INTERNATIONAL STANDARD

ISO 12743

Fourth edition
2021-05

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

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

Reference number
ISO 12743:2021(E)

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

Published in Switzerland

Contents

Page

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.

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 documents 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This fourth edition cancels and replaces the third edition (ISO 12743:2018), which has been technically revised. The main changes to the previous edition are as follows:

— The minimum cutting aperture for cross-belt cutters in 8.3.2.3 i) has been reduced to 30 mm.

— A NOTE has been added to 15.4.10 indicating that ribbons with smaller dimensions can be formed depending on the mass of sample to be divided, and that the ribbon division method is particularly suitable for dividing chemical analysis samples.

— The requirements for preparation of chemical analysis samples in 16.2 have been expanded.

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 — Sampling procedures for determination of metal and moisture content

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 to ensure compliance with any other restrictions.

1 Scope

This document sets out the basic methods for sampling copper, lead, zinc and nickel 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, a common sample that is sufficiently representative, i.e. unbiased and sufficiently precise, is used for moisture determination and chemical analysis to eliminate bias (see ISO 10251). 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 document.

Stopped-belt sampling is the reference method for collecting concentrate samples against which mechanical and manual-sampling procedures can 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 document for sampling from stationary lots only minimize some of the systematic sampling errors.

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 10251, *Copper, lead, zinc and nickel concentrates — Determination of mass loss of bulk material on drying*

ISO 12744, *Copper, lead, zinc and nickel concentrates — Experimental methods for checking the precision of sampling*

ISO 13292, *Copper, lead, zinc and nickel concentrates — Experimental methods for checking the bias of sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
representative sample
quantity of concentrate representing a larger mass of concentrate with both *precision* (3.29) and *bias* (3.28) within acceptable limits

3.2
lot
quantity of concentrate to be sampled

3.3
lot sample
quantity of concentrate representative of the *lot* (3.2)

3.4
sub-lot
subdivided parts of a *lot* (3.2) which are processed separately, each of them producing a *subsample* (3.5) which is analysed separately, for example for moisture determination

3.5
subsample
quantity of concentrate representative of the *sub-lot* (3.4)

3.6
sampling
sequence of operations aimed at obtaining a sample representative of a *lot* (3.2)

Note 1 to entry: It comprises a series of sampling stages, each stage usually comprising operations of selection and preparation.

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

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

3.9
division
operation of decreasing sample mass, without change of particle size, where a representative part of the sample is retained

3.10
constant-mass division
method of *division* (3.9) in which the retained portions from individual *increments* (3.8) or *subsamples* (3.5) are of uniform mass

3.11
proportional division
method of *division* (3.9) in which the retained portions from individual *increments* (3.8) or *subsamples* (3.5) are a constant proportion of their original mass

3.12
preparation
nonselective operation without *division* (3.9) such as sample transfer, drying, comminution or homogenization

3.13
sample processing
whole sequence of selection and preparation operations which transforms a *stage i sample* (3.15) into a *test sample* (3.19)

3.14
comminution
operation of reducing particle size by crushing, grinding or pulverisation

3.15
stage *i* sample
sample obtained at the *i*th stage of the sampling scheme

3.16
moisture sample
representative quantity of concentrate from which *test portions* (3.20) are taken for moisture determination

Note 1 to entry: Alternatively, the whole moisture sample may be dried to determine its moisture content.

3.17
laboratory sample
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* (3.19) for analysis

3.18
common sample
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* (3.19) for chemical analysis

3.19
test sample
representative quantity of concentrate obtained from a *laboratory sample* (3.17) when additional preparation, such as drying or hygroscopic moisture determination, is needed prior to the selection of one or more *test portions* (3.20)

3.20
test portion
representative quantity of concentrate taken from a *moisture sample* (3.16), a *laboratory sample* (3.17) or a *test sample* (3.19) which is submitted to moisture determination or analysis in its entirety

3.21
systematic sampling
selection of *increments* (3.8) in which the concentrate being sampled is divided into equal strata and the first *increment* (3.8) is taken at random within the first stratum, the interval between subsequent *increments* (3.8) being equal to the stratum size

3.22
stratified random sampling
selection of *increments* (3.8) in which the concentrate being sampled is divided into equal strata, each *increment* (3.8) being taken at random within each stratum

3.23
agglomerate
cluster of particles that are held together by chemical or physical phenomena

3.24
nominal top size
aperture size of a test sieve that retains 5 % of the mass of concentrate

3.25
moisture determination
quantitative measurement of the mass loss of the moisture *test portion* (3.20) under the conditions of drying specified in ISO 10251

3.26
chemical analysis
quantitative determination of the required chemical constituents of the analysis *test portion* (3.20)

3.27
error
difference between the true value and the value obtained for an individual measurement in any quantitative measurement

3.28
bias
statistically significant difference between the mean of the test results and an accepted reference value

Note 1 to entry: See also ISO 13292.

3.29
precision
closeness of agreement between independent test results obtained under stipulated conditions

Note 1 to entry: See also ISO 12744.

3.30
interleaved samples
samples constituted by placing consecutive primary *increments* (3.8) 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 shall 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 is always to reduce the total variance to an acceptable level, while at the same time eliminating any significant biases, for example minimizing degradation of samples used for determination of size distribution.

Moisture samples shall be processed as soon as possible and test portions shall be weighed immediately. If this is not possible, samples shall be stored in impervious airtight 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 (e.g. chemical analysis, moisture determination, determination of particle size distribution) and is given by Formula (1):

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

where

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

s_A^2 is the analytical variance.

In Formula (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 shall be 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, which reduces the total variance. In this case, if r replicate analyses are made, the total variance is given by Formula (2):

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

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, and shall be carried out as specified in Annex A. The total variance is then given by Formula (3):

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

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. In this case, the total variance is given by Formula (4):

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

where

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

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

s^2_A is the analytical variance, including selection of the test portion [at stage u in Formula (3)].

The primary sampling variance is identical to the sampling variance for stage 1 in Formula (3), while s^2_P 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 Formula (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^2_T can be estimated experimentally by collecting interpenetrating 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 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, but it can be used to monitor whether a sampling scheme is in control.

4.3 Sampling-stage method of estimating sampling and total variance

The sampling variance for stage i is given by Formula (5) (see Annex A):

$$s^2_{S_i} = \frac{s^2_{b_i}}{n_i} \tag{5}$$

where

$s^2_{b_i}$ 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, $s^2_{b_i}$, can be estimated using Formula (6):

$$s^2_{b_i} = \frac{\sum_{j=1}^{n}(x_j - \bar{x})^2}{n_i - 1} - s^2_{PA} \tag{6}$$

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 Formula (6) to obtain an unbiased estimate of $s^2_{b_i}$.

NOTE Care is needed in subtracting variances. The difference is significant only when 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 Formula (7):

$$s^2_S = \sum_{i=1}^{u-1} \frac{s^2_{b_i}}{n_i} \tag{7}$$

Combining Formulae (2) and (7) provides the total variance s^2_T, which is given by Formula (8):

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

For a three-stage sampling scheme (including selection of the test portion), Formula (8) reduces to Formula (9):

$$s^2_T = \frac{s^2_{b_i}}{n_1} + \frac{s^2_{b_2}}{n_2} + \frac{s^2_A}{r} \tag{9}$$

The best way of reducing the value of s^2_T to an acceptable level is to reduce the largest terms in Formula (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 Formula (6) where appropriate:

$s_{b_1} = 0{,}3\,\%\,\text{Cu}$

$s_{b_2} = 0{,}2\,\%\,\text{Cu}$

$s_{b_3} = 0{,}1\,\%\,\text{Cu}$

$s_A = 0{,}05\,\%\,\text{Cu}$

NOTE Many measurements might 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

Formula (5) gives:

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

Stage 2

The primary increments are collected in a hopper and then fed to the secondary cutter at a 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

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

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 eight 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

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

Stage 4

Dry the sample and then pulverize it 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:

$s_A = 0,05\ \%\ Cu$

The total variance is given by:

$$s^2_T = s^2_{S_1} + s^2_{S_2} + s^2_{S_3} + s^2_A$$

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

$$= 0,006$$

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 Formula (5), the primary sampling variance $s^2_{S_1}$ is given by Formula (10):

$$s^2_{S_1} = \frac{s^2_{b_1}}{n_1} \tag{10}$$

where