INTERNATIONAL STANDARD

ISO 13547-1

First edition
2014-11-01

Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic —

Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method

Concentrés sulfurés de cuivre, de plomb et de zinc — Dosage de l'arsenic —

Partie 1: Méthode par digestion acide, co-précipitation avec le fer et plasma induit par haute fréquence

Reference number
ISO 13547-1:2014(E)

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Published in Switzerland

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

ISO 13547 consists of the following parts, under the general title *Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic:*

— *Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method*

— *Part 2: Acid digestion and inductively coupled plasma atomic emission spectrometric method*

Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic —

Part 1:
Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method

WARNING — This International Standard may involve hazardous materials, operations, 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 iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric (ICP-AES) method for the determination of the mass fraction of arsenic in copper, lead, zinc, and nickel sulfide concentrates as follows:

a) for copper sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 2,0 %;

b) for lead sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 1,0 %;

c) for zinc sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 0,6 %;

d) for nickel sulfide concentrates, the method is applicable to the determination of mass fraction of arsenic from 0,05 % to 1,0 %.

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 648, *Laboratory glassware — Single-volume pipettes*

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

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 and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*

ISO 12743:2006, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

ISO Guide 35:2006, *Reference materials — General and statistical principles for certification*

3 Principle

The test portion is decomposed in nitric and sulphuric acids. Arsenic is coprecipitated in ammonia solution by the carriers of iron hydroxide to be separated from the matrix. The arsenic content is determined by comparison against iron-matched standards using ICP-AES.

4 Reagents

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

4.1 Sodium hydroxide (NaOH), AR grade.

4.2 Arsenic trioxide (As_2O_3), AR grade.

4.3 Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), AR grade (<0,000 05 % arsenic).

4.4 Ammonium chloride (NH_4Cl), AR grade.

4.5 Ammonium acetate, AR grade.

4.6 Nitric acid (ρ_{20} 1,42 g/mL), AR grade.

4.7 Nitric acid, dilute (1 + 1).

Slowly, add 50 ml of nitric acid (4.6) to 50 ml of water while stirring.

4.8 Hydrochloric acid (ρ_{20} 1,16 g/mL), AR grade.

4.9 Hydrochloric acid, dilute (1 + 1).

Slowly add 50 ml of hydrochloric acid (4.8) to 50 ml of water while stirring.

4.10 Sulphuric acid (ρ_{20} 1,840 g/mL), AR grade.

4.11 Sulphuric acid, dilute (1 + 1).

Slowly add 50 ml of sulphuric acid (4.10) to 50 ml of water while stirring.

4.12 Sulphuric acid, dilute (1 + 50).

Slowly add 20 ml of sulphuric acid (4.10) to 1 000 ml of water while stirring.

The addition of sulphuric acid to water generates heat and shall be performed with adequate precautions.

4.13 Hydrofluoric acid (ρ_{20} 1,13 g/mL).

4.14 Aqueous ammonia (ρ_{20} 0,91 g/mL).

4.15 Ammonia solution (1 + 99).

4.16 Iron(III) chloride solution (10 g/l).

Dissolve 48,4 g iron (III) chloride hexahydrate (4.3) in 200 ml of water. Add 10 ml of hydrochloric acid (4.8) and make up to 1 000 ml with water and mix.

4.17 Sodium hydroxide solution (20 % w/v).

Weigh 20,0 g of sodium hydroxide (4.1) in a polytetrafluoroethylene beaker (5.6). Add 50 ml of water and allow the solid to dissolve. Dilute the solution to 100 ml and store in a labelled polyethylene container.

4.18 Saturated solution of potassium chlorate in nitric acid.

Superfluous potassium chlorate is added into the nitric acid and then the deposited solution is allowed to form the saturated solution.

4.19 Ammonium acetate (25 % w/v).

Dissolve 25,0 g of ammonium acetate (4.5) into 100 ml water.

4.20 Arsenic standard solution, 1 ml contains 1 mg of As.

Weigh 1 320,3 g of arsenic trioxide (4.2) to the nearest 0,1 mg. Transfer to a 400 ml beaker and add 5 ml of sodium hydroxide solution (4.17). Warm slightly to dissolve. When dissolution is complete, cool and add 40 ml of nitric acid (4.6). Transfer to a 1 000 ml volumetric flask containing 60 ml of nitric acid (4.6). Fill up nearly to the mark with water, mix, and equilibrate at room temperature then fill up exactly to the mark and mix again. Store in a labelled container.

Alternatively, purchase a suitable high quality prepared standard.

Before use, compare this International Standard against a traceable National Standard to ensure suitability for use.

4.21 Arsenic standard solution, 1 ml contains 0,2 mg of As.

Pipette 40 ml of arsenic standard solution (4.20) into a 200 ml volumetric flask containing 2 ml hydrochloric acid (4.8). Fill up nearly to the mark with water, mix, and equilibrate at room temperature then fill up exactly to the mark and mix again. Store in a labelled glass container.

5 Apparatus

All laboratory glassware and equipment shall be shown to be free of arsenic contamination.

5.1 Balance, sensitive to ±0,000 1 g.

5.2 Ordinary laboratory glassware, complying with ISO 648 and ISO 1042 and used in accordance with ISO 4787.

5.3 Inductively coupled plasma atomic emission spectrometer (ICP-AES).

The emission wavelength should be set to 193,696 nm or 197,197 nm.

5.4 Insoluble filter paper, Whatman®[1)] No. 40 or equivalent.

5.5 Coprecipitation filter paper, Whatman®[1)] No. 1 or equivalent.

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO.

5.6 Polytetrafluoroethylene beaker, 200 ml capacity.

6 Sample

6.1 Laboratory sample

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

As arsenic may evaporate from samples at elevated temperatures, consideration should be given to preparing a separate chemical analysis sample as described in ISO 12743:2006, 16.2.

6.2 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 to be used (see Annex A).

6.3 Test portion

Taking multiple increments, extract approximately 0,5 g from the test sample and weigh to the nearest 0,1 mg. At the same time, as 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 A may be used to prepare pre-dried test portions directly from the laboratory sample.

Obtain an approximate concentration for the iron in the sample as required in step 7.6.

7 Procedure

7.1 Number of determinations

Carry out the determinations, 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.

7.2 Blank test

Carry out a blank test in parallel with the analysis using the same quantities of all reagents, but omitting the test portion. The purpose of the blank test in this method is to check the quality of reagents. If a significant value is obtained as a result of the blank test, check all reagents and rectify the problem.

7.3 Dissolution of the test portion

Transfer the test portion into a 300 ml narrow-necked conical beaker. Moisten with 5 ml of water. Add 15 ml of the saturated solution of potassium chlorate in nitric acid (4.18), cover the beaker with a watch glass, place the conical beaker on a hotplate, and heat gently until all nitrogen oxides are expelled. Add 15 ml of diluted sulphuric acid (4.11) and heat the solution until strong white fumes have evolved then cool.

If the residue appears dark (indicating the presence of carbon), slowly add a small amount of nitric acid (4.6) to the hot solution until the solution becomes colourless, then heat until strong white fumes have evolved.

Add 50 ml of water and bring to boil, then allow cooling.

Filter the solution through an insoluble filter paper (5.4) in a 250 ml conical beaker. Wash the 300 ml conical beaker and filter paper thoroughly with dilute sulphuric acid (4.12) and collect the filtrate in the same 250 ml conical beaker.

If acid-insoluble material is present, treat this residue as the procedure in 7.5, otherwise proceed to step 7.6.

In the case of lead concentrates, proceed to step 7.4.

7.4 Removal of lead sulfate

Quantitatively, transfer the precipitate into a 250 ml conical beaker with small amount of water. Add 15 ml of ammonium acetate solution (4.19) and place the conical beaker on the hotplate and heat until dissolution of the $PbSO_4$.

Cool the solution to room temperature and filter through an insoluble filter paper (5.4), then wash the insoluble residue with dilute sulphuric acid (4.12). Reject the filtrate and washing solution. If acid insoluble material is present, then treat this residue as the procedure in 7.5, otherwise proceed to step 7.6.

7.5 Dissolution of the insoluble residue

Quantitatively, transfer the insoluble residue into a 200 ml polytetrafluoroethylene beaker (5.6) with a small quantity of deionised water. Add 5 ml of nitric acid (4.6), 2 ml of dilute sulphuric acid (4.11) and 3 ml to 5 ml of hydrofluoric acid (4.8). Heat the solution until the evolution of white sulphuric acid fumes to remove silicon dioxide then allow to cool. Dissolve the soluble salts with 10 ml of deionised water and 10 ml of nitric acid (4.6). Proceed to step 7.6.

7.6 Separation of arsenic

Quantitatively, transfer the solution from step 7.5 and the filtrate from step 7.3 into a 300 ml conical beaker and dilute to approximately 150 ml with water. Add 5 g of ammonium chloride (4.4) to the solution and agitate to dissolve. Add sufficient iron chloride solution (4.16) to give a total mass of 150 mg of iron in the filtrate.

If the mass of iron from the test portion is above 150 mg, then do not add any iron chloride solution (4.16). Add sufficient aqueous ammonia (4.14) to effect complete precipitation then add an extra 10 ml of aqueous ammonia. Bring it to boil and leave boiling for 1 min.

Immediately, filter through the coprecipitation filter paper (5.5) and wash several times with warm ammonia solution (4.15). Store the coprecipitation filter paper in the original conical beaker.

Repeat the separation on the filtrate by evaporating the filtrate down to approximately 150 ml. Add 15 ml of dilute sulfuric acid (4.12) and mix, then add 5 g of ammonium chloride (4.4) to the solution and agitate to dissolve. Add sufficient iron chloride solution (4.16) to give a total mass of 150 mg iron in the filtrate. Add sufficient aqueous ammonia (4.14) to effect complete precipitation then add an extra 10 ml of aqueous ammonia. Bring to boil and leave boiling for 1 min.

Immediately, filter through the co-precipitation filter paper (5.5) and wash several times with warm ammonia solution (4.15). Reject the filtrate and washing solution.

Wash both iron precipitates from filter paper into a 400 ml beaker with water. Remove all the iron from the papers by gently adding 20 ml of hydrochloric acid (4.8) over the whole paper to dissolve the iron.

Complete the transfer of iron by washing the filter paper with small quantities of warm water. Complete the dissolution of all the iron precipitate by swirling and if necessary, very gentle heating.

7.7 Preparation of test solutions

Quantitatively, transfer the solutions from step 7.6 to a 200 ml volumetric flask and dilute to approximately 150 ml of deionized water. Allow this solution to cool to room temperature, then dilute to 200 ml of water and mix again.

7.8 Preparation of calibration solutions

Using arsenic standard (4.21), prepare a series of calibration standards as per Table 1. Transfer the appropriate volumes of arsenic standard (4.21) using pipettes into 100 ml volumetric flasks containing 10 ml of hydrochloric acid (4.8) and 15 ml of iron chloride solution (4.16). Fill up nearly to the mark with water, mix, and equilibrate at room temperature, then fill up exactly to the mark and mix again.

Table 1 — Calibrating solutions

Volume of arsenic standard ml	Arsenic mass µg	Concentration of arsenic µg/ml
0	0	0
0,5	100	1,0
1	200	2,0
2	400	4,0
5	1 000	10,0
10	2 000	20,0
20	4 000	40,0
30	6 000	60,0
35	7 000	70,0

7.9 Preparation of arsenic calibration curve

Set up the ICP-AES (5.3) according to the guidelines set out in 5.3. Adjust the instrument read-out scale to zero. Aspirate each calibration solution as prepared in step 7.8 through the ICP-AES and record the emission intensities. Manually or electronically plot a curve of the average emission intensities versus the mass (µg) of arsenic.

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

7.10 Determination of arsenic content in test solutions

After calibrating the ICP-AES (5.3), determine the arsenic content in the test solutions (7.8). Adjust the instrument read-out scale to zero. Aspirate the first duplicate of each sample through the ICP-AES and record the emission intensities checking the zero regularly.

Check the calibration of the instrument, then aspirate the second duplicate of each sample in the reverse order through the ICP-AES and record the emission intensities checking the zero regularly.

Determine the mass of arsenic in each test sample (F_{As}, in µg) from the curve determined in 7.9.

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

8 Expression of results

The mass fraction of arsenic in the test portion (w_{As}), expressed as a percentage, is given by the following formula:

$$w_{As} = \frac{F_{As} - F_{As\ blank}}{M \times 10^6} \times 100\,K \quad (1)$$

where

F_{As} is the mass of arsenic found in test sample aliquot, in µg;

$F_{As\ blank}$ is the mass of arsenic found in the blank test, in µg;

M is the mass of the test portion, in g.

K is the hygroscopic moisture conversion factor, calculated using the following formula:

$$K = 100 \div (100 - H) \quad (2)$$

where

H is the hygroscopic moisture content of the sample, expressed as a percentage, determined using ISO 9599.

NOTE If pre-dried test samples are used, $H = 0$.

9 Precision

9.1 Expression of precision

The precision of this analytical method is expressed by the following formulae.

a) For copper sulfide concentrates

$$s_r = 0{,}003\,8\bar{X} + 0{,}002\,6 \quad (3)$$

$$s_L = 0{,}015\,5\bar{X} + 0{,}010\,2 \quad (4)$$

b) For lead sulfide concentrates

$$s_r = 0{,}009\bar{X} + 0{,}002 \quad (5)$$

$$s_L = 0{,}040\bar{X} + 0{,}001 \quad (6)$$

c) For zinc sulfide concentrates

$$s_r = 0{,}014\bar{X} \quad (7)$$

$$s_L = 0{,}047\bar{X} \quad (8)$$

d) For nickel sulfide concentrates

$$s_r = 0{,}009\,7\bar{X} + 0{,}001\,5 \quad (9)$$

$$s_L = 0{,}026\,5\bar{X} + 0{,}001\,8 \quad (10)$$

where

$\bar{X}$ is the mean mass fraction of arsenic in the sample, expressed as a percentage;

s_r is the within-laboratory standard deviation, expressed as a percentage by mass;

s_L is the between-laboratories standard deviation, expressed as a percentage by mass.

9.2 Procedure for obtaining the final result

Calculate the following quantities from the duplicate results x_1 and x_2 (%) and process according to the flowchart in Annex B.

a) Mean of duplicates

$$\bar{X} = \frac{x_1 + x_2}{2} \quad (11)$$

Within laboratory standard deviation (s_r), using Formula (3) for copper sulfide concentrates, Formula (5) for lead sulfide concentrates, Formula (7) for zinc sulfide concentrates, or Formula (9) for nickel sulfide concentrates.

b) Repeatability limit

$$R = 2{,}8\,s_r \quad (12)$$

9.3 Between-laboratories precision

The 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:

a) Mean of final results

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (13)$$

Between-laboratories standard deviation (s_L) by substituting μ_{12} for $\bar{X}$ in using Formula (4) for copper sulfide concentrates, Formula (6) for lead sulfide concentrates, Formula (8) for zinc sulfide concentrates, and Formula (10) for nickel sulfide concentrates.

Within-laboratory standard deviation (s_r) by substituting μ_{12} for $\bar{X}$ in using Formula (3) for copper sulfide concentrates, Formula (5) for lead sulfide concentrates, Formula (7) for zinc sulfide concentrates, and Formula (10) for nickel sulfide concentrates.

b) Permissible tolerance

$$P = 2{,}8 \times \sqrt{s_L^2 + \frac{s_r^2}{2}} \quad (14)$$

c) Range

$$E = |\mu_1 - \mu_2| \quad (15)$$

where

μ_1 is the final result reported by laboratory 1, expressed as a percentage by mass of arsenic;

μ_2 is the final result reported by laboratory 2, expressed as a percentage by mass of arsenic.

If E is ≤ P, the final results are in agreement.

9.4 Check of trueness

9.4.1 General

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:

a) $|\mu_c - A_c| \leq C \quad (16)$

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

b) $|\mu - A_c| > C \quad (17)$

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

where

μ_c is the final result, expressed as a percentage by mass of arsenic of the certified reference material;

A_c is the certified value, expressed as a percentage by mass of arsenic of the certified reference material;

C is a quantity, expressed as a percentage by mass of arsenic depending on the type of certified reference material used as defined in 9.4.2.

9.4.2 Type of certified reference material (CRM) or reference material (RM)

9.4.2.1 General

The reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35:2006.

9.4.2.2 Reference material certified/characterized by inter-laboratory test programme

The quantity C (see 9.4.1), expressed as a percentage by mass of arsenic, is given by the following formula:

$$C = 2 \times \sqrt{s_L^2 + \frac{s_r^2}{n} + s^2(A_c)} \quad (18)$$

where

$s^2(A_c)$ is the variance of the certified value;

n is the number of replicate determinations.

9.4.2.3 Reference material certified/characterized by one laboratory

The quantity C (see 9.4.1), expressed as a percentage by mass of arsenic, is given by the following formula:

$$C = \sqrt{2\,s_L^2 + \frac{s_r^2}{n}} \quad (19)$$

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

10 Test report

The test report shall contain the following information:

a) a reference to this International Standard (i.e. ISO 13547-1);

b) identification of the sample;

c) mass fraction of arsenic in the sample, expressed as a percentage;

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.

Annex A
(normative)

Procedure for the preparation and determination of the mass of a pre-dried test portion

A.1 General

This annex specifies a method for the preparation and determination of the mass of a pre-dried test portion in the analysis of copper, lead, zinc, and nickel sulfide concentrates. The method is applicable to sulfide concentrates not susceptible to oxidation and with hygroscopic moisture contents ranging from 0,05 % to 2 %.

A.2 Principle

The test portion to be used for analysis is dried in air in an oven maintained at 105°C ± 5°C. The dried test portion is then weighed and used for the analysis. No correction for moisture is required.

A.3 Reagents

A.3.1 Desiccant, such as self-indicating silica gel or anhydrous magnesium perchlorate.

WARNING — Care needs to be taken whenever disposing of exhausted magnesium perchlorate and all other laboratory chemicals. Environmental regulations often apply. Users should seek specialist's advice to determine an appropriate, effective, health-conscious, safety-conscious, and environmentally sound means of disposal.

A.4 Apparatus

Ordinary laboratory equipment and the following.

A.4.1 Analytical balance, sensitive to 0,1 mg.

A.4.2 Weighing vessels, of glass or silica or corrosion resistant metal with externally fitting airtight covers. For small test portions (of mass less than 3 g), the mass of the vessel should be as small as possible (i.e. less than 20 g).

A.4.3 Laboratory oven, capable of maintaining a temperature of 105°C ± 5°C.

A.5 Procedure

A.5.1 Preparation of the weighing vessel

Dry the weighing vessel and its cover (A.4.2) by heating in a laboratory oven (A.4.3) at 105°C ± 5°C for 1 h. Transfer the vessel and its cover to a desiccator containing suitable fresh desiccant (A.3.1) and allow cooling to ambient temperature.

A.5.2 Test portion

Tare the dried weighing vessel and vessel cover (A.5.1). Immediately, add the mass of laboratory sample specified for analysis. An accurate total mass of the test portion and weighing vessel is not required at this point.

A.5.3 Determination of the test portion dry mass

Transfer the uncovered weighing vessel, the test portion, and the vessel cover to the laboratory oven (A.4.3) 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 cooling to ambient temperature in the desiccator. When cool, remove the weighing vessel containing the dry test portion and the vessel cover from the desiccator and weigh to the nearest 0,1 mg (m_{1a}) after slightly lifting the cover and quickly replacing it.

Transfer the test portion into the appropriate analytical apparatus and immediately reweigh the empty weighing vessel and vessel cover. Record the mass (m_2) to the nearest 0,1 mg.

For new concentrates of unknown characteristics, it is advisable to repeat the drying for another 2 h at 105°C ± 5°C and to reweigh the weighing vessel and test portion plus vessel cover to the nearest 0,1 mg (m_{1b}). The test portion can be considered stable if the difference between m_{1a} and m_{1b} is within ±0,5 mg. If this condition is not achieved, the drying and weighing steps should be repeated.

A.6 Calculation of the test portion dry mass

The dry mass of the test portion (m_3) in grams is calculated using the following formula:

$$m_3 = m_{1a} - m_2 \tag{A.1}$$

where

m_{1a} is the mass of the dried test portion plus weighing vessel and its vessel cover, in grams;

m_2 is the mass of the empty weighing vessel plus its cover, in grams.

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.