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

*Concentrés sulfurés de cuivre, de plomb et de zinc — Détermination  
de la perte de masse au séchage du matériau en vrac*

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## Foreword

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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 10251 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes A and B form an integral part of this International Standard. Annex C is for information only.

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## Introduction

Reference to the percentage mass loss as moisture content is appropriate, because, although oxidation, decomposition or sublimation of elemental sulfur may contribute, most of the mass loss on drying is due to loss of moisture.

When oxidation, decomposition or sublimation of elemental sulfur has been shown to occur or volatile organic flotation reagents such as kerosene are present, the chemical analysis test sample should be prepared from the dried moisture test portions. Under these circumstances, the sampling scheme established in accordance with ISO 12743 must ensure that moisture samples and test portions are sufficiently representative for subsequent chemical analysis. When oxidation is a problem, an inert atmosphere may also be used during the drying stage. Annex A provides a procedure by which it can be determined whether or not a concentrate is susceptible to oxidation, decomposition or sublimation.

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# Copper, lead and zinc sulfide concentrates — Determination of mass loss of bulk material on drying

## 1 Scope

This International Standard specifies methods for the determination of moisture content of a lot of copper, lead or zinc sulfide concentrate, defined as the percentage mass loss of the moisture test portion under the conditions of drying specified in this document.

NOTE 1 In order to obtain an unbiased estimate of the metal content of the lot, it is important that the same drying conditions are used for the determination of bulk and hygroscopic moisture or for preparing a predried test portion.

This International Standard is not applicable to drying samples used for determination of volatile elements such as mercury and sulfur. Such samples are allowed to dry at ambient temperature and a hygroscopic moisture determination is carried out in accordance with ISO 9599 at the time of chemical analysis.

## 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 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 9599: 1991, *Copper lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method.*

ISO 12743:—<sup>1)</sup>, *Copper, lead and zinc sulfide concentrates — Sampling procedures for determination of metal and moisture content.*

## 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.

**3.3 lot sample:** A quantity of concentrate which is representative of the lot.

1) To be published.

**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 which is representative of the sub-lot.

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

**3.7 moisture sample:** A representative quantity of concentrate from which test portions are taken for moisture determination.

NOTE 2 Alternatively, the whole moisture sample may be dried to determine its moisture content.

**3.8 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 chemical analysis.

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

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

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

## 4 Drying method

### 4.1 General

Test portions are dried at  $105\text{ °C} \pm 5\text{ °C}$  until constant mass is obtained and the moisture content determined as the percentage mass loss on drying. However, drying to constant mass can be difficult or impossible if the concentrate is susceptible to oxidation, decomposition or sublimation of elemental sulfur or volatile organic flotation reagents such as kerosene are present (see annex A). Under these circumstances, a common sample for moisture determination and chemical analysis, drying in an inert atmosphere, or interruption of drying is applied. If a common sample is used, the moisture samples and test portions must be sufficiently representative for subsequent chemical analysis.

One of the following drying methods is selected to suit the particular concentrate. A flowsheet for selecting the correct drying method is given in figure 1.

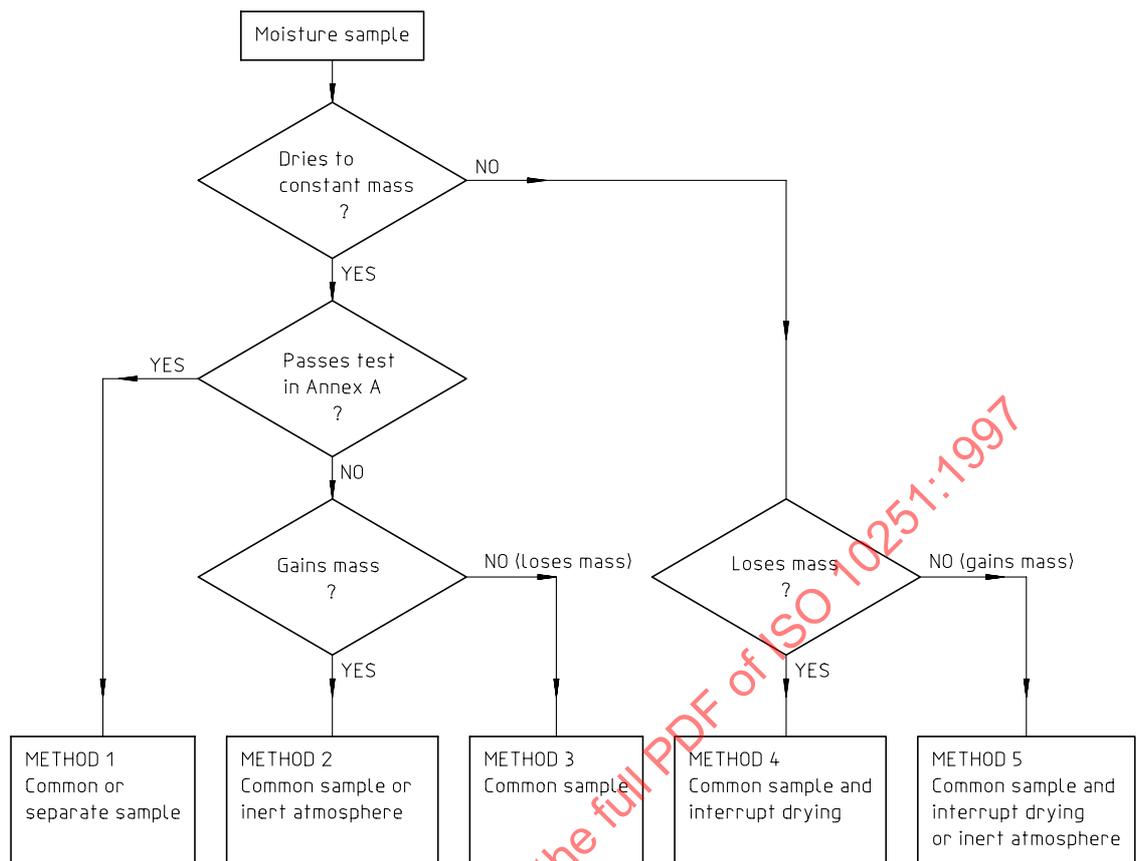


Figure 1 - Procedure for selection of appropriate drying method

#### 4.2 Method 1

Where the test portion can be dried to constant mass and the concentrate passes the test in annex A, a separate sample for moisture determination or a common sample is dried to constant mass.

#### 4.3 Method 2

Where the test portion can be dried to constant mass, but the test in annex A results in a higher mass, indicating that the concentrate is susceptible to oxidation, moisture and chemical analysis samples are dried to constant mass in an inert atmosphere, or a common sample is dried to constant mass.

#### 4.4 Method 3

Where the test portion can be dried to constant mass, but the test in annex A results in a lower mass, indicating that the concentrate may be losing organics over an extended period, a common sample is dried to constant mass.

#### 4.5 Method 4

Where the test portion cannot be dried to constant mass and continues to lose mass over long periods, indicating that the concentrate may be losing hydrated water, decomposing or subliming, a common sample is used, with interruption of drying after a period determined in accordance with clause 8 for each concentrate type and no further drying prior to analysis.

#### 4.6 Method 5

Where the test portion cannot be dried to constant mass and after the initial drying period continues to gain mass, indicating that the concentrate may be oxidizing even when dry, moisture and chemical analysis samples are dried to constant mass in an inert atmosphere, or a common sample is used with interruption of drying after a period determined in accordance with clause 8 for each concentrate type and no further drying prior to analysis.

### 5 Apparatus

**5.1 Drying oven**, ventilated, with forced circulation of air or inert gas, regulated at a temperature of  $105\text{ °C} \pm 5\text{ °C}$ .

**5.2 Top-loading balance**, having a minimum precision of 0,01 % as specified in table 1.

**5.3 Drying trays**, having dimensions that permit the sample to be spread to a thickness of less than 30 mm. The trays shall be made of corrosion-resistant and heat-resistant material such as stainless steel, glass or enamel plate.

**Table 1 - Mass of sample and minimum precision and weighing**

Mass of sample kg	Minimum precision of balance and weighing g
1	0,1
2	0,2
5	0,5
10	1
20	2
50	5

NOTE 3 The weighing platform should be protected from heat transfer by a suitable insulating material, e.g. a 13 mm layer of polystyrene.

### 6 Processing of samples

Samples for moisture determination shall be taken and processed in accordance with ISO 12743.

If the concentrate is cohesive or excessively wet, the sample may be predried until sample preparation can be conducted without difficulty. The predried moisture content and the total moisture content of the sample shall be determined by the procedure specified in annex B.

## 7 Samples for moisture determination

### 7.1 General

Samples for moisture determination shall be taken as close to the point (position and time) of mass determination as practicable. Samples shall then be prepared and the test portions weighed immediately to minimize bias.

Breaking up of agglomerates by screening is not permitted, because this will result in a change in moisture content.

NOTE 4 If agglomerates are present, the minimum mass of the test portion specified in 7.2, 7.3 and 7.4 or the number of moisture determinations may have to be increased to obtain the required precision.

### 7.2 From a single lot sample

Where a single lot sample is obtained from a lot, four test portions of not less than 1 kg shall be taken as specified in table 2 and two of these shall be submitted initially for the determination of moisture content. The two reserve test portions shall be weighed in accordance with the procedure specified in clause 8 and set aside on a covered tray.

**Table 2 - Minimum number of test portions for moisture determination**

Type of sample	Number of test portions	Number of subsamples per lot
Lot sample	4	-
Subsample	2	2 to 3
	1	≥ 4
Increment	1	-

### 7.3 From subsamples

Where subsamples from a lot are not combined into a single lot sample, the minimum number of test portions specified in table 2 shall be taken from each subsample and submitted for the determination of moisture content. Each test portion shall be not less than 1 kg in mass.

Where a subsample is prepared from each sub-lot, the mass of each sub-lot shall be recorded at the same time for calculation of the moisture content of the lot.

### 7.4 From increments

Where moisture determination is conducted on each increment, one test portion of not less than 1 kg shall be taken from each increment as specified in table 2 and submitted for the determination of moisture content.

The mass of each stratum shall be recorded at the same time for calculation of the moisture content of the lot.

## 8 Procedure for drying samples to constant mass

Weigh the drying tray and record the mass ( $m_1$ ) to the precision specified in table 1. Transfer the test portion (not less than 1 kg) to the drying tray, spreading it evenly using a suitable implement, and weigh immediately. Record the mass ( $m_2$ ) to the precision specified in table 1.

Place the tray and test portion in the drying oven and dry at  $105\text{ °C} \pm 5\text{ °C}$  for a predetermined time.

### NOTES

5 The initial drying time has to be determined empirically. Sixteen hours is sufficient for most cases and can be taken as a guide.

6 Once the drying period has commenced, no other wet samples should be placed in the same drying oven.

Remove the tray and test portion from the oven and weigh while still hot. Dry the tray and test portion at  $105\text{ °C} \pm 5\text{ °C}$  for an additional 4 h. Remove from the oven and while still hot weigh again. Repeat this step as necessary until two successive determinations of the mass of the tray plus dried test portion agree within 0,05 % of the initial mass of the test portion. Record the mass of the dried test portion plus tray ( $m_3$ ) to the precision specified in table 1.

NOTE 7 Any crusts or cakes that form on the surface of the test portion after initial drying should be broken up to enable easier release of moisture.

If after two successive 4 h drying periods the masses of the tray plus dried test portion do not agree to within 0,05 % of the initial mass of the test portion ( $m_2 - m_1$ ) or the mass  $m_3$  has started to increase, one of the conditions described in 4.4 or 4.5 exists. At this stage the drying process shall be interrupted and the mass of the tray plus dried test portion recorded as the final mass ( $m_3$ ). The total drying period, i.e. 24 h assuming a 16 h initial drying period, shall be used in future when drying this type of concentrate.

## 9 Calculation

### 9.1 Moisture content of test portion

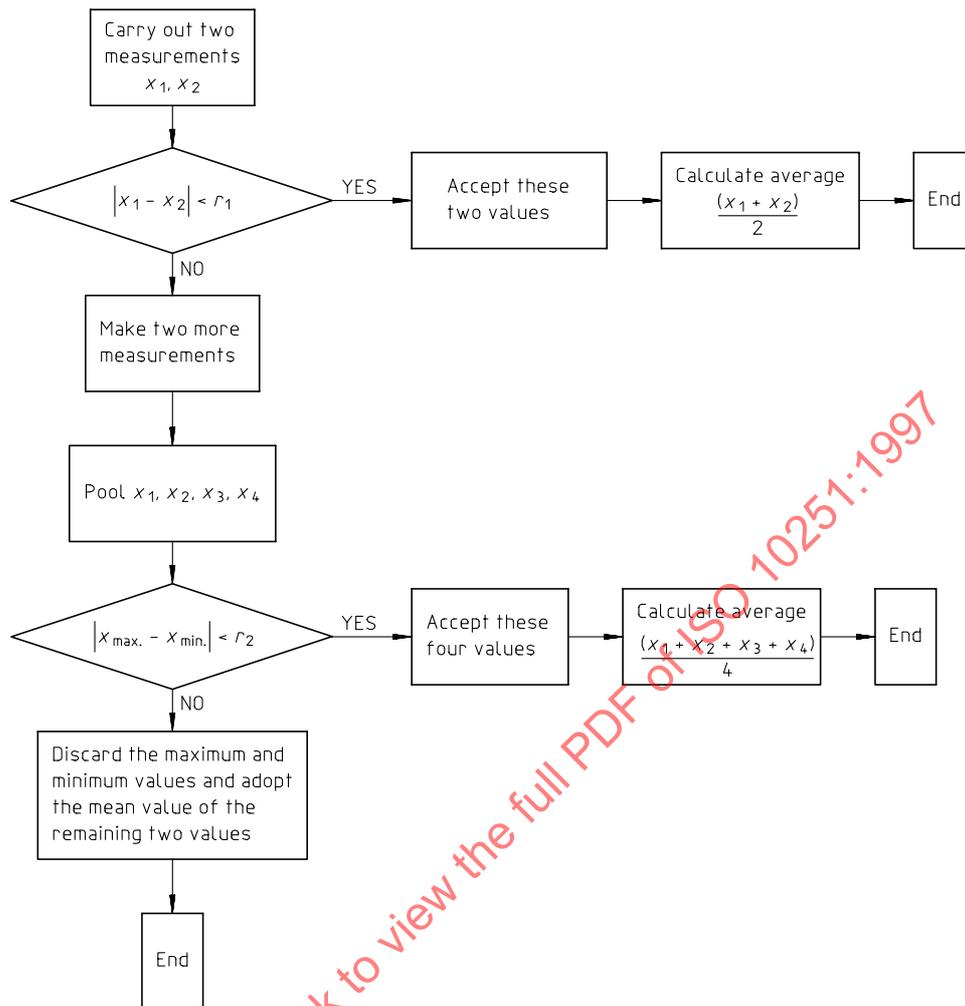
The moisture content of the test portion, expressed as a percentage by mass, is calculated using the equation:

$$x = \frac{100(m_2 - m_3)}{m_2 - m_1} \quad \dots (1)$$

where

- $x$  is the moisture content as a percentage of the test portion;
- $m_1$  is the mass of the drying tray, in grams;
- $m_2$  is the mass of the drying tray plus wet test portion, in grams;
- $m_3$  is the mass of the drying tray plus dried test portion, in grams.

An example of a work sheet for reporting the results is given in annex C.



## NOTE

- a)  $r_1 = 0,14 \%$  Pb, Zn concentrates  
Moisture < 6 %  
 $r_1 = 0,20 \%$  All other cases
- b)  $r_2 = 0,18 \%$  Pb, Zn concentrates  
Moisture < 6 %  
 $r_2 = 0,25 \%$  All other cases

**Figure 2 - Flow chart of procedure for the processing of the result of moisture determination on a lot sample**

## 9.2 Moisture content of lot

### 9.2.1 From a single lot sample

The moisture content of the lot shall be calculated by the procedure set out in figure 2.

**9.2.2 From several subsamples**

Where several subsamples have been extracted and a single test portion is taken from each subsample, the moisture content of the subsample shall be taken as the moisture content of the sub-lot. However, if two test portions have been taken from each subsample, the mean of the duplicate measurements shall be taken as the moisture content of the sub-lot. The moisture content of the lot, expressed as a percentage by mass, is calculated using the equation:

$$W_L = \frac{\sum_{i=1}^k W_i m_i}{\sum_{i=1}^k m_i} \quad \dots (2)$$

where

- $W_L$  is the moisture content as a percentage of the lot;
- $k$  is the number of sub-lots;
- $W_i$  is the moisture content as a percentage of the  $i$ th sub-lot;
- $m_i$  is the mass of the  $i$ th sub-lot, in tonnes.

**9.2.3 From each increment**

Where moisture determination has been conducted on each increment, the moisture content of the lot, expressed as a percentage by mass, is calculated using the equation:

$$W_L = \frac{\sum_{i=1}^n W_i m_{s,i}}{\sum_{i=1}^n m_{s,i}} \quad \dots (3)$$

where

- $n$  is the number of increments;
- $W_i$  is the moisture content as a percentage of the  $i$ th increment;
- $m_{s,i}$  is the mass of the  $i$ th stratum, in tonnes.

Where the masses of the strata are approximately equal, the above equation can be simplified as follows:

$$w_L = \frac{\sum_{i=1}^n W_i}{n} \quad \dots (4)$$

## 10 Test report

The test report shall contain the following information:

- a) identification of the sample;
- b) reference to this International Standard, i.e., ISO 10251;
- c) a statement regarding use of an inert atmosphere or a common moisture/chemical analysis sample;
- d) number of moisture determinations;
- e) moisture content of the lot, expressed as a percentage by mass;
- f) dates of sampling and completion of tests.

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## Annex A (normative)

### Test for susceptibility of concentrates to oxidation, decomposition and sublimation

#### A.1 Introduction

Some sulfide concentrate samples are susceptible to oxidation or decomposition on drying in air at  $105\text{ °C} \pm 5\text{ °C}$  and a few concentrates will lose sulfur at this temperature.

#### A.2 Procedure

Select at random, a moisture test portion. After determining the mass ( $m_3$ ) of the dried test portion plus tray in accordance with clause 8, add an amount of water (without mixing or disturbing the sample in any way) approximately equal to the observed drying loss ( $m_2 - m_3$ ) and repeat the drying steps described in clause 8 until a constant mass ( $m_4$ ) is obtained.

- a) The concentrate is susceptible to oxidation if:

$$m_4 - m_3 > \frac{m_2 - m_1}{2\,000} \quad \dots \text{(A.1)}$$

where

$m_1$  is the mass of the drying tray, in grams;

$m_2$  is the mass of the drying tray plus wet test portion, in grams;

$m_3$  is the mass of the drying tray plus dried test portion, in grams;

$m_4$  is the mass of the drying tray plus the re-dried test portion, in grams.

- b) The concentrate is susceptible to decomposition and/or sublimation of elemental sulfur if:

$$m_3 - m_4 > \frac{m_2 - m_1}{2\,000} \quad \dots \text{(A.2)}$$