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Lead sulfide concentrates — Determination of lead content — EDTA titration method after acid digestion

Concentrés sulfurés de plomb — Dosage du plomb — Méthode par titrage à l'EDTA après digestion acide

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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

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

International Standard ISO 13545 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes A and B form a normative part of this International Standard. Annex C is for information only.

Lead sulfide concentrates — Determination of lead content — EDTA titration method after acid digestion

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 a lead sulfate precipitation EDTA titrimetric method after acid decomposition for determination of the lead content of lead sulfide concentrates.

The method is applicable to lead sulfide concentrates having lead content in the range 50 % (*m/m*) to 80 % (*m/m*). The method is not applicable to lead concentrates containing more than 1 % (*m/m*) of barium.

2 Normative references

The following normative documents contain certain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*.

ISO 9599:1991, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*.

ISO Guide 35:1989, *Certification of reference materials — General and statistical principles*.

3 Principle

Decomposition of the test portion in nitric and sulfuric acids and bromine, and removal of arsenic, antimony and tin by hydrobromic acid treatment. Separation of lead from interfering elements by precipitation of lead sulfate. Dissolution of the precipitate in an ammonium acetate solution. Titration of the solution with EDTA using xylenol orange as the indicator.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water that complies with grade 2 of ISO 3696.

4.1 Dilute hydrochloric acid (1+1)

To 500 ml of water slowly add 500 ml of hydrochloric acid (ρ_{20} 1,16 g/ml to 1,19 g/ml).

4.2 Nitric acid, ρ_{20} 1,42 g/ml

4.3 Dilute nitric acid, (1+3)

To 300 ml of water slowly add 100 ml of nitric acid (4.2).

4.4 Dilute nitric acid, (1+9)

To 450 ml of water slowly add 50 ml of nitric acid (4.2).

4.5 Perchloric acid, ρ_{20} 1,70 g/ml

4.6 Hydrofluoric acid, ρ_{20} 1,14 g/ml

4.7 Hydrobromic acid, ρ_{20} 1,50 g/ml

4.8 Dilute sulfuric acid, (1+1)

To 500 ml of water slowly add, with stirring and cooling, 500 ml of sulfuric acid (ρ_{20} 1,84 g/ml).

4.9 Dilute sulfuric acid, (1+99)

To 490 ml of water slowly add 10 ml of dilute sulfuric acid (4.8).

4.10 Dilute sulfuric acid washing solution

To 500 ml of sulfuric acid (4.9) add 50 ml of ethanol or methanol (4.21) and cool.

4.11 Nitration mixture

To 250 ml of nitric acid (4.2) slowly add, with stirring and cooling, 250 ml of sulfuric acid (ρ_{20} 1,84 g/ml).

4.12 Dilute ammonium hydroxide, (1+1)

To 500 ml of water slowly add 500 ml of ammonium hydroxide (ρ_{20} 0,90 g/ml).

4.13 Lead metal, minimum 99,99 % purity

The surface of the metal shall be free from oxide prior to use and may be cleaned by immersing the metal in nitric acid (4.4) for 1 min, washing well with water followed by acetone and drying in an oven at 50 °C.

4.14 Bromine

4.15 Sodium chloride

4.16 Potassium chloride

4.17 Sodium peroxide

4.18 Fusion mixture, one part sodium carbonate mixed with one part potassium carbonate.

4.19 Ammonium acetate solution

Dissolve 250 g of ammonium acetate in water and dilute to 1 000 ml. Add 25 ml of acetic acid to the solution and mix.

4.20 Ammonium acetate washing solution

To 950 ml of water, add 50 ml of ammonium acetate solution (4.19).

4.21 Ethanol [99,5 % (*V*/*V*)] or **methanol** [99,5 % (*V*/*V*)].

4.22 L (+) - ascorbic acid

4.23 Iron(III) solution, (10 mg Fe/ml)

Dissolve 1 g of iron in 30 ml of dilute hydrochloric acid (4.1) by heating. Add 5 ml of nitric acid (4.2) to oxidize to iron(III) ion, cool and dilute to 100 ml with water. This solution shall be used for matrix matching.

4.24 Zinc solution, (10 mg Zn/ml)

Dissolve 1 g of zinc [minimum purity 99,9 % and maximum lead content 0,001 % (*m*/*m*)] in 20 ml of dilute hydrochloric acid (4.1) by heating, cool and dilute to 100 ml with water. This solution shall be used for matrix matching.

4.25 Lead standard solution, (0,1 mg Pb/ml)

Weigh 0,1 g of clean lead metal (4.13), accurately to the nearest 0,1 mg, transfer it to a 200 ml beaker and decompose with 20 ml of dilute nitric acid (4.3). Heat to remove nitrogen oxides, cool to room temperature and add about 50 ml of water. Transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

4.26 Disodium ethylenediaminetetraacetic acid solution, (0,01 mol/l EDTA solution)

Dissolve 0,38 g of disodium ethylenediaminetetraacetate (dihydrate) in 100 ml of water. This solution is used for titration of bismuth in the alternative titration described in 7.6.

4.27 Disodium ethylenediaminetetraacetic acid volumetric standard solution, (0,025 mol/l EDTA standard solution).

4.27.1 Preparation

Dissolve 9,4 g of disodium ethylenediaminetetraacetate (dihydrate) in 1 000 ml of water; 1 ml of this solution corresponds to about 5,2 mg of lead. Standardize by the procedure specified in 4.27.2.

4.27.2 Standardization

Accurately weigh three samples of clean lead metal (4.13), each of 200 mg, to the nearest 0,1 mg, and transfer them to three 400 ml conical beakers. Record these masses as m_1, m_2 and m_3.

NOTE The mass of lead metal should match the estimated mass of lead in the test portion.

Add 20 ml of dilute nitric acid (4.3), cover with a watch glass and heat to complete dissolution. After rinsing the underside of the watch glass and inside of the conical beakers with water, boil gently to expel nitrogen oxides and cool. Add 30 ml of ammonium acetate solution (4.19) and dilute the solution to about 150 ml with water. Adjust the pH of the solution to a value between 5,5 and 5,7 with dilute ammonium hydroxide (4.12) using a pH meter (5.6). Continue the standardization in accordance with the procedure specified in 7.6. Record the volume of EDTA standard solution used in the titration as V_1, V_2 and V_3.

Calculate the factors f_1, f_2 and f_3 using the following equation:

$$f_1 = \frac{m_1}{V_1} \qquad f_2 = \frac{m_2}{V_2} \qquad f_3 = \frac{m_3}{V_3} \tag{1}$$

Calculate, to four significant figures, the mean factor, f, for the EDTA standard solution provided that the range of the values of f_1, f_2 and f_3 does not exceed 0,02 mg/ml. If this range is exceeded, repeat the standardization.

4.29 Xylenol orange solution, (1 g/l)

Dissolve 0,1 g of xylenol orange in 100 ml of water.

5 Apparatus

Ordinary laboratory equipment and

5.1 Volumetric glassware, class A, complying with ISO 385-1, ISO 648, and ISO 1042 and used in accordance with ISO 4787.

5.2 Analytical balance, sensitive to 0,1 mg.

5.3 Platinum crucibles or **zirconium crucibles**.

5.4 Atomic absorption spectrometer (AAS), with lead hollow cathode lamp.

Instrument conditions:

— flame – air/acetylene;

— wavelength – 217 or 283,3 nm.

5.5 ICP atomic emission spectrometer (ICP), optional.

5.6 pH meter.

6 Sampling

6.1 Test sample

Prepare an air-equilibrated test sample in accordance with ISO 9599.

NOTE An air-equilibrated test sample is not required if predried test portions are to be used (see annex A).

6.2 Test portion

Taking multiple increments, extract a mass of test portion from the test sample and weigh 0,25 g to 0,5 g to the nearest 0,1 mg. The test portion weighed shall contain about 200 mg lead. 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 predried test portions directly from the laboratory sample.

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.

7.3 Decomposition of test portion

Transfer the test portion to a 400 ml conical beaker and moisten with 5 ml of water. Add 10 ml to 20 ml of nitric acid (4.2) and 1 ml to 2 ml of bromine (4.14) in small portions, cover with a watch glass and heat gradually to decompose the test portion completely. Cool slightly, rinse the underside of the watch glass and the inside of the conical beaker with a minimum volume of warm water. Add 10 ml of dilute sulfuric acid (4.8) and heat until strong white fumes are evolved, then cool.

If the residue appears dark (presence of carbon), slowly add a small amount of the nitration mixture (4.11) to the hot solution until the solution is colourless or bluish and heat until strong white fumes are evolved, then cool.

Carefully add 5 ml of water and 10 ml of hydrobromic acid (4.7), heat gradually until strong white fumes are evolved then cool. Add 5 ml of dilute sulfuric acid (4.8) and 10 ml of hydrobromic acid (4.7), again heat gradually until strong white fumes are evolved then cool.

NOTE The above step is not required if the sample contains less than 0,5 % (*m/m*) of arsenic, tin, antimony and/or selenium.

7.4 Formation of lead sulfate

Carefully rinse the wall of the conical beaker with water and adjust the volume to about 100 ml with water. Heat gradually until boiling to dissolve the salts and cool to room temperature. Add 10 ml of ethanol or methanol (4.21), allow the lead sulfate precipitate to settle for at least 2 h.

NOTE It is preferable to allow the lead to settle overnight using the method described in NOTE 1 under 7.5.

7.5 Separation and dissolution of lead sulfate

Filter the precipitate through a fine porosity filter paper and wash the inside of the beaker two or three times with dilute sulfuric acid washing solution (4.10). Wash the precipitate on the filter paper three or four times with dilute sulfuric acid washing solution (4.10), then with a small amount of water. Reserve the filtrate and washing in a beaker (400 ml) for determination of lead by AAS (as described in 7.7). Rinse the precipitate into the original 400 ml conical beaker with warm water.

NOTE 1 Instead of using the above steps, the following method is available:

Initial decanting of liquid through a fine porosity filter paper leaving precipitate in the beaker. Washing the precipitate three or four times with dilute sulfuric acid washing solution (4.10) in the beaker by decantation, then with water. Washing the precipitate on the filter paper three or four times with dilute sulfuric acid washing solution (4.10) then with water. Reserving the filtrate and washing in a beaker (400 ml) for determination of lead by AAS (as described in 7.7).

Place the original conical beaker containing the main precipitate under the original funnel. Add 30 ml of warm ammonium acetate solution (4.19) to the filter paper in order to dissolve the lead sulfate on the filter paper, then wash with hot water. Collect the washing in the original conical beaker. Wash around the inside of the beaker with

small amounts of hot ammonium acetate solution (4.19). Heat to boil for a few minutes to dissolve the lead sulfate. Filter the solution through the original filter paper, wash well with warm ammonium acetate washing solution (4.20). Collect the filtrate and washing in a 400 ml conical beaker, dilute to about 150 ml with water and cool.

NOTE 2 If the solution contains a small amount of Fe(III) ion, the end point of titration is not clear. In such a case, about 0,2 g of L (+) - ascorbic acid (4.22) should be added to the solution prior to titration to cause reduction of Fe(III) to Fe(II).

Reserve the residue and the filter paper for AAS determination of lead (as described in 7.8) unless it has been proven, through previous testing, that the lead in the sample is completely soluble in the initial decomposition (7.3).

7.6 Titration

Add 0,5 ml of xylenol orange solution (4.28) as the indicator, and immediately titrate with 0,025 mol/l EDTA standard solution (4.27) until the colour of the solution changes from red to yellow. Record the volume of 0,025 mol/l EDTA standard solution used in the titration as V_4 for test portion analysis and as V_5 for blank test.

If the test portion contains more than 0,1 % (*m/m*) of bismuth, titrate as follows:

Adjust pH of the solution (from 7.5) to approximately 1,5 with nitric acid (4.2) using the pH meter (5.6) and add 0,5 ml of xylenol orange solution (4.28) as the indicator. Immediately titrate with 0,01 mol/l EDTA solution (4.26) until the colour of the solution changes from reddish violet to yellow. Adjust the pH to 5,5 to 5,7 with dilute ammonium hydroxide (4.12) using the pH meter (5.6), then titrate with 0,025 mol/l EDTA standard solution (4.27) until the colour of the solution changes from red to yellow.

7.7 Determination of lead in filtrate

Heat the reserved filtrate and washing (from 7.5) to evaporate to about 100 ml. Add 10 ml of dilute hydrochloric acid (4.1). After cooling transfer the solution to a 200 ml volumetric flask and dilute to the mark with water.

Prepare calibration solutions by adding 0 ml; 0,5 ml; 1 ml; 3 ml and 5 ml of lead standard solution (4.25) to separate 200 ml volumetric flasks from a pipette or a micro-burette. Add 10 ml of dilute hydrochloric acid (4.1), 10 ml of dilute sulfuric acid (4.8) and estimated mass of iron and zinc in the test portion to each volumetric flask and dilute to the mark with water.

Aspirate the test solutions with the calibration solutions into the air/acetylene flame of the atomic absorption spectrometer (5.4) and measure the absorbance of each at wavelength of 217 nm or 283,3 nm with background correction.

Prepare calibration graphs of mass of lead in the calibration solutions versus absorbance and read the mass of lead in the test solution from the calibration graph.

NOTE Alternatively an ICP atomic emission spectrometer (5.5) can be used for the determination of lead at a wavelength of 220,35 nm.

7.8 Determination of lead in insoluble residue

Place the retained residue and filter paper in a zirconium crucible (5.3), dry and ignite at 700 °C to 750 °C. Allow the crucible to cool, add 4 g of fusion mixture (4.18) and 1 g of sodium peroxide (4.17) and heat on the burner until molten. After cooling, place the crucible and its contents in a 400 ml beaker. Add 50 ml of water, cover the beaker with a watch glass and allow to stand in order to dissolve the melt. Heat until gently boiling to decompose peroxide. Carefully add 25 ml of dilute hydrochloric acid (4.1). When reaction has ceased, wash the crucible out with water. Transfer the solution to a 250 ml volumetric flask and dilute to the mark with water.

Prepare calibration solutions by adding 0 ml, 0,5 ml, 2 ml and 5 ml of lead standard solution (4.25) to separate 250 ml volumetric flasks from a pipette or a micro-burette, add 10 ml of dilute hydrochloric acid (4.1), 2,1 g of potassium chloride (4.16) and 3,7 g of sodium chloride (4.15) to each flask and dilute to the mark with water.

If the residue (see 7.5) contains a small quantity of lead, instead of using the above steps, the following method is available:

Place the retained residue and filter paper (see 7.5) in a platinum crucible (5.3), dry and ash at low temperature, then ignite at 700 °C to 750 °C. Allow the crucible to cool, add 3 ml of nitric acid (4.2), 2 ml to 5 ml of hydrofluoric acid (4.6) and 5 ml of perchloric acid (4.5). Heat in order to evaporate to near dryness and volatilize the silicon as silicon tetrafluoride. Dissolve in a small quantity of water and 10 ml of dilute hydrochloric acid (4.1) by heating. Transfer the solution to a 100 ml volumetric flask and dilute to the mark with water.

Prepare calibration solutions by adding 0 ml, 0,5 ml, 2 ml and 5 ml of lead standard solution (4.25) to separate 100 ml volumetric flasks from a pipette or a micro-burette, add 10 ml of dilute hydrochloric acid (4.1) to each flask and dilute to the mark with water.

Aspirate the test solutions and the calibration solutions in the atomic absorption spectrometer (5.4) using air/acetylene flame and a wavelength of 217 nm or 283,3 nm with background correction. Prepare calibration graphs of mass of lead in the calibration solutions versus absorbance and read the mass of lead in the test solution from the calibration graph.

NOTE Alternatively, an ICP atomic emission spectrometer (5.5) can be used for the determination of lead at a wavelength of 220,35 nm.

8 Expression of results

The lead content of the test portion, expressed as a percentage by mass to two decimal places, is calculated from the following equation:

$$\text{Pb}\ \%\ (m/m) = \frac{(V_4 - V_5) \times f + m_4 + m_5}{m} \times 100 \times \frac{100}{100 - H} \qquad (2)$$

where

V_4 is the volume, in millilitres, of EDTA standard solution used for the sample solution;

V_5 is the volume, in millilitres, of EDTA standard solution used for the blank test solution;

f is the factor, in milligrams of lead per millilitre, for the EDTA standard solution;

m_4 is the mass, in milligrams, of lead in the insoluble residue;

m_5 is the mass, in milligrams, of lead in the filtrate;

m is the mass, in milligrams, of the test portion;

H is the hygroscopic moisture, in percent by mass, of the test portion. In the case of a predried test portion being used, H = 0.

9 Accuracy

9.1 Precision

9.1.1 Expression of precision

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

$$s_r = 0{,}065\ 8 \quad (3)$$

$$s_R = 0{,}135\ 7 \quad (4)$$

where

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

s_R is the between-laboratories standard deviation, expressed as a percentage by mass of lead.

NOTE Additional information is given in annex C.

9.1.2 Method for obtaining the final result

See annex B.

Calculate the following quantities from the duplicate results x_1 and x_2, and process according to the flowchart in annex B:

Mean of duplicates

$$x = \frac{(x_1 + x_2)}{2} \quad (5)$$

Repeatability limit

$$r = 2{,}8\ s_r = 0{,}184\ 2 \quad (6)$$

9.1.3 Precision between laboratories

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

Calculate the following quantities:

Mean final results

$$\mu_{1,2} = \frac{(\mu_1 + \mu_2)}{2} \quad (7)$$

Between-laboratories standard deviation

$$s_R = 0{,}135\ 7 \quad (4)$$

Within-laboratory standard deviation

$$s_r = 0{,}065\ 8 \quad (3)$$

Permissible difference

$$P = 2{,}8\sqrt{s_R^2 + \left(s_r^2/2\right)} \quad (8)$$

Range

$$E = |\mu_1 - \mu_2| \quad (9)$$

where

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

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

If $E \leqslant P$, the final results are in agreement.

9.2 Trueness

9.2.1 Check of trueness

The trueness of an analytical result can be checked by applying it to a certified reference material (CRM). The procedure is the same as that described in clause 7. When the precision has been confirmed, the final laboratory result can be compared with the certified value, A_c.

The following two possibilities exist;

$$|\mu_c - A_c| \leqslant C \quad (10)$$

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

$$|\mu_c - A_c| > C \quad (11)$$

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

In equations (10) and (11), the symbols have the following meanings:

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

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

C is a quantity, expressed as a percentage by mass of lead, depending on the type of the certified reference material used, as defined in 9.2.2.

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

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

a) Reference material certified/characterized by an inter-laboratory test programme

The quantity C (see 9.2.1), expressed as percentage by mass of lead, is given by the following equation:

$$C = 2\sqrt{s_R^2 + \left(s_r^2/n\right) + s^2\{A_c\}} \quad (12)$$

where

$s^2\{Ac\}$ is the variance of the certified value;

n is the number of replicate determinations.

b) Reference material certified/characterized by one laboratory

The quantity C (see 9.2.1), expressed as percentage by mass of lead is given by the following equation:

$$C = 2\sqrt{s_R^2 + \left(s_r^2 / n\right)} \qquad (13)$$

NOTE It is recommended that this type of certified reference material 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) identification of the sample;

b) reference to this International Standard, i.e. ISO 13545;

c) lead content of the sample, expressed as a percentage by mass;

d) date on which the test was carried out;

e) any occurrences noted 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 predried test portion

A.1 Scope

This annex specifies a method for the preparation and determination of the mass of a predried test portion in the analysis of lead sulfide concentrates.

The method is applicable to lead sulfide concentrates not susceptible to oxidation and having 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 Chemicals

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

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

A.4 Apparatus

Ordinary laboratory equipment and

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 air-tight covers. For small test portions of mass (less than 3 g), the mass of the vessel shall 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 vessel cover (A.4.2) by heating in the laboratory oven (A.4.3) at 105 °C ± 5 °C for 1 h. Transfer the vessel and its cover to a desiccator containing a suitable fresh desiccant (A.3.1) and allow to cool to ambient temperature.

A.5.2 Test portion

Determine the tare weight of the vessel and its cover (A.4.2). 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 containing 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 to cool 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_4) after slightly lifting the cover and quickly replacing it.

Transfer the test portion to the appropriate analytical apparatus and immediately reweigh the empty weighing vessel and its cover. Record the mass (m_5) to the nearest 0,1 mg.

NOTE 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 containing the test portion and the vessel cover to the nearest 0,1 g (m'_4). The mass of the test portion can be considered to be constant if the difference ($m_4 - m'_4$) is less than or equal to 0,05 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_6, in grams, is given by the following equation:

$$m_6 = m_4 - m_5 \qquad \text{(A.1)}$$

where

m_4 is the mass, in grams, of the dried test portion plus the weighing vessel and its cover;

m_5 is the mass, in grams, of the empty weighing vessel plus its cover.

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

Annex B
(normative)

Flowchart of the procedure for the acceptance of analytical values for test samples