INTERNATIONAL STANDARD

ISO 9599

First edition
1991-02-15

Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method

Concentrés sulfurés de cuivre, de plomb et de zinc — Détermination de l'humidité hygroscopique dans l'échantillon pour analyse — Méthode gravimétrique

INTERNATIONAL ISO

Reference number
ISO 9599:1991(E)

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 9599 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annex A forms an integral part of this International Standard. Annex B is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method

1 Scope

This International Standard specifies a gravimetric loss-in-mass method for the determination of the hygroscopic moisture content in analysis samples of copper, lead and zinc sulfide concentrates.

The method is applicable to copper, lead and zinc sulfide concentrates free from volatile organic flotation reagents, for example kerosene, and with hygroscopic moisture contents between 0,05 % (m/m) and 2 % (m/m). It is used to correct the analysis results from the equilibrated moisture level to the dry basis.

NOTE 1 The result of the determination of hygroscopic moisture using this International Standard should not be reported as part of the analysis of a concentrate sample. When the bulk moisture content of a commercial shipment of concentrate is required, the method specified [1] should be used. The determination of hygroscopic moisture and the determination of bulk moisture content are connected with each other. In both determinations the same state of dryness has to be achieved, in order to ascertain the correct metal content of a lot.

This method is not applicable to sulfide concentrates which are susceptible to oxidation (see 6.3, note 2). Annex A sets out a modified procedure which can be used in this case.

2 Principle

Drying of a weighed test portion in air in an oven maintained at 105 °C ± 5 °C and calculation of the percentage moisture content from the loss in mass.

3 Reagents

3.1 Desiccant, such as self-indicating silica gel or anhydrous magnesium perchlorate.

WARNING — Exhausted magnesium perchlorate must be disposed of by carefully washing it down the sink with a stream of water.

4 Apparatus

Ordinary laboratory equipment, and

4.1 Analytical balance, sensitive to 0,1 mg.

4.2 Laboratory oven, capable of maintaining a temperature of 105 °C ± 5 °C.

4.3 Weighing vessels, shallow, of glass or silica or corrosion-resistant metal, with externally fitting air-tight covers of approximately 50 mm diameter.

4.4 Flat dish, or **tray**.

5 Sampling and samples

5.1 Laboratory sample

Use a sample of minus 150 µm particle size.

5.2 Preparation of the test sample

Take a sufficient mass of the laboratory sample for the required chemical analysis and moisture determination and transfer to a flat dish or tray (4.4). Spread the sample evenly in a thin layer about 3 mm to 5 mm thick. Cover the dish to protect the sample from dust, allowing a free flow of air across the top of the sample. Allow the test sample to equilibrate with the laboratory atmosphere for 2 h or for long enough to achieve equilibration.

Equilibration is achieved when the change in mass of the test sample over a 2 h period of exposure is less than 0,1 %.

6 Procedure

6.1 Preparation of the weighing vessel

Dry a weighing vessel and cover (4.3) by heating in the laboratory oven (4.2) at 105 °C ± 5 °C for 1 h. Transfer the weighing vessel and cover to a desiccator containing a suitable fresh desiccant (3.1), and allow to cool to ambient temperature. Remove the weighing vessel and cover from the desiccator and weigh to the nearest 0,1 mg (mass m_1) after slightly lifting the cover and quickly replacing it.

6.2 Test portion

Transfer approximately 10 g of the equilibrated test sample (5.2) directly to the dried and tared weighing vessel (6.1), spreading in an even layer about 3 mm to 5 mm thick. Record the mass of the weighing vessel and cover plus test portion to the nearest 0,1 mg (mass m_2). Within the following 5 min, also weigh the test portions required for the determination of constituents for which correction of the analytical values to a dry basis is required and transfer such test portions to the weighing vessels (4.3).

6.3 Determination

Transfer the uncovered weighing vessel containing the test portion and the vessel cover to the laboratory oven (4.2) and dry at 105 °C ± 5 °C for 2 h. After the 2 h period, remove the weighing vessel containing the dry test portion from the oven, replace the cover and allow to cool to ambient temperature in the desiccator. When cool, remove the weighing vessel containing the dry test portion and the vessel cover from the desiccator and reweigh to the nearest 0,1 mg, after slightly lifting the cover and quickly replacing it.

Repeat the drying at 105 °C ± 5 °C for another 2 h, cool to ambient temperature in the desiccator and weigh to determine if constant mass (± 1 mg) has been achieved. If constant mass has not been achieved, repeat the drying and weighing steps described above. Record the constant mass (m_3).

NOTE 2 If constant mass (± 1 mg) is not achieved after three drying periods of 2 h, then the method specified in annex A should be used.

7 Expression of results

The hygroscopic moisture content, H, expressed as a percentage by mass, is calculated from the formula

$$H = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the dried weighing vessel plus cover;

m_2 is the mass, in grams, of the weighing vessel plus cover plus test portion before drying;

m_3 is the mass, in grams, of the weighing vessel plus cover plus test portion after drying.

Calculate the hygroscopic moisture content of the sample to the second decimal place.

8 Test report (for internal laboratory use only)

The test report shall contain the following information:

a) identification of the sample;

b) reference to this International Standard;

c) hygroscopic moisture content of the sample;

d) date on which the test was carried out.

Annex A
(normative)

Method for samples susceptible to oxidation — Drying in nitrogen

A.1 Principle

Drying of a weighed test portion in oxygen-free dry nitrogen in an oven maintained at 105 °C ± 5 °C and calculation of the percentage moisture content from the loss in mass.

A.2 Reagents

A.2.1 Nitrogen, dry gas containing less than 30 µl of oxygen per litre.

A.2.2 Desiccant, such as self-indicating silica gel or anhydrous magnesium perchlorate.

WARNING — Exhausted magnesium perchlorate must be disposed of by carefully washing it down the sink with a stream of water.

A.3 Apparatus

Ordinary laboratory equipment, and

A.3.1 Analytical balance, sensitive to 0,1 mg.

A.3.2 Minimum-free-space oven, capable of maintaining a temperature of 105 °C ± 5 °C, with provision for pre-heated nitrogen (A.2.1) to pass through it at 15 to 20 oven-volumes per hour. A suitable oven is shown in figure A.1.

A.3.3 Weighing vessel, shallow, of glass or silica or corrosion-resistant metal, with an externally fitting air-tight cover of approximately 50 mm diameter.

A.3.4 Flowmeter, capable of measuring the rate of flow of nitrogen (A.2.1) through the oven (A.3.2).

A.3.5 Drying tower, of approximately 250 ml capacity, packed with anhydrous magnesium perchlorate (A.2.2) for drying the nitrogen (A.2.1).

A.4 Procedure

A.4.1 Preparation of the weighing vessel

Raise the temperature of the oven (A.3.2) to 105 °C ± 5 °C while passing nitrogen (A.2.1) through it at the rate of 15 to 20 oven-volumes per hour.

Dry a weighing vessel and cover (A.3.3) by heating in the oven at 105 °C ± 5 °C for 1 h. Transfer the weighing vessel and cover to a desiccator containing a suitable fresh desiccant (A.2.2), and allow to cool to ambient temperature. Remove the weighing vessel and cover from the desiccator and weigh to the nearest 0,1 mg (mass m_1), after slightly lifting the cover and quickly replacing it.

A.4.2 Test portion

Transfer approximately 10 g of the equilibrated test sample (5.2) directly to the dried and tared weighing vessel (A.4.1), spreading it in an even layer about 3 mm to 5 mm thick. Record the mass of the weighing vessel and cover plus test portion to the nearest 0,1 mg (mass m_2).

A.4.3 Determination

Transfer the uncovered weighing vessel containing the test portion and the vessel cover to the oven (A.3.2) and dry at 105 °C ± 5 °C until constant mass (± 1 mg) is achieved (see note 3).

Remove the weighing vessel containing the dry test portion from the oven, replace the cover and allow to cool to ambient temperature in the desiccator. When cool, remove the weighing vessel containing the test portion and the vessel cover from the desiccator and reweigh to the nearest 0,1 mg (mass m_3), after slightly lifting the cover and quickly replacing it.

NOTE 3 Under the conditions described above, drying should be complete in between 1,5 h and 3 h. Constancy in mass (± 1 mg) should be established by reheating at 105 °C ± 5 °C for a further period of 30 min, followed by cooling and weighing as described.

A.5 Expression of results

The hygroscopic moisture content, H, expressed as a percentage by mass, is calculated from the formula

$$H = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the dried weighing vessel plus cover;

m_2 is the mass, in grams, of the weighing vessel plus cover plus test portion before drying;

m_3 is the mass, in grams, of the weighing vessel plus cover plus test portion after drying.

Calculate the hygroscopic moisture content of the sample to the second decimal place.

A.6 Test report (for internal laboratory use only)

The test report shall contain the following information:

a) identification of the sample;

b) reference to this International Standard;

c) hygroscopic moisture content of the sample;

d) date on which the test was carried out.

Dimensions in millimetres

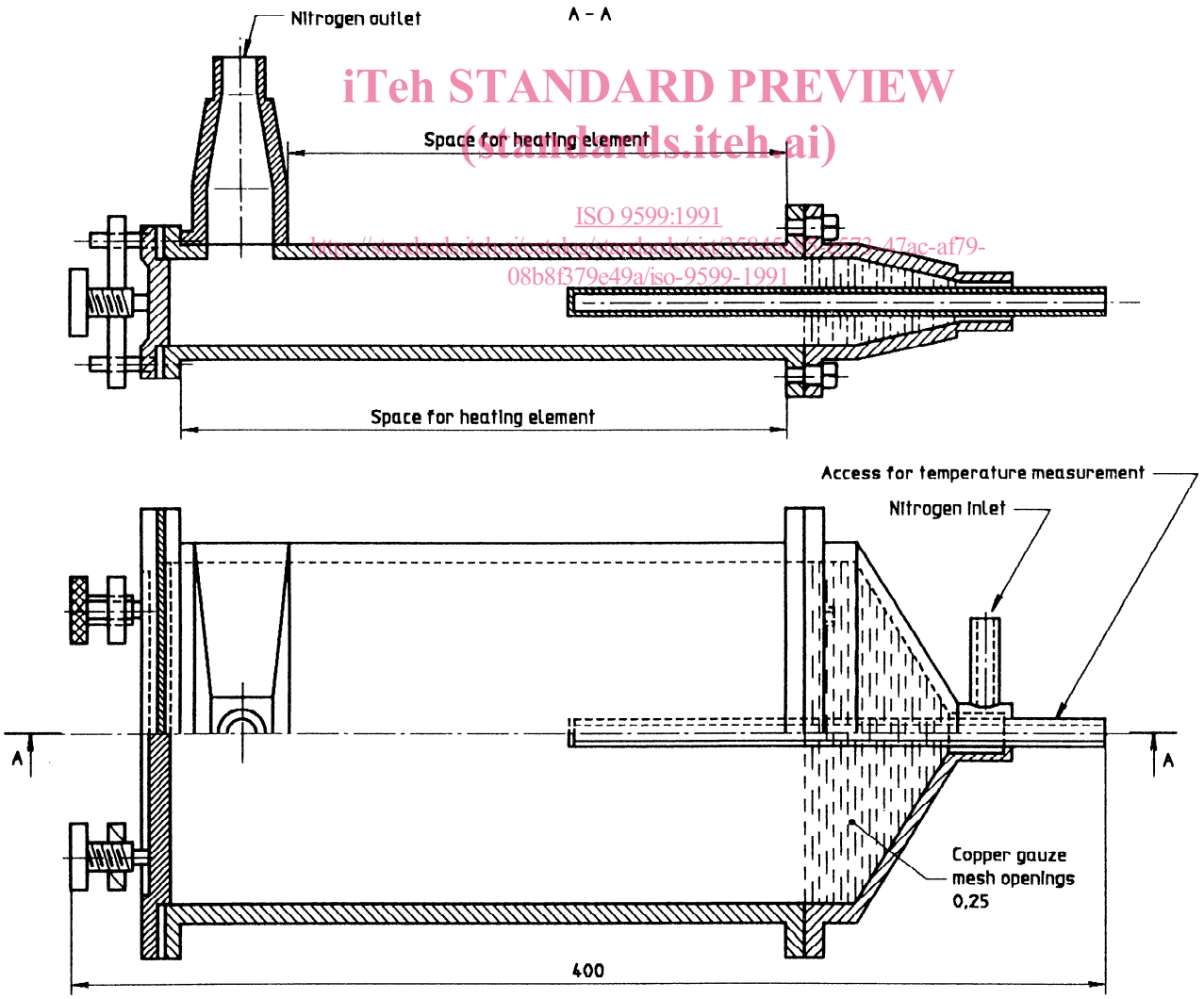

Figure A.1 — Suitable nitrogen oven

Annex B
(informative)

Bibliography

[1] ISO 10251:—[1], *Copper, lead and zinc sulfide concentrates — Determination of moisture in the bulk material.*

1) To be published.

UDC 549.321 + .328 + .332:543.71

Descriptors: copper ores, lead ores, zinc ores, concentrates, copper inorganic compounds, lead inorganic compounds, zinc inorganic compounds, sulphides, tests, hygroscopic tests, determination, humidity, gravimetric analysis.

Price based on 5 pages