SLOVENSKI STANDARD
SIST ISO 12743:2000

01-junij-2000

Koncentrati sulfidov bakra, svinca in cinka - Postopki vzorļenja za določļevanje kovine in vlage

Copper, lead and zinc sulfide concentrates -- Sampling procedures for determination of metal and moisture content

Concentrés sulfurés de cuivre, de plomb et de zinc -- Procédures d'échantillonnage pour la détermination de la teneur en métal et de l'humidité

Ta slovenski standard je istoveten z: ISO 12743:1998

ICS:

73.060.99 Druge rude Other metalliferous minerals

SIST ISO 12743:2000 **en**

INTERNATIONAL STANDARD

ISO 12743

First edition
1998-05-01

Copper, lead and zinc sulfide concentrates — Sampling procedures for determination of metal and moisture content

Concentrés sulfurés de cuivre, de plomb et de zinc — Procédures d'échantillonnage pour la détermination de la teneur en métal et de l'humidité

Reference number
ISO 12743:1998(E)

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Annexes

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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 12743 was prepared by Technical Committee ISO/TC 183, *Copper lead and zinc and concentrates*.

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

Copper, lead and zinc sulfide concentrates — Sampling procedures for determination of metal and moisture content

1 Scope

This International Standard sets out the basic methods for sampling copper, lead and zinc concentrates from moving streams and stationary lots, including stopped-belt sampling, to provide samples for chemical analysis, physical testing and determination of moisture content in accordance with the relevant International Standards. Where the concentrates are susceptible to significant oxidation or decomposition, it is necessary to use a common sample for moisture determination and chemical analysis to eliminate bias (see ISO 10251). In such cases, the common sample needs to be sufficiently representative, i.e. unbiased and sufficiently precise, for chemical analysis and determination of moisture content. Any large agglomerates (>10 mm) present in the primary sample are crushed prior to further sample processing. Sampling of concentrates in slurry form is specifically excluded from this International Standard.

Stopped-belt sampling is the reference method for collecting concentrate samples against which mechanical and manual sampling procedures may be compared. Sampling from moving streams is the preferred method. Both falling-stream and cross-belt samplers are described.

Sampling from stationary lots is used only where sampling from moving streams is not possible. The procedures described in this International Standard for sampling from stationary lots only minimize some of the systematic sampling errors.

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 applying the most recent editions of standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 10251:—[1)], *Copper, lead and zinc sulfide concentrates - Determination of mass loss of bulk material on drying*.

ISO 12744:1997, *Copper, lead and zinc sulfide concentrates - Experimental methods for checking the precision of sampling*.

ISO 13292:—[1)], *Copper, lead and zinc sulfide concentrates - Experimental methods for checking the bias of sampling*.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 representative sample: A quantity of concentrate representing a larger mass of concentrate with both precision and bias within acceptable limits.

3.2 lot: A quantity of concentrate to be sampled.

1) To be published.

3.3 lot sample: A quantity of concentrate representative of the lot.

3.4 sub-lot: Subdivided parts of a lot which are processed separately, each of them producing a subsample which is analysed separately, e.g. for moisture determination.

3.5 subsample: A quantity of concentrate representative of the sub-lot.

3.6 sampling: A sequence of operations aimed at obtaining a sample representative of a lot. It comprises a series of sampling stages, each stage usually comprising operations of selection and preparation.

3.7 selection: The operation by which a smaller quantity of concentrate is taken from a larger quantity of concentrate.

3.8 increment: A quantity of concentrate selected by a sampling device in one operation.

3.9 increment selection: A selection process that consists of extracting from the lot or from an intermediate sample successive increments which can be combined to constitute a sample.

3.10 division: The operation of decreasing sample mass, without change of particle size, where a representative part of the sample is retained.

3.11 constant-mass division: A method of division in which the retained portions from individual increments or subsamples are of uniform mass.

3.12 proportional division: A method of division in which the retained portions from individual increments or subsamples are a constant proportion of their original mass.

3.13 preparation: A non-selective operation without division such as sample transfer, drying, comminution or homogenization.

3.14 sample processing: The whole sequence of selection and preparation operations which transforms a stage *i* sample into a test sample.

3.15 comminution: The operation of reducing particle size by crushing, grinding or pulverization.

3.16 stage *i* sample: A sample obtained at the *i*th stage of the sampling scheme.

3.17 moisture sample: A representative quantity of concentrate from which test portions are taken for moisture determination. Alternatively, the whole moisture sample may be dried to determine its moisture content.

3.18 laboratory sample: A sample that is processed so that it can be sent to the laboratory and used for further processing and selection of one or more test samples for analysis.

3.19 common sample: A representative quantity of concentrate which is dried to determine its mass loss and subsequently used for further processing and selection of one or more test samples for chemical analysis.

3.20 test sample: A representative quantity of concentrate obtained from a laboratory sample when additional preparation, such as drying or hygroscopic moisture determination, is needed prior to the selection of one or more test portions.

3.21 test portion: A representative quantity of concentrate taken from a moisture sample, a laboratory sample or a test sample which is submitted for moisture determination or analysis in its entirety.

3.22 systematic sampling: The selection of increments in which the concentrate being sampled is divided into equal strata and the first increment is taken at random within the first stratum, the interval between subsequent increments being equal to the stratum size.

3.23 stratified random sampling: The selection of increments in which the concentrate being sampled is divided into equal strata, each increment being taken at random within each stratum.

3.24 homogenization: A preparation operation that reduces the distribution heterogeneity of the concentrate.

3.25 agglomerate: A cluster of particles that are held together by chemical or physical phenomena.

3.26 nominal top size: The aperture size of a test sieve that retains 5 % of the mass of concentrate.

3.27 moisture determination: The quantitative measurement of the mass loss of the moisture test portion under the conditions of drying specified in ISO 10251.

3.28 chemical analysis: The quantitative determination of the required chemical constituents of the analysis test portion.

3.29 error: In any quantitative measurement, the difference between the true value and the value obtained for an individual measurement.

3.30 bias: The statistically significant difference between the mean of the test results and an accepted reference value (see also ISO 13292).

3.31 precision: The closeness of agreement between independent test results obtained under stipulated conditions (see also ISO 12744).

3.32 interleaved samples: Samples constituted by placing consecutive primary increments alternately into two separate sample containers.

4 Sampling theory

4.1 General

The basic rule for a correct sampling method is that all possible increments from the concentrate stream or stratum have the same probability of being selected and appearing in the sample. Any deviation from this basic requirement can result in a bias. An incorrect sampling scheme cannot be relied on to provide representative samples.

Sampling should preferably be carried out on a systematic basis, either on a mass basis (see 7.2) or on a time basis (see 7.3), but only where it can be shown that no systematic error (or bias) could be introduced due to any periodic variation in quality or quantity that may coincide with, or approximate to, any multiples of the proposed sampling interval. In such cases, it is recommended that stratified random sampling within fixed time or mass intervals be carried out (see 7.4).

The methods for sampling, including sample processing, depend on the final choice of the sampling scheme and on the steps necessary to minimize possible systematic errors. The aim always is to reduce the total variance to an acceptable level while at the same time eliminating any significant biases, e.g. minimizing degradation of samples used for determination of size distribution.

Moisture samples shall be processed as soon as possible and test portions weighed immediately. If this is not possible, samples shall be stored in impervious air-tight containers with a minimum of free air space to minimize any change in moisture content, but should be prepared without delay.

4.2 Total variance

The general aim of a sampling scheme is to provide one or several test portions, sufficiently representative of a lot, for determination of the quality characteristics of the lot. The total variance of the final result, denoted by s_T^2 consists of the variance of sampling (including sample processing) plus the variance of analysis (chemical analysis, moisture determination, determination of particle size distribution, etc.) as follows:

$$s_T^2 = s_S^2 + s_A^2 \quad \ldots (1)$$

where

s_S^2 is the sampling variance (including sample processing);

s_A^2 is the analytical variance.

In equation 1, the sampling variance includes the variances due to all sampling (and sample processing) steps except selection of the test portion. The variance due to selection of the test portion is included in the analytical variance, s_A^2, which is determined in accordance with ISO 12744, because it is difficult to determine separately the "true" analytical variance.

Often replicate analyses of quality characteristics are carried out, reducing the total variance. In this case, if r replicate analyses are made:

$$s_T^2 = s_S^2 + \frac{s_A^2}{r} \quad \ldots (2)$$

The estimation or measurement of the total variance can be carried out in several ways, depending on the purpose of the exercise. In many respects the different approaches are complementary.

The first method, which was developed by Gy, [3],[4], is to break up the sampling variance into its components for each sampling stage (see annex A). The total variance is then given by:

$$s_T^2 = s_{S_1}^2 + \ldots + s_{S_i}^2 + \ldots + s_{S_{u-1}}^2 + \frac{s_A^2}{r} \quad \ldots (3)$$

where

$s_{S_1}^2$ is the sampling variance for stage 1, i.e. the primary sampling variance;

$s_{S_i}^2$ is the sampling variance for stage i;

$s_{S_{u-1}}^2$ is the sampling variance for stage u-1, the second last stage;

u is the number of sampling stages, stage u corresponding to selection of the test portion.

This is referred to as the "sampling stage" method (see 4.3) and provides very detailed information on the variance components, which is particularly useful for designing and assessing sampling schemes. However, to obtain maximum benefit, it is necessary to collect data at each sampling stage.

The second method, called the "simplified" method (see 4.4), is to break up the total variance into primary sampling, sample processing and analytical variances only as follows:

$$s_T^2 = s_{S_1}^2 + s_P^2 + \frac{s_A^2}{r} \quad \ldots(4)$$

where

$s_{S_1}^2$ is the primary sampling variance;

s_P^2 is the variance due to all subsequent sampling steps, i.e. sample processing, except selection of the test portion;

s_A^2 is the analytical variance, including selection of the test portion (at stage u in equation 3).

The primary sampling variance is identical to the sampling variance for stage 1 in equation 3, while s_P^2 is equal to the total sampling variance for the remaining sampling stages, except for selection of the test portion which is included in the analytical variance. The relative magnitudes of the variance components in equation 4 indicate where additional effort is required to reduce the total variance. However, it is not possible to separate the variances of the separate sample processing stages. This method is suitable for estimating the total variance for new sampling schemes based on the same sample processing procedures, where the numbers of primary increments, sample processings and analyses are varied.

Finally, the total variance s_T^2 can be estimated experimentally by collecting interleaved duplicate samples (see 4.5). This is called the "interleaved sample" method and gives valuable information on the total variance actually achieved for a given sampling scheme with no extra effort, provided that facilities are available for collecting duplicate samples (Merks [5]). It gives no information on variance components, but the total variance can be compared with the analytical variance to ascertain whether the sampling scheme used was optimized or not. It is therefore of limited use for designing sampling schemes.

4.3 Sampling stage method of estimating sampling and total variance

The sampling variance for stage i is given by (see annex A):

$$s_{S_i}^2 = \frac{s_{b_i}^2}{n_i} \quad \ldots(5)$$

where

$s_{b_i}^2$ is the variance between increments for stage i;

n_i is the number of increments for stage i.

The variance between increments for stage i, $s_{b_i}^2$, can be estimated using the following equation:

$$s_{b_i}^2 = \frac{\sum_{j=i}^{n} \left(x_j - \bar{x}\right)^2}{n_i - 1} - s_{PA}^2 \quad \ldots(6)$$

where

x_j is the test result for increment j;

$\bar{x}$ is the mean test result for all increments;

s^2_{PA} is the variance of subsequent sample processing and analysis.

The variance of subsequent sample processing and analysis of each increment, s^2_{PA}, has been taken into account in equation 6 to obtain an unbiased estimate of $s^2_{b_i}$.

NOTE — Care is needed in subtracting variances. The difference is significant only if the *F* ratio of the variances being subtracted is statistically significant.

Remembering that the variance due to selection of the test portion is included in the analytical variance s^2_A, the total sampling variance is given by:

$$s^2_S = \sum_{i=1}^{u-1} \frac{s^2_{b_i}}{n_i} \quad \ldots(7)$$

Combining equations 2 and 7 gives the total variance s^2_T as follows:

$$s^2_T = \sum_{i=1}^{u-1} \frac{s^2_{b_i}}{n_i} + \frac{s^2_A}{r} \quad \ldots(8)$$

For a three-stage sampling scheme (including selection of the test portion), equation 8 reduces to:

$$s^2_T = \frac{s^2_{b_1}}{n_1} + \frac{s^2_{b_2}}{n_2} + \frac{s^2_A}{r} \quad \ldots(9)$$

The best way of reducing the value of s^2_T to an acceptable level is to reduce the largest terms in equation 8 first. Clearly $s^2_{b_i}/n_i$ for a given sampling stage can be reduced by increasing the number of increments n_i or reducing $s^2_{b_i}$ by homogenizing the concentrate prior to sampling. The last term can be reduced by reducing the particle size prior to selection of the test portion, or performing replicate analyses. Selecting the optimum number of increments n_i for each sampling stage may require several iterations to obtain the required total variance s^2_T.

Example

Consider a four-stage sampling scheme for determining the metal content of a copper concentrate containing 31,2 % Cu. Assume that the concentrate is being conveyed at 500 t/h on a conveyor belt, that the lot size is 500 t, and that the following parameters have been determined using equation 6 where appropriate:

s_{b_1} = 0,3 % Cu

s_{b_2} = 0,2 % Cu

s_{b_3} = 0,1 % Cu

s_A = 0,05 % Cu

NOTE — Many measurements may be required to obtain good estimates of s_{b_1}, s_{b_2}, s_{b_3} and s_A.

Stage 1
Assume that the primary cutter takes increments of 12 kg mass at 2 min intervals. Thus:

$n_1 = 30$

Primary sample mass = 360 kg

Equation 5 gives:

$s_{S_1}^2 = (0,3)^2/30 = 0,003\,0$

Stage 2
The primary increments are collected in a hopper, and then fed to the secondary cutter at the rate of 360 kg/h. Secondary increments of 0,01 kg are taken at 30 s intervals. Thus:

$n_2 = 120$

Divided sample mass = 1,2 kg

$s_{S_2}^2 = (0,2)^2/120 = 0,000\,333$

Stage 3
The 1,2 kg sample is transported to the sample processing laboratory and fed through a rotary sample divider with a sample collection canister divided into 8 equal sectors rotating at 30 rev/min (0,5 s^{-1}). Sample division takes 2 min. Thus:

$n_3 = 60$

Divided sample mass = 150 g

$s_{S_3}^2 = (0,1)^2/60 = 0,000\,167$

Stage 4
Dry the sample and then pulverize to 150 µm. Select a 1 g test portion by taking 10 increments of 0,1 g with a spatula and conduct a single analysis. Thus:

$s_A = 0,05$ % Cu

Total variance
The total variance is given by:

$$s_T^2 = s_{S_1}^2 + s_{S_2}^2 + s_{S_3}^2 + s_A^2$$

$$= 0,003\,0 + 0,000\,333 + 0,000\,167 + 0,002\,5$$

$$= 0,006$$

Hence:

$s_T = 0,077$ % Cu

In this example, the largest components of variance are due to primary sampling and analysis.

Consequently, the total variance can be reduced by increasing the number of primary increments and conducting replicate analyses.
An example of the application of the sampling stage method of estimating total variance to sampling from grabs is given in annex B.

4.4 Simplified method of estimating sampling and total variance

While it is not possible to partition, i.e. separate, the variances of the individual sample processing stages, the simplified method is suitable for estimating the total variance for new sampling schemes based on the same sample processing procedures, where the numbers of primary increments, sample processings and analyses are varied.

Using equation 5, the primary sampling variance $s_{S_1}^2$ is given by:

$$s_{S_1}^2 = \frac{s_{b_1}^2}{n_1} \qquad \ldots (10)$$

where

n_1 is the number of primary increments;

$s_{b_1}^2$ is the primary variance between increments determined using equation 6.

The primary sampling variance can be reduced by increasing the number of primary increments n_1.

The sample processing variance s_P^2 and analytical variance s_A^2 are determined experimentally by duplicate sample processing and determination of quality characteristics in accordance with ISO 12744. The analytical variance s_A^2 can also be obtained by carrying out duplicate analyses on test samples.

Multiple sample processings and analyses are often carried out to reduce the total variance. In this case, combining equations 4 and 10 gives:

a) Where a single sample is constituted for the lot and *r* replicate analyses are carried out on the test sample:

$$s_T^2 = \frac{s_{b_1}^2}{n_1} + s_P^2 + \frac{s_A^2}{r} \qquad \ldots (11)$$

b) Where the lot is divided into *k* sub-lots, a subsample is constituted for each sub-lot, and *r* replicate analyses are carried out on each resultant test sample:

$$s_T^2 = \frac{s_{b_1}^2}{n_1} + \frac{s_P^2}{k} + \frac{s_A^2}{rk} \qquad \ldots (12)$$

c) Where sample processing and analysis is carried out on each increment taken from the lot and *r* replicate analyses are carried out:

$$s_T^2 = \frac{s_{b_1}^2 + s_P^2 + \dfrac{s_A^2}{r}}{n_1} \qquad \ldots (13)$$

Example

Assume that 50 primary increments are taken from a zinc concentrate lot that has been divided into two sub-lots. The resultant two subsamples are processed separately and analysed in duplicate. Assume that the primary increment, sample processing and analytical standard deviations have been determined experimentally as follows:

s_{b_1} = 0,3 % Zn

s_P = 0,1 % Zn

s_A = 0,05 % Zn

Using equation 12, the total variance is given by:

$$s_T^2 = (0,3)^2/50 + (0,1)^2/2 + (0,05)^2/(2 \times 2)$$

$$= 0,001\,8 + 0,005 + 0,000\,625$$

$$= 0,007\,43$$

Hence:

s_T = 0,086 % Zn

In this example, the major component of variance is sample processing. This component could be reduced by dividing the lot into a larger number of sub-lots, and constituting a subsample for each sub-lot.

4.5 Interleaved sample method of measuring total variance

The total variance s_T^2 actually achieved for a given sampling operation can be estimated experimentally by collecting interleaved duplicate samples as shown in figure 1. The odd and even numbered increments from two adjacent lots are separately combined to give samples A and B for the two lots (each sample essentially representing a lot twice the size). Samples A and B are then separately submitted to sample processing and analysis.

This procedure is repeated until sampling has been completed. The total variance for a single lot is then given by:

$$s_T^2 = \frac{\pi}{4}\left[\frac{\sum_{i=1}^{N}\left|x_{A_i} - x_{B_i}\right|}{N}\right]^2 \quad \ldots (14)$$

where

x_{A_i} and x_{B_i} are the analyses for each pair of samples A_i and B_i;

N is the number of pairs (in the range 10 to 20);

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