

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

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



Reference number  
ISO 10251:2006(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

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

This second edition cancels and replaces the first edition (ISO 10251:1997), which has been technically revised.

## 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. Where 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, zinc and nickel concentrates — Determination of mass loss of bulk material on drying

**WARNING** — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety issues associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

## 1 Scope

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

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 according to ISO 9599 at the time of chemical analysis.

## 2 Normative references

The following referenced documents are indispensable for the application 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 12743, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

## 3 Terms and definitions

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

### 3.1

#### **representative sample**

quantity of concentrate representing a larger mass of concentrate with both precision and bias within acceptable limits

### 3.2

#### **lot**

quantity of concentrate to be sampled

### 3.3

#### **lot sample**

quantity of concentrate that is representative of the lot

**3.4**

**sub-lot**

subdivided parts of a lot that are processed separately, each of them producing a subsample which is analysed separately, e.g. for moisture determination

**3.5**

**subsample**

quantity of concentrate that is representative of the sub-lot

**3.6**

**increment**

quantity of concentrate selected by a sampling device in one operation

**3.7**

**moisture sample**

representative quantity of concentrate from which test portions are taken for moisture determination

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

**3.8**

**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 for chemical analysis

**3.9**

**common sample**

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**

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**

representative quantity of concentrate taken from a moisture sample, a laboratory sample or a test sample that is submitted to moisture determination or analysis in its entirety

## **4 Drying method**

### **4.1 General**

Test portions are dried at  $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  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, using a common sample for moisture determination and chemical analysis, drying in an inert atmosphere, or interrupting the drying is applied. Of these alternatives, the use of a common sample is the best approach, although care needs to be taken to ensure that the moisture samples and test portions are sufficiently representative for subsequent chemical analysis.



One of the following drying methods is selected to suit the particular concentrate. A flow chart for selecting the correct drying method is given in Figure 1, noting that use of a common sample is applicable in all cases.

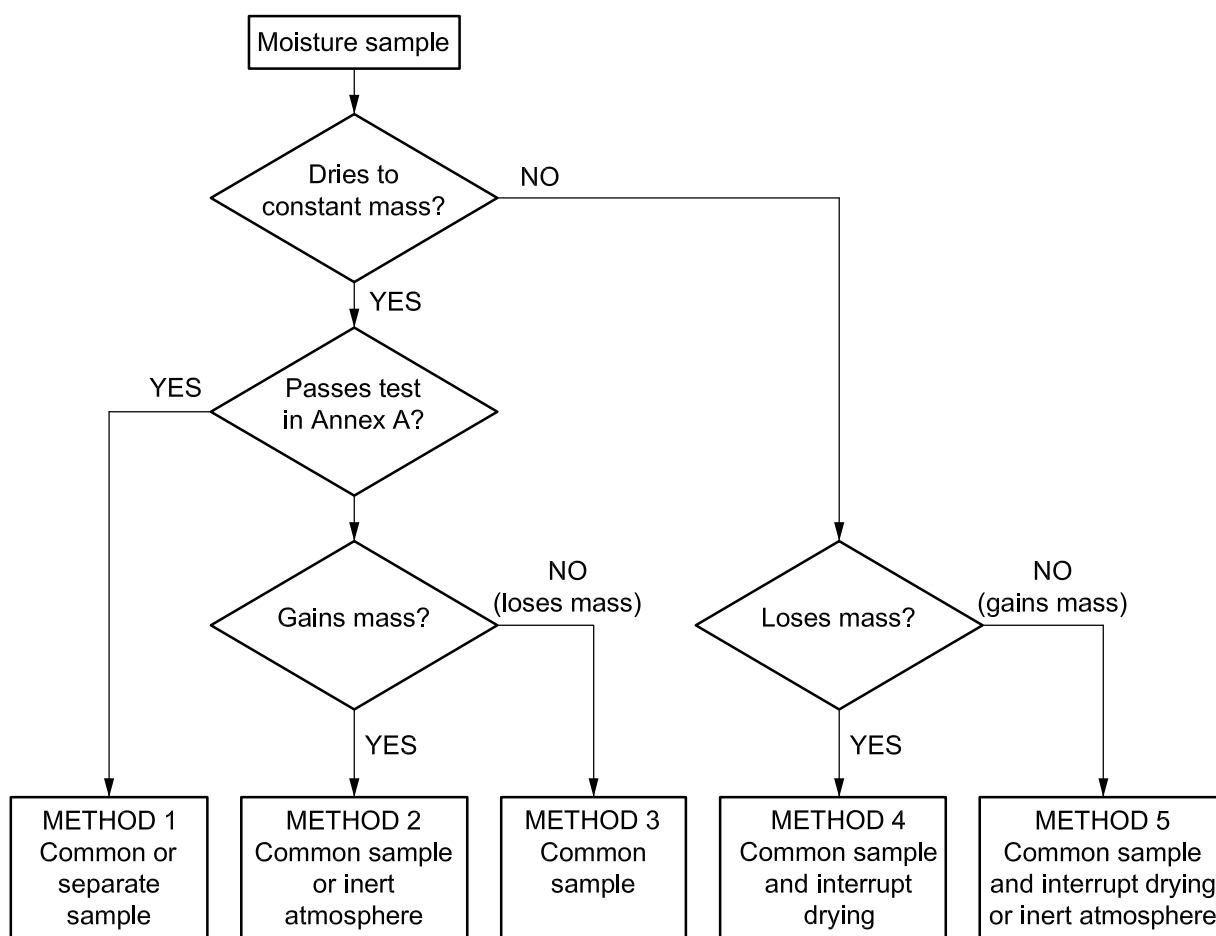


Figure 1 — Procedure for selection of appropriate drying method

#### 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  $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ .

**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 of balance 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

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

### 6 Processing of samples

Moisture samples 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 Moisture samples

#### 7.1 General

Moisture samples 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 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	$\geq 4$
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  $105\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$  for a predetermined time.

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

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  $105\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$  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 to 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.

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 4.5 or 4.6 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 Equation (1):

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

where

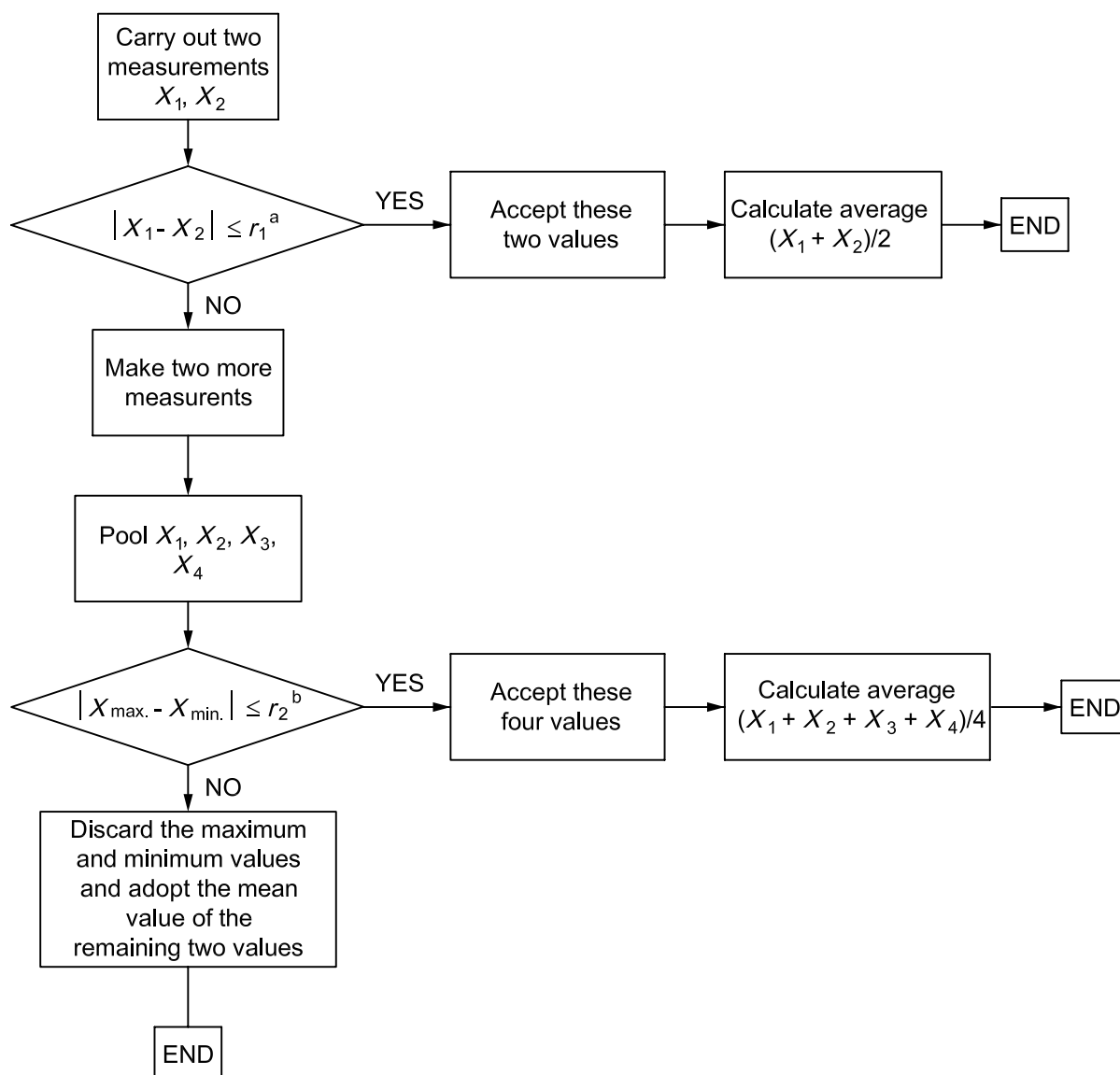
- $X$  is the moisture content as a percentage of the test portion;
- $m_1$  is the mass of the drying tray, in grams;
- $m_2$  is the mass of the drying tray plus wet test portion, in grams;
- $m_3$  is the mass of the drying tray plus dried test portion, in grams.

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

### 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.



<sup>a</sup>  $r_1 = 0,14$  % Pb, Zn concentrates  
Moisture < 6 %

$r_1 = 0,20$  % All other cases

<sup>b</sup>  $r_2 = 0,18$  % 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**

### 9.2.2 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 Equation (2):

$$w_L = \frac{\sum_{i=1}^k w_i m_i}{\sum_{i=1}^k m_i} \quad (2)$$

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 Equation (3):

$$w_L = \frac{\sum_{i=1}^n w_i m_{s,i}}{\sum_{i=1}^n m_{s,i}} \quad (3)$$

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:

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

## 10 Test report

The test report shall contain the following information:

- a) identification of the sample;
- b) a 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) the number of moisture determinations;
- e) the moisture content of the lot, expressed as a percentage by mass;
- f) dates of sampling and completion of tests.

## Annex A (normative)

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

#### A.1 Introduction

Some concentrate samples are susceptible to oxidation or decomposition on drying in air at  $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  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.

a) The concentrate is susceptible to oxidation if:

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

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 or sublimation if:

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



## Annex B (normative)

### Determination of moisture content of cohesive or wet concentrates

#### B.1 Introduction

Where it is difficult to conduct division, owing to the moisture sample being adhesive or excessively wet, the moisture sample may be dried in air until preparation can be conducted without any trouble. In this case, the moisture content of the lot shall be determined using the predrying method specified below.

#### B.2 Procedure

- a) Determine the initial mass of the moisture sample.
- b) Spread the moisture sample to a uniform thickness and dry it by air-drying or in a drying apparatus at a temperature no higher than 105 °C. The sample shall only be dried as far as is necessary for a representative test portion to be taken for completion of the moisture determination.
- c) After predrying, determine the mass of the moisture sample again.
- d) Calculate the predried moisture content, expressed as a percentage by mass, using Equation (B.1).

$$w_p = \frac{100 (m'_i - m'_p)}{m'_i} \quad (\text{B.1})$$

where

$w_p$  is the predried moisture content as a percentage of the moisture sample;

$m'_i$  is the initial mass of the moisture sample, in grams;

$m'_p$  is the mass of the moisture sample after predrying, in grams.

- e) Prepare the test portions for moisture measurement from the predried moisture sample without delay according to ISO 12743.
- f) Determine the drying loss of the test portion according to Clause 8 and calculate the additional moisture content, expressed as a percentage by mass, according to 9.1.
- g) Calculate the total (as received) moisture content, expressed as a percentage by mass, using Equation (B.2):

$$w_T = w_p + \frac{(100 - w_p)w}{100} \quad (\text{B.2})$$

where

$w_p$  is the predried moisture content as a percentage of the moisture sample;

$w_T$  is the total moisture content as a percentage of the moisture sample;

$w$  is the additional moisture content as a percentage obtained according to 9.1 after predrying.

- h) Determine the moisture content of the lot, expressed as a percentage by mass, according to 9.2.

## Annex C (informative)

### Example of a work sheet for the determination of moisture content of each test portion in accordance with this International Standard

Type and grade of concentrate:			
Lot identification:			
Sub-lot No:	Sample No:		
	Date:		
	<b>Notation</b>	<b>Result</b>	
Total mass before drying (g)	(1)	2 756,6	
Mass of drying tray (g)	(2)	912,4	
Initial mass of test portion (g)	(3) = (1) – (2)	1 844,2	
Value of 0,05 % of initial mass of sample (g)	(4) = (3) ÷ 2 000	0,9	
Total mass after 16 h drying (g)	(5)	2 618,4	
Total mass after another 4 h drying (g)	(6)	2 616,0	
Additional drying loss (g)	(5) – (6)	2,4	
Total mass after another 4 h drying (g)	(7)	2 615,8	
Additional drying loss (g)	(6) – (7)	0,2	
Final drying loss (g) <sup>a</sup>	(8) = (1) – (7)	140,8	
Moisture content $X$ (%)	(9) = [(8) ÷ (3)] × 100	7,63	
Remarks:			
Analyst:			
<sup>a</sup> Terminate drying when the additional drying loss becomes equal to (4) or less.			

## Bibliography

- [1] ISO 9599, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*

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