

**Copper and zinc sulfide concentrates — Determination of silver content —  
Acid digestion and flame atomic absorption spectrometric or inductively  
coupled plasma atomic emission spectrometric method**

The parts changed from the 7th edition are shown in red.

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# Copper and zinc Concentrates - Determination of silver content - Acid digestion and flame atomic absorption spectrometric or inductively coupled plasma atomic emission spectrometric method

**WARNING** — This Standard may involve hazardous materials and equipment. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices, and determine the applicability of regulatory limitations prior to use.

## 1 Scope

This International Standard specifies an acid digestion and flame atomic absorption spectrometry (AAS) method, or inductively coupled plasma atomic emission spectrometry (ICP-AES) method for the determination of the mass fraction of silver in copper and zinc sulfide concentrates as follows:

The method is applicable to concentrates having silver contents in the following ranges

Copper concentrates	10 g/t to 800 g/t
Zinc concentrates	10 g/t to 800 g/t

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Working Draft. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Working Draft are encouraged to investigate the possibility of applying the most recent editions of the Standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648, Laboratory glassware — Single-volume pipettes

ISO 1042, Laboratory glassware — One-mark volumetric flasks

ISO 3696, Water for analytical laboratory use—Specification and test methods.

ISO 4787, Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use

ISO 8466-2, Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions

ISO 9599, Copper, lead, zinc and nickel sulfide concentrates — Determination of hygroscopic moisture content of the analysis sample — Gravimetric method

ISO 12743, Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content

ISO Guide 35, Reference materials — General and statistical principles for certification

### 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at [https:// www .iso .org/ obp](https://www.iso.org/obp)

— IEC Electropedia: available at [http:// www .electropedia .org/](http://www.electropedia.org/)

### 4 Principle

The test portion is decomposed by acid digestion using nitric and hydrochloric acids. Silver is determined by comparison against matrix-matched standards using AAS or ICP-AES.

### 5 Reagents

During the analysis only reagents of analytical grade and grade 2 water of ISO 3696 shall be used. Particular care should be taken to ensure all reagents are silver free except for 5.1.1, 5.3.1, 5.3.2 and 5.3.3.

#### 5.1 Solids

5.1.1 Silver metal, 99,99 %

5.1.2 Copper metal, 99,99% purity, (<0,000 05% silver)

Should be taken care in choice of the copper metal. Because normal copper cathode contain about 0,001% silver

5.1.3 Zinc metal ( $\geq 99,99$  %), (<0,000 05% silver)

5.1.4 Iron metal ( $\geq 99,99$  %), (<0,000 05% silver)

#### 5.2 Solutions

5.2.1 Nitric acid ( $\rho_{20} = 1,38$  g/ml)

5.2.2 Nitric acid, dilute (1 + 1)

Slowly add 500 ml of nitric acid (5.2.1) to 500 ml of water while stirring.

5.2.3 Hydrochloric acid ( $\rho_{20} = 20,16$  g/ml)

5.2.4 Hydrochloric acid, dilute (1 +1)

Slowly add 250 ml of hydrochloric acid (5.2.3) to 250 ml of water while stirring.

5.2.5 Hydrochloric acid, dilute (1 +3)

Slowly add 250 ml of hydrochloric acid (5.2.3) to 750 ml of water while stirring.

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

### 5.2.7 Sulphuric acid, dilute (1 + 1)

Slowly add 100 ml of sulphuric acid (5.2.6) to 100 ml of water while stirring. The addition of sulphuric acid to water generates heat and shall be performed with adequate precautions.

### 5.2.8 Perchloric acid ( $\rho_{20} = 1.77$ g/ml)

### 5.2.9 Hydrofluoric acid ( $\rho_{20}= 1,13$ g/ml)

## 5.3 Standard Solutions

### 5.3.1 Primary silver standard (1000 $\mu\text{g/ml}$ )

Weigh 1,000 g of silver metal (5.1.1) into a 300 ml beaker. Add 50 ml of dilute nitric acid (5.2.2) and 50 ml of water and swirl to mix. Heat gently until nitrogen oxides are generated. Cool to room temperature. **Quantitatively transfer this solution into a 1000 ml volumetric flask**, dilute to the mark with water, mix. And store in an amber-colored glass container.

**Alternatively, purchase a suitable high-quality prepared standard solution.**

**NOTE :** There are the reference solutions with the metrological traceability secured.

### 5.3.2 Working silver standard A (100 $\mu\text{g/ml}$ )

**Pipette 20 ml of primary silver standard (5.3.1) into a 200 ml volumetric flask containing 100 ml of hydrochloric acid (5.2.3) and 50 ml of water and mix well. Allow to the solution to cool and then dilute to volume with water. Mix and store in an amber-colored glass container.**

**This solution should be freshly prepared. If any turbidity is present, then remake this standard.**

### 5.3.3 Working silver standard B (10 $\mu\text{g/ml}$ )

**Pipette 5 ml of primary silver standard (5.3.1) into a 500 ml volumetric flask containing 250 ml of dilute hydrochloric acid (5.2.4) and mix well. Allow to the solution to cool and then dilute to volume with water. Mix and store in an amber-colored glass container.**

**This solution should be freshly prepared. If any turbidity is present, then remake this standard.**

### 5.3.4 Copper matrix/interference standard solution (25,0 mg/ml).

Dissolve 5.00 g of copper metal (5.1.3) with 50 ml of dilute nitric acid (5.2.2). Heat and evaporate to approximately 25 ml to remove nitrogen oxides. Transfer to a 200 ml volumetric flask, add 100 ml of dilute hydrochloric acid (5.2.4), then fill up with water nearly to the mark, mix and cool to room temperature, then fill up exactly to the mark and mix again.

### 5.3.5 Zinc matrix/interference standard solution (25,0 mg/ml).

Dissolve 5.00 g of zinc metal (5.1.3) with 50 ml of dilute hydrochloric acid (5.2.4). Transfer to a 200 ml volumetric flask, add 50 ml of dilute hydrochloric acid (5.2.4), then fill up with water nearly to the mark, mix and cool to room temperature, then fill up to the mark and mix again.

### 5.3.6 Iron matrix/interference standard solution (25 mg/ml)

Dissolve 5,00 g of Iron metal (5.1.4) with 50 ml of dilute hydrochloric acid (5.2.4). Heat gently until the reaction is complete. Add 5 ml of nitric acid (5.2.1) and heat. Transfer to a 200 ml volumetric flask, add 50 ml of dilute hydrochloric acid (5.2.4), then fill up with water nearly to the mark, mix and cool to room temperature, then fill up to the mark and mix again.

## 6 Apparatus

Use laboratory glassware and equipment that are free of silver contamination.

### 6.1 Balance

Accurate and readable to  $\pm 0,0001$  g

### 6.2 Laboratory hot plate with temperature controller

### 6.3 Normal laboratory glassware

Complying with ISO 648 and ISO 1042 and used in accordance with ISO 4787.

### 6.4 Polytetrafluoroethylene beaker

200 ml capacity.

### 6.5 Insoluble filter paper

Whatman® No. 40 or equivalent.

### 6.6 Atomic absorption spectrometer (AAS)

### 6.7 Inductively coupled plasma atomic emission spectrometer (ICP-AES)

## 7 Instrument operating parameters

### 7.1 Analytical Parameters

A variety of AAS and ICP-AESs are available and the manufacturer's instructions supplied with the instrument should provide adequate information for their operation. Refer to the manufacturer's instructions for optimising AAS and ICP-AES. The instrument should be optimized to give maximum sensitivity and as near as practical to a linear relationship between absorbance or emission intensity and concentration.

### 7.2 The flame of AAS

When using the AAS, use air-acetylene (oxidizing) flame.

### 7.3 Measurement wavelength

The recommended measurement wavelengths are shown below.

AAS : 328,1 nm



ICP-AES : 328,068 nm

NOTE 1: The wavelength resolution of the ICP-AES differs depending on the type of device. Therefore, it is recommend that check for interference in advance and select an appropriate measurement wavelength. Higher order spectral lines may be selected in a device that should measure higher order spectral lines. When a suitable emission line without interference can not be selected, the interference amount shall be suitably corrected.

NOTE 2: The ICP-AES measurement wavelength which gives the emission intensity suitable for aiming determination range should be selected among the measurement wavelength of respective element. In this case, the detection limit, measuring accuracy, etc. should be sufficiently investigated.

NOTE 3 : There are two types of observation method of the ICP-AES from the emission part, which are the horizontal observation method and the axially observation method, and both of them may be used. However, the axially observation method has a narrower linear range of the calibration curve and more interference than the horizontal observation method.

#### **7.4 The background correction mechanism**

When using the AAS, a background correction mechanism shall be used. On the other hand, when using the ICP-AES, a background correction mechanism may be used.

### **8 Sample and sample preparation**

#### **8.1 Laboratory sample**

Laboratory samples shall be taken and prepared in accordance with the procedures described in ISO 12743.

#### **8.2 Test sample**

Prepare an air-equilibrated test sample and a hygroscopic moisture test sample in accordance with ISO 9599.

NOTE : A test sample is not required if pre-dried test portions are used, see Annex B.

### **9 Procedure**

#### **9.1 Number of Determinations**

Carry out the determination at least in duplicate, as far as possible under repeatability conditions, on each test sample.

NOTE : Repeatability conditions exist where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

#### **9.2 Test portion**

Taking multiple increments, extract approximately 1,0 g from the test sample and weigh to the nearest 0,1 mg. At the same time, as the test portions are being weighed for analysis, weigh test portions for the determination of hygroscopic moisture in accordance with ISO 9599.

Alternatively, the method specified in Annex B may be used to prepare pre-dried test portions directly from the laboratory sample.

Obtain an approximate content for the copper, zinc, and iron in the sample as required in step 9.8.

### 9.3 Blank test

Carry out a blank test in parallel with the analysis using the same quantities of all reagents but omitting the sample. **The result in  $\mu\text{g}$  of silver in the blank test will be subtracted from the result of samples.**

The purpose of the blank test is to check the atmosphere used in the test, apparatus contamination and reagent purity.

### 9.4 Dissolution of the test portion

**Decompose the test portion in accordance with step 9.4.1 or 9.4.2.**

#### 9.4.1 Dissolution of the test portion with nitric acid and hydrochloric acid

Quantitatively, transfer the test portion into a 300 ml beaker. Add cautiously 20 ml of dilute nitric acid (5.2.2). Cover with a watch glass and leave the sample at room temperature for 30 min, then place the beaker on the hotplate and heat slowly from room temperature to 90 °C. Expel oxides of nitrogen. Rinse the watch glass with a small amount of water and collect the washing solutions to the dissolved solutions. Remove the watch glass, then evaporate to approximately 10 ml. Add 5 ml of hydrochloric acid (5.2.3), and heat gently and evaporate to near dryness. Remove the beaker from the hotplate.

NOTE 1: Rapid heating may cause the free sulfur to deposit, the free sulfur holding some of the silver ions.

In the case of free sulfur be deposited with gently heating, should be carried out the dissolution in step 9.4.2.

NOTE 2: Silver oxide may form if heating is continued to the point of dryness of the solution. Silver oxide is difficult to dissolve by the procedure described in the next step.

**Cover with watch glass again.** Cool and cautiously add 20 ml of dilute hydrochloric acid (5.2.5). Heat the solution to boiling, then cool to room temperature. Rinse the watch glass with dilute hydrochloric acid (5.2.5) and collect the washing solutions to the dissolved solutions, then remove the watch glass. Filter the solution through an insolubles filter paper (6.5) into a clean volumetric flask that is specified in step 9.6. Thoroughly wash the 300 ml beaker used to digest the test portion and filter paper with dilute hydrochloric acid (5.2.5) and collect the washing solutions into the same volumetric flask.

If acid insoluble material is present, then treat this residue as the procedure in 9.5, otherwise proceed to step 9.6.

#### 9.4.2 Alternative dissolution of the test portion

Quantitatively transfer the test portion into a 300 ml beaker. Add cautiously 20 ml of dilute nitric acid (5.2.2). Cover with a watch glass and place the beaker on the hotplate and heat gently until all nitrogen oxides are expelled. Cautiously add 5 ml of perchloric acid (5.2.8). Continue heating until dense fumes of perchloric acid condense and drain down the sides of the beaker. Remove the watch glass and evaporate to dryness, then remove the beaker from the hotplate immediately.

NOTE 1: Ensure that the temperature of the hotplate is between 200 °C and 220 °C.

NOTE 2: Silver oxide may form if heating is continued even after the point of dryness of the solution. Silver oxide is difficult to dissolve by the procedure described in the next step.

**Cover with watch glass again.** Cool and cautiously add 20 ml of dilute hydrochloric acid (5.2.5). Heat the solution to boiling, then cool to room temperature. Rinse the watch glass with dilute hydrochloric acid (5.2.5) and collect the washing solutions to the dissolved solutions, then remove the watch glass. Filter the solution through an insolubles filter paper (6.5) into a clean volumetric flask that is specified in step 9.6. Thoroughly wash the 300 ml beaker used to digest the test portion and filter paper with dilute hydrochloric acid (5.2.5) and collect the washing solutions into the same volumetric flask.

## 9.5 Dissolution of the insoluble residue

Quantitatively, transfer the acid insoluble residue into a 200 ml polytetrafluoroethylene beaker (6.4) with a small quantity of water. Add 5 ml of nitric acid (5.2.1), 5 ml of dilute sulphuric acid (5.2.7) and 3 ml to 5 ml of hydrofluoric acid (5.2.9). Heat the solution until enough white sulfuric acid gas is evolved to remove the silicon dioxide, then allow to cool. Add 20 ml of dilute hydrochloric acid (5.2.5) and cover the beaker with a watch glass. Heat the solution to boiling, then cool to room temperature. Rinse the watch glass and collect the washing solutions to the dissolved solutions.

If the insoluble material remains after this treatment, filter through an insoluble filter paper(6.5), then wash the insoluble material and the filter paper with dilute hydrochloric acid (5.2.5), and collect the filtrate and the washing solution to the dissolved solutions. Reject insoluble material and the filter paper.

NOTE : Should the absence of silver in the acid-insoluble residue be confirmed, the procedure of dissolution of the insoluble residue may be omitted.

Proceed to step 9.6.

## 9.6 Preparation of test solutions

Prepare the test solutions in accordance with step 9.6.1 or 9.6.2.

### 9.6.1 Atomic absorption spectrometer (AAS)

Quantitatively transfer the solution from 9.4.1 or 9.4.2 and 9.5 to volumetric flask of the volume specified in Table 1. Dilute to about 3/4 with dilute hydrochloric acid (5.2.5). Allow the solution to cool to room temperature, then dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

Table 1 — Volume of test solution for AAS

Silver content of sample (g/t)	Volume of volumetric flask (ml)
≤200	100
200 to 400	200
400 to 800	500

### 9.6.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES)

Quantitatively transfer the solution from 9.4.1 or 9.4.2 and 9.5 to 100 ml volumetric flask. Dilute to about 3/4 with dilute hydrochloric acid (5.2.5). Allow the solution to cool to room temperature, then dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

## 9.7 Preparation of the calibration solutions

Prepare the calibration solutions in accordance with 9.7.1 or 9.7.2.

### 9.7.1 Atomic absorption spectrometer (AAS)

Using working silver standard B (5.3.3), prepare a series of calibration solutions as per Table 2. Transfer the appropriate volumes of working standard solutions using pipettes, into each 100 ml volumetric flasks containing:

1) sufficient matrix/interference standard to match the major element in the concentrate being measured. For copper sulfide concentrates, use copper matrix/interference standard (5.3.4). For zinc sulfide concentrates, use zinc matrix/interference standard (5.3.5).

2) sufficient iron matrix/interference standard (5.3.6) to match the iron content of the samples;

Dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

NOTE1: Where silver is determined using AAS, if the interference from matrix is not appeared, addition of matrix / interference standard may be omitted.

NOTE 2: The linearity of the calibration curve of the AAS differs depending on the type of device. Therefore, it is recommend to confirm the linearity in advance, taking into consideration the concentration range of the analytical sample. Depending on the result, the number and the concentration of the calibration solutions in Table 2 may be changed.

**Table 2 — Example of calibration Solutions for AAS**

<b>Volume of working silver standard B (5.3.3), ml</b>	<b>Silver mass, µg</b>	<b>Concentration of silver, µg/ml</b>
0	0	0
5	50	0,50
10	100	1,00
15	150	1,50
20	200	2,00

**9.7.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES)**

Using working silver standard A (5.3.2) and working silver standard B (5.3.3) , prepare a series of calibration solutions as per Table 3. Transfer the appropriate volumes of working standard solutions using pipettes, into each 100 ml volumetric flasks containing:

1) sufficient matrix/interference standard to match the major element in the concentrate being measured. For copper sulfide concentrates, use copper matrix/interference standard (5.3.4). For zinc sulfide concentrates, use zinc matrix/interference standard (5.3.5).

2) sufficient iron matrix/interference standard (5.3.6) to match the iron content of the samples;

Dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

NOTE : The linearity of the calibration curve of the ICP-AES differs depending on the type of device. Therefore, it is recommend to confirm the linearity in advance, taking into consideration the concentration range of the analytical sample. Depending on the result, the number and the concentration of the calibration solutions in Table 3 may be changed.

**Table 3 — Example of calibration Solutions for ICP-AES**

<b>Volume of working silver standard A (5.3.2), ml</b>	<b>Volume of working silver standard B (5.3.3), ml</b>	<b>Silver mass, µg</b>	<b>Concentration of silver, µg/ml</b>
-	0	0	0
-	10	100	1,00
-	20	200	2,00
5	-	500	5,00
7	-	700	7,00
10	-	1000	10,00

## 9.8 Preparation of silver calibration curve

Prepare the silver calibration curve in accordance with 9.8.1 or 9.8.2.

### 9.8.1 Atomic absorption spectrometer (AAS)

Set up the AAS (5.6) according to the guidelines set out in Step 7.

Adjust the instrument read-out scale to zero. Atomize each calibration solutions as prepared in step 9.7.1 into air-acetylene flame of an AAS and record the absorbance of silver. Manually or electronically plot a relation curve between the absorbance and the mass of silver (in µg) as calibration curve graph .

Use ISO 8466-2 as a guide to determine the acceptability of the calibration curve.

### 9.8.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES)

Set up the ICP-AES (5.7) according to the guidelines set out in Step 7.

Atomize each calibration solutions as prepared in step 9.7.2 into argon plasma of an ICP-AES and measure the emission intensities of silver. Manually or electronically plot a relation curve between the emission intensities and the mass of silver (in µg) as calibration curve graph. It is recommended that the solution introduction unit be rinsed with dilute hydrochloric acid (5.2.5) after each calibration solutions measurement.

Use ISO 8466-2 as a guide to determine the acceptability of the calibration curve.

## 9.9 Determination of silver content in test solutions

Determine the silver content in test solutions in accordance with 9.9.1 or 9.9.2.

### 9.9.1 Atomic absorption spectrometer (AAS)

Immediately after calibrating the AAS, determine the silver content in the test solutions from step 9.6.1

Adjust the instrument read-out scale to zero. Atomize each test solutions as prepared in step 9.6.1 into air-acetylene flame of an AAS and record the absorbance of silver.

NOTE 1: The calibration curve may vary due to long-term continuous operation of AAS or the accumulated number of measurements. In such a case, for accurate determination, the calibration curve should be corrected by measuring the solution for correction of calibration curve at constant intervals or every certain number of measurements.

NOTE 2: If more than 10 µg of silver is found for the blank test, then this shall be investigated and the results for the samples shall not be reported.

### 9.9.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES)

Immediately after calibrating the ICP-AES, determine the silver content in the test solutions from step 9.6.2

Atomize each test solutions as prepared in step 9.6.2 into argon plasma of an ICP-AES and record the emission intensities of silver. It is recommended that the solution introduction unit be rinsed with dilute hydrochloric acid (5.2.5) after each test solution measurement.

NOTE 1: The calibration curve may vary due to long-term continuous operation of ICP-AES or the accumulated number of measurements. In such a case, for accurate determination, the calibration curve should be corrected by measuring the solution for correction of calibration curve at constant intervals or every certain number of measurements.

NOTE 2: If more than 10 µg of silver is found for the blank test, then this shall be investigated and the results for the samples shall not be reported.

## 10 Calculations

Calculate the silver content of the sample ( $W_{Ag}$ ) according to the following formula:

$$W_{Ag}(g/t) = (F_E * V/100 - F_{E\ blank})/M * K \quad \dots (1)$$

Where

$F_E$  = is the mass of silver found in the test solutions, in µg. (9.9)

$F_{E\ blank}$  = is the mass of silver found in the blank test, in µg. (9.3)

$V$  = is the final volume of the test solutions, in ml. (9.6)

100 = is the final minimum volume of the test solutions, in ml. (9.6)

$M$  = is the mass of the test portion, in g. (9.2)

$K$  = Hygroscopic moisture conversion factor determined as below:

$$K = 100/(100 - H) \quad \dots (2)$$

Where

$H$  = The hygroscopic moisture content of the sample determined by ISO 9599

If pre-dried test samples are used (according to Annex B)  $H = 0$

## 11 Procedure for obtaining the final result

Process the duplicate results for each test portion of the sample according to the chart in Annex A and repeat the determination for a sample as necessary.

## 12 Test report

12.1 Once all the requirements in Clause 10.1 have been achieved the result can be reported.

12.2 The test report shall contain the following information:

- a) Identification of the sample;
- b) Reference to this International Standard;
- c) Silver content of the sample, expressed as g/t;
- d) Date on which the test was carried out'
- e) Any occurrences noticed during the determination which may have had an influence on the results.

## 13 Precision and accuracy

### 13.1 Expression of precision

The precision of this analytical method is expressed by the following equations:

$$S_r = aX + b \quad \dots (3)$$

$$S_L = a|X + b| \quad \dots (4)$$

where

$X$  is the concentration of silver, in g/t, in the sample.

$S_r$  is the within laboratory standard deviation, in g/t.

$S_L$  is the between laboratories standard deviation, in g/t.

NOTE: Additional information is given in Annex D.

### 13.2 Procedure for obtaining the final result (Annex A)

Calculate the following quantities from the duplicate results  $X_1$  and  $X_2$  (g/t) and process according to the chart in Annex A:

$$\text{Mean of duplicates:} \quad x = (X_1 + X_2)/2 \quad \dots (5)$$

$$\text{Within laboratory standard deviation (repeatability)} \quad S_r = aX + b \quad \dots (6)$$

$$\text{Repeatability limit} \quad r = 2,8S_r \quad \dots (7)$$

### 13.3 Between laboratories precision

Between laboratories precision is used to determine the agreement between the results reported by two (or more) laboratories. The assumption is that all laboratories followed the same procedure.

Calculate the following quantities:

$$\text{Mean of final results:} \quad \mu_{1,2} = (\mu_1 + \mu_2)/2 \quad \dots (8)$$

$$\text{Between laboratories standard} \quad S_L = a|\mu_{1,2} + b| \quad \dots (9)$$

$$\text{Within laboratory standard deviation} \quad S_r = a\mu_{1,2} + b \quad \dots (10)$$

$$\text{Permissible tolerance} \quad P = 2,8\sqrt{S_L^2 + S_r^2}/2 \quad \dots (11)$$

$$\text{Range} \quad E = |\mu_1 - \mu_2| \quad \dots (12)$$

where

$\mu_1$  is the final result, in g/t, reported by laboratory 1

$\mu_2$  is the final result, in g/t, reported by laboratory 2.

If E is equal to or less than P, the final results are in agreement.

### 13.4 Check of trueness

The trueness of the analytical method can be checked by applying it to a certified reference material (CRM). When the precision has been confirmed, the final laboratory result can be compared with the certified value,  $A_c$ . There are two possibilities as follows:

$$|\mu_c - A_c| \leq C \quad \dots (13)$$

If this condition exists, the difference between the reported result and the certified value is statistically significant.

$$|\mu_c - A_c| > C \quad \dots (14)$$

If this condition exists, the difference between the reported results and the certified value is statistically insignificant.

where

$\mu_c$  is the final result, in g/t, of the certified reference material.

$A_c$  is the certified value, in g/t, of the certified reference material.

C is a quantity, in g/t, depending on the type of certified reference material used as defined in Clause 13.4.1.1.

#### 13.4.1 Type of certified reference material (CRM) or reference material (RM)

Reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35.



**13.4.1.1 Reference material certified/characterised by inter-laboratory test programme**

$$C = 2\sqrt{S_L^2 + S_r^2/n + S^2\{A_c\}} \quad \dots (15)$$

where

$S^2\{A_c\}$  is the variance of the certified value

$n$  is the number of replicate determinations

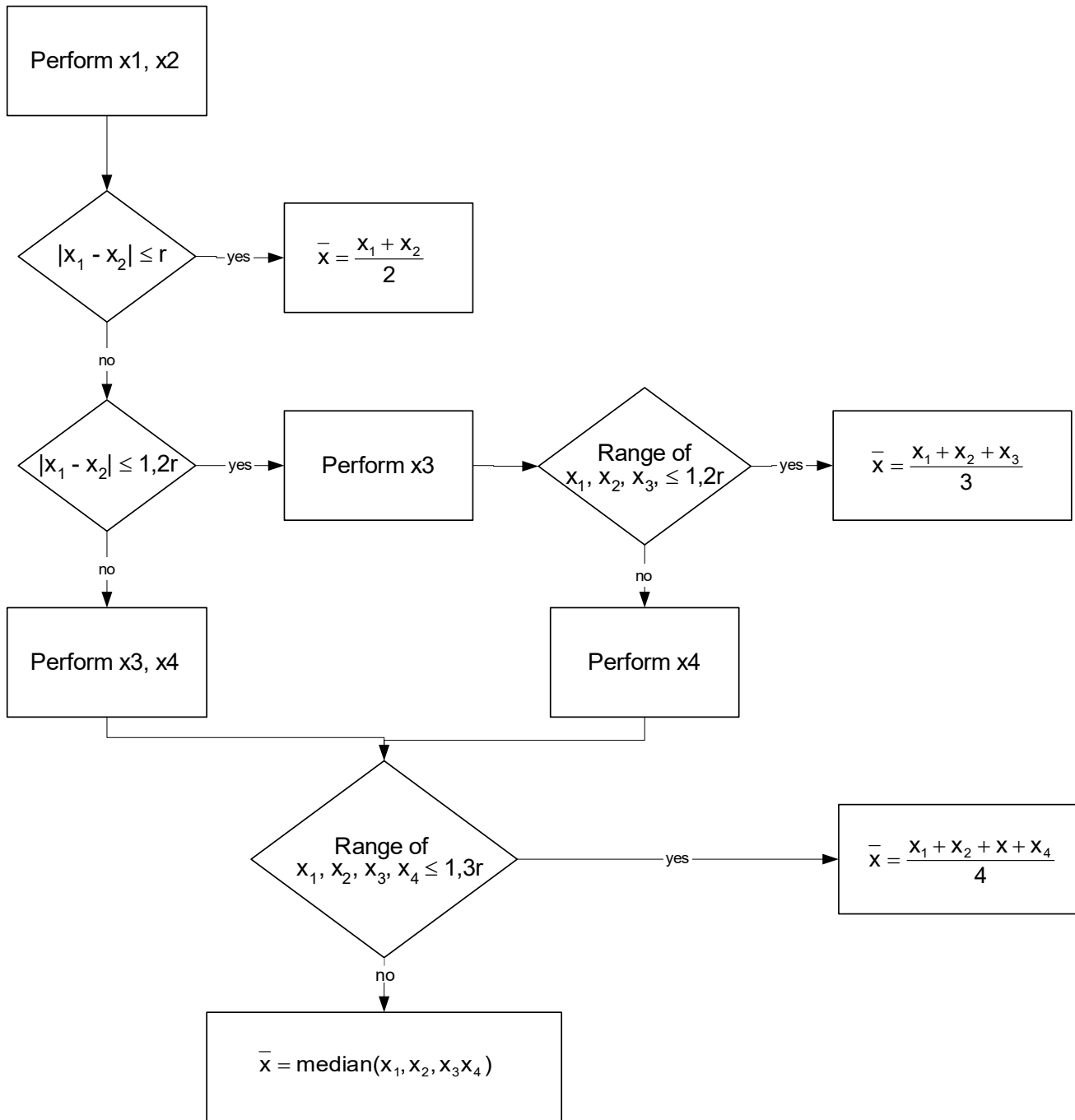
**13.4.1.2 Reference material certified/characterised by one laboratory**

$$C = 2\sqrt{2S_L^2 + S_r^2/n} \quad \dots (16)$$

NOTE: It is recommended that this type of certified reference material should be avoided unless the particular CRM is known to have unbiased certified value.

**Annex A  
(normative)**

**Procedure for Obtaining the Final Results**



## **Annex B (normative)**

### **Procedure for the preparation and determination of the mass of a predried test portion**

#### **B0 Scope**

This procedure sets out a method for the preparation and determination of the mass of a predried test portion in the analysis of copper, lead and zinc sulphide concentrates. The method is applicable to copper, lead and zinc sulphide concentrates not susceptible to oxidation and with hygroscopic moisture contents ranging from 0.05 % to 2 %.

#### **B.1 Principle**

The test portion to be used for analysis is dried in air in an oven maintained at  $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$ . The dried test portion is then weighed and used for the analysis. No correction for moisture is required.

#### **B.2 Reagent**

**B.2.1** Desiccant such as self-indicating silica gel or anhydrous magnesium perchlorate.

**WARNING:** Care should be taken when disposing of exhausted magnesium perchlorate. It should be washed down the sink with a stream of running water.

#### **B.3 Apparatus**

Ordinary laboratory equipment and

**B.3.1** Analytical balance, sensitivity to 0.1 mg.

**B.3.2** Weighing vessel, of glass or silica or corrosion resistant metal with externally-fitting air-tight covers. For small test portions (less than 3 g) the mass of the vessel should be as small as possible, i.e. less than 20 g.

**B.3.3** Laboratory oven, capable of maintaining a temperature of  $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$ .

#### **B.4 Procedure**

##### **B.4.1 Preparation of the weighing vessel**

Dry the weighing vessel and vessel cover (B.3.2) by heating in a laboratory oven (B.3.3) at  $105^{\circ}\text{C}$  for 1h. Transfer the vessel and vessel cover to a desiccator containing suitable fresh desiccant (B.2.1) and allow to cool to ambient temperature.

##### **B.4.2 Test portion**

Tare the dried weighing vessel and vessel cover (B.4.1). Immediately add a portion of the laboratory sample to provide a suitable predried test portion. An accurate mass of the test portion and weighing vessel is not required at this point.

**B.4.3 Determination of the test portion dry mass**

Transfer the uncovered weighing vessel and test portion and vessel cover to the laboratory oven (B.3.3) and dry at 105°C ± 5°C for 2 h. After the 2 h period, remove the weighing vessel and dry test portion from the oven, replace the vessel cover and allow to cool to ambient temperature in the desiccator. When cool, remove the weighing vessel and dry test portion and vessel cover from the desiccator and weigh to the nearest 0.1 mg (M<sub>1a</sub>) after slightly lifting the cover and quickly replacing it. Transfer the test portion into the appropriate analytical apparatus and immediately re-weigh the empty weighing vessel and vessel cover. Record the mass to the nearest 0.1 mg (M<sub>2</sub>).

NOTE:B1 For new concentrates of unknown characteristics it is advisable to repeat the drying for another 2 h at 105°C ± 5°C and to re-weigh the weighing vessel and test portion plus vessel cover to the nearest 0.1 mg (M<sub>1b</sub>). The test portion can be considered stable if the difference between M<sub>1a</sub> and M<sub>1b</sub> is within ± 0.5 mg. If this condition is not achieved, the drying and weighing steps should be repeated.

**B.5 Calculation of the test portion dry mass**

The dry mass of the test portion (M<sub>3</sub>) is calculated from the formula:

$$M_3 = M_{1a} - M_2 \qquad \dots (B1)$$

where

M<sub>1a</sub> = mass, in grams, of the dried test portion plus weighing vessel plus cover

M<sub>2</sub> = mass, in grams, of the empty weighing vessel plus vessel cover

M<sub>3</sub> = mass, in grams, of the dry test portion

The mass of the dry test portion is the mass to be used to calculate the element concentration in the laboratory sample on a dry basis. No correction for hygroscopic moisture is required.