

INTERNATIONAL STANDARD

ISO
12744

First edition
1997-05-01

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

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



Reference number
ISO 12744:1997(E)

Contents

Page

1	Scope.....	1
2	Normative reference.....	1
3	General conditions.....	1
4	Definition of symbols.....	2
5	Method of experiment.....	2
6	Evaluation of experimental data	5
7	Assessment of results and action	8
8	Recording of data	8

Annexes

A	Recording of sampling data	9
B	Bibliography	12

© ISO 1997

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
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 12744 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes A and B of this International Standard are for information only.

STANDARDSISO.COM : Click to view the full PDF of ISO 12744:1997

This page intentionally left blank

STANDARDSISO.COM : Click to view the full PDF of ISO 12744:1997

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

1 Scope

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

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 12743:—¹⁾, *Copper, lead and zinc sulfide concentrates — Sampling procedures for determination of metal and moisture content*.

3 General conditions

3.1 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.

3.2 Number of lots

It is recommended that pairs of interleaved samples 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 this requirement is met.

3.3 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 must be divided by $\sqrt{2}$ to obtain the sampling precision for lot samples comprising n increments.

3.4 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.

3.5 Frequency of precision checks

It is recommended that, even after a precision check has been conducted, further checks 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 be carried out as a part of routine sampling and analysis.

1) To be published.

4 Definition of symbols

Symbol	Term
k	Number of lots
n	Number of increments
R_1	Absolute difference between duplicates for interleaved samples A and B
$\bar{R}_1$	Mean absolute difference between duplicates for interleaved samples A and B for k lots
R_2	Absolute difference between means for divided interleaved samples A_1 and A_2 , and B_1 and B_2
$\bar{R}_2$	Mean absolute difference between means for divided interleaved samples A_1 and A_2 , and B_1 and B_2 , for k lots
R_3	Absolute difference between means for interleaved sample A and interleaved sample B
$\bar{R}_3$	Mean absolute difference between means for interleaved sample A and interleaved sample B for k lots
s	Estimated value of standard deviation, σ
s_1^2	Estimated variance from $\bar{R}_1$
s_2^2	Estimated variance from $\bar{R}_2$
s_3^2	Estimated variance from $\bar{R}_3$
s_A	Estimated standard deviation of analysis
s_P	Estimated standard deviation of sample processing
s_{S_1}	Estimated standard deviation of primary sampling
s_{SP}	Estimated standard deviation of primary sampling and sample processing
s_T	Estimated total standard deviation of primary 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 of duplicate results
$\bar{\bar{\bar{x}}}$	Mean of $\bar{\bar{x}}$ values, and grand mean for sample processing method 3
$\bar{\bar{\bar{\bar{x}}}}$	Grand mean of all results for sample processing methods 1 and 2

5 Method of experiment

5.1 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.

5.2 Sample processing and analysis

The pairs of interleaved samples A and B taken in accordance with 5.1 shall be divided separately and subjected to either method 1, method 2 or method 3 sample processing and analysis as described in 5.2.1, 5.2.2 or 5.2.3.

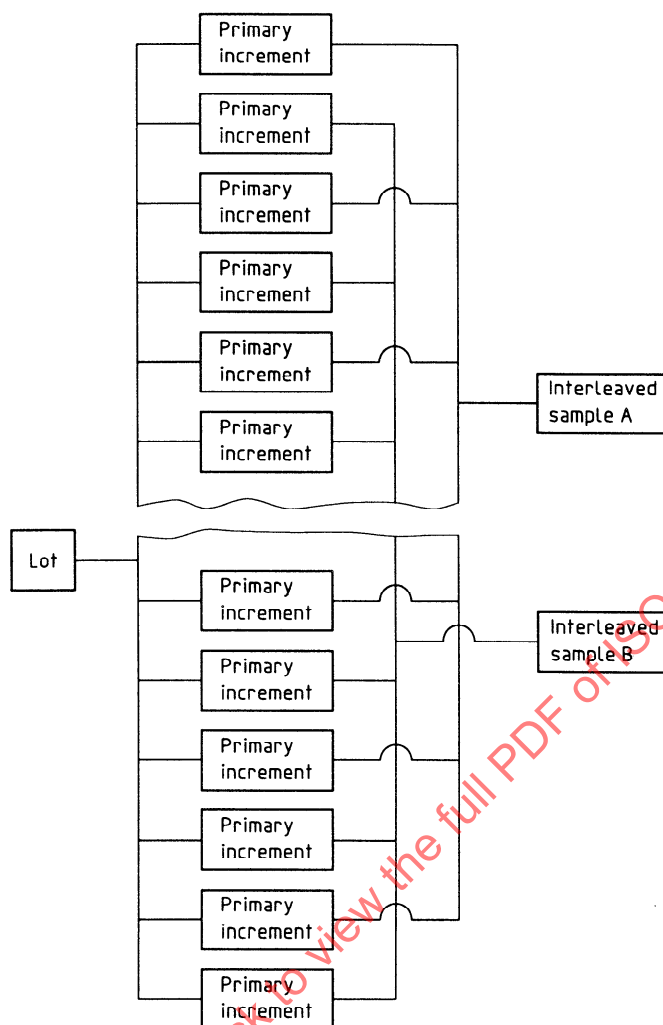


Figure 1 — Example of a plan for interleaved duplicate sampling

5.2.1 Sample processing method 1 (see figure 2)

The two interleaved samples A and B shall be divided separately to prepare four laboratory samples, A_1 , A_2 , and B_1 , B_2 . These laboratory samples shall each be analysed in duplicate, and the duplicates designated 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 and x_{221} and x_{222} for sample B_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 1 By using method 1, the estimated precisions of primary sampling, sample processing and analysis can be obtained separately.

5.2.2 Sample processing method 2 (see figure 3)

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 x_{111} and x_{112} for sample A_1 , x_{121} and x_{122}

for sample A_2 , and x_{21} and x_{22} for sample B. The six determinations shall be run in random order by the same analyst on the same day using the same analytical equipment.

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

5.2.3 Sample processing method 3 (see figure 4)

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 x_{11} and x_{12} for sample A and x_{21} and x_{22} for sample B. The four determinations shall be run in random order by the same analyst on the same day using the same analytical equipment.

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

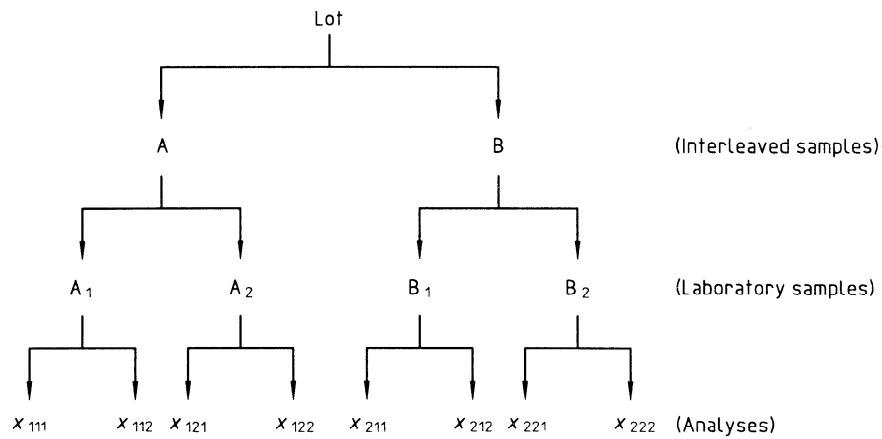


Figure 2 — Flowsheet for sample processing method 1

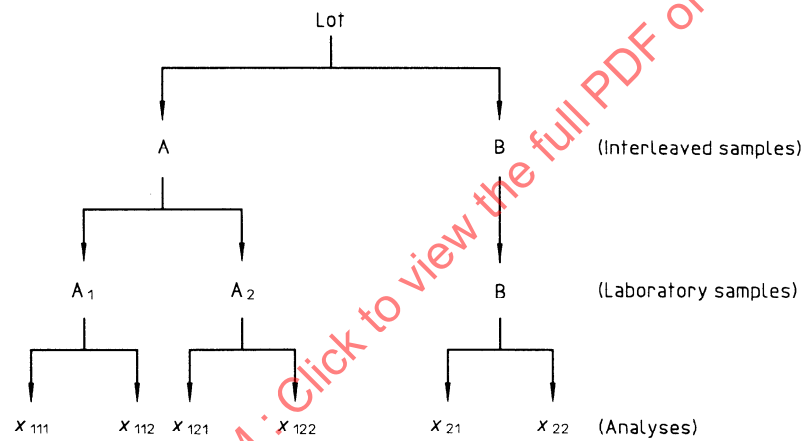


Figure 3 — Flowsheet for sample processing method 2

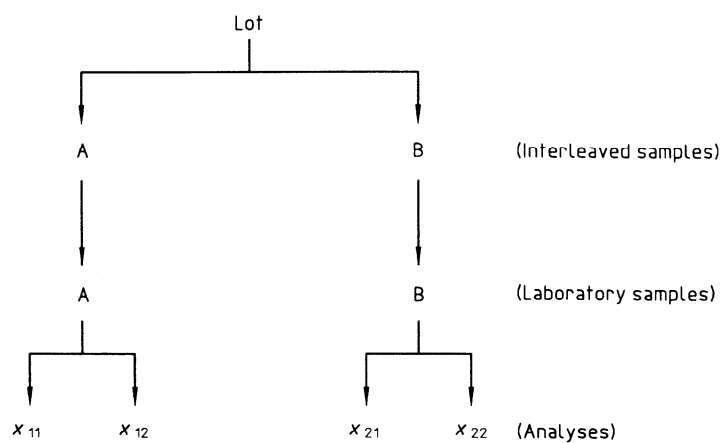


Figure 4 — Flowsheet for sample processing method 3

6 Evaluation of experimental data

6.1 General

The method for evaluation of experimental data shall be as specified below, depending on the method of sample processing selected.

6.2 Sample processing method 1

The estimated standard deviations of primary sampling, sample processing and analysis shall be calculated in accordance with the following procedure.

- a) Calculate the mean and range for each pair of duplicates:

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

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

where

$i = 1$ and 2 representing interleaved samples A and B respectively;

$j = 1$ and 2 representing laboratory samples A₁ and A₂ or B₁ and B₂.

- b) Calculate the mean of the mean and range for each pair of duplicates:

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

$$R_2 = |\bar{x}_{i1} - \bar{x}_{i2}| \quad \dots (4)$$

- c) Calculate the mean and range for each pair of interleaved samples, A and B:

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

$$R_3 = |\bar{\bar{x}}_1 - \bar{\bar{x}}_2| \quad \dots (6)$$

- d) Calculate the grand mean and the means of ranges R_1 , R_2 and R_3 :

$$\bar{\bar{\bar{x}}} = \frac{1}{k} \sum \bar{\bar{x}} \quad \dots (7)$$

$$\bar{R}_1 = \frac{1}{4k} \sum R_1 \quad \dots (8)$$

$$\bar{R}_2 = \frac{1}{2k} \sum R_2 \quad \dots (9)$$

$$\bar{R}_3 = \frac{1}{k} \sum R_3 \quad \dots (10)$$

where k is the number of lots.

- e) Calculate the variances s_1^2 , s_2^2 and s_3^2 from the means of ranges $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$:

$$s_1^2 = \frac{\pi}{4} (\bar{R}_1)^2 \quad \dots (11)$$

$$s_2^2 = \frac{\pi}{4} (\bar{R}_2)^2 \quad \dots (12)$$

$$s_3^2 = \frac{\pi}{4} (\bar{R}_3)^2 \quad \dots (13)$$

where $\pi/4$ is a statistical factor relating range to variance for a pair of measurements.

- f) Conduct F -tests on the variances s_1^2 , s_2^2 and s_3^2 to determine whether their differences are statistically significant using the following procedure:

- 1) calculate the variance ratios s_2^2/s_1^2 and s_3^2/s_2^2 ;
- 2) compare these ratios with the 95 % confidence F -ratios given in table 1 for the number of degrees of freedom applicable to each variance estimate;
- 3) if the calculated variance ratio exceeds the F -ratio, partition the two variances into variance components as their difference is statistically significant.

If the calculated variance ratio does not exceed the F -ratio, the variances s_2^2 and/or s_3^2 cannot be meaningfully partitioned into variance components and more data need to be collected.

- g) Assuming the F -tests conducted in f) indicate that the differences between the variances s_1^2 , s_2^2 and s_3^2 are significant, calculate the estimated values of the variance of analysis (s_A^2), sample processing (s_P^2) and primary sampling ($s_{S_1}^2$) as follows:

$$s_A^2 = s_1^2 \quad \dots (14)$$

$$s_P^2 = s_2^2 - \frac{1}{2}s_1^2 \quad \dots (15)$$

$$s_{S_1}^2 = s_3^2 - \frac{1}{2}s_2^2 \quad \dots (16)$$

- h) Calculate the total variance of primary sampling, sample processing and analysis (s_T^2) as follows:

$$s_T^2 = s_{S_1}^2 + s_P^2 + s_A^2 \quad \dots (17)$$

Table 1 — *F*-ratios at the 95 % confidence level for comparison of two variances

Degrees of freedom (smaller variance)	Degrees of freedom (larger variance)						
	20	24	30	40	60	120	∞
20	2,12	2,08	2,04	1,99	1,95	1,90	1,84
24	2,03	1,98	1,94	1,89	1,84	1,79	1,73
30	1,93	1,89	1,84	1,79	1,74	1,68	1,62
40	1,84	1,79	1,74	1,69	1,64	1,58	1,51
60	1,75	1,70	1,65	1,59	1,53	1,47	1,39
120	1,66	1,61	1,55	1,50	1,43	1,35	1,25
∞	1,57	1,52	1,46	1,39	1,32	1,22	1,00

- i) Calculate the estimated values of the total standard deviation (s_T) and the standard deviations of primary sampling (s_{S_1}), sample processing (s_P) and analysis (s_A).

- j) Compare the values of s_T , s_{S_1} , s_P and s_A thus obtained with the desired standard deviations.

$$R_3 = \left| \bar{x} - \frac{1}{2}(x_{21} + x_{22}) \right| \quad \dots (23)$$

- d) Calculate the grand mean and the means of ranges R_1 , R_2 and R_3 :

$$\bar{\bar{x}} = \frac{1}{k} \sum \bar{x} \quad \dots (24)$$

$$\bar{R}_1 = \frac{1}{3k} \sum R_1 \quad \dots (25)$$

$$\bar{R}_2 = \frac{1}{k} \sum R_2 \quad \dots (26)$$

$$\bar{R}_3 = \frac{1}{k} \sum R_3 \quad \dots (27)$$

6.3 Sample processing method 2

The estimated standard deviations of primary sampling, sample processing and analysis shall be calculated in accordance with the following procedure.

- a) Calculate the mean and range for each pair of duplicates:

$$\bar{x} = \frac{1}{2}(x_{111} + x_{112}), \frac{1}{2}(x_{121} + x_{122}), \quad \dots (18)$$

$$R_1 = \left| x_{111} - x_{112} \right|, \left| x_{121} - x_{122} \right|, \quad \dots (19)$$

- b) Calculate the mean and range for duplicates A_1 and A_2 :

$$\bar{\bar{x}} = \frac{1}{4}(x_{111} + x_{112} + x_{121} + x_{122}) \quad \dots (20)$$

$$R_2 = \frac{1}{2} \left| (x_{111} + x_{112}) - (x_{121} + x_{122}) \right| \quad \dots (21)$$

- c) Calculate the mean and range for each pair of interleaved samples, A and B:

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

where k is the number of lots.

- e) Calculate the variances s_1^2 , s_2^2 and s_3^2 from the means of ranges $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$:

$$s_1^2 = \frac{\pi}{4} (\bar{R}_1)^2 \quad \dots (28)$$

$$s_2^2 = \frac{\pi}{4} (\bar{R}_2)^2 \quad \dots (29)$$

$$s_3^2 = \frac{\pi}{4} (\bar{R}_3)^2 \quad \dots (30)$$

where $\pi/4$ is a statistical factor relating range to variance for a pair of measurements.

- f) Conduct *F*-tests on the variances s_1^2 , s_2^2 and s_3^2 to determine whether their differences are statistically significant using the following procedure:

- 1) calculate the variance ratios s_2^2/s_1^2 and s_3^2/s_2^2 ;
- 2) compare these ratios with the 95 % confidence *F*-ratios given in table 1 for the num-

ber of degrees of freedom applicable to each variance estimate;

- 3) if the calculated variance ratio exceeds the F -ratio, partition the two variances into variance components as their difference is statistically significant.

If the calculated variance ratio does not exceed the F -ratio, the variances s_2^2 and/or s_3^2 cannot be meaningfully partitioned into variance components and more data need to be collected.

- g) Assuming the F -tests conducted in f) indicate that the differences between the variances s_1^2 , s_2^2 and s_3^2 are significant, calculate the estimated values of the variance of analysis (s_A^2), sample processing (s_P^2) and primary sampling ($s_{S_1}^2$) as follows:

$$s_A^2 = s_1^2 \quad \dots (31)$$

$$s_P^2 = s_2^2 - \frac{1}{2}s_1^2 \quad \dots (32)$$

$$s_{S_1}^2 = s_3^2 - \frac{3}{4}s_2^2 \quad \dots (33)$$

- h) Calculate the total variance of primary sampling, sample processing and analysis (s_T^2) as follows:

$$s_T^2 = s_{S_1}^2 + s_P^2 + s_A^2 \quad \dots (34)$$

- i) Calculate the estimated values of the total standard deviation (s_T) and the standard deviations of primary sampling (s_{S_1}), sample processing (s_P) and analysis (s_A).
- j) Compare the values of s_T , s_{S_1} , s_P and s_A thus obtained with the desired standard deviations.

6.4 Sample processing method 3

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

- a) Calculate the mean and range for each pair of duplicates:

$$\bar{x}_i = \frac{1}{2}(x_{i1} + x_{i2}) \quad \dots (35)$$

$$R_1 = |x_{i1} - x_{i2}| \quad \dots (36)$$

where $i=1$ and 2 representing interleaved samples A and B respectively.

- b) Calculate the mean and range for each pair of interleaved samples, A and B:

$$\bar{\bar{x}} = \frac{1}{2}(\bar{x}_1 + \bar{x}_2) \quad \dots (37)$$

$$R_3 = |\bar{x}_1 - \bar{x}_2| \quad \dots (38)$$

- c) Calculate the grand mean and the means of ranges R_1 and R_3 :

$$\bar{\bar{\bar{x}}} = \frac{1}{k} \sum \bar{\bar{x}} \quad \dots (39)$$

$$\bar{R}_1 = \frac{1}{2k} \sum R_1 \quad \dots (40)$$

$$\bar{R}_3 = \frac{1}{k} \sum R_3 \quad \dots (41)$$

where k is the number of lots.

- d) Calculate the variances s_1^2 and s_3^2 from the means of ranges $\bar{R}_1$ and $\bar{R}_3$:

$$s_1^2 = \frac{\pi}{4} (\bar{R}_1)^2 \quad \dots (42)$$

$$s_3^2 = \frac{\pi}{4} (\bar{R}_3)^2 \quad \dots (43)$$

where $\pi/4$ is a statistical factor relating range to variance for a pair of measurements.

- e) Conduct F -tests on the variances s_1^2 and s_3^2 to determine whether their differences are statistically significant using the following procedure:

- 1) calculate the variance ratio s_3^2/s_1^2 ;
- 2) compare this ratio with the 95 % confidence F -ratio given in table 1 for the number of degrees of freedom applicable to each variance estimate;
- 3) if the calculated variance ratio exceeds the F -ratio, partition the two variances into variance components as their difference is statistically significant.

If the calculated variance ratio does not exceed the F -ratio, the variance s_3^2 cannot be meaningfully partitioned into variance components and more data need to be collected.

- f) Assuming the F -tests conducted in e) indicate that the difference between the variances s_1^2 and s_3^2 are significant, calculate the estimated values of the variance of analysis (s_A^2) and the variance of primary sampling and sample processing (s_{SP}^2) as follows:

$$s_A^2 = s_1^2 \quad \dots (44)$$

$$s_{SP}^2 = s_3^2 - \frac{1}{2}s_1^2 \quad \dots (45)$$

- g) Calculate the total variance of primary sampling, sample processing and analysis (s_T^2) as follows:

$$s_T^2 = s_{SP}^2 + s_A^2 \quad \dots (46)$$

- h) Calculate the estimated values of the total standard deviation (s_T) and the standard deviations of primary sampling and sample processing (s_{SP}) and analysis (s_A).
- i) Compare the values of s_T , s_{SP} and s_A thus obtained with the desired standard deviations.

7 Assessment of results and action

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

7.1 Primary sampling

Check for a change in variance between increments of the concentrate in accordance with the procedure in

ISO 12743. Where a significant change is confirmed, revise the number of increments taken from the lot accordingly.

NOTE 4 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 primary 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 primary sampling.

7.2 Sample processing

Check the variance at each stage of sample processing in accordance with the procedure 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.

7.3 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.

8 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 B.

Annex A (informative)

Recording of sampling data

A.1 Scope

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. Table A.1 summarizes sampling particulars and results for the determination of Cu content (*m/m*). Table A.2 illustrates the recommended procedure for recording data and calculating s_A , s_P and s_{S_1} .

A.2 Comment on data

The estimated variances obtained from the mean ranges $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$ are as follows (see table A.2):

$$s_1^2 = 0,000\ 38 \quad (79 \text{ degrees of freedom})$$

$$s_2^2 = 0,000\ 66 \quad (39 \text{ degrees of freedom})$$

$$s_3^2 = 0,002\ 73 \quad (19 \text{ degrees of freedom})$$

Hence,

$$s_2^2/s_1^2 = 1,74 \text{ } F\text{-ratio from table 1} = 1,59$$

$$s_3^2/s_2^2 = 4,14 \text{ } F\text{-ratio from table 1} = 1,84$$

In both cases the difference between the estimated variances is significant at the 95 % confidence level.

Using equations (14), (15) and (16) in 6.2, the following values are obtained for the estimated precisions of analysis, sample processing and primary sampling:

- a) Standard deviation of analysis
 $s_A = 0,019 \text{ } \%$ Cu (*m/m*)
- b) Standard deviation of sample processing
 $s_P = 0,022 \text{ } \%$ Cu (*m/m*)
- c) Standard deviation of primary sampling
 $s_{S_1} = 0,049 \text{ } \%$ Cu (*m/m*)

Of the three, the standard deviation of primary sampling s_{S_1} 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: Copper content

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 flowrate: 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, % (m/m):

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

Estimated standard deviations:

$s_A = 0,019$ % Cu (m/m)

$s_P = 0,022$ % Cu (m/m)

$s_{S_1} = 0,049$ % Cu (m/m)

$s_T = 0,057$ % Cu (m/m)

Comments and remarks:

Date: Reported by:

(Name of supervisor of experiment)

Table A.2 — Example of data sheet for checking precision (% Cu, m/m)

Lot	A ₁				A ₂				A		B ₁				B ₂				B		R ₃	
	x ₁₁₁	x ₁₁₂	x̄ ₁₁	R ₁	x ₁₂₁	x ₁₂₂	x̄ ₁₂	R ₁	x̄ ₁	R ₂	x ₂₁₁	x ₂₁₂	x̄ ₂₁	R ₁	x ₂₂₁	x ₂₂₂	x̄ ₂₂	R ₁	x̄ ₂	R ₂		
1	23,10	23,05	23,08	0,05	23,06	23,09	23,08	0,03	23,08	0,00	23,07	23,06	23,07	0,01	23,06	23,04	23,05	0,02	23,06	0,02	23,07	0,02
2	23,09	23,06	23,08	0,03	23,09	23,09	23,09	0,00	23,09	0,01	23,12	23,12	23,12	0,00	23,14	23,14	23,14	0,00	23,13	0,02	23,11	0,04
3	23,12	23,14	23,13	0,02	23,10	23,12	23,11	0,02	23,12	0,02	23,16	23,10	23,13	0,06	23,12	23,12	23,12	0,00	23,13	0,01	23,13	0,01
4	23,07	23,04	23,06	0,03	23,04	23,03	23,04	0,01	23,05	0,02	23,10	23,10	23,10	0,00	23,12	23,09	23,11	0,03	23,11	0,01	23,08	0,06
5	22,97	23,00	22,99	0,03	22,99	22,98	22,99	0,01	22,99	0,00	23,05	22,98	23,02	0,07	23,02	23,08	23,05	0,06	23,04	0,03	23,02	0,05
6	23,00	22,99	23,00	0,01	22,95	23,00	22,98	0,05	22,99	0,02	22,88	22,95	22,02	0,07	22,94	22,94	22,94	0,00	22,93	0,02	22,96	0,06
7	22,94	22,89	22,92	0,05	22,84	22,87	22,86	0,03	22,89	0,06	22,92	22,91	22,92	0,01	22,86	22,90	22,88	0,04	22,90	0,04	22,90	0,01
8	23,02	23,05	23,04	0,03	23,10	23,07	23,09	0,03	23,07	0,05	23,00	23,02	23,01	0,02	23,05	22,99	23,02	0,06	23,02	0,01	23,05	0,05
9	22,99	22,99	22,99	0,00	23,02	22,99	23,01	0,03	23,00	0,02	23,06	23,03	23,05	0,03	23,01	23,05	23,03	0,04	23,04	0,02	23,02	0,04
10	23,08	23,05	23,07	0,03	23,04	23,04	23,04	0,00	23,06	0,03	23,09	23,08	23,09	0,01	23,10	23,08	23,09	0,02	23,09	0,00	23,08	0,03
11	23,17	23,14	23,16	0,03	23,12	23,13	23,13	0,01	23,15	0,03	23,00	23,00	23,00	0,00	23,03	23,04	23,04	0,01	23,02	0,04	23,09	0,13
12	23,04	23,05	23,05	0,01	23,06	23,05	23,06	0,01	23,06	0,01	23,06	23,05	23,06	0,01	23,07	23,09	23,08	0,02	23,07	0,02	23,07	0,01
13	23,20	23,20	23,20	0,00	23,11	23,08	23,10	0,03	23,15	0,10	23,03	23,00	23,02	0,03	23,07	23,05	23,06	0,02	23,04	0,04	23,10	0,11
14	22,81	22,82	22,82	0,01	22,87	22,90	22,89	0,03	22,86	0,07	22,92	22,93	22,93	0,01	22,92	22,88	22,90	0,04	22,92	0,03	22,89	0,06
15	23,08	23,07	23,08	0,01	23,05	23,02	23,04	0,03	23,06	0,04	23,04	22,99	23,02	0,05	22,92	22,96	22,94	0,04	22,98	0,08	23,02	0,08
16	22,96	22,96	22,96	0,00	22,88	22,92	22,90	0,04	22,93	0,06	22,75	22,72	22,74	0,03	22,73	22,75	22,74	0,02	22,74	0,00	22,84	0,19
17	22,98	22,99	22,99	0,01	22,95	22,96	22,96	0,01	22,98	0,03	22,96	22,97	22,97	0,01	22,99	23,01	23,00	0,02	22,99	0,03	22,99	0,01
18	22,85	22,85	22,85	0,00	22,90	22,87	22,89	0,03	22,87	0,04	22,89	22,90	22,90	0,01	22,91	22,89	22,90	0,02	22,90	0,00	22,89	0,03
19	22,99	22,96	22,98	0,03	22,98	22,98	22,98	0,00	22,98	0,00	23,05	23,04	23,05	0,01	23,00	22,98	22,99	0,02	23,02	0,06	23,00	0,04
20	23,01	22,98	23,00	0,03	22,97	22,96	22,97	0,01	22,99	0,03	22,82	22,82	22,82	0,00	22,85	22,87	22,86	0,02	22,84	0,04	22,92	0,15
Mean	23,02	23,01	23,02	0,020 5	23,01	23,01	23,01	0,020 5	23,02	0,032 0	23,00	22,99	23,00	0,022 0	23,00	23,00	23,00	0,025 0	23,00	0,026 0	23,01	0,059 0

$$s_A^2 = s_1^2 = 0,000 38$$

$$s_A = 0,019$$

$$s_P^2 = s_2^2 - s_1^2/2 = 0,000 66 - 0,000 19 = 0,000 47$$

$$s_P = 0,022$$

$$s_{S_1}^2 = s_3^2 - s_2^2/2 = 0,002 73 - 0,000 33 = 0,002 40$$

$$s_{S_1} = 0,049$$

$$\bar{R}_1 = (0,020 5 + 0,020 5 + 0,022 0 + 0,026 0)/4 = 0,022 0$$

$$\bar{R}_2 = (0,032 0 + 0,026 0)/2 = 0,029 0$$

$$\bar{R}_3 = 0,059 0$$

$$s_1^2 = \pi/4 (\bar{R}_1)^2 = 0,785 4 \times 0,022 0^2 = 0,000 38$$

$$s_2^2 = \pi/4 (\bar{R}_2)^2 = 0,785 4 \times 0,029 0^2 = 0,000 66$$

$$s_3^2 = \pi/4 (\bar{R}_3)^2 = 0,785 4 \times 0,059 0^2 = 0,002 73$$

Annex B

(informative)

Bibliography

- [1] ISO 10258:1994, *Copper sulfide concentrates — Determination of copper content — Titrimetric methods.*
- [2] ISO 10378:1994, *Copper sulfide concentrates — Determination of gold and silver contents — Fire assay gravimetric and atomic absorption spectrometric method.*
- [3] ISO 10469:1994, *Copper sulfide concentrates — Determination of copper content — Electrogravimetric method.*
- [4] ISO 11441:1995, *Lead sulfide concentrates — Determination of lead content — Back titration of EDTA after precipitation of lead sulfate.*
- [5] ISO 12739:—²⁾, *Zinc sulfide concentrates — Determination of zinc content — Ion-exchange method.*
- [6] ISO 13291:—²⁾, *Zinc sulfide concentrates — Determination of zinc content — Solvent extraction EDTA titrimetric method.*

2) To be published.