard solution (4.17) into a 500 ml one-mark volumetric flask and add 12,5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,010 mg of manganese.

## 5 Apparatus

All volumetric glassware shall be class A in accordance with ISO 385, ISO 648, or ISO 1042 as appropriate.

Ordinary laboratory apparatus and the following.

### 5.1 **Atomic absorption spectrometer**.

**WARNING — The manufacturer's recommendations should be closely followed and particular attention is drawn to the following safety points.**

a) The explosive nature of acetylene and regulations concerning its use.

b) The need to shield the eyes of the operator from ultraviolet radiation by means of tinted glass.

c) The need to keep the burner clear of deposits because a badly clogged burner can cause a flashback.

d) The need to ensure that the liquid trap is filled with water.

e) The need to spray distilled water between the test solutions, blank solution, and/or calibration solutions.

The atomic absorption spectrometer used shall be checked according to the instrument performance parameters given in Annex A.

The instrument shall be equipped with burners suitable for both an air/acetylene and a nitrous oxide/acetylene flame.

The instrument should be suitable for operating with single or multi-element element hollow cathode lamps powered at currents recommended by the manufacturer.

## 6 Sampling and sample preparation

Sampling and sample preparation shall be carried out by normal agreed procedures or, in case of dispute, by appropriate National or International Standards.

The laboratory sample normally is in the form of millings or drillings and no further preparation of the sample is necessary.

If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned with high purity acetone and then dried in air.

If the laboratory sample contains particles or pieces of widely varying sizes, the test sample should be obtained by riffling.

# 7 Procedure

## 7.1 Test portion

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

## 7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

## 7.3 Dissolution

Transfer the test portion (7.1) into a 600 ml beaker. Add 20 ml of the nitric–hydrochloric acids mixture (4.5) and cover with a watch glass. Apply sufficient heat to initiate and maintain the reaction until dissolution is complete. If the alloy resists dissolution, some adjustment of the acids mixture may be required. Add hydrochloric acid (4.3) in 1 ml increments and continue heating to dissolve the sample.

Using a low heat, evaporate the solution just to dryness. Do not bake. Cool to about 50 °C, add 25 ml of hydrochloric acid (4.3), and evaporate again to dryness. Add a new 25 ml of hydrochloric acid (4.3) portion and repeat the evaporation.

NOTE Some alloys having high copper contents can be easily dissolved in nitric acid solution (4.2). For some alloys, an acid mixture containing 30 ml hydrochloric acid (4.3) and 2 ml of nitric acid (4.1) is more effective.

If sample inhomogeneity is suspected, a larger mass of sample may be taken for analysis. However, an aliquot portion corresponding to a 1 g sample shall be taken from this test solution and processed in accordance with the procedure given.

## 7.4 Determination of cobalt

### 7.4.1 Preparation of the test solution

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

#### 7.4.1.1 Cobalt contents between 0,01 % and 0,10 %

Transfer 25 ml of the test solution (7.4.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 4 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.4.1.2 Cobalt contents between 0,1 % and 1 %

Transfer 2,5 ml of the test solution (7.4.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.4.1.3 Cobalt contents between 1 % and 4 %

Transfer 5 ml of the test solution (7.4.1) into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Transfer 10 ml of this solution into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

### 7.4.2 Preparation of the calibration solutions

#### 7.4.2.1 Cobalt contents between 0,01 % and 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.9) shown in Table 1. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 1 — Calibration for cobalt contents between 0,01 % and 0,10 % percentage (mass fraction)**

| **Cobalt standard solution volume** (4.9) (ml) | **Corresponding cobalt mass** (mg) | **Corresponding content of cobalt in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 1 | 0,025 | 0,01 |
| 2 | 0,050 | 0,02 |
| 4 | 0,100 | 0,04 |
| 6 | 0,150 | 0,06 |
| 8 | 0,200 | 0,08 |
| 10 | 0,250 | 0,10 |

#### 7.4.2.2 Cobalt contents between 0,1 % and 1 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.9) shown in Table 2. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 2 — Calibration for cobalt contents between 0,1 % and 1 % percentage (mass fraction)**

| **Cobalt standard solution volume** (4.9) (ml) | **Corresponding cobalt mass** (mg) | **Corresponding content of cobalt in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 1 | 0,025 | 0,1 |
| 2 | 0,050 | 0,2 |
| 4 | 0,100 | 0,4 |
| 6 | 0,150 | 0,6 |
| 8 | 0,200 | 0,8 |
| 10 | 0,250 | 1,0 |

#### 7.4.2.3 Cobalt contents between 1 % and 4 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.9) shown in Table 3. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 3 — Calibration for cobalt contents between 1 % and 4 % percentage (mass fraction)**

| Cobalt standard solution volume (4.9) (ml) | Corresponding cobalt mass (mg) | Corresponding the content of cobalt in sample percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2 | 0,050 | 1,0 |
| 3 | 0,075 | 1,5 |
| 4 | 0,100 | 2,0 |
| 5 | 0,125 | 2,5 |
| 6 | 0,150 | 3,0 |
| 7 | 0,175 | 3,5 |
| 8 | 0,200 | 4,0 |

## 7.5 Determination of chromium

### 7.5.1 Preparation of the test solution

#### 7.5.1.1 Chromium contents between 0,01 % and 0,10 %

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6), dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

#### 7.5.1.2 Chromium contents between 0,1 % and 1 %

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

Transfer 10 ml of this solution into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 4,5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.5.1.3 Chromium contents between 1 % and 4 %

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

Transfer 2,5 ml of this solution into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

### 7.5.2 Preparation of the calibration solutions

#### 7.5.2.1 Chromium contents between 0,01 % and 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of chromium standard solution (4.11) shown in Table 4. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 4 — Calibration for chromium contents between 0,01 % and 0,10 % percentage (mass fraction)**

| **Chromium standard solution volume** (4.11) (ml) | **Corresponding chromium mass** (mg) | **Corresponding content of chromium in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 1 | 0,1 | 0,01 |
| 2 | 0,2 | 0,02 |
| 4 | 0,4 | 0,04 |
| 6 | 0,6 | 0,06 |
| 8 | 0,8 | 0,08 |
| 10 | 1,0 | 0,10 |

#### 7.5.2.2 Chromium contents between 0,1 % and 1 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of chromium standard solution (4.11) shown in Table 5. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 5 — Calibration for chromium contents between 0,1 % and 1 % percentage (mass fraction)**

| **Chromium standard solution volume** (4.11) (ml) | **Corresponding chromium mass** (mg) | **Corresponding content of chromium in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 1 | 0,1 | 0,1 |
| 2 | 0,2 | 0,2 |
| 4 | 0,4 | 0,4 |
| 6 | 0,6 | 0,6 |
| 8 | 0,8 | 0,8 |
| 10 | 1,0 | 1,0 |

#### 7.5.2.3 Chromium contents between 1 % and 4 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of chromium standard solution (4.11) shown in Table 6. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 6 — Calibration for chromium contents between 1 % and 4 % percentage (mass fraction)**

| **Chromium standard solution volume** (4.11) (ml) | **Corresponding chromium mass** (mg) | **Corresponding content of chromium in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2,5 | 0,25 | 1,0 |
| 4,0 | 0,40 | 1,6 |
| 6,0 | 0,60 | 2,4 |
| 8,0 | 0,80 | 3,2 |
| 10,0 | 1,00 | 4,0 |

## 7.6 Determination of copper

### 7.6.1 Preparation of the test solution

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

#### 7.6.1.1 Copper contents between 0,01 % and 0,10 %

Transfer 50 ml of the test solution (7.6.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 2,5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.6.1.2 Copper contents between 0,1 % and 0,6 %

Transfer 10 ml of the test solution (7.6.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 4,5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.6.1.3 Copper contents between 0,5 % and 4 %

Transfer 2 ml of the test solution (7.6.1) into a 200 ml one-mark volumetric flask, add 8 ml of strontium chloride solution (4.6) and 10 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

### 7.6.2 Preparation of the calibration solutions

#### 7.6.2.1 Copper contents between 0,01 % and 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of copper standard solution (4.14) shown in Table 7. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 7 — Calibration for copper contents between 0,01 % and 0,10 % percentage (mass fraction)**

| **Copper standard solution volume** (4.14) (ml) | **Corresponding copper mass** (mg) | **Corresponding content of copper in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2 | 0,050 | 0,01 |
| 4 | 0,100 | 0,02 |
| 8 | 0,200 | 0,04 |
| 12 | 0,300 | 0,06 |
| 16 | 0,400 | 0,08 |
| 20 | 0,500 | 0,10 |

### 7.6.2.2 Copper contents between 0,1 % and 0,6 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of copper standard solution (4.13) shown in Table 8. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 8 — Calibration for copper contents between 0,1 % and 0,6 % percentage (mass fraction)**

| **Copper standard solution volume** (4.13) (ml) | **Corresponding copper mass** (mg) | **Corresponding content of copper in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 1 | 0,1 | 0,1 |
| 2 | 0,2 | 0,2 |
| 4 | 0,4 | 0,4 |
| 5 | 0,5 | 0,5 |
| 6 | 0,6 | 0,6 |

### 7.6.2.3 Copper contents between 0,5 % and 4 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of copper standard solutions (4.13) or (4.14) shown in Table 9. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 9 — Calibration for copper contents between 0,5 % and 4 % percentage (mass fraction)**

| **Copper standard solutions volume** (ml) | | **Corresponding copper mass** (mg) | **Corresponding content of copper in the sample** percentage (mass fraction) |
|---|---|---|---|
| (4.13) | (4.14) | | |
| | 0 | 0 | 0 |
| | 2 | 0,050 | 0,50 |
| | 3 | 0,075 | 0,75 |
| | 4 | 0,100 | 1,00 |
| 2 | | 0,200 | 2,00 |
| 3 | | 0,300 | 3,00 |
| 4 | | 0,400 | 4,00 |

## 7.7 Determination of iron

### 7.7.1 Preparation of the test solution

#### 7.7.1.1 Iron contents between 0,01 % and 0,10 %

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6), dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

#### 7.7.1.2 Iron contents between 0,1 % and 0,8 %

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, add 20 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

Transfer 25 ml of this solution into a 50 ml one-mark volumetric flask, add 2 ml of strontium chloride solution (4.6) and 1,5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.7.1.3 Iron contents between 0,5 % and 4 %

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, add 20 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

Transfer 10 ml of this solution into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

### 7.7.2 Preparation of the calibration solutions

#### 7.7.2.1 Iron contents between 0,01 % and 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of iron standard solution (4.16) shown in Table 10. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 10 — Calibration for iron contents between 0,01 % and 0,10 % percentage (mass fraction)**

| Iron standard solution volume (4.16) (ml) | Corresponding iron mass (mg) | Corresponding content of iron in the sample percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2 | 0,10 | 0,010 |
| 5 | 0,25 | 0,025 |
| 10 | 0,50 | 0,050 |
| 15 | 0,75 | 0,075 |
| 20 | 1,00 | 0,100 |

#### 7.7.2.2 Iron contents between 0,1 % and 0,8 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of iron standard solution (4.16) shown in Table 11. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 11 — Calibration for iron contents between 0,1 % and 0,8 % percentage (mass fraction)**

| Iron standard solution volume (4.16) (ml) | Corresponding iron mass (mg) | Corresponding content of iron in the sample percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2 | 0,10 | 0,10 |
| 4 | 0,20 | 0,20 |
| 8 | 0,40 | 0,40 |
| 12 | 0,60 | 0,60 |
| 16 | 0,80 | 0,80 |

#### 7.7.2.3 Iron contents between 0,5 % and 4 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of iron standard solution (4.16) shown in Table 12. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 12 — Calibration for iron contents between 0,5 % and 4 % percentage (mass fraction)**

| Iron standard solution volume (4.16) (ml) | Corresponding iron mass (mg) | Corresponding content of iron in the sample percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2 | 0,10 | 0,50 |
| 4 | 0,20 | 1,00 |
| 6 | 0,30 | 1,50 |
| 8 | 0,40 | 2,00 |
| 10 | 0,50 | 2,50 |
| 12 | 0,60 | 3,00 |
| 14 | 0,70 | 3,50 |
| 16 | 0,80 | 4,00 |

## 7.8 Determination of manganese

### 7.8.1 Preparation of the test solution

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, 0,5 ml of hydrogen peroxide (4.7), and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

#### 7.8.1.1 Manganese contents between 0,01 % and 0,10 %

Transfer 20 ml of the test solution (7.8.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 4 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.8.1.2 Manganese contents between 0,1 % and 2,0 %

Transfer 2,5 ml of the test solution (7.8.1) into a 250 ml one-mark volumetric flask, add 10 ml of strontium chloride solution (4.6) and 12,5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

#### 7.8.1.3 Manganese contents between 2 % and 4 %

Transfer 5 ml of the test solution (7.8.1) into a 200 ml one-mark volumetric flask, add 8 ml of strontium chloride solution (4.6) and 10 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

### 7.8.2 Preparation of the calibration solutions

#### 7.8.2.1 Manganese contents between 0,01 % and 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.19) shown in Table 13. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 13 — Calibration for manganese contents between 0,01 % and 0,10 % percentage (mass fraction)**

| **Manganese standard solution volume** (4.19) (ml) | **Corresponding manganese mass** (mg) | **Corresponding content of manganese in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 2 | 0,02 | 0,010 |
| 5 | 0,05 | 0,025 |
| 7 | 0,07 | 0,035 |
| 10 | 0,10 | 0,050 |
| 15 | 0,15 | 0,075 |
| 20 | 0,20 | 0,100 |

#### 7.8.2.2 Manganese contents between 0,1 % and 2,0 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.19) shown in Table 14. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 14 — Calibration for manganese contents between 0,1 % and 2,0 % percentage (mass fraction)**

| **Manganese standard solution volume** (4.19) (ml) | **Corresponding manganese mass** (mg) | **Corresponding content of manganese in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 1 | 0,01 | 0,10 |
| 2 | 0,02 | 0,20 |
| 5 | 0,05 | 0,50 |
| 7 | 0,07 | 0,70 |
| 10 | 0,10 | 1,00 |
| 15 | 0,15 | 1,50 |
| 20 | 0,20 | 2,00 |

#### 7.8.2.3 Manganese contents between 2 % and 4 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of manganese standard solution (4.18) shown in Table 15. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

**Table 15 — Calibration for manganese contents between 2 % and 4 % percentage (mass fraction)**

| **Manganese standard solution volume** (4.18) (ml) | **Corresponding manganese mass** (mg) | **Corresponding content of manganese in the sample** percentage (mass fraction) |
|---|---|---|
| 0 | 0 | 0 |
| 5 | 0,5 | 2,0 |
| 6 | 0,6 | 2,4 |
| 7 | 0,7 | 2,8 |
| 8 | 0,8 | 3,2 |
| 9 | 0,9 | 3,6 |
| 10 | 1, 0 | 4,0 |

## 7.9 Adjustment of atomic absorption spectrometer

Fit the appropriate hollow cathode lamp into the atomic absorption spectrometer (5.1), switch on the current, and allow it to stabilize. Adjust to minimum absorbance the wavelength in the region of the following:

— 240,7 nm, for cobalt;

— 357,9 nm, for chromium;

— 324,8 nm, for copper;

— 248,3 nm, for iron;

— 279,5 nm for the manganese determination between 0,01 and 2 % and of 403,0 nm for the manganese determination between 2 % and 4 %.

Following manufacturer's instructions, fit

— the air/acetylene burner for cobalt, copper, iron, and manganese,

— the nitrous oxide/acetylene burner for chromium, and

— light the flame (see 5.1) and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

Set the absorbance value at zero using water.

Choose an integration time to give a signal stable enough to fulfil the precision criteria given in Annex A. Evaluate the criteria of A.2 to ensure that the instrument is suitable for the determination of the element selected.

NOTE Scale expansion may have to be used to obtain the required readability.

## 7.10 Spectrometric measurements

Depending on the expected content of the element being determined, nebulize the relevant series of calibration solutions in succession (7.4.2.1, 7.4.2.2, or 7.4.2.3 for cobalt; 7.5.2.1, 7.5.2.2, or 7.5.2.3 for chromium; 7.6.2.1, 7.6.2.2, or 7.6.2.3 for copper; 7.7.2.1, 7.7.2.2, or 7.7.2.3 for iron; and 7.8.2.1, 7.8.2.2, or 7.8.2.3 for manganese) into the flame and measure the absorbance for each.

Take care to keep the nebulization rate constant throughout the preparation of the calibration curve. Nebulize water through the burner after each measurement (see Note).

NOTE For certain types of apparatus, instead of water, it is preferable to use a solution containing the attack reagents in the same concentrations as in the test portion solutions.

Nebulize the test solution(s) and note the reading to determine the approximate concentration of the test solutions. Nebulize water through the burner after each measurement (see Note).

For each test solution and for the blank test, select two calibration solutions, one having an absorbance just below that of the test solution and one just above. Nebulize these first in ascending order, then in descending order, with the test solution as the middle solution in each case measuring the absorption in relation to water.

For each test solution, repeat the measurements at least once.

### 7.11 Calibration curves

For each element and each range of contents, establish the calibration curve using measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or an offline computer for regression calculations or prepare a graphical representation.

Some instruments may be adjusted to give a read-out directly in concentration of the analyte. A graph of instrument response versus concentration should be plotted to check the validity of the readings.

### 7.12 Expression of results

Using the appropriate calibration curve, determine the concentration of the element being determined in the test solution and convert it in milligrams per litre.

Calculate the element being determined [mass fraction ($W_X$) in percent (%)] according to Formula (1):

$$Wx = \frac{\rho \times V \times F}{m} \times 10^{-4} \tag{1}$$

where

$\rho$ is the analyte concentration, in milligrams per litre, found in the test solution less the blank;

$V$ is the volume, in millilitres, of the initial test solution;

$F$ is the dilution factor;

$m$ is the mass, in grams, of the test portion.

### 7.13 Precision

#### 7.13.1 General

The approximate composition, expressed as a percentage (mass fraction), of the samples used for testing the methods described is given in Table 16.

**Table 16 — Composition of the samples**

| Sample | Al | Co | Cr | Cu | Fe | Mn | Ni | Si | Ti |
|---|---|---|---|---|---|---|---|---|---|
| 825 | 0,2 | 0,07 | 21 | 1,6 | 30 | 0,7 | Reminder | 0,4 | 1,1 |
| 902 | 0,4 | 0,05 | 5 | 0,04 | 48 | 0,4 | Reminder | 0,35 | 2,5 |
| 3 920 | 0,15 | 2 | 19 | 0,1 | 3 | 0,3 | Reminder | 0,6 | 2,3 |
| 3 927 | 0,1 | 1 | 20 | 0,05 | 44 | 0,4 | Reminder | 0,8 | 0,6 |
| 7 013 | 1,5 | 17 | 20 | 0,2 | 0,2 | 0,05 | Reminder | 0,7 | 2,4 |
| 7 049 | 1 | 0,01 | 15 | 0,15 | 7 | 0,8 | Reminder | 0,3 | 2,3 |

For each element, samples were analyzed in duplicate on different days.

The data were tested for statistical outliers by the Cochran and Dixon tests described in ISO 5725. The principle of Cochran test is that a set of results is an outlier if the within-laboratory variance is too large in relation to others. Dixon’s test is to determine if the mean from a laboratory is too far from the other laboratory means.

Both tests were applied at the 95 % confidence level.

Repeatability and reproducibility were also calculated according to ISO 5725 at the 95 % confidence level.

For each element, the mean values and the precision data are expressed as a percentage (mass fraction).

### 7.13.2 Precision for cobalt

12 laboratories in six countries participated in the testing of this procedure using five samples (see Table 16).

The precision data are summarized in Table 17.

**Table 17 — Precision data**

| Sample | Mean | Within-laboratory standard deviation | Between laboratory standard deviation | Repeatability | Reproducibility |
|---|---|---|---|---|---|
| 825 | 0,067 0 | 0,000 6 | 0,002 1 | 0,001 8 | 0,006 2 |
| 902 | 0,046 0 | 0,001 2 | 0,001 7 | 0,003 5 | 0,005 0 |
| 3 920 | 2,010 | 0,029 | 0,029 | 0,085 | 0,085 |
| 3 927 | 1,030 | 0,016 | 0,022 | 0,047 | 0,065 |
| 7 049 | 0,007 2 | 0,000 2 | 0,000 9 | 0,000 6 | 0,002 7 |

### 7.13.3 Precision for chromium

There is no precision data available.

### 7.13.4 Precision for copper

12 laboratories in six countries participated in the testing of this procedure using six samples (see Table 16).

The precision data are summarized in Table 18.

**Table 18 — Precision data**

| Sample | Mean | Within-laboratory standard deviation | Between laboratory standard deviation | Repeatability | Reproducibility |
|---|---|---|---|---|---|
| 825 | 1,620 | 0,006 | 0,018 | 0,018 | 0,053 |
| 902 | 0,036 0 | 0,000 4 | 0,001 2 | 0,001 2 | 0,003 5 |
| 3 920 | 0,110 | 0,003 | 0,005 | 0,009 | 0,015 |
| 3 927 | 0,054 0 | 0,000 9 | 0,002 2 | 0,002 7 | 0,006 5 |
| 7 013 | 0,200 | 0,002 | 0,004 | 0,006 | 0,012 |
| 7 049 | 0,160 | 0,003 | 0,005 | 0,009 | 0,015 |

### 7.13.5 Precision for iron

11 laboratories in six countries participated in the testing of this procedure using two samples (see Table 16).

The precision data are summarized in Table 19.

**Table 19 — Precision data**

| Sample | Mean | Within-laboratory standard deviation | Between laboratory standard deviation | Repeatability | Reproducibility |
|---|---|---|---|---|---|
| 3 920 | 3,00 | 0,03 | 0,05 | 0,09 | 0,15 |
| 7 013 | 0,210 | 0,002 | 0,006 | 0,006 | 0,018 |

### 7.13.6 Precision for manganese

11 laboratories in six countries participated in the testing of this procedure using six samples (see Table 16).

The precision data are summarized in Table 20.

**Table 20 — Precision data**

| Sample | Mean | Within-laboratory standard deviation | Between laboratory standard deviation | Repeatability | Reproducibility |
|---|---|---|---|---|---|
| 825 | 0,740 | 0,015 | 0,027 | 0,044 | 0,080 |
| 902 | 0,370 | 0,003 | 0,017 | 0,009 | 0,050 |
| 3 920 | 0,280 | 0,004 | 0,009 | 0,012 | 0,027 |
| 3 927 | 0,390 | 0,005 | 0,016 | 0,015 | 0,047 |
| 7 013 | 0,058 0 | 0,000 7 | 0,001 6 | 0,002 1 | 0,004 7 |
| 7 049 | 0,810 | 0,005 | 0,007 | 0,015 | 0,021 |

## 8 Test report

The test report shall contain the following information:

a) method used by reference to this part of ISO 7530, i.e. ISO 7530-1;

b) all information necessary for the identification of the sample, the laboratory, and the date of analysis or of the test report;

c) results and the units in which they are expressed;

d) any unusual characteristics noted during the determination;

e) any operation not specified in this part of ISO 7530 or any operation which might have influenced the results;

f) signature of the responsible person.

# Annex A
(normative)

# Procedures for checking the instrumental criteria

## A.1 General

The performance of atomic absorption spectrometers of the same or different manufactures may vary from instrument to instrument. It is therefore essential to establish that a particular instrument meets certain performance requirements before it is used for the determinations described in this part of ISO 7530.

## A.2 Spectrometer performance check

### A.2.1 Determination of minimum precision

Spray the most concentrated calibration solution 10 times to obtain 10 individual absorbance readings, $A_{\mathrm{X,max,i}}$, and calculate the corresponding mean value, $A_{\mathrm{X,max}}$, and standard deviation, $s_{\mathrm{X,max}}$.

Spray the least concentrated calibration solution (excluding the zero member) 10 times to obtain 10 individual absorbance readings, $A_{\mathrm{X,min,I}}$, and calculate the corresponding mean value, $A_{\mathrm{X,min}}$, and standard deviation, $s_{\mathrm{X,min}}$.

The minimum precisions of the most and least concentrated calibration solutions are obtained from $s_{\mathrm{X,max}} \times 100/A_{\mathrm{X,max}}$ and $s_{\mathrm{X,min}} \times 100/A_{\mathrm{X,min}}$, respectively.

The standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution shall not exceed 1,5 % of the corresponding mean absorbance.

The standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

### A.2.2 Determination of limit of detection

The limit of detection is a number, expressed in units of concentration (or amount), that describes the lowest concentration level (or amount) of an element that can be determined to be statistically different from an analytical blank.

Prepare two solutions each containing the same matrix concentration as the sample solution, but with the element of interest (X) at the following known concentrations:

- $C_{\mathrm{X,A0,01}}$ (µg/ml) to give an absorbance, $A_{\mathrm{X,A0,01}}$, of approximately 0,01;
- the “zero member” giving an absorbance, $A_{\mathrm{X,0}}$.

Alternatively, spray the “zero member” solution and the $C_{\mathrm{X,A0,01}}$ solution 10 times recording each reading for about 10 s and using sufficient scale expansion to make the fluctuations in signal clearly visible.

Calculate the following:

- the mean absorbance ($A_{\mathrm{X,0}}$) and standard deviation ($s_{\mathrm{X,0}}$) corresponding to the “zero member” solution readings;
- the mean absorbance ($A_{\mathrm{X,A0,01}}$) corresponding to the $C_{\mathrm{X,A0,01}}$ solution readings.

Taking into account the statistical risks α and β each of them at the 95 % level (4,65), calculate the limit of detection ($C_{\mathrm{lim}}$) according to Formula (A.1).

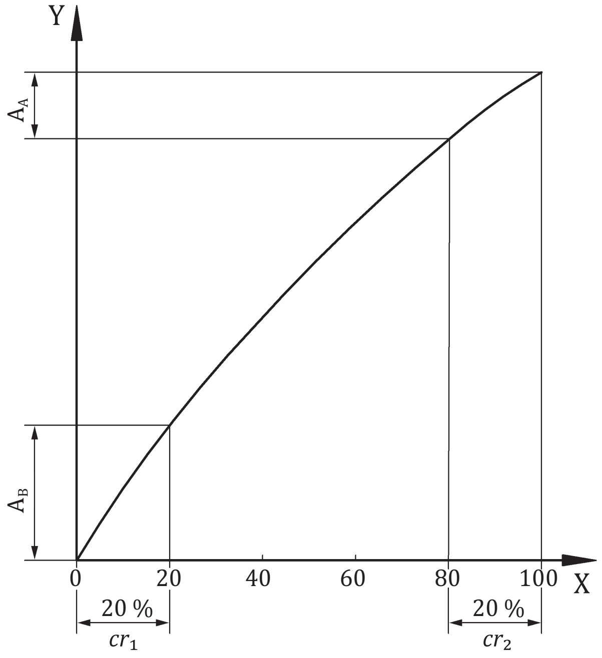
$$C_{\mathrm{lim}} = \frac{4{,}65 \times s_{\mathrm{X,0}} \times C_{\mathrm{X,A0,01}}}{A_{\mathrm{X,A0,01}} - A_{\mathrm{X,0}}} \qquad \text{(A.1)}$$

The value placed on this detection limit should be specified with reference to the lowest concentration of the element of interest likely to be encountered in the application envisaged. In the ideal case, the specified limit of detection should be less than one-tenth of the lowest concentration level to be determined and should be measured in the same matrix.

### A.2.3 Criterion for graph linearity

Having established the calibration graph (see Figure A.1) before the application of any curve straightening device, obtain the net absorbance value, $A_{\mathrm{A}}$, corresponding to the top 20 % of the concentration range and the net absorbance, $A_{\mathrm{B}}$, corresponding to the bottom 20 % of the concentration range.

Calculate $A_{\mathrm{A}}/A_{\mathrm{B}}$. This should not be less than 0,7. If this ratio is less than 0,7, further adjustments to the instrument may give acceptable results. Otherwise, the operating range of the method shall be reduced by lowering the concentration of the calibration solution of highest concentration.



**Key**

X net absorbance

Y concentration scale

$cr_1$ bottom 20 % of the concentration range

$cr_2$ top 20 % of the concentration range

Figure A.1 — Linearity criterion

### A.2.4 Determination of characteristic concentration, $c_{\mathrm{X,cc}}$

Prepare a solution containing the same matrix concentration as the sample solution, but with the element of interest at a known concentration, $C_{\mathrm{X,A0,1}}$ (μg/ml), to give an absorbance, $A_{\mathrm{X,A0,1}}$, of approximately 0,1.

Spray the $C_{X,A0,1}$ and the “zero member” solutions without scale expansion and measure the absorbances $A_{X,A0,1}$ and $A_{X,0}$.

The characteristic concentration, $c_{X,cc}$, is given by Formula (A.2).

$$C_{X,cc} = \frac{C_{X,A0,1} \times 0,0044}{A_{X,A0,1} - A_{X,0}} \qquad \text{(A.2)}$$

NOTE It has been stated in the literature that the optimum working range of an atomic absorption spectrometer is from 20 to 125 times the characteristic concentration.

# Bibliography

[1] ISO 5725:1986[1], *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*

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1) This standard is withdrawn and replaced by ISO 5725:1994.

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