
**Zinc sulfide concentrates — Determination
of zinc content — Solvent extraction and
EDTA titrimetric method**

*Concentrés sulfurés de zinc — Dosage du zinc — Méthode par extraction à
l'aide d'un solvant et titrage à l'EDTA*

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X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

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 13291 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

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

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Zinc sulfide concentrates — Determination of zinc content — Solvent extraction and EDTA titrimetric method

1 Scope

This International Standard specifies a solvent extraction/titrimetric method for the determination of the zinc content of zinc sulfide concentrates.

The method is applicable to zinc sulfide concentrates with zinc content in the range from 11 % (m/m) to 62 % (m/m).

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:—¹⁾, *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

A test portion of zinc concentrate is dissolved in bromine and nitric acid. Any remaining insoluble residue is dissolved in hydrofluoric and perchloric acids. A zinc thiocyanate complex is selectively extracted into methyl isobutyl ketone after screening of some elements with thiourea and citrate ions. Partially extracted cadmium is screened with iodide ions before the final titration as explained in annex C. Cobalt is extracted and must be determined separately if present at concentration levels exceeding 0,05 %. Zinc is determined by titration with EDTA solution at pH 5,5.

1) To be published. (Revision of ISO 1042:1983)

4 Reagents

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

4.1 Zinc metal, minimum purity 99,99 %, free from oxide prior to use.

The surface of the metal may be cleaned by immersing the metal in hydrochloric acid (4.2), diluted 1+9 for 1 min, washed well with water followed by an acetone rinse and dried in an oven at 50°C.

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

4.3 Bromine

4.4 Nitric acid, (ρ_{20} 1,42 g/ml)

4.5 Hydrochloric acid, (ρ_{20} 1,16 g/ml to 1,19 g/ml) diluted (1+4)

Add 200 ml of hydrochloric acid (4.2) to 800 ml of water.

4.6 Hydrofluoric acid, (ρ_{20} 1,13 g/ml to 1,15 g/ml)

4.7 Perchloric acid, (ρ_{20} 1,54 g/ml)

4.8 Ammonia, (ρ_{20} 0,89 g/ml)

4.9 4-methyl-2-pentanone (methyl isobutyl ketone)

4.10 Ethanol, pure or denatured

4.11 Screening solution

Dissolve 60 g of thiourea, 100 g of di-ammonium citrate and 200 g of ammonium thiocyanate in water and dilute to 1 l. Filter if necessary.

4.12 Sodium fluoride solution (20 g/l)

Dissolve 20 g of sodium fluoride in water. Dilute to 1 l.

4.13 Thiourea solution (100 g/l)

Dissolve 100 g of thiourea in water and dilute to 1 l.

4.14 Buffer solution

Dissolve 250 g of hexamethylenetetramine in water. Add 60 ml of acetic acid (ρ_{20} 1,05 g/ml) and dilute to 1 l.

4.15 EDTA standard volumetric solution (0,05 mol/l)

Dissolve 18,6 g of the di-sodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA) in water. Dilute to 1 l.

4.16 Potassium iodide solution (1 000 g/l)

Dissolve 100 g of potassium iodide in water and dilute to 100 ml.

Prepare fresh on the day of use.

4.17 Xylenol orange indicator (1 % m/m)

Mix 1 g of the sodium salt of xylenol orange with 99 g of potassium nitrate crystals by gently grinding in a ceramic mortar with a pestle. Mixing is considered complete when the colour is uniform throughout.

4.18 Iron stock solution

Dissolve 45 g of iron(III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) in water and dilute to 1 l.

5 Apparatus

5.1 Ordinary laboratory equipment and

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

5.3 Platinum crucibles, of 25 ml capacity.

5.4 Balance, capable of being read to 0,1 mg.

5.5 Oven, with temperature controlled at $105\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$.

5.6 Muffle furnace, having a maximum required operating temperature higher than $800\text{ }^\circ\text{C}$.

5.7 Laboratory hotplate.

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

Take multiple increments, extract a test portion from the test sample in such a manner that it is representative of the dish or tray. Weigh, to the nearest 0,1 mg, 2,5 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 Blank test

Determine a reagent blank. It is advisable to perform duplicate blank determinations every time an analysis is carried out on a laboratory sample. The blank samples are carried through the whole procedure apart from where no laboratory sample test portion is required. However, 5 ml of iron stock solution (4.18) should be added to assist in the determination of turbidity. The volume of EDTA titrant used is V_b .

7.3 Dissolution of test portion

Place the test portion in a 300 ml narrow-necked conical flask. Moisten the material with about 20 ml of water and add 2 ml to 3 ml of bromine (4.3). Allow to react at ambient temperature for 15 min swirling the flask and contents from time to time. Add 15 ml of nitric acid (4.4) and leave for a further 15 min. Place the flask on a hotplate (5.7) and bring gently to the boil to expel all bromine vapours. Cool, add 100 ml of water, heat to boiling, cool.

If no insoluble material is present, transfer the liquid to a 500 ml one-mark volumetric flask, rinsing the conical flask thoroughly. Make up to the mark with water.

If insoluble material is present, filter through a medium speed filter paper into a 500 ml one-mark volumetric flask. Rinse the filter thoroughly with water. Place the filter and insoluble residue in a platinum crucible (5.3) (see Note) and gently ash the paper in the muffle furnace (5.6) set at 800 °C. Add 2 ml of hydrofluoric acid (4.6) and then 5 ml of perchloric acid (4.7). Heat until dense perchloric acid fumes appear. Allow to cool, dilute with about 25 ml of water. Filter if a lead sulfate precipitate appears. Transfer the filtrate to the same one-mark volumetric flask used above. Rinse the filter paper thoroughly and make up to the mark with water.

NOTE — If the sample contains lead, damage to the platinum crucible may occur. In this case, fusion with sodium peroxide in a zirconium crucible is preferable.

7.4 Extraction

Pipette 50,00 ml of the solution obtained in 7.3 into a 250 ml separating funnel. Add ammonia (4.8) dropwise until a slight turbidity develops. Add 5 ml of dilute hydrochloric acid (4.5) and 50 ml of screening solution (4.11). Mix well. Add 80 ml of 4-methyl-2-pentanone (4.9) and shake for 1 min. Allow the phases to separate and slowly transfer the lower aqueous phase to another separating funnel. Perform a second extraction with 20 ml of 4-methyl-2-pentanone (4.9). Separate the phases and discard the aqueous phase. Combine the two separate organic phases in a 400 ml low-form beaker.

Place 1 ml of dilute hydrochloric acid (4.5) and 70 ml of ethanol (4.10) into each of the two separating funnels. Shake well and discharge the contents from both funnels into the 400 ml low-form beaker.

7.5 Titration

Add successively 10 ml of sodium fluoride solution (4.12), 10 ml of thiourea solution (4.13), 20 ml of buffer solution (4.14), 5 ml of potassium iodide solution (4.16) and 0,1 g of xylenol orange indicator (4.17).

Titrate with EDTA solution (4.15) until the colour changes from red to yellow. Titrate very slowly when approaching the equivalence point. Note the volume, V_t , of titrant used.

7.6 Determination of the titration factor of the EDTA standard solution

NOTE — In order to obtain a relative accuracy of between 0,1 % and 0,2 % it is necessary to standardize the EDTA solution with zinc at the same time and under the same conditions as the analysis. It is thus advisable for the calibration to follow the complete set of operating conditions set down for the analysis. Likewise, to improve the repeatability of the calibration it is useful to prepare several zinc reference solutions. Iron is added to the standard solution to assist in the determination of turbidity in 7.4.

The EDTA standard solution should be standardized as follows:

- Weigh into three separate 300 ml conical flasks, between 0,25 g and 1,625 g of zinc (4.1), to the nearest 0,000 1 g, depending on the zinc content of the test sample. Record these masses as m_1 , m_2 and m_3 .
- Add 15 ml of water, 15 ml of nitric acid (4.4) and 5 ml of iron stock solution (4.18). After dissolution of the zinc, boil gently to expel nitrogen oxide gases.
- Cool and transfer to a 500 ml one-mark volumetric flask. Rinse the initial flask thoroughly and add the washings to the volumetric flask. Make up to the mark. Continue the procedure as described in 7.4 and 7.5. Record the volumes of EDTA standard solution used in the titrations as V_1 , V_2 and V_3 .

Calculate the intermediate factor, f_{i_x} , for each beaker using the following equation:

$$f_{i_x} = m_x / V_x \quad \text{for } x = 1 \text{ to } 3 \quad \text{--- (1a)}$$

where

f_{i_x} is the factor obtained from the titration;

m_x is the mass of zinc weighed, in grams;

V_x is the volume of EDTA solution, in millilitres.

If the range of values for f_{i_1} , f_{i_2} , f_{i_3} exceeds 0,000 01 g/ml then repeat the standardization. Otherwise calculate the mean factor as follows:

$$f = \frac{f_{i_1} + f_{i_2} + f_{i_3}}{3} \quad \text{... (1b)}$$

8 Expression of results

The zinc content of the test portion, W_{Zn} , expressed as a percentage by mass, is given by the following equation:

$$W_{Zn} = \frac{(V_t - V_b) \times f \times 100}{m} \times \frac{100}{100 - H} \quad \text{... (2)}$$

where

f is the zinc equivalence factor, in grams per millilitre, determined in 7.6;

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

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

V_b is the volume of EDTA solution (4.15), in millilitres, used to titrate the blank solution;

V_t is the volume of EDTA solution (4.15), in millilitres, used to titrate the sample solution.

Calculate the zinc content of the test portion to the second decimal place.

9 Precision

9.1 Expression of precision

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

$$s_r = 0,000\ 8\bar{X} + 0,038\ 2 \quad \dots (3)$$

$$s_L = 0,001\ 6\bar{X} + 0,053\ 9 \quad \dots (4)$$

where

\bar{X} is the mean content of zinc, expressed as a percentage by mass, in the sample;

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.

NOTE — Additional information is given in annex D.

9.2 Procedure 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:
$$\bar{X} = \frac{(X_1 + X_2)}{2} \quad \dots (5)$$

Within-laboratory standard deviation (repeatability)
$$s_r = 0,000\ 8\bar{X} + 0,038\ 2 \quad \dots (3)$$

Repeatability limit
$$r = 2,8s_r \quad \dots (6)$$

9.3 Between-laboratories precision

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

Calculate the following quantities:

Mean of final results:
$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad \dots (7)$$

Between-laboratories standard deviation
$$s_L = 0,001\ 6\mu_{12} + 0,053\ 9 \quad \dots (8)$$

Within-laboratory standard deviation
$$s_r = 0,000\ 8\mu_{12} + 0,038\ 2 \quad \dots (9)$$

Permissible difference
$$P = 2,8 \sqrt{s_L^2 + \frac{s_r^2}{2}} \quad \dots (10)$$

Range
$$E = |\mu_1 - \mu_2| \quad \dots (11)$$

where

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

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

If E is equal to or less than 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). When the precision has been confirmed, the final laboratory result can be compared with the certified value, A_c . The following two possibilities exist:

$$|\mu_c - A_c| \leq C \quad \dots (12)$$

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

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

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

In equations (12) and (13), the symbols are defined as follows:

μ_c is the final result, as a percentage of zinc mass of the certified reference material;

A_c is the certified value, as a percentage of zinc mass of the certified reference material;

C is a quantity, as a percentage of zinc mass, depending on the type of certified reference material used as defined in 9.4.1.

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

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

9.4.1.1 Reference material certified/characterized by an interlaboratory test programme

The quantity C (see 9.4), expressed as a percentage by mass of zinc, is given by the following equation:

$$C = 2 \sqrt{s_L^2 + \frac{s_r^2}{n} + s^2 \{A_c\}} \quad \dots (14)$$

where

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

n is the number of replicate determinations.

9.4.1.2 Reference material certified/characterised by one laboratory

The quantity C (see 9.4), expressed as a percentage by mass of zinc, is given by the following equation:

$$C = 2\sqrt{s_L^2 + \frac{s_r^2}{n}} \quad \dots (15)$$

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 13291;
- c) zinc content of the sample, expressed as a percentage by mass;
- d) date on which the test was carried out;
- e) any occurrence noticed during the determination which may have had an influence on the results.

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Annex A (normative)

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

A.1 Scope

This method is applicable to zinc 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 $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. 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 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 (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 $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

A.5 Procedure

A.5.1 Preparation of the weighing vessel

Dry the weighing vessel and vessel cover (A4.2) by heating in a laboratory oven (A4.3) at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 1 h. Transfer the vessel and vessel cover to a desiccator containing suitable fresh desiccant (A3.1) and allow to cool to ambient temperature.

A.5.2 Test portion

Weigh the dried weighing vessel and vessel cover (A5.1). Immediately add a portion 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.

A5.3 Determination of the test portion dry mass

Transfer the uncovered weighing vessel and test portion and vessel cover to the laboratory oven (A4.3) and dry at $105\text{ °C} \pm 5\text{ °C}$ 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_4) 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_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 $105\text{ °C} \pm 5\text{ °C}$ and to re-weigh the weighing vessel and test portion plus vessel cover to the nearest 0,1 mg (m_4'). The mass of the test portion can be considered to be constant if the difference between m_4 and m_4' 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_6 , in grams, given by the following equation:

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

where

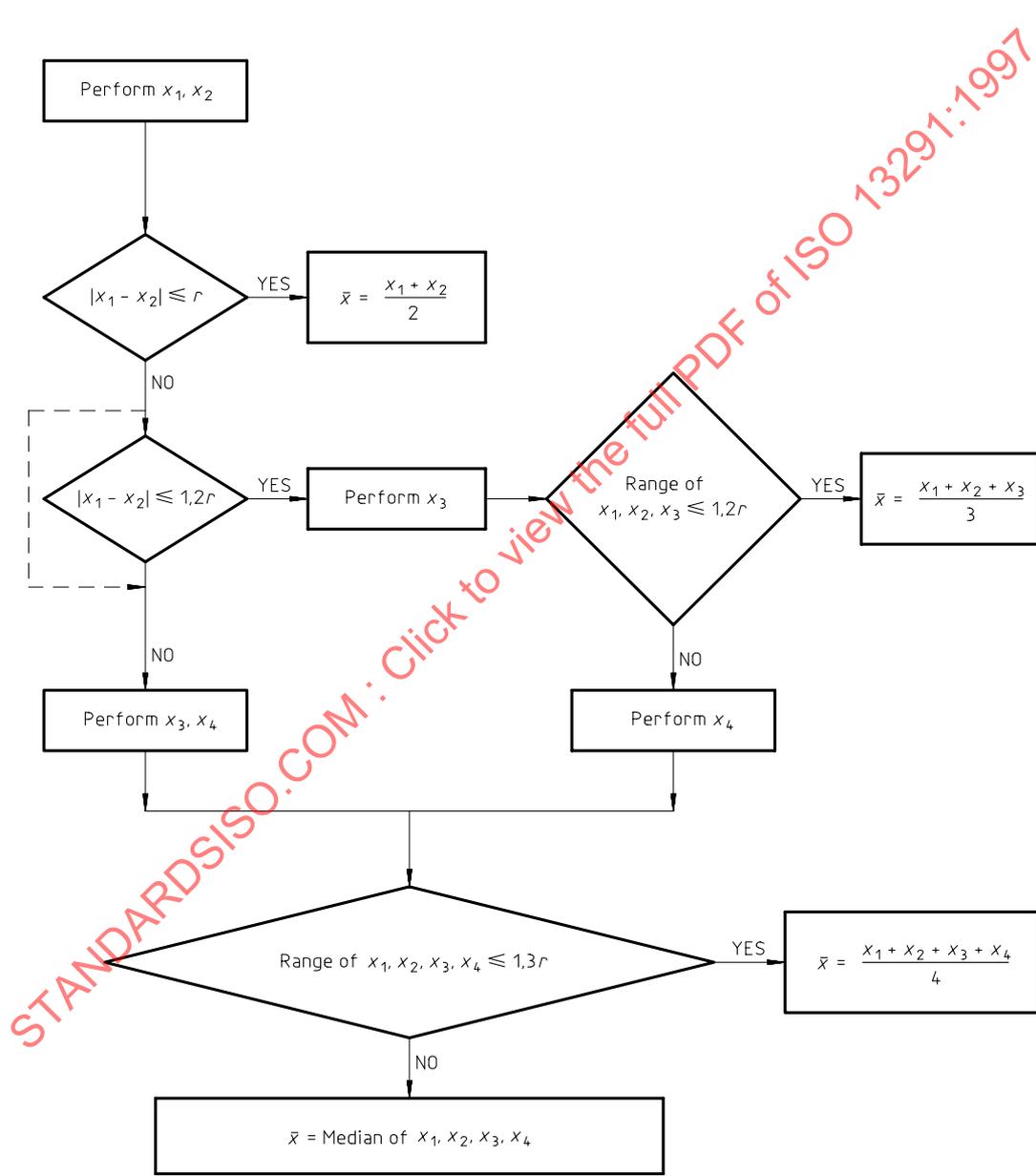
m_4 is the mass, in grams, of the dried test portion plus weighing vessel and its cover;

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



r: defined in 9.2

Annex C (normative)

Elements that interfere with this method

C.1 Cadmium

Cadmium is largely extracted at the same time as zinc and must be screened with potassium iodide before the final titration.

C.2 Cobalt

A large part of any cobalt present in the test sample is extracted with the zinc as evidenced by the blue colour taken on by the organic phase. If the cobalt content is less than 0,05 % its influence is negligible. If the cobalt content is higher it should be determined after the EDTA titration by atomic absorption spectrometry. The amount of cobalt found is then subtracted from the zinc value by multiplying the cobalt concentration to obtain an equivalent zinc concentration.

C.3 Lead

Lead can interfere at values higher than 7 %. It is precipitated as lead sulfate in the dissolution stage and can be removed by filtration.

C.4 Manganese

Manganese interferes at values greater than 2 % by lowering the result. It is rare to have an internationally traded zinc concentrate with this level of manganese.

C.5 Magnesium

Interference occurs for concentrations of magnesium greater than 5 % by lowering the result. The highest value reported for internationally traded zinc concentrates is 0,8 %.

C.6 Titanium and vanadium

Titanium and vanadium interfere only when present in concentrations greater than 0,2 %. No such levels are known to exist in internationally traded zinc concentrates.

C.7 Other elements

It has been established that other elements do not interfere at values less than the following concentrations:

Al : 2 % Ba : 10 % Cu : 10 % Ni : 1 %

As : 0,5 % Ca : 5 % Fe : 15 % Sr : 2 %

Annex D (informative)

Derivation of precision equations

D.1 Introduction

This International Standard was tested in an interlaboratory test programme involving nine countries and twenty two laboratories. Five samples of zinc concentrate covering the range 11 % (m/m) to 62 % (m/m) were analysed for zinc content. The test programme was designed to determine the repeatability and within-laboratory and between-laboratories reproducibilities in general, using the principle of ISO 5725-2 [1].

D.2 Design of the test programme

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

D.3 Test samples

This test programme used five samples of zinc concentrate. The composition of these samples is shown in table D.1.

D.4 Statistical evaluation

The procedure for statistical evaluation is illustrated schematically in figure D.1. The results of the statistical evaluation are summarized in tables D.2 and D.3. The estimated precision (s_r , s_L , r and P) are plotted against their corresponding sample means on a graph as shown in figure D.2, and the regression equations of these precisions against sample means were computed and are presented in table D.2.

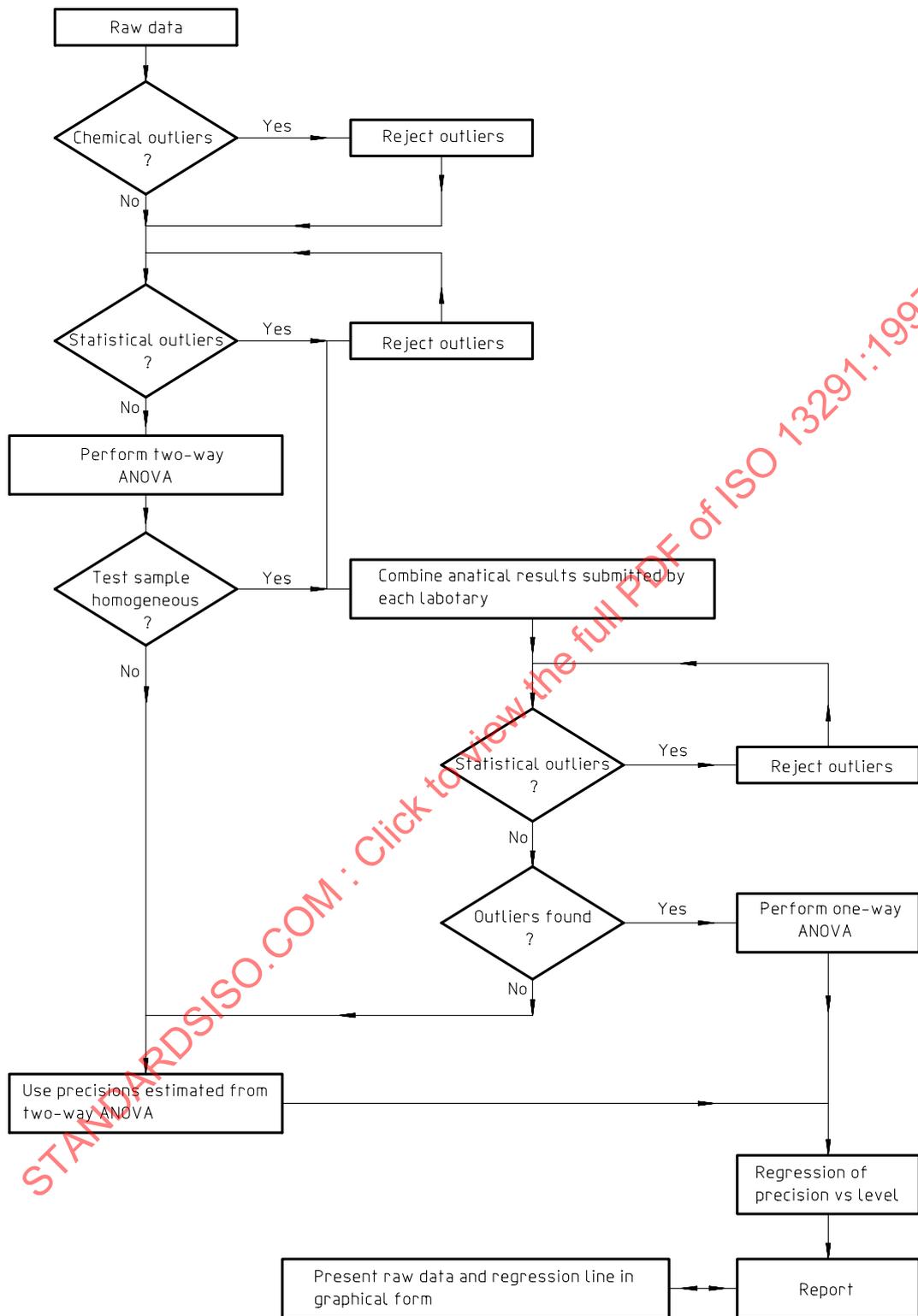


Figure D.1 — Flowchart of procedure for statistical evaluation of analytical data resulting from international tests

Table D.1 — Composition of zinc concentrate samples

Element	Unit	Sample numbers ¹⁾				
		90-1	90-2	90-3	91-9	91-10
Zn	% (m/m)	50	33	62	11	55
S	% (m/m)	30	30	30	20	31
Fe	% (m/m)	10	14	2	8	8
Pb	% (m/m)	3	14	1	53	2
Si	% (m/m)	2	2	-	2	1
Al	% (m/m)	0,5	0,6	0,3	0,2	0,1
Cu	% (m/m)	0,2	0,5	0,1	0,8	0,1
Ca	% (m/m)	0,4	0,5	0,3	0,1	0,1
Mg	% (m/m)	0,3	0,3	0,2	0,8	0,2
Cd	% (m/m)	0,2	0,1	0,2	0,02	0,2
As	g/t	300	550	-	250	100
Sb	g/t	250	700	-	300	20
Ag	g/t	125	400	60	380	160
Co	g/t	40	90	<1	60	20
Tl	g/t	25	60	1	-	10
Ni	g/t	2	8	2	-	8
Bi	g/t	<1	<1	<1	1 600	<1

1) 90-1MIM (Australia)
90-2MIM (Australia)
90-3Polaris (Canada)
91-9Cobar (Australia)
91-10Hilton (Australia)