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Zinc sulfide concentrates — Determination of silver and gold contents — Fire assay and flame atomic absorption spectrometric method using scorification or cupellation

Concentrés sulfurés de zinc — Dosage de l'argent et de l'or — Méthode par voie sèche et spectrométrie d'absorption atomique dans la flamme à partir d'une scorification ou d'une coupellation

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

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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.

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.

International Standard ISO 15248 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores.*

Annexes A to D form an integral part of this International Standard, annexes E and F are for information only.

Zinc sulfide concentrates — Determination of silver and gold contents — Fire assay and flame atomic absorption spectrometric method using scorification or cupellation

1 Scope

This International Standard specifies a fire assay and flame atomic absorption spectrometric procedure for the determination of silver and gold contents of zinc sulfide concentrates.

The method is applicable to the determination of silver and gold in zinc sulfide concentrates containing up to 60 % (m/m) zinc in the form of zinc blende and related materials.

The method is applicable to silver contents from 10 g/t to 500 g/t and gold contents from 0,1 g/t to 12 g/t.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 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*.

3 Principle

3.1 Scorification

Fire assay fusion of a test portion to produce a lead button, which is scorified to reduce it to a mass of 2 g to 5 g.

Retreatment fusion of the primary fusion and scorification slags to produce a low-silver content lead button which is scorified to approximately 2 g to 5 g.

Dissolution of both lead buttons in nitric acid and filtration of the solution. Dissolution of the filter paper plus gold and determination of silver and gold by flame atomic absorption spectrometry.

3.2 Cupellation

Fire assay fusion of a test portion to produce a lead button, which is cupelled to produce a bead of silver and gold.

Retreatment fusion of the primary fusion slag and spent cupel to produce a second bead of silver and gold.

Dissolution of both beads in dilute nitric acid and filtration of the solution. Dissolution of the filter paper plus gold and determination of silver and gold by flame atomic absorption spectrometry.

4 Reagents

During the analysis, use only reagents of a recognized analytical grade and water that complies with grade 2 of ISO 3696. Wash all glassware with aqua regia followed by water and 25 % ammonia. A chloride-free environment is recommended.

4.1 Sodium carbonate, anhydrous

4.2 Litharge, assay reagent grade having silver content less than 0,2 g/t, and gold content less than 0,01 g/t

4.3 Silica, precipitated grade

4.4 Potassium nitrate or sodium nitrate

4.5 Flour

4.6 Nitric acid (ρ_{20} 1,42 g/ml), chloride content < 0,5 µg/ml

4.7 Nitric acid wash solution, (5 ml/l)

To 995 ml of water add 5 ml of nitric acid (4.6).

4.8 Nitric acid, (250 ml/l)

To 1 500 ml of water carefully add, with stirring, 500 ml of nitric acid (4.6) and cool.

4.9 Hydrochloric acid, (ρ_{20} 1,16 g/ml to 1,19 g/ml).

4.10 Borax, fused anhydrous sodium tetraborate

4.11 Silver metal, minimum 99,99 % purity

4.12 Gold metal, minimum 99,99 % purity

4.13 Aqua regia

Mix 3 parts of hydrochloric acid (4.9) with 1 part of nitric acid (4.6); prepare freshly as required.

4.14 Ammonia solution

Add 500 ml of ammonia solution (ρ_{20} 0,89 g/ml) to 500 ml of water.

4.15 Standard solutions

NOTE Standard solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.15.1 Silver standard solutions

4.15.1.1 Silver standard solution, (1 000 µg/ml)

Weigh 0,500 0 g of silver metal (4.11) into a 400 ml beaker, add 100 ml of nitric acid (4.8), cover and heat gently until the metal dissolves. Continue heating to remove oxides of nitrogen. Cool, and transfer to a 500 ml volumetric flask. Dilute to volume with water and mix thoroughly.

This solution should be stored in a brown bottle.

4.15.1.2 Silver standard solution, (50 µg/ml)

Pipette 10 ml of silver standard solution (4.15.1.1) into a 200 ml volumetric flask, add 40 ml of nitric acid (4.8), dilute to volume and mix thoroughly.

This solution should be stored in a brown bottle.

4.15.2 Gold standard solutions

4.15.2.1 Gold standard solution, (1 000 µg/ml)

Weigh 0,100 g of gold metal (4.12) into a 50 ml beaker, add 5 ml of aqua regia solution (4.13), cover and heat to dissolve the gold. Wash and remove the cover and evaporate to near dryness (do not allow to go to dryness, as gold may precipitate.) Cool, add 5 ml of hydrochloric acid (4.9), transfer the solution quantitatively to a 100 ml volumetric flask, dilute to volume with water, stopper and mix thoroughly. Store the solution in a brown bottle.

4.15.2.2 Gold standard solution, (100 µg/ml)

Pipette 20 ml of gold standard solution (4.15.2.1) into a 100 ml volumetric flask, add 10 ml of hydrochloric acid (4.9), dilute to volume with water, stopper and mix thoroughly.

4.16 Calibration solutions

NOTE Calibration solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.16.1 Silver calibration solutions

To five 100 ml volumetric flasks, add from a burette (5.6) 0 ml, 2 ml, 4 ml, 6 ml and 8 ml of silver standard solution (4.15.1.2); add 20 ml of nitric acid (4.8), dilute to volume with water and mix thoroughly.

These standards contain 0 µg, 1 µg, 2 µg, 3 µg and 4 µg of silver per ml and shall be freshly prepared.

4.16.2 Gold calibration solutions

To six 100 ml volumetric flasks, add from a burette (5.6) 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of gold standard solution (4.15.2.2); add 10 ml of aqua regia solution (4.13), dilute to volume with water and mix thoroughly.

These standards contain 0 µg, 2 µg, 4 µg, 6 µg, 8 µg and 10 µg of gold per ml and shall be freshly prepared.

5 Apparatus

Ordinary laboratory equipment plus the following.

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

5.2 Conventional fire assay equipment

5.2.1 Assay crucible furnace, having a maximum required operating temperature of 1 100°C.

5.2.2 Muffle furnace, having a maximum required operating temperature of 1 100°C; temperature indication, automatic temperature control and controlled air flow are desirable.

5.2.3 Crucibles, made of fire clay, of nominal capacity 300 ml to 500 ml, capable of withstanding corrosion by the sample and fluxes at 1 050°C. The crucible shall be of such a size that the charge does not fill the crucible to a depth of greater than 3/4 the depth of the crucible.

5.2.4 Scorifiers, made of fire clay, of approximately 75 mm diameter, preferably not shallow form.

5.2.5 Cupels, made of magnesium oxide, or bone ash cupels having a nominal capacity of 50 g of molten lead. The inside bottom of the cupel shall be concave.

5.2.6 Conical mould, made of cast iron, of sufficient capacity to contain all of the molten lead plus slag from the crucible fusion.

5.2.7 Pulverizer

5.3 Balances

5.3.1 Top loading, capable of being read to 1 mg

5.3.2 Precision analytical, capable of being read to 0,1 mg

5.4 Atomic absorption spectrometer (AAS), equipped with background correction and a glass bead in the spray chamber rather than a flow spoiler

5.5 Filtration equipment

5.5.1 Membrane filter - cellulose nitrate/cellulose acetate, diameter 25 mm, mesh size 0,45 µm

5.5.2 Vacuum flask, of 250 ml capacity, buchner filter flask having a wide neck

5.5.3 Membrane filter funnel, suitable for use with 25 mm membrane filters

5.6 Burette, A grade 10 ml capacity, capable of being read to 0,02 ml

6 Sample

6.1 Test sample

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

NOTE A 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 test portion from the test sample in such a manner that it is representative of the whole contents of the dish or tray. Weigh to the nearest 0,1 mg approximately 20 g of test sample. At the same time as the test portion is weighed, 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 Trial fusion

Carry out a trial fusion as described in annex B to ensure that the mass of the lead button is between 28 g and 35 g.

NOTE It is essential that this trial fusion be performed.

7.3 Blank tests

Carry out a duplicate reagent blank test as described in annex C in parallel with each batch of samples fired, using the same quantities of all reagents with the addition of sufficient flour (4.5) to the flux to give a button size of 28 g to 35 g. Omit the sample and the potassium nitrate.

NOTE If the blank solutions (annex C) exceed 0,05 µg/ml for gold, the reagents should be checked and the problem rectified. Otherwise the blank is subtracted from the result.

7.4 Charge preparation

Determine the mass of potassium nitrate (4.4) required in the charge as indicated by the trial fusion (see annex B) and include this reagent in the flux mixture (see table 1).

Thoroughly mix the test portion with a flux of the composition specified in table 1.

Table 1 — Recommended masses of flux components for preparation of charges

Flux components	Mass g
Sodium carbonate (4.1)	30
Litharge (4.2)	120
Silica (4.3)	10
Borax (4.10)	10
Potassium nitrate (4.4)	–

Place the mixture in the assay crucible (5.2.3).

NOTE1 The intimate mixing of fluxes and samples is very important. All flux components should be in a finely divided state, with a preferred particle size of less than 0,5 mm.

NOTE 2 The quantities of oxidizing reagent in the mixture are dependent upon the reducing power of the test sample. The trial fusion (see annex B) will determine the mass of potassium nitrate necessary to yield a button of mass 28 g to 35 g. The oxidizing reagent should not be included in a bulk flux. It should be weighed up individually to ensure the repeatability of the mass of the lead button, which, if fusion conditions are uniformly repeated, should agree within ± 1 g of lead, thus decreasing the variability of finishing times of scorification or cupellation.

NOTE 3 If difficulties are experienced in producing repeatable and predictable masses of lead button, a lid for the crucible may be of assistance.

7.5 Primary fusion

Place the crucible in the furnace (5.2.1) preheated to approximately 900 °C.

Slowly raise the furnace temperature to 1 050°C. Maintain this temperature until fusion has been calm for at least 10 min.

Pour the fusion into a dry conical mould (5.2.6), taking care to ensure that no loss of lead or slag occurs. Reserve the crucible for retreatment fusion.

Allow the mixture to cool and carefully separate the lead button from the slag. Hammer the lead button as necessary to remove any small particles of adhering slag. Reserve the slag for retreatment.

Weigh the lead button. If the button weighs less than 28 g or more than 35 g, discard the button and slag and repeat the assay using less oxidizing agent or adding flour (see annex B).

NOTE 1 If oil-fired or gas-fired furnaces are used, the fuel should be turned off immediately before opening the furnace.

NOTE 2 To minimize crucible corrosion and build-up of impurities in the lead button, the overall fusion time should not exceed 40 min.

NOTE 3 Buttons weighing less than 28 g show poor collection efficiencies, and those exceeding 35 g contain higher amounts of impurities such as copper and may also cause problems in obtaining the required mass of lead after scorification.

7.6 Selection of recovery procedure

The recovery of the silver and gold in the lead button from the primary fusion can use *either* scorification (see 7.7) *or* cupellation (see 7.8). Individual analysts should select the technique most familiar to their respective laboratories. The final determination of silver and gold by AAS is the same in each case.

7.7 Scorification

7.7.1 General

Place the lead button from the primary fusion (see 7.5) in a preheated scorifier containing approximately 0,5 g of silica (4.3) in a muffle furnace (5.2.2) at 900 °C.

Allow the scorification to proceed at a muffle temperature of approximately 900 °C with steady air flow until the visible lead melt is approximately 10 mm in diameter (approximately 30 min depending on the mass of the lead button).

Toward the end of the scorification, increase the muffle temperature to 950 °C to decrease the viscosity for clean pouring. The required lead mass is 2 g to 5 g.

Pour the mixture into a dry conical mould (5.2.6), allow to cool, remove and weigh the lead button. Reserve the slag for retreatment fusion. Discard the scorifier.

7.7.2 Retreatment fusion

Place the slags from the primary fusion (see 7.5) and the scorification (see 7.7.1) in a grinder (5.2.7) and pulverize for a period of about 20 s. Thoroughly clean the pulverizer by grinding a silver- and gold-free medium between each sample.

Intimately mix the pulverized slag with a flux of the following composition:

- litharge (4.2) 50 g;
- sodium carbonate (4.1) 50 g;
- silica (4.3) 50 g;
- borax (4.10) 50 g;
- flour (4.5) sufficient to produce a 28 g to 35 g lead button (usually approximately 4 g).

Place the mixture into the original assay crucible from 7.5.

Carry out a fusion as detailed in 7.5.

Pour the fusion into a dry conical mould (5.2.6) taking care that no loss of lead occurs. Discard the crucible and slag.

7.7.3 Scorification

Place the lead button from the retreatment fusion, 7.7.2, in a new preheated scorifier containing approximately 0,5 g of silica (4.3) in the muffle furnace (5.2.2) at 950 °C. The button is added to the scorifier in the muffle furnace.

Proceed as in 7.7.1. Discard the slag and scorifier.

7.8 Cupellation

7.8.1 General

Place the lead button from the primary fusion, 7.5, into a preheated cupel in a muffle furnace (5.2.2) at 900 °C. The button is added to the cupel in the muffle furnace.

Allow the cupellation to proceed at a lower muffle temperature of approximately 860 °C with steady air flow until the visible lead melt is approximately 10 mm in diameter (approximately 30 min depending on the mass of lead button).

Raise the muffle furnace temperature to 900 °C to finish the cupellation.

Remove the cupel and allow to cool. Take the silver and gold bead and remove any attached cupel material with a brush. Weigh the bead. Reserve the cupel for retreatment fusion.

7.8.2 Retreatment fusion

Place the slag from the primary fusion, 7.5, and the cupel from the recovery procedure, 7.8.1 in a grinder (5.2.7) and pulverize for about 20 s to reduce the material to minus 150 µm. Longer grinding may cause caking of the material.

Thoroughly clean the pulverizer by grinding a silver- and gold-free medium between each sample.

Intimately mix the pulverized slag and cupel with a flux of the following composition:

- Litharge (4.2) 50 g;
- Sodium carbonate (4.1) 50 g;
- Silica (4.3) 50 g;
- Borax (4.10) 50 g;
- Flour (4.5) sufficient to produce a 28 to 35 g lead button (usually approximately 4 g).

Place the mixture into the original assay crucible from 7.5.

Carry out a fusion as detailed in 7.5.

Pour the fusion into a dry conical mould (5.2.6), taking care that no loss of lead occurs. Discard the crucible and the slag.

7.8.3 Cupellation

Place the lead button from the retreatment fusion, 7.8.2, into a new preheated cupel in a muffle furnace (5.2.2) at 900 °C. The button is added to the cupel in the furnace.

Proceed as in 7.8.1. Discard the cupel.

7.9 Treatment of the lead buttons or silver/gold beads

Place *either* the cleaned lead buttons (see 7.7.1 and 7.7.3) into one 250 ml beaker *or* the silver and gold beads (see 7.8.1 and 7.8.3) into one 250 ml beaker.

Add 100 ml of nitric acid (4.8), cover and heat to dissolve all the zinc or the bead. Boil gently to remove oxides of nitrogen.

NOTE Any black material remaining after the button or bead has been dissolved is due to precious metals other than silver.

Remove the beaker from the hotplate, allow to cool slightly, wash and remove cover, and vacuum filter through a 25 mm, 0,45 µm filter (5.5.1) into a 250 ml buchner flask (5.5.2). Use approximately 50 ml of hot nitric acid (4.7) to wash the beaker and filter paper. Give one 10 ml wash with warm ammonia solution (4.14) to dissolve any traces of silver chloride and one water wash.

Remove the top section of the filter funnel and carefully wash around the base of the top section of the filter funnel into the original 250 ml beaker to remove any adhering particles of gold. Transfer the filter paper to the original 250 ml beaker, add 2 ml of nitric acid (4.6), place on a low heat hotplate and evaporate to near dryness. Do not bake. Reserve the beaker for the determination of gold.

7.10 Determination of silver in the test solution by flame atomic absorption spectrometry

Transfer the filtrate (see 7.9) quantitatively to a 500 ml volumetric flask using nitric acid (4.7). Dilute to volume and mix thoroughly.

If the silver content exceeds 100 g/t, dilute by pipetting 20 ml of the above test solution into a 100 ml volumetric flask, add 10 ml of nitric acid (4.8), dilute to volume with water and mix thoroughly.

Determine the silver concentration by flame atomic absorption spectrometry using calibration solutions in accordance with 4.16.1. As a guide, the following atomic absorption settings are recommended; however, the instrument should be optimized to give maximum sensitivity and as near as practical to a linear relationship between absorbance and concentration.

- flame: air-acetylene (oxidizing);
- wavelength: 328,1 nm;
- lamp current: 5 mA;
- background corrector: off;
- aspiration rate: optimize for maximum signal;
- integration time: 3 s;
- number of integrations: 5.

Perform three measurements on each standard solution. Calculate, to three significant figures, the mean absorbance for each standard solution provided that the range of values does not exceed 0,003 absorbance units. If this range is exceeded, repeat the calibrations.

The test solutions should be treated in the same manner. Plot a calibration graph of absorbance versus concentration.

NOTE During all FAAS determinations, the test solutions and calibration solutions should have the same temperature as well as the same acid concentrations.

7.11 Determination of gold in the test solution by flame atomic absorption spectrometry

Add 5 ml of aqua regia (4.13) to the beaker containing the gold (see 7.9), cover and gently heat to dissolve the gold. Transfer the test solution quantitatively to a 25 ml volumetric flask. Dilute to volume with water and mix thoroughly.

Determine the gold concentration by flame atomic absorption spectrometry using calibration solutions in accordance with 4.16.2. As a guide, the following atomic absorption settings are recommended; however, the instrument should be optimized to give maximum sensitivity and as near as practical to a linear relationship between absorbance and concentration.

- flame: air/acetylene oxidizing;
- wavelength: 242,8 nm;
- lamp current: 4 mA;
- background corrector: on;

— aspiration rate: optimize for maximum signal;

— integration time: 3 s;

— number of integrations: 5.

Perform three measurements on each standard solution. Calculate to three significant figures the mean absorbance for each standard solution, provided that the range of values does not exceed 0,003 absorbance units. If this range is exceeded, repeat the calibrations.

The test solutions should be treated in the same manner. Prepare a calibration graph of absorbance versus concentration.

NOTE During all FAAS determinations, the test solutions and calibration solutions should have the same temperature as well as the same acid concentrations.

8 Expression of results

8.1 Silver content

The silver content of the test portion w_{Ag}, expressed in grams per tonne, is given by one of the following equations:

$$w_{\mathrm{Ag}} = \frac{(C_1 - B_1)500}{m} \times \frac{100}{100 - H} \qquad \text{. . . (1)}$$

or

$$w_{\mathrm{Ag}} = \frac{(C_1 - B_1)500 \times 100}{m \times 20} \times \frac{100}{100 - H} \qquad \text{. . 2)}$$

where

C_1 is the silver content of the analysis solution, in micrograms per millilitre;

B_1 is the silver content of the blank solution, in micrograms per millilitre;

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

H is the hygroscopic moisture content, as a percentage of the test portion (in the case of a predried test portion being used, $H = 0$).

8.2 Gold content

The gold content of the test portion w_{Au}, expressed in grams per tonne, is given by the following equation:

$$w_{\mathrm{Au}} = \frac{(C_2 - B_2) \times V}{m} \times \frac{100}{100 - H} \qquad \text{. . .. (3)}$$

where

C_2 is the gold content of the analysis solution, in micrograms per millilitre;

B_2 is the gold content of the blank solution, in micrograms per millilitre;

V is the volume of the analysis solution (see 7.11), in millilitres;

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

H is the hygroscopic moisture content, as a percentage of the test portion (in the case of a predried test portion being used, $H = 0$).

9 Precision

9.1 Expression of precision

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

9.1.1 Silver

$$s_r = 0{,}010\,5\,X + 0{,}321\,7 \quad \text{. . . (4)}$$

$$s_L = 0{,}016\,X + 3{,}475\,6 \quad \text{. . . (5)}$$

9.1.2 Gold

$$s_r = 0{,}025\,5\,X + 0{,}027\,1 \quad \text{. . . (6)}$$

$$s_L = 0{,}057\,1\,X + 0{,}084 \quad \text{. . . (7)}$$

where

s_r is the within-laboratory standard deviation, in grams of silver or gold per tonne;

s_L is the between-laboratories standard deviation, in grams of silver or gold per tonne;

X is the mean content of silver or gold, in grams per tonne, in the sample.

See annex E.

9.2 Method 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 D.

Mean of duplicates $X = (X_1 + X_2)/2$ . . . (8)

Within-laboratory standard deviation:

Silver determination $s_r = 0{,}010\,5\,X + 0{,}321\,7$ . . . (4)

Gold determination $s_r = 0{,}025\,5\,X + 0{,}027\,1$ . . . (6)

Repeatability limit $r = 2{,}8 s_r$ . . . (9)

9.3 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 laboratories followed the same procedure.

Calculate the following quantities:

Mean of final results $\mu_{1,2} = (\mu_1 + \mu_2)/2$ . . . (10)

Between-laboratories standard deviation

Silver determination $s_L = 0{,}016\,X + 3{,}475\,6$ . . . (5)

Gold determination $s_L = 0{,}057\,1\,X + 0{,}084$ . . . (7)

Within-laboratory standard deviation

Silver determination $s_r = 0{,}010\,5\,X + 0{,}321\,7$ . . . (4)

Gold determination $s_r = 0{,}025\,5\,X + 0{,}027\,1$ . . . (6)

Permissible difference $P = 2{,}8\sqrt{\left(s_L^2 + s_r^2/2\right)}$ . . . (11)

Range $E = \left|\mu_1 - \mu_2\right|$ . . . (12)

where

μ_1 is the final result, in grams of silver or gold per tonne, reported by laboratory 1;

μ_2 is the final result, in grams of silver or gold per tonne, reported by laboratory 2.

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

9.4 Check of trueness

The trueness of the analytical method 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:

$\left|\mu_c - A_c\right| \leqslant C$ . . . (13)

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

$\left|\mu_c - A_c\right| > C$ . . . (14)

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

In equations (13) and (14), the symbols are defined as follows:

μ_c is the final result, in grams of silver or gold per tonne of the certified reference material;

A_c is the certified value, in grams of silver or gold per tonne of the certified reference material;

C is a quantity of silver or gold, in grams per tonne, depending on the type of the certified reference material used.

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

Where the reference material has been certified/characterized by an interlaboratory test programme, the quantity C expressed in grams per tonne of silver or gold, is given by the following equation:

$C = 2\sqrt{s_L^2 + \left(s_r^2/n\right) + s^2\left\{A_c\right\}}$ . . . (15)

where

$s^2\left\{A_c\right\}$ is the variance of the certified value;

n is the number of replicate determinations.

Where the reference material has been certified/characterized by one laboratory, the quantity *C* in grams per tonne of silver or gold, is given by the following equation:

$$C = 2\sqrt{2s_\mathrm{L}^2 + \left(s_\mathrm{r}^2/n\right)} \qquad \ldots (16)$$

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 test sample;

b) reference to this International Standard, i.e. ISO 15248;

c) silver content of the sample, expressed in grams per tonne;

d) gold content of the sample, expressed in grams per tonne;

e) date on which the test was carried out;

f) any occurrence noticed during the determination that 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 procedure sets out a method for the preparation and determination of the mass of a predried test portion in the analysis of zinc sulfide concentrates. The method is applicable to zinc 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 Reagents

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

WARNING — Care should be taken when disposing of exhausted magnesium perchlorate. It shall 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, having 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

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 a laboratory oven (A.4.3) at 105 °C ± 5 °C for 1 h. Transfer the vessel and vessel cover to a desiccator containing suitable fresh desiccant (A.3.1) and allow to cool to ambient temperature.

A.5.2 Test portion

Tare the dried weighing vessel and vessel cover. Immediately add a proportion of the laboratory sample to provide a suitable predried test portion. 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 and test portion and vessel cover to the laboratory oven (A.4.3) and dry at 105 °C ± 5 °C for 2 h. After the two hour 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_1) after slightly lifting the cover and quickly replacing it. Transfer the test portion to the appropriate analytical apparatus and immediately re-weigh the empty weighing vessel and vessel cover. Record the mass (m_2) 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 re-weigh the weighing vessel and test portion plus vessel cover to the nearest 0,1 mg (m'_1). The mass of the test portion can be considered to be constant if the difference between m_1 and m'_1 is less than or equal to 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 given by the following equation:

$$m_3 = m_1 - m_2 \qquad \text{. . . (A.1)}$$

where

m_1 is the mass, in grams, of the dried test portion plus weighing vessel and its cover;

m_2 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)

Trial fusion

B.1 Introduction

The mass of oxidizing agent, potassium or sodium nitrate (4.4), in the charge is dependent upon the reducing power of the test sample. A trial is carried out to ensure that the test fusion yields a lead button of mass between 28 g and 35 g.

B.2 Procedure

Mix a 10 g test portion with the appropriate quantities of fluxes shown in table 1.

Carry out a trial fusion using the procedure described in 7.5.

Weigh the lead button obtained from the fusion. If the button weighs less than 28 g or more than 35 g, an adjustment of the oxidizing/reducing properties of the fusion mixture may be made using the following approximations.

a) the addition of 1 g of potassium nitrate will decrease the button mass by about 4 g;

b) 1 g flour produces approximately 10 g of lead from litharge.

Annex C
(normative)

Blank determination

C.1 Introduction

A blank determination is carried out to determine the silver and gold content of the assay reagents, principally litharge. It is recommended that the blank determination be performed in duplicate.

C.2 Procedure

Thoroughly mix the flux components shown in table 1 together with sufficient flour (4.5), usually 2,5 g to 3 g, as a reducing agent to produce a lead button of 28 g to 35 g. Omit the addition of potassium nitrate (4.4).

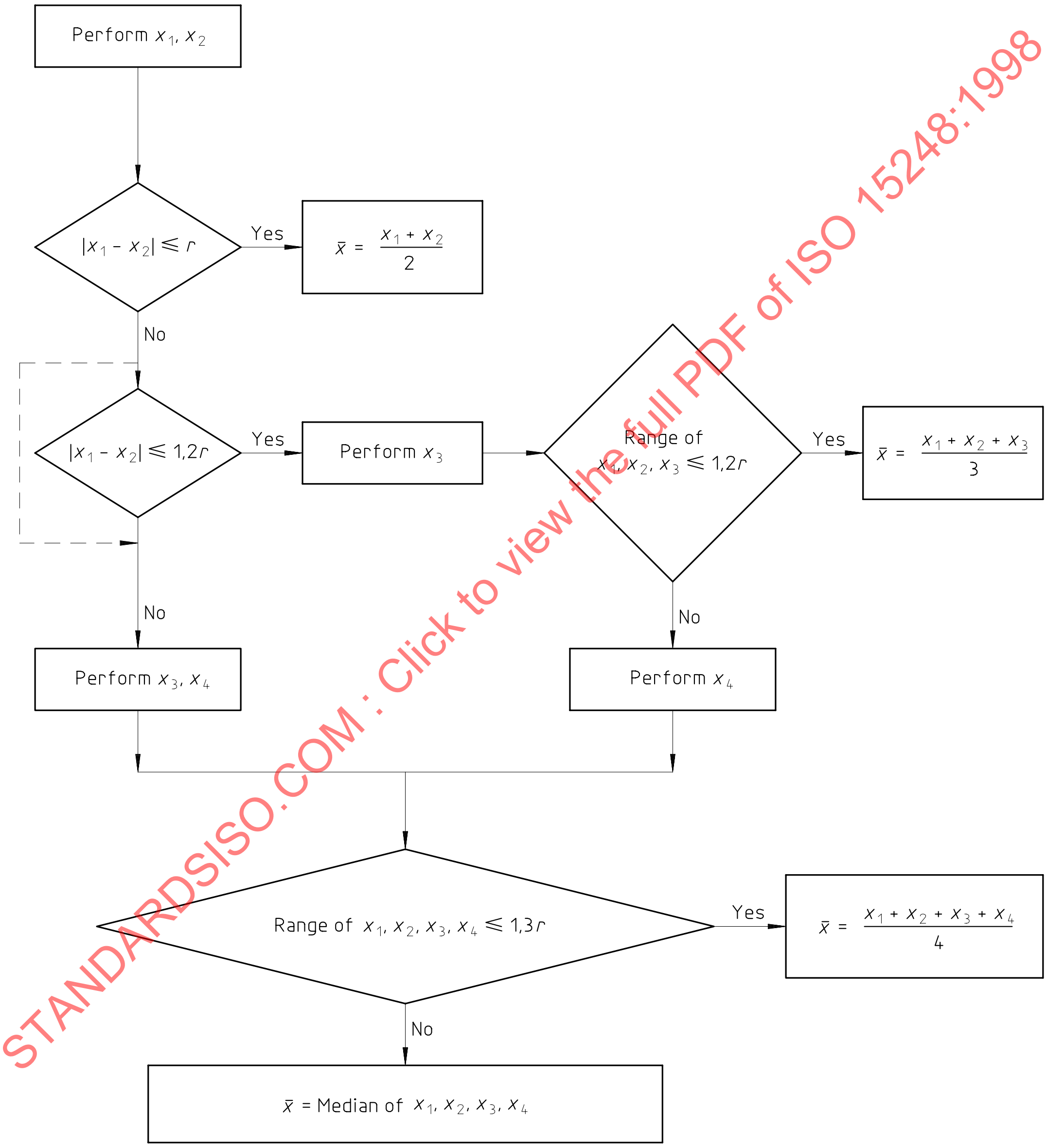
Continue the blank determination in accordance with 7.5 to 7.9.

Determine the silver and gold content of the blank solutions in accordance with 7.10 and 7.11 respectively.

Annex D

(normative)

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



r: defined in 9.2

Annex E

(informative)

Derivation of precision equations

E.1 Introduction

This International Standard was tested in an interlaboratory test programme involving five countries and fifteen laboratories. Six samples of zinc concentrates covering the range up to 60 % (*m/m*) were analysed to determine the silver and gold content. The test programme was designed to determine the repeatability and within-laboratory and between-laboratories reproducibilities in general, using the principles of ISO 5725:1986.

E.2 Design of test programme

The analytical test programme was designed with the aim of providing maximum information. Each laboratory used two samples (two bags) of each concentrate and each sample was analysed twice independently.

E.3 Test samples

This test programme used six samples of zinc concentrates. The composition of these samples is shown in table E.1.

E.4 Statistical evaluation

The procedure for statistical evaluation is illustrated schematically in figure E.1. The results of the statistical evaluation are summarized in table E.2.

The estimated precisions (s_r, s_L, r and P) are plotted against their corresponding sample means on a graph as shown in figure E.2 and the regression equations of these precisions against sample means were computed and are presented in table E.2.

Table E.1 — Composition of zinc concentrate samples

Element	Sample numbers					
	89-1 1)	**89-2** 1)	**89-3** 1)	**89-10** 2)	**91-16**	**93-1** 3)
Cu % (*m*/*m*)	0,61	0,34	0,22	0,12	0,53	0,23
Pb % (*m*/*m*)	3,50	3,01	5,54	1,32	3,72	3,24
Zn % (*m*/*m*)	47,14	53,69	50,15	52,0	46,76	46,68
Au g/t	10	3	0,5	0,2	7	2
Ag g/t	300	130	340	30	19	15
S % (*m*/*m*)	31,25	31,44	26,68	33,0	30,28	26,51
Fe % (*m*/*m*)	8,83	5,09	5,18	11,6	10,51	6,70
SiO_2 % (*m*/*m*)	3,42	4,35	7,33	1,0	3,78	12,74
Al_2O_3 % (*m*/*m*)	0,73	0,31	0,46	0,00	0,42	1,20
CaO % (*m*/*m*)	0,78	0,86	0,78	0,26	0,60	0,72
MgO % (*m*/*m*)	1,15	1,00	1,08	0,03	1,18	1,51
K_2O % (*m*/*m*)	0,32	0,16	0,16	0,00	0,22	0,31
MnO % (*m*/*m*)	0,55	0,64	0,30	0,00	0,14	0,15

1) Cominco (Canada)

2) Zinc Corporation (Australia)

3) Peak (Australia)

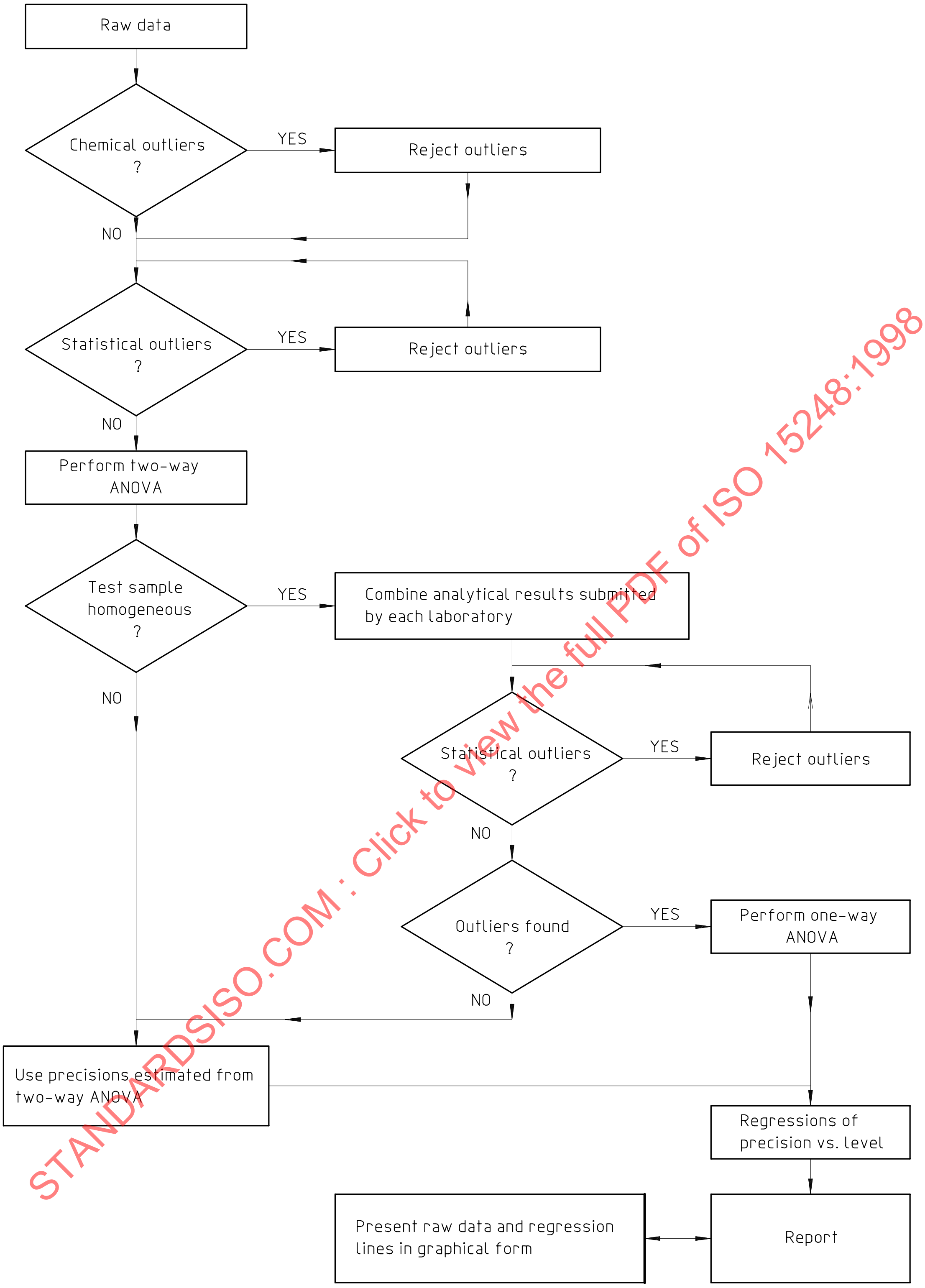


Figure E.1 — Flowsheet of procedure for statistical evaluation of analytical data resulting from international tests