BS ISO 15247:2015

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Zinc sulfide concentrates — Determination of silver content — Acid dissolution and flame atomic absorption spectrometric method

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee NFE/36, Copper lead and zinc ores and concentrates.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Second edition
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Zinc sulfide concentrates — Determination of silver content — Acid dissolution and flame atomic absorption spectrometric method

Concentrés sulfurés de zinc — Dosage de l'argent — Méthode par dissolution acide et spectrométrie d'absorption atomique dans la flamme

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Foreword

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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

The committee responsible for this document is ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

This second edition cancels and replaces the first edition (ISO 15247:1999), of which the warning in A.3.1 in Annex A has been revised.

Zinc sulfide concentrates — Determination of silver content — Acid dissolution and flame atomic absorption spectrometric method

1 Scope

This International Standard specifies an acid dissolution and flame atomic absorption spectrometric method for the determination of silver content of zinc sulfide concentrates.

The method is applicable to the determination of silver 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.

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

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

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

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

3 Principle

Decomposition of the concentrate in hydrochloric and nitric acids. Dissolution of the digestion residue in hydrochloric acid and measurement by flame atomic absorption at 328,1 nm.

4 Reagents

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

4.1 Silver metal, minimum 99,99 % purity.

4.2 Nitric acid, (ρ_{20} 1,42 g/ml), chloride content < 0,5 mg/ml.

4.3 Nitric acid, (500 ml/l).

To 250 ml of water carefully add, with stirring, 250 ml of nitric acid (4.2).

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

4.5 Hydrochloric acid, (200 ml/l).

To 800 ml of water carefully add, with stirring, 200 ml of hydrochloric acid (4.4).

4.6 Ammonia solution, (ρ_{20} 0,89 g/ml).

4.7 Ammonia solution, (250 ml/l).

To 750 ml of water add, with stirring, 250 ml of ammonia solution (4.6).

4.8 Silver standard solutions

4.8.1 Silver standard solution, (1 000 µg/ml).

Weigh 0,500 0 g of silver metal (4.1) into a 250 ml conical beaker, add 50 ml of nitric acid (4.3), cover, and heat gently until the metal dissolves. Remove the cover and evaporate gently to near dryness. Add 250 ml of hydrochloric acid (4.4) and warm until the solution clears. Cool, and transfer to a 500 ml volumetric flask. Dilute to volume with water and mix thoroughly.

This solution should be freshly prepared, unless it is being used on a regular basis.

4.8.2 Silver standard solution, (100 µg/ml).

Pipette 10 ml of silver standard solution (4.8.1) into a 100 ml volumetric flask containing 45 ml of hydrochloric acid (4.4). Dilute to volume with water and mix thoroughly.

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

Silver standard solutions should be stored in brown glass bottles.

4.9 Calibration solutions

To six 100 ml volumetric flasks, each containing 20 ml of hydrochloric acid (4.4), add from a burette (5.2) 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, and 5 ml of silver standard solution (4.8.2). Dilute to volume with water and mix thoroughly.

These standards contain 0 µg, 1 µg, 2 µg, 3 µg, 4 µg, and 5 µg of silver per ml and shall be freshly prepared.

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

5 Apparatus

Ordinary laboratory apparatus and the following.

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

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

5.3 Atomic absorption spectrometer (AAS), equipped with a glass bead in the spray chamber rather than a flow spoiler.

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

6 Samples

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 1 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 can 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 and 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 all reagents specified in the determination but omitting the test portion. The purpose of the blank test in this method is to check the quality of the reagents. If a significant blank value is obtained as a result of the blank test, check all reagents and rectify the problem.

7.3 Decomposition of test portion

Transfer the test portion to a 250 ml conical beaker and moisten with 1 ml of water.

All glassware should be washed in ammonia (4.7) and rinsed with water prior to use to remove any silver adhering to the glass surface.

Add 35 ml of nitric acid (4.3), cover with a watch glass, and heat at a low temperature until the reaction ceases.

Add 10 ml of hydrochloric acid (4.4), raise the cover slightly, and evaporate to dryness. Remove from the hotplate and cool.

Add a further 10 ml of hydrochloric acid (4.4) and again evaporate to dryness.

Rapid heating can cause samples to splatter or spit. Care should be taken to ensure that this does not occur.

Re-dissolve in 25 ml of hydrochloric acid (4.5). Heat to boiling, remove from the hotplate and cool.

Transfer the solution to the volumetric flask indicated in Table 1 by washing and diluting to volume with hydrochloric acid (4.5).

Table 1 — Volumetric flask size

Ag content g/t	**Volumetric flask** ml
10 to 250	50
250 to 500	100

7.4 Determination of silver

Determine the silver content of the test portion by flame atomic absorption spectrometry using calibration solutions (4.9). 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 correction: none

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 calibration.

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

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

The silver content of the test portion, w_{Ag}, expressed in grams per tonne, is given by Formula (1):

$$w_{Ag} = \frac{C \times V}{m} \times \frac{100}{100 - H} \quad (1)$$

where

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

V is the volume of the analysis solution, in millilitres (see 7.3);

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 pre-dried test portion being used, $H = 0$).

9 Precision

9.1 Expression of precision

The precision of this analytical method is expressed by Formulae (2) and (3):

$$s_r = 0{,}009\,5\,\overline{X} + 0{,}182\,6 \tag{2}$$

$$s_L = 0{,}031\,1\,\overline{X} + 0{,}881\,3 \tag{3}$$

where

$\overline{X}$ is the mean content of silver, in grams per tonne, in the sample;

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

s_R is the between-laboratories standard deviation, in grams of silver per tonne.

NOTE Additional information is given in Annex C.

9.2 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:

a) Mean of duplicates:

$$\overline{x} = \frac{x_1 + x_2}{2} \tag{4}$$

b) Within-laboratory standard deviation:

$$s_r = 0{,}009\,5\,\overline{X} + 0{,}182\,6$$

c) Repeatability limit:

$$r = 2{,}8\,s_r \tag{5}$$

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:

a) Mean of final results:

$$\mu_{1,2} = \frac{\mu_1 + \mu_2}{2} \tag{6}$$

b) Within-laboratory standard deviation:

$$s_L = 0{,}031\,1\,\mu_{1,2} + 0{,}881\,3 \tag{7}$$

c) Between-laboratories standard deviation:

$$s_r = 0{,}009\,5\,\mu_{1,2} + 0{,}182\,6 \tag{8}$$

d) Permissible difference:

$$P = 2{,}8\sqrt{s_L^2 + \frac{s_r^2}{2}} \tag{9}$$

e) Range:

$$E = |\mu_1 - \mu_2| \tag{10}$$

where

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

μ_2 is the final result, in grams of silver per tonne, 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) 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:

a) $$|\mu_C - A_C| \leq C \tag{11}$$

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

b) $$|\mu_C - A_C| > C \tag{12}$$

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

where

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

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

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

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

Where the reference material is certified/characterized by an interlaboratory test programme, the quantity C, in grams of silver per tonne, is given by Formula (13):

$$C = 2\sqrt{s_{\mathrm{L}}^2 + \frac{s_{\mathrm{r}}^2}{n} + s^2\{A_{\mathrm{c}}\}} \tag{13}$$

where

$s^2\{A_{\mathrm{c}}\}$ is the variance of the certified value;

n is the number of replicate determinations.

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

$$C = 2\sqrt{2\,s_{\mathrm{L}}^2 + \frac{s_{\mathrm{r}}^2}{n}} \tag{14}$$

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 reports

The test report shall include the following information:

a) identification of the sample;

b) a reference to this International Standard, i.e. ISO 15247;

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

d) date on which the test was carried out;

e) any occurrences noticed during the determination which could 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 General

This Annex 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 Reagent

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 (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 its 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

Tare the dried weighing vessel and vessel cover (A.5.1). Immediately add a proportion of the laboratory sample to provide a suitable pre-dried 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 cooling 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 into 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.

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 dry mass of the test portion

The dry mass of the test portion m_3, in grams, is given by Formula (A.1):

$$m_3 = m_1 - m_2 \tag{A.1}$$

where

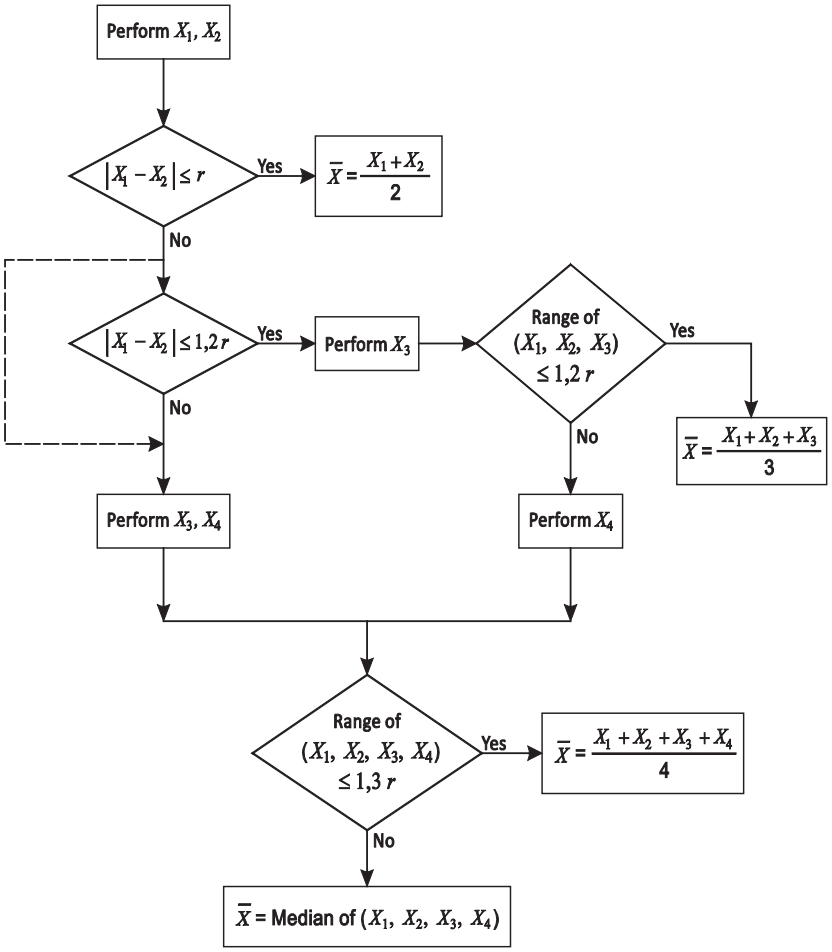
m_1 is the mass, in grams, of the dried test portion plus the weighing vessel and its cover;

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)

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



NOTE r is defined in 9.2.

Figure B.1 — Flowsheet of the procedure for the acceptance of analytical values for test samples

Annex C
(informative)

Derivation of precision equations

C.1 General

This International Standard was tested in an interlaboratory test programme involving eight countries and 15 laboratories. Five samples of zinc concentrate covering the range up to 60 % (m/m) were analysed to determine the silver content. The test programme was designed to determine the repeatability and within-laboratory and between laboratories reproducibilities in general, using the principles of ISO 5725-2.

C.2 Design of the 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.

C.3 Test samples

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

Table C.1 — Composition of copper concentrated samples

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

[a] Cominco (Canada).

[b] Peak (Australia).

C.4 Statistical evaluation

The procedure for statistical evaluation is illustrated schematically in Figure C.1. The results of the statistical evaluation are summarized in Table C.2.

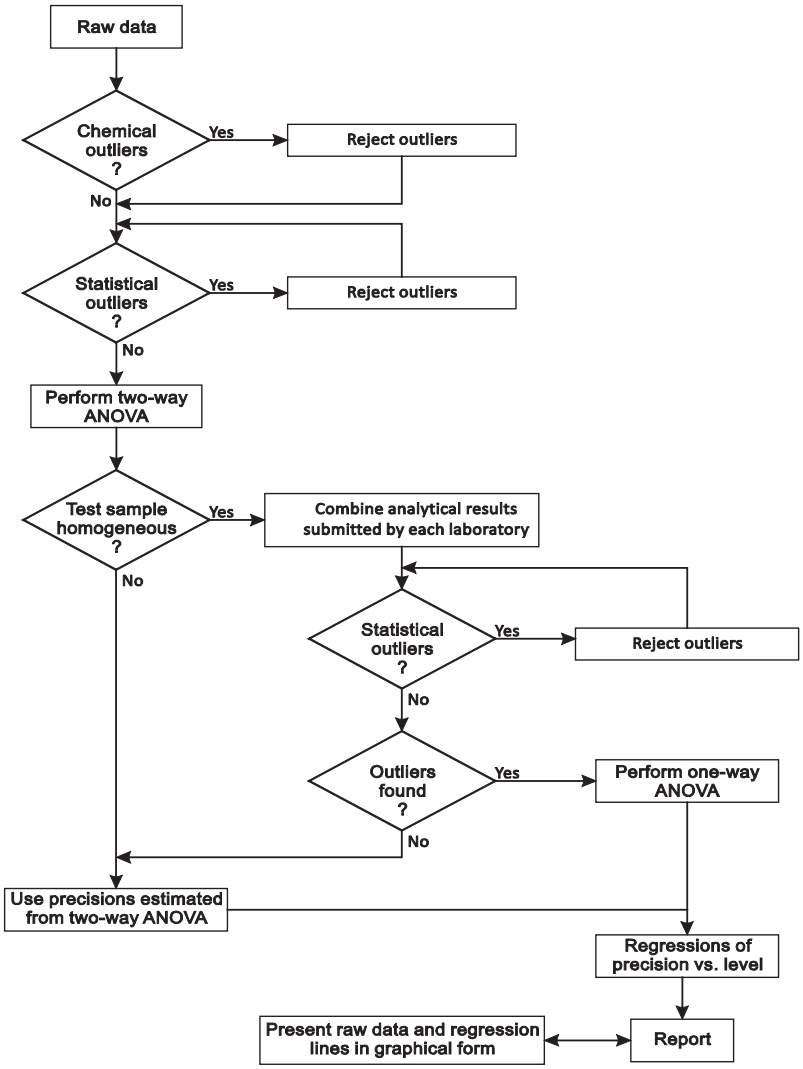


Figure C.1 — Flowsheet of procedure for statistical evaluation of analytical data resulting from international tests

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

Table C.2 — Summary of precisions for all samples — Silver

Sample number (see Table C.1)	k_0	k	n_0	n	$\overline{\overline{X}}$	r	P	s_r	s_L	s_L/s_r
89–1	15	15	58	58	300,066	86,044	256,032	30,404	90,471	2,98
89–2	15	15	57	57	131,741	50,124	168,962	17,712	59,704	3,37
89–3	15	15	58	58	338,620	92,183	345,901	32,573	120,813	3,71
91–16	15	15	58	55	18,877	0,769 7	46,059	0,272 0	16,275	5,98
93–1	15	15	58	44	14,999	0,549 2	19,689	0,194 1	0,695 7	3,58

Regression equations	Correlation coefficient
$r = 0{,}026\,8\,\overline{X} + 0{,}881\,3$	0,991
$P = 0{,}088\,8\,\overline{X} + 2{,}451\,5$	0,982
$S_r = 0{,}009\,5\,\overline{X} + 0{,}182\,6$	0,991
$S_L = 0{,}031\,1\,\overline{X} + 0{,}881\,3$	0,983

k_0 is the total number of participating laboratories

k is the number of participating laboratories used for computation of precision

n_0 is the total number of analytical results

n is the number of analytical results used for computation of precision

$\overline{\overline{X}}$ is the overall mean of the silver content, in grams per tonne

r is the permissible within-laboratory tolerance (repeatability), in grams of silver per tonne

P is the permissible between-laboratories tolerance, in grams of silver per tonne

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

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

$\overline{X}$ is the mean content of silver, in grams per tonne of the sample

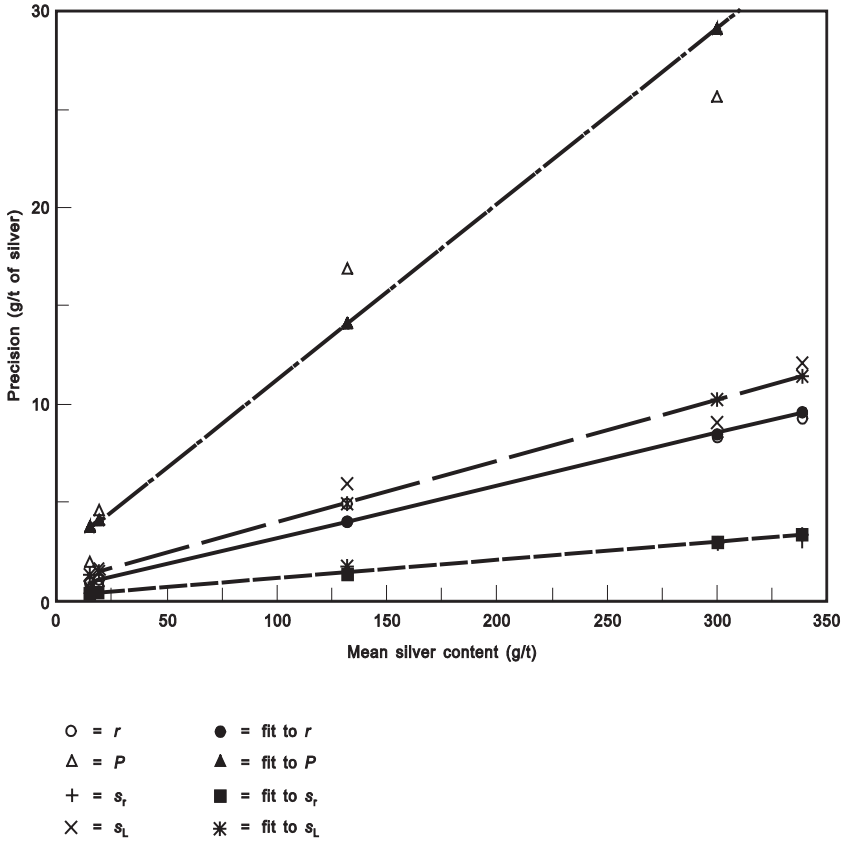


Figure C.2 — Least-squares fit of precision against the mean silver content

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[1] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[2] ISO Guide 35, *Reference materials — General and statistical principles for certification*

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