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Copper, lead, zinc and nickel concentrates — Experimental methods for checking the precision of sampling

Concentrés de cuivre, de plomb, de zinc et de nickel — Méthodes expérimentales de contrôle de la fidélité de l'échantillonnage

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

This third edition cancels and replaces the second edition (ISO 12744:2006), which has been technically revised.

The main changes are as follows:

— the precisions of sampling, sample preparation and measurement are now estimated from the mean squared differences between duplicates rather than simply the mean differences, which provides a better unbiased estimate of precision.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Copper, lead, zinc and nickel concentrates — Experimental methods for checking the precision of sampling

WARNING — This document can involve hazardous materials, operations and equipment. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies methods for checking the precision of primary sampling, sample processing, chemical analysis, physical testing and determination of moisture content of copper, lead, zinc and nickel concentrates being carried out in accordance with the methods specified in ISO 12743, expressed in terms of standard deviations.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10258, *Copper sulfide concentrates — Determination of copper content — Titrimetric methods*

ISO 11441, *Lead sulfide concentrates — Determination of lead content — Back titration of EDTA after precipitation of lead sulfate*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Symbols

k	number of lots
n	number of increments
R_1	absolute difference between duplicates for interleaved samples A and B
R_2	absolute difference between means for divided interleaved samples A_1 and A_2, and B_1 and B_2
R_3	absolute difference between means for interleaved sample A and interleaved sample B
s	estimated value of standard deviation, σ

s_1^2 estimated variance from R_1^2

s_2^2 estimated variance from R_2^2

s_3^2 estimated variance from R_3^2

s_A estimated standard deviation of analysis

s_P estimated standard deviation of sample processing

s_S estimated standard deviation of sampling

s_{SP} estimated standard deviation of sampling and sample processing

s_T estimated total standard deviation of sampling, sample processing and analysis

x_{i1} first duplicate result for interleaved sample, where i = 1 and 2 and indicates interleaved sample A or B

x_{i2} second duplicate result for interleaved sample, where i = 1 and 2 and indicates interleaved sample A or B

x_{ij1} first duplicate result for interleaved sample, where i = 1 and 2 and indicates interleaved sample A or B, and j = 1 or 2 and indicates laboratory samples A_1 or A_2, and B_1 or B_2

x_{ij2} second duplicate result for sample, where i = 1 and 2 and indicates interleaved sample A or B, and j = 1 or 2 and indicates laboratory samples A_1 or A_2, and B_1 or B_2

$\bar{x}$ mean value of duplicate results

$\bar{\bar{x}}$ mean of mean value of duplicate results

$\bar{\bar{\bar{x}}}$ mean of $\bar{\bar{x}}$ values, and grand mean for sample processing method 3

$\bar{\bar{\bar{x}}}$ grand mean of all results for sample processing methods 1 and 2

5 General conditions

5.1 General

The determination of precision of primary sampling is based on collecting pairs of interleaved samples from each lot. If sample processing and measurement are also carried out in duplicate, it is possible to determine the precision of sample processing and analysis.

5.2 Number of lots

It is recommended that pairs of interleaved samples should be collected from more than 20 lots of the same type of concentrate, in order to reach a reliable conclusion. The lot size shall be chosen to ensure that more than 20 lots are available for the precision determination.

5.3 Number of increments and number of samples

The minimum number of increments for checking precision should preferably be twice the number determined in accordance with ISO 12743. Hence, if the number of increments required for routine sampling is n and one lot sample is constituted, the minimum number of increments should be $2n$, and two interleaved samples shall be constituted.

Alternatively, if the precision is being checked as part of routine sampling, n increments may be taken and two interleaved samples constituted, each comprising $n/2$ increments. The sampling precision thus obtained shall be divided by $\sqrt{2}$ to obtain the sampling precision for lot samples comprising n increments.

5.4 Sample processing and analysis

Sample processing shall be carried out in accordance with ISO 12743. The analysis of samples shall be carried out according to the methods specified in the relevant International Standards, such as ISO 10258, ISO 11441 and ISO 12739.

5.5 Frequency of precision checks

It is recommended that, even after a precision check has been conducted, further checks should be carried out at regular intervals. Precision checks should also be carried out when there is a change in equipment.

Because of the large amount of work involved in checking precision, it is recommended that checks should be carried out as a part of routine sampling and analysis.

6 Method of experiment

6.1 Interleaved samples

Each alternate primary increment shall be diverted so that pairs of interleaved samples A and B are formed. The number of divided increments per primary increment should be the same as for routine sampling. An example of a sampling plan for producing pairs of interleaved samples A and B is shown in Figure 1.

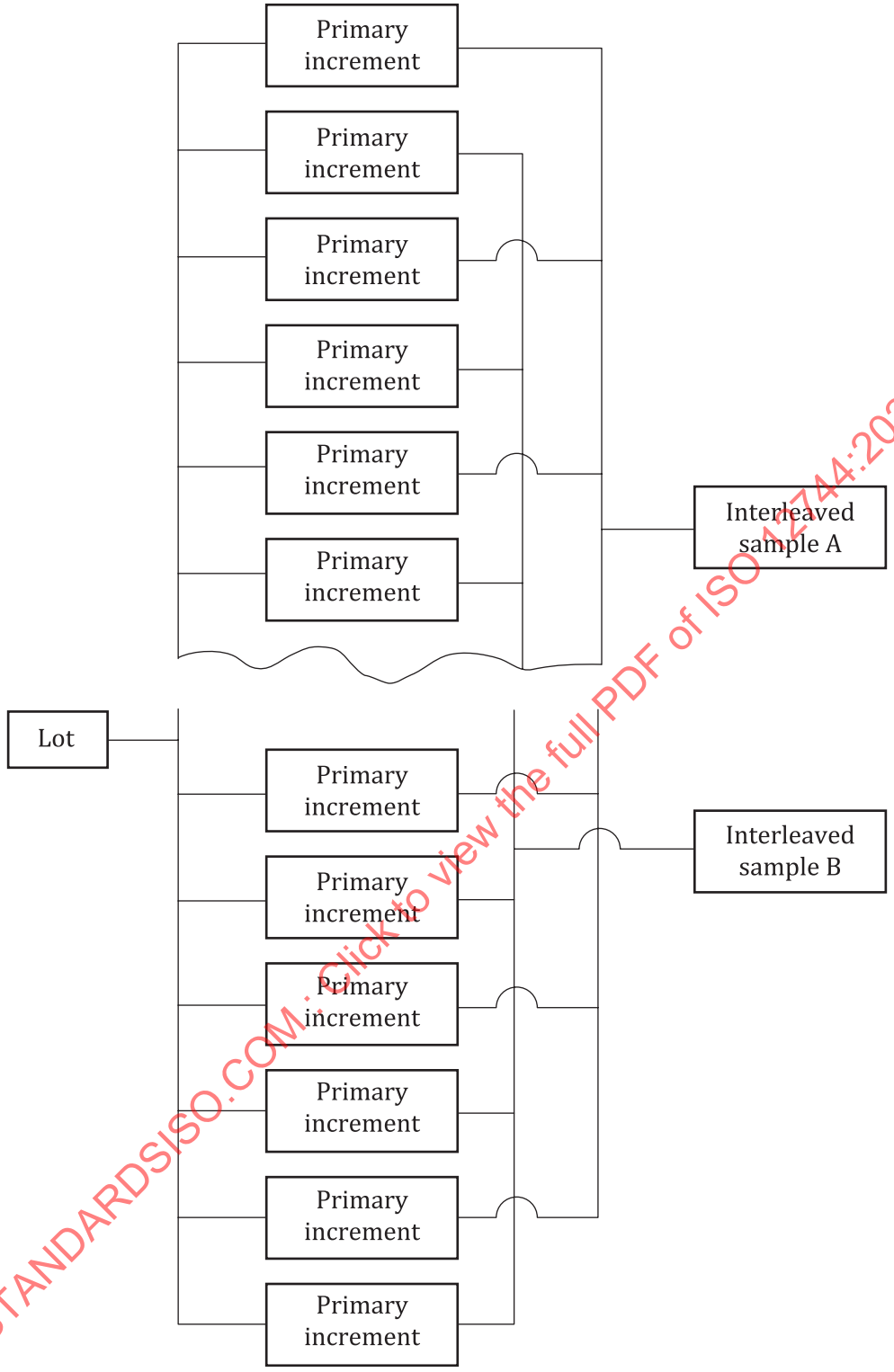

Figure 1 — Example of a plan for interleaved duplicate sampling

6.2 Sample processing and analysis

6.2.1 General

The pairs of interleaved samples A and B taken in accordance with 6.1 shall be divided separately and subjected to method 1, method 2 or method 3 sample processing and analysis as described in 6.2.2, 6.2.3 or 6.2.4.

6.2.2 Sample processing method 1

The two interleaved samples A and B shall be divided separately to prepare four laboratory samples: A_1, A_2, B_1 and B_2. These laboratory samples shall each be analysed in duplicate, and the duplicates designated as follows:

— x_{111} and x_{112} for sample A_1;

— x_{121} and x_{122} for sample A_2;

— x_{211} and x_{212} for sample B_1;

— x_{221} and x_{222} for sample B_2.

See Figure 2.

The eight determinations shall be run in random order, by the same analyst on the same day using the same analytical equipment. An example is given in Annex A.

NOTE By using method 1, the estimated precisions of sampling, sample processing and analysis can be obtained separately.

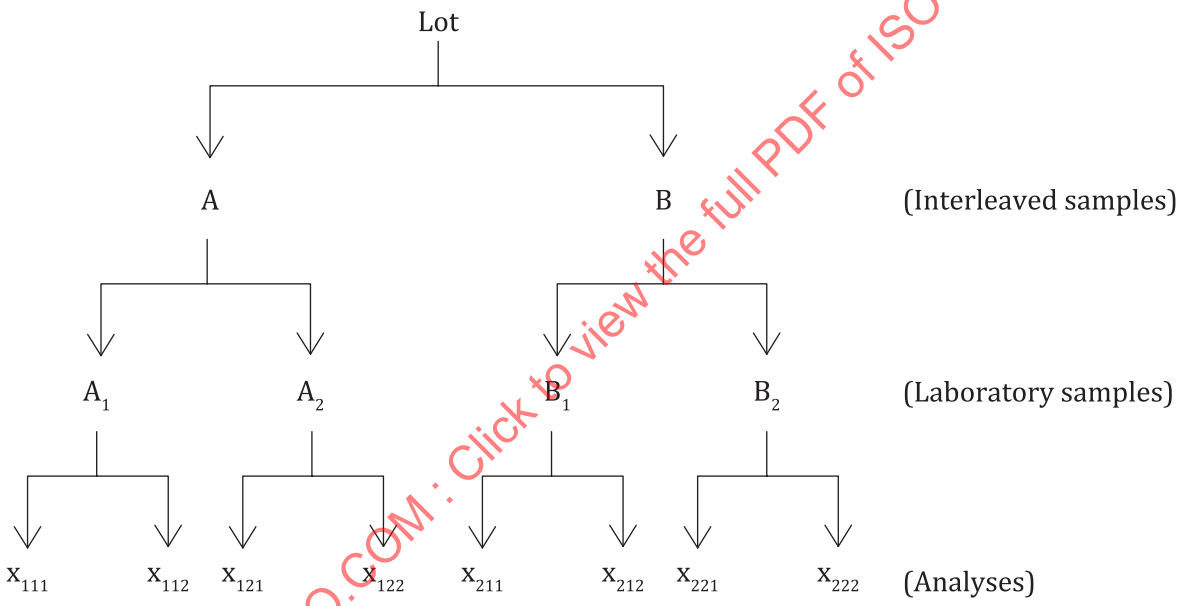

Figure 2 — Flowsheet for sample processing method 1

6.2.3 Sample processing method 2

Sample A shall be divided to prepare two laboratory samples: A_1 and A_2. From sample B, only one laboratory sample shall be prepared. The laboratory samples shall each be analysed in duplicate, and the duplicates designated as follows:

— x_{111} and x_{112} for sample A_1;

— x_{121} and x_{122} for sample A_2;

— x_{21} and x_{22} for sample B.

See Figure 3.

The six determinations shall be run in random order, by the same analyst on the same day using the same analytical equipment.

NOTE By using method 2, the estimated precisions of sampling, sample processing and analysis can be obtained separately. However, the estimated values will be less precise than those obtained using method 1.

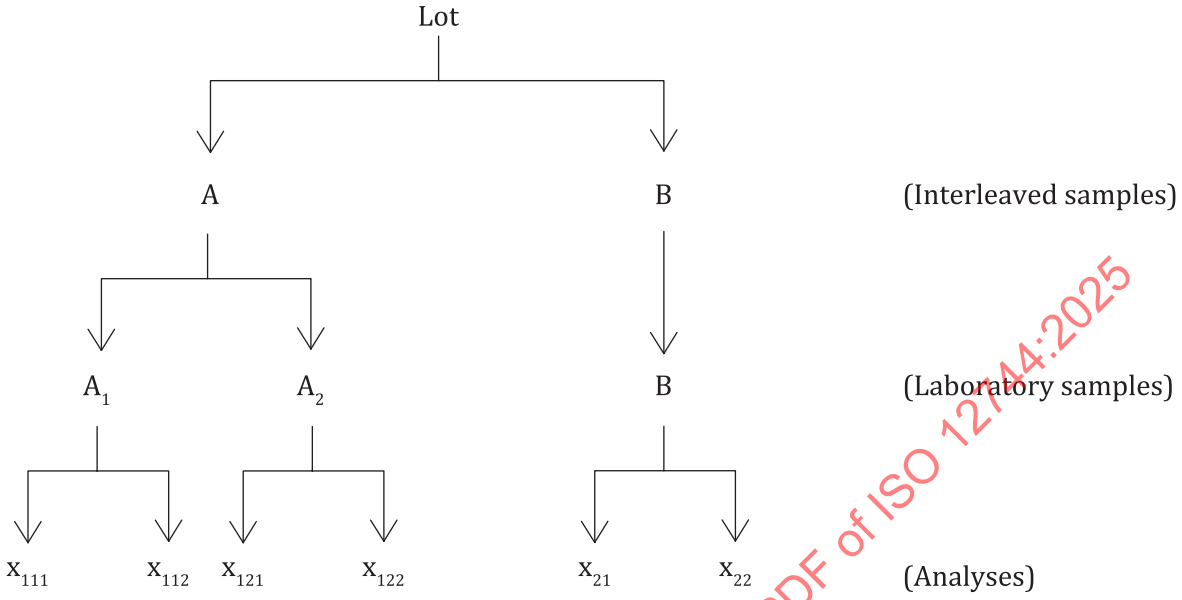

Figure 3 — Flowsheet for sample processing method 2

6.2.4 Sample processing method 3

From each of the two interleaved samples A and B, one laboratory sample shall be prepared. The two laboratory samples A and B shall be analysed in duplicate, and the measurements obtained shall be designated as follows:

— x_{11} and x_{12} for sample A;

— x_{21} and x_{22} for sample B.

See Figure 4.

The four determinations shall be run in random order, by the same analyst on the same day using the same analytical equipment.

NOTE By using method 3, only the estimated precision of analysis and the combined precision of sampling and sample processing are obtained.

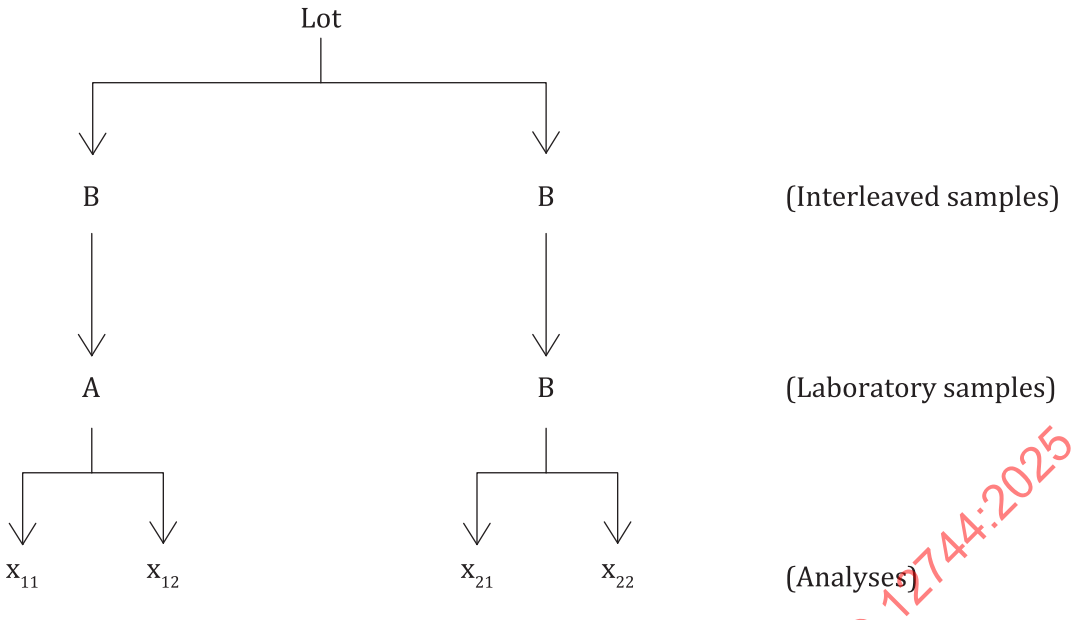

Figure 4 — Flowsheet for sample processing method 3

7 Evaluation of experimental data

7.1 General

The method for evaluation of experimental data shall be as specified in 7.2, 7.3 or 7.4, depending on the method of sample processing selected.

7.2 Sample processing method 1

7.2.1 The estimated standard deviations shall be calculated in accordance with the procedure given in 7.2.2 to 7.2.9.

7.2.2 Calculate the mean and range for each pair of duplicates using Formulae (1) to (2):

$$\bar{x}_{ij} = \frac{1}{2}\left(x_{ij1} + x_{ij2}\right) \quad (1)$$

$$R_1 = \left|x_{ij1} - x_{ij2}\right| \quad (2)$$

where

i is 1 and 2, representing interleaved samples A and B, respectively;

j is 1 and 2, representing laboratory samples A_1 and A_2 or B_1 and B_2.

7.2.3 Calculate the mean of the mean value and range for each pair of duplicates using Formulae (3) to (4):

$$\bar{\bar{x}}_i = \frac{1}{2}\left(\bar{x}_{i1} + \bar{x}_{i2}\right) \quad (3)$$

$$R_2 = \left|x_{i1} - x_{i2}\right| \quad (4)$$

7.2.4 Calculate the mean and range for each pair of interleaved samples, A and B using Formulae (5) to (6):

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(\bar{\bar{x}}_1 + \bar{\bar{x}}_2) \tag{5}$$

$$R_3 = |\bar{\bar{x}}_1 - \bar{\bar{x}}_2| \tag{6}$$

7.2.5 Calculate the grand mean and the variances s_1^2, s_2^2 and s_3^2 using Formulae (7) to (10):

$$\bar{\bar{\bar{x}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \tag{7}$$

$$s_1^2 = \frac{1}{8k}\Sigma R_1^2 \tag{8}$$

$$s_2^2 = \frac{1}{4k}\Sigma R_2^2 \tag{9}$$

$$s_3^2 = \frac{1}{2k}\Sigma R_3^2 \tag{10}$$

where k is the number of lots.

7.2.6 Calculate the estimated values of the variance of analysis (s_A^2), sample processing (s_P^2) and sampling (s_S^2) using Formulae (11) to (13):

$$s_A^2 = s_1^2 \tag{11}$$

$$s_P^2 = s_2^2 - \frac{1}{2}s_1^2 \tag{12}$$

$$s_S^2 = s_3^2 - \frac{1}{2}s_2^2 \tag{13}$$

7.2.7 Calculate the total variance of sampling, sample processing and analysis (s_T^2) using Formula (14):

$$s_T^2 = s_S^2 + s_P^2 + s_A^2 \tag{14}$$

7.2.8 Calculate the estimated values of the total standard deviation (s_T) and the standard deviations of sampling (s_S), sample processing (s_P) and analysis (s_A).

7.2.9 Compare the values of s_T, s_S, s_P and s_A thus obtained with the desired standard deviations.

7.3 Sample processing method 2

7.3.1 The estimated standard deviations of sampling, sample processing and analysis shall be calculated in accordance with the procedure given in 7.3.2 to 7.3.9.

7.3.2 Calculate the mean and range for each pair of duplicates using Formulae (15) to (16):

$$\bar{x} = \frac{1}{2}(x_{111} + x_{112}), \frac{1}{2}(x_{121} + x_{122}), \frac{1}{2}(x_{21} + x_{22}) \tag{15}$$

$$R_1 = |x_{111} - x_{112}|, |x_{121} - x_{122}|, |x_{21} - x_{22}| \quad (16)$$

7.3.3 Calculate the mean and range for duplicates A_1 and A_2 using Formulae (17) to (18):

$$\bar{\bar{\bar{x}}} = \frac{1}{4}(x_{111} + x_{112} + x_{121} + x_{122}) \quad (17)$$

$$R_2 = \frac{1}{2}|(x_{111} + x_{112}) - (x_{121} + x_{122})| \quad (18)$$

7.3.4 Calculate the mean and range for each pair of interleaved samples, A and B using Formulae (19) to (20):

$$\bar{\bar{\bar{x}}} = \frac{1}{2}\left[\bar{\bar{x}} + \frac{1}{2}(x_{21} + x_{22})\right] \quad (19)$$

$$R_3 = \left|\bar{x} - \frac{1}{2}(x_{21} + x_{22})\right| \quad (20)$$

7.3.5 Calculate the grand mean and the variances s_1^2, s_2^2 and s_3^2 using Formulae (21) to (24):

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \quad (21)$$

$$s_1^2 = \frac{1}{6k}\sum R_1^2 \quad (22)$$

$$s_2^2 = \frac{1}{2k}\sum R_2^2 \quad (23)$$

$$s_3^2 = \frac{1}{2k}\sum R_3^2 \quad (24)$$

where k is the number of lots.

7.3.6 Calculate the estimated values of the variance of analysis (s_A^2), sample processing (s_P^2) and sampling (s_S^2)using Formulae (25) to (27):

$$s_A^2 = s_1^2 \quad (25)$$

$$s_P^2 = s_2^2 - \frac{1}{2}s_1^2 \quad (26)$$

$$s_S^2 = \frac{9}{8}s_3^2 - \frac{3}{2}s_2^2 \quad (27)$$

7.3.7 Calculate the total variance of sampling, sample processing and analysis (s_T^2)using Formula (28):

$$s_T^2 = s_S^2 + s_P^2 + s_A^2 \quad (28)$$

7.3.8 Calculate the the estimated values of the total standard deviation (s_T) and the standard deviations of sampling (s_S), sample processing (s_P) and analysis (s_A).

7.3.9 Compare the values of s_T, s_S, s_P and s_A thus obtained with the desired standard deviations.

7.4 Sample processing method 3

7.4.1 Although the estimated standard deviation of analysis can be obtained, the estimated standard deviations of sampling and sample processing cannot be separated using sample processing method 3. The estimated standard deviations shall be calculated in accordance with the procedure given in 7.4.2 to 7.2.9.

7.4.2 Calculate the mean and range for each pair of duplicates using Formulae (29) to (30):

$$\bar{x}_i = \frac{1}{2}(x_{i1} + x_{i2}) \tag{29}$$

$$R_1 = |x_{i1} - x_{i2}| \tag{30}$$

where i = 1 and 2 represent interleaved samples A and B, respectively.

7.4.3 Calculate the mean and range for each pair of interleaved samples, A and B using Formulae (31) to (32):

$$\bar{\bar{x}} = \frac{1}{2}(\bar{x}_1 + \bar{x}_2) \tag{31}$$

$$R_3 = |\bar{x}_1 - \bar{x}_2| \tag{32}$$

7.4.4 Calculate the grand mean and the variances s_1^2 and s_3^2 using Formulae (33) to (35):

$$\bar{\bar{\bar{x}}} = \frac{1}{k}\sum \bar{\bar{x}} \tag{33}$$

$$s_1^2 = \frac{1}{4k}\Sigma R_1^2 \tag{34}$$

$$s_3^2 = \frac{1}{2k}\Sigma R_3^2 \tag{35}$$

where k is the number of lots.

7.4.5 Calculate the estimated values of the variance of analysis (s_A^2) and the variance of sampling and sample processing (s_{SP}^2) using Formulae (36) to (37):

$$s_A^2 = s_1^2 \tag{36}$$

$$s_{SP}^2 = s_3^2 - \frac{1}{2}s_1^2 \tag{37}$$

7.4.6 Calculate the total variance of sampling, sample processing and analysis (s_T^2) using Formula (38):

$$s_T^2 = s_{SP}^2 + s_A^2 \tag{38}$$

7.4.7 Calculate the estimated values of the total standard deviation (s_T) and the standard deviations of sampling and sample processing (s_{SP}) and analysis (s_A).

7.4.8 Compare the values of s_T, s_{SP} and s_A thus obtained with the desired standard deviations.

8 Assessment of results and action

8.1 General

When the standard deviations of sampling, sample processing and/or analysis exceed the desired values, the relevant procedures shall be modified as outlined in 8.2 to 8.4.

8.2 Sampling

Check for a change in variance between increments of the concentrate in accordance with the procedure given in ISO 12743. Where a significant change is confirmed, revise the number of increments taken from the lot accordingly.

NOTE For systematic or stratified random sampling where a greater number (denoted by n_1) of increments is collected from a lot, the standard deviation of sampling is improved in proportion to $\sqrt{n/n_1}$.

An alternative is to increase the mass of increments. There will, however, be a limit above which increasing the sample mass does not significantly improve the standard deviation of sampling.

8.3 Sample processing

Check the variance at each stage of sample processing in accordance with the procedure given in ISO 12743. Reduce the major variance components by reducing the particle size of the concentrate prior to division, or increasing the mass of divided sample.

8.4 Analysis

Check that the specified analytical procedures are being followed. Other factors, such as the fineness and homogeneity of the laboratory samples, should also be checked.

9 Recording of data

Detailed records of sampling data should be kept in a standardized form to avoid errors and omissions and for future reference. An example is given in Annex A.

Annex A
(informative)

Recording of sampling data

This annex provides an example for recording sampling data and calculation of the standard deviations of sampling, sample processing and analysis for systematic sampling using sample processing method 1 (see 7.2). Table A.1 summarizes sampling particulars and results for the determination of the mass fraction of Cu. Table A.2 illustrates the recommended procedure for recording data and calculating s_A, s_P and s_{S_1}.

The estimated variances obtained from the mean square differences between duplicates are as follows (see Table A.2):

s_1^2 = 0,000 396 (79 degrees of freedom)

s_2^2 = 0,000 684 (39 degrees of freedom)

s_3^2 = 0,002 93 (19 degrees of freedom)

Using Formulae (11), (12) and (13) in 6.2, the following values are obtained for the estimated precisions of analysis, sample processing and primary sampling:

a) Standard deviation of analysis:

s_A = a mass fraction of 0,020 % Cu

b) Standard deviation of sample processing:

s_P = a mass fraction of 0,022 % Cu

c) Standard deviation of primary sampling:

s_S = a mass fraction of 0,051 % Cu

Of the three, the standard deviation of sampling s_S is the largest component. If this is considered to be unsatisfactory, the number of increments should be increased.

Table A.1 — Record of experimental results

(Name of the Company and Works)

Report on checking the precision of sampling

Period of experiment: .....

Site of experiment: (Location identification) .....

Characteristic measured: Mass fraction of copper

Lots investigated

Source and type of concentrate: .....

Loading point: .....

Transportation method: (Ship, wagon, truck, etc.)

Number of lots: 20

Mass of lots: 500 t

Particulars of sampling

Nominal top size of concentrate: 1 mm

Concentrate flow rate: 500 t/h

Conveyor speed: 2 m/s

Type of cutter: Mechanical diverter cutter

Cutter aperture: 50 mm

Nominal mass of increment: 12 kg

Number of increments: 50

Processing of samples

Method of constituting interleaved samples: Place alternate individual increments in containers A and B, to constitute samples A and B, each comprising 25 increments.

Mass of samples: 300 kg

Method of processing samples: Sample processing method 1

Measurements of Cu, % (mass fraction):

Statistic	Experimental results	Commercial determination	Manifested at loading point
Mean	23,01	—	—
Minimum	22,72	—	—
Maximum	23,20	—	—

Estimated standard deviations:

s_A = a mass fraction of 0,020 % Cu

s_P = a mass fraction of 0,022 % Cu

s_S = a mass fraction of 0,051 % Cu

s_T = a mass fraction of 0,059 % Cu

Comments and remarks: .....

.....

Date: ..... Reported by: .....

(Name of supervisor of experiment)