INTERNATIONAL STANDARD

ISO 13292

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

Concentrés sulfurés de cuivre, de plomb et de zinc — Méthodes expérimentales de contrôle de l'erreur systématique d'échantillonnage

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13292 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.

1 Scope

This International Standard specifies methods for checking whether there is any bias in the sampling of copper, lead and zinc sulfide concentrates, where the sampling is carried out in accordance with the methods specified in ISO 12743. These methods can also be used for checking whether there is any bias in sample processing and for checking possible significant differences in the analyses of exchange samples and lot samples taken at different places, e.g., at loading and discharge points. Numerical examples are given in annex A.

2 Normative reference

The following standards 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 edition of the standard 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 requirements and recommendations

The procedures specified in this International Standard are applicable to paired data only. The results obtained from the method to be checked for bias (referred to as Method B) are compared with the results for a reference method (referred to as Method A), which is considered to produce unbiased results from technical and empirical viewpoints. If there is no significant difference between the results obtained using Method B and Method A, then Method B may be adopted as a routine method.

While the procedures specified in clause 5 are principally designed for checking bias against a reference method, separate measurements of quality characteristics, e.g., at loading (Method A) and discharge (Method B), or analyses of exchange samples may also be compared to check whether there is a statistically significant difference between the results.

Mechanical sampling systems or manual sampling methods are tested for bias by comparing the test results for final system or manually collected samples (Method B) with test results for reference increments collected from a stopped conveyor belt (Method A). Analytical methods or test procedures are checked against certified reference materials.

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1) To be published.

The standard method of taking reference increments from a stopped conveyor belt presents operational difficulties, even if the handling system is capable of being restarted with a fully loaded belt. The main problems are losses in production tonnage and the difficulty experienced in sequence starting the handling system. During a ship loading or unloading operation, this can cause delays in the turnaround time of the ship.

Alternative reference methods, which are also expensive, divert the ore flow onto a transfer conveyor belt to produce a material bed section identical to that from the main belt on which the mechanical primary sampler (Method B) is installed. Stopped belt sampling is then carried out on the transfer belt to collect reference increments (Method A). The transfer conveyor belt should be of sufficient length to allow establishment of a material bed section which is not influenced by any longitudinal segregation introduced by the diversion plate. The primary sampler and the point of diversion to the transfer belt should be as close as possible.

Sampling and sample processing procedures are more prone to bias than analytical techniques and test methods. As system samples and reference increments are progressively reduced in mass and particle size, subsamples and test samples become increasingly vulnerable to sample loss and contamination. Therefore, systematic errors occur more frequently during sample processing than during sampling, so different stages of the sample processing procedure may require testing for bias.

When testing for bias, the number of paired sets of measurements (data sets) shall be not less than 20. The number of data sets required depends on the variance of the differences between the two methods and on the value of the bias, δ, to be detected.

Any chemical or physical characteristic may be used to check whether any bias is present, e.g., copper, lead, zinc or moisture content. Bias may not always be present for just one parameter. Therefore several parameters, preferably including all those which are likely to be of interest, should be examined to determine whether bias exists. Characteristics to be tested for the presence of bias need to be decided before the test work begins.

It is recommended that increments for Method A and Method B be taken from closely adjacent portions of the concentrate, and that sample processing and analysis be carried out on each increment individually. This is likely to reduce the number of data sets required to detect a given bias.

Even after a bias check has been conducted, further checks should be carried out at regular intervals. Bias checks should also be carried out when sampling equipment is changed.

4 Sampling and sample processing methods

4.1 Sampling

4.1.1 General

The method to be checked (Method B) shall be compared with a designated reference method (Method A) using the same type of concentrate. As specified in clause 3, at least 20 pairs of samples shall be collected following the procedures detailed in ISO 12743.

4.1.2 Checking bias in mechanical sampling

Method A: Reference increments are taken by stopped-belt sampling.

Method B: System increments are taken from a moving stream with a mechanical sampler.

4.1.3 Checking bias between sampling at different locations

Method A: Increments are taken from a moving stream with a mechanical sampler at a loading location.

Method B: Increments are taken from a moving stream with a mechanical sampler at a discharge location.

4.2 Sample processing

The procedure for constituting pairs of samples and carrying out the subsequent sample processing and analysis shall be as follows:

a)   Constitute two samples (sample A and sample B), consisting of one or a number of individual increments obtained in accordance with Method A and Method B.

b)   Subject sample A and sample B to similar sample processing and analysis procedures, as specified in ISO 12743 and other relevant International Standards, to obtain sets of measurements for the characteristics of interest.

5   Analysis of experimental data

5.1   Statistical basis

It is helpful at this point to review the definitions of Type I and Type II risks in bias testing. Consider the null hypothesis, H_0, that there is no positive or negative bias in Method B compared with Method A. The alternative hypothesis, H_1, is that there is either positive or negative bias. A Type I error occurs if H_0 is rejected on the basis of the sample data falling into the critical region of a statistical test when, in reality, H_0 is true. A Type II error occurs if H_0 is accepted on the basis of the statistical test on the sample data when, in reality, H_1 is true.

In this International Standard, the statistical test for accepting H_0 is that the absolute value of the difference between the mean of the quality characteristic for the reference set of increments (Method A) and the mean for the corresponding set of system increments (Method B) is smaller than a specified bias detection limit, BDL.

A bias can be either positive or negative, so for a positive bias:

$$\overline{x}\,(\text{B}) - \overline{x}\,(\text{A}) > + \text{BDL} \qquad\qquad\qquad ... (1)$$

while for a negative bias:

$$\overline{x}\,(\text{B}) - \overline{x}\,(\text{A}) < - \text{BDL} \qquad\qquad\qquad ... (2)$$

where

> $\overline{x}\,(\text{A})$ and $\overline{x}\,(\text{B})$ are the means for the reference set of increments (Method A) and the system set of increments (Method B), respectively.

The bias detection limit is defined as the minimum bias that can be detected at the 95 % confidence level using the data available. It is calculated using the method described in 5.3.

By convention, a symmetrical, two-sided 5 % probability ($\alpha = 0,05$) is assigned to the risk (Type I) of rejecting the null hypothesis, H_0 , when in fact it is true [1, 2]. In addition, an asymmetrical 10 % probability ($\beta = 0,10$) is assigned to the risk of accepting the null hypothesis, H_0, when in fact it is not true. Thus, sufficient data sets shall be collected to ensure that there is a small risk $\alpha = 0,05$ of concluding that there is a statistical difference between Method A and Method B when none exists (Type I risk), and a small risk $\beta = 0,10$ of concluding that there is no statistical difference when a bias δ is present (Type II risk). A Student's t-test (two-sided) shall then be applied at the 5 % level of significance. If the result is significant, it is concluded that a bias exists. However, if the result is not significant, it is concluded that no bias as large as δ exists.

5.2   Determination of the mean difference and its standard deviation

Determine the standard deviation of the differences between Method A and Method B using the following procedure.

a) Denote individual measurements for the ith sample pair using Method A and Method B as x_{A_i} and x_{B_i}, respectively.

b) Calculate the difference, d_i, between x_{B_i} and x_{A_i}, i.e.,

$$d_i = x_{B_i} - x_{A_i} \qquad ..(3)$$

where

$i = 1, 2, ..., k$

k = the number of paired sets of measurements.

c) Calculate the mean, $\bar{d}$, of the differences using the following equation:

$$\bar{d} = \frac{\sum_{i=1}^{k} d_i}{k} = \bar{x}(B) - \bar{x}(A) \qquad ... (4)$$

d) Calculate the sum of the squares, SS_d, and the standard deviation, s_d, of the difference using the following equations, where summations are over $i = 1,2,..., k$:

$$SS_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 \qquad ... (5)$$

$$s_d = \sqrt{\frac{SS_d}{k-1}} \qquad ... (6)$$

5.3 Determination of the required number of data sets

a) Specify the magnitude of the bias, δ, to be detected.

b) Calculate the bias detection limit for the combined risk of Type I and Type II errors using the following equation:

$$\text{BDL} = \left(t_{0,05;\ k-1} + t_{0,10;\ k-1}\right)\frac{s_d}{\sqrt{k}} \qquad ... (7)$$

where

$t_{0,05;k-1}$ and $t_{0,10;k-1}$ are the tabulated Student's t-values for $k-1$ degrees of freedom corresponding to $\alpha = 0,05$ and $\beta = 0,10$ given in table 1.

The bias detection limit, BDL, is the minimum bias that can be detected using the data available.

c) If BDL $\leq \delta$, the number of data sets is sufficient and the statistical test in 5.4 can be applied.

d) If BDL $> \delta$, calculate the standardized difference, D, and the required number of data sets, n_r, corresponding to this value of D using equations 8 and 9 below. These equations are based on the strategy of adjusting the number of data sets in equation 7 until BDL $= \delta$, while avoiding the rigour of adjusting the corresponding degrees of freedom in the t-tests:

$$D = \frac{\delta}{s_{\mathrm{d}}} \qquad \qquad \text{.. (8)}$$

$$n_{\mathrm{r}} = \frac{\left(t_{0,05;k-1} + t_{0,10;k-1}\right)^2}{D^2} \qquad \qquad \text{... (9)}$$

Calculated values of n_{r} for $k = 20$ and different values of the standardized difference D are given in table 2.

e) Collect an additional ($n_{\mathrm{r}} - k$) data sets and repeat step (b) − (d) until the number of data sets exceeds n_{r}.

Table 1 - Values of t at 5 % and 10 % levels of significance (two-sided test)

Number of data sets, k	$t_{0,05;k-1}$	$t_{0,10;k-1}$
20	2,093	1,729
21	2,086	1,725
22	2,080	1,721
23	2,074	1,717
24	2,069	1,714
25	2,064	1,711
26	2,060	1,708
27	2,056	1,706
28	2,052	1,703
29	2,048	1,701
30	2,045	1,699
31	2,042	1,697
41	2,021	1,684
61	2,000	1,671
121	1,980	1,658
∞	1,960	1,645

**Table 2 - Required number of data sets determined from the initial 20 measurements
for given standardized differences   and $\alpha = 0{,}05$ and $\beta = 0{,}10$**

Standardized difference D	Required number of data sets n_{r}
0,35	119
0,40	91
0,45	72
0,50	58
0,55	48
0,60	41
0,65	35
0,70	30
0,75	26
0,80	23
0,85	20

NOTE — The required number of data sets in table 2 is calculated using equation 9 with $t_{0,05;19} = 2{,}093$ and $t_{0,10;19} = 1{,}729$ (from table 1).

D

5.4 Statistical test

Calculate the value of a parameter t_0 (which is assumed to have a t distribution with $k-1$ degrees of freedom) using the following equation:

$$t_0 = \frac{\bar{d}\,\sqrt{k}}{s_\mathrm{d}}$$

.. (10)

When the absolute value of t_0 is smaller than the value of $t_{0,05;k-1}$ given in table 1 for k data sets ($k-1$ degrees of freedom), the difference between Method A and Method B is not larger than δ, the value of the bias to be detected. Therefore, Method B could be adopted as a routine method.

However, when the absolute value of t_0 is larger than the value of $t_{0,05;k-1}$ in table 1, there is significant bias in Method B and action to eliminate this bias should be taken.

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Annex A
(informative)

Numerical examples of determining the bias of sampling

A.1 Example 1 (δ = 0,2 % copper)

The numerical example shown in table A.1 is the result of an experiment comparing stopped-belt sampling with a mechanical sampler carried out in accordance with 4.1. The magnitude of bias to be detected is 0,2 % copper (*m/m*).

Table A.1 - Numerical example 1 (copper concentrate)

Data set	Copper content (%, *m/m*)		$d_i = x_{B_i} - x_{A_i}$	d_i^2
	x_{B_i}	x_{A_i}		
1	29,20	29,00	0,20	0,040 0
2	29,75	29,67	0,08	0,006 4
3	31,00	30,74	0,26	0,067 6
4	31,62	32,16	-0,54	0,291 6
5	30,96	31,26	-0,30	0,090 0
6	30,02	29,92	0,10	0,010 0
7	31,17	31,11	0,06	0,003 6
8	31,91	31,87	0,04	0,001 6
9	29,98	30,42	-0,44	0,193 6
10	31,21	31,13	0,08	0,006 4
11	31,26	31,30	-0,04	0,001 6
12	28,98	29,22	-0,24	0,057 6
13	28,95	29,09	-0,14	0,019 6
14	31,97	31,89	0,08	0,006 4
15	29,36	28,88	0,48	0,230 4
16	30,74	31,24	-0,50	0,250 0
17	30,74	31,14	-0,40	0,160 0
18	30,47	30,33	0,14	0,019 6
19	30,55	31,03	-0,48	0,234 4
20	30,80	30,94	-0,14	0,019 6
Sum			-1,70	1,706 0
Mean	30,53	30,62	-0,085	

Substituting into equations 4 and 5 gives:

$$\bar{d} = \frac{1}{k} \sum d_i = \frac{-1,70}{20} = -0,085$$

$$SS_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 1,706\ 0 - \frac{(-1,70)^2}{20} = 1,561\ 5$$