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**Iron ores — Determination of  
hygroscopic moisture in analytical  
samples — Gravimetric, Karl Fischer and  
mass-loss methods**

*Minerais de fer — Détermination de l'humidité hygroscopique dans les  
échantillons pour analyse — Méthodes gravimétrique, selon Karl  
Fischer et par perte de masse*

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## Foreword

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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 2596 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 2596:1994), which has been technically revised.

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## Introduction

In the analysis of iron ores, the reporting limit of the analytical value of each constituent on a dry sample basis can be achieved by using predried samples. However, with certain ore types, where the constituent being determined is above a certain concentration level as specified in the scope, this technique can produce erroneous results. In these cases, for the calculation of analytical values of the other constituents in the ore to a dry sample basis, a direct determination of the hygroscopic moisture content becomes necessary.

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# Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods

## 1 Scope

This International Standard specifies the following four test methods for the determination of the hygroscopic moisture content of test samples:

Method 1 — Gravimetric method;

Method 2 — Karl Fischer volumetric method;

Method 3 — Karl Fischer coulometric method;

Method 4 — Mass-loss method.

Any of these methods is applicable wherever the analytical value of a chemical constituent is to be calculated to a dried sample basis in the following ore types.

- a) Processed ores containing metallic iron (direct reduced iron).
- b) Natural or processed ores in which the sulfur content is greater than 0,2 % (mass fraction).
- c) Natural or processed ores in which the combined water is greater than 2,5 % (mass fraction).

Any of these methods is applicable to a concentration range of 0,05 % (mass fraction) to 4,5 % (mass fraction) hygroscopic moisture.

NOTE 1 Where the reportable moisture content of a commercial consignment of ore is required, the procedure in ISO 3087 is used.

NOTE 2 With natural or processed ores outside the field of application specified in a), b) or c), a determination of a constituent at any level of concentration may be conducted using any of these methods, or as specified in ISO 7764.

NOTE 3 Alternatively, the loss on ignition content may be measured as an estimate of the combined water content.

## 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 760, *Determination of water — Karl Fischer method (General method)*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

### 3 Method 1 — Gravimetric method

#### 3.1 Principle

The hygroscopic moisture content of an environmentally equilibrated test portion is determined at  $105\text{ °C} \pm 2\text{ °C}$  within a drying tube, into which dry nitrogen is passed (at a rate of 100 ml/min to 200 ml/min). Evolved moisture (swept by gas) is collected in an absorption tube containing anhydrous magnesium perchlorate, and the percentage moisture content is determined via the corrected mass increase of the tube.

#### 3.2 Reagents

##### 3.2.1 Desiccant

Anhydrous magnesium perchlorate  $\text{Mg}(\text{ClO}_4)_2$  of size 0,80 mm to 1,25 mm, to ensure carrier-gas water-vapour pressures below  $5\text{ }\mu\text{g H}_2\text{O/l}$ .

As measurement accuracy and precision are highly dependent on blank determinations, the stability of residual background moisture shall be controlled as a function of combining capacity.

The combining capacity ( $C_c$ ) is calculated as a percentage of desiccant and residual carrier-gas moisture content using the following equation:

$$C_c = \frac{(m_1)L \cdot N}{m_2} \tag{1}$$

where

$m_1$  is the mass of residual carrier-gas moisture content, in centigrams per litre, where

$m_1 = 0,000\ 1\text{ cg/l}$  for aluminium calcium silicate molecular sieves,  
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$0,000\ 2\text{ cg/l}$  for silica gel desiccant;

$L$  is the number of litres of gas per bottle;

$N$  is the number of gas bottles consumed;

$m_2$  is the mass, in grams, of desiccant added to drying tower.

For successful drying, the combining capacity should be restricted to 10 % (mass fraction).

**WARNING** Magnesium perchlorate is a powerful oxidant and cannot be allowed to be exposed to organic materials. When exhausted, it should not be discarded into waste bins, but should be washed down the sink.

##### 3.2.2 Aluminium calcium silicate molecular sieves, made of 1/16 in pellets.

Before use, sieves shall be dried by heating to  $400\text{ °C}$  for 4 h.

NOTE This limit is based on extending the service life of secondary desiccants (magnesium perchlorate), by limiting input stream contaminants.

##### 3.2.3 Silica gel desiccant, blue self-indicating.

Before use, gel shall be dried by heating to  $105\text{ °C}$  for 4 h.



**3.2.4 Copper(II) sulfate pentahydrate analytical reagent grade (AR)** ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), free-flowing crystalline material, press-crushed if necessary under a pestle by hand, without grinding, to a size of approximately 1 mm.

**3.2.5 Nitrogen**, filtered, predried, oil-free, containing less than 10  $\mu\text{l}$  of oxygen per litre at a pressure of approximately 35 kPa to 50 kPa above atmospheric pressure.

### 3.3 Apparatus

NOTE A suitable apparatus for the determination is shown diagrammatically in Annex A.

**3.3.1 Balance**, capable of reading the mass of the absorption vessel to 0,1 mg.

**3.3.2 Oven**, preferably of the aluminium metal-block type, capable of accommodating one, but preferably several, glass drying tubes (3.3.3) and of maintaining a temperature within the range  $105\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$  over a minimum tube length of 160 mm.

**3.3.3 Borosilicate glass drying tubes and connections**, fitted with Viton "O"-ring-seal pushrod cap assemblies.

NOTE A suitable drying tube is shown diagrammatically in Annex B.

**3.3.4 Drying towers**, of capacity 250 ml, one filled with molecular sieves (3.2.2) or silica gel (3.2.3) and the other packed with magnesium perchlorate desiccant (3.2.1), to dry the stream of nitrogen (3.2.5) entering the drying tubes.

Molecular sieves (3.2.2) and silica gel (3.2.3) drying towers shall be repacked with freshly dried desiccants fortnightly.

**3.3.5 Flowmeters**, capable of measuring a flow rate within the range 100  $\text{cm}^3/\text{min}$  to 200  $\text{cm}^3/\text{min}$ .

If a pressure drop over a constriction is used as a means of measuring flow rate, the manometer liquid shall be a non-volatile oil.

**3.3.6 Absorption tubes**, manufactured of chemically inert conducting material to minimize static charging effects (titanium is preferred), with pan-balance location pads to minimize corner-load weighing errors.

NOTE A suitable tube is shown in Annex C.

Tubes shall be of suitable design (8 mm ID  $\times$  300 mm) to contain sufficient desiccant (3.2.1) to remove the moisture completely from the stream of nitrogen (3.2.5).

The tubes should have sealable inlet and outlet connections and the direction of gas flow should be unambiguously identified. Desiccants shall be firmly packed to prevent "channelling" and be retained in position with glass-wool plugs.

**3.3.7 Guard tubes**, of a suitable design, containing magnesium perchlorate desiccant (3.2.1) to prevent back diffusion of moisture into absorption tubes.

**3.3.8 Sample boats**, of an inert and stable material, such as glass, stainless steel or glazed porcelain.

Approximate dimensions are 100 mm  $\times$  20 mm  $\times$  10 mm. Before use, boats should be dried at approximately  $105\text{ }^\circ\text{C}$ , and then cooled to ambient temperature in a desiccator. Boats shall be stored in a desiccator prior to use.

**3.3.9 Filter discs, of sintered metal, sintered glass or similar**, inserted in the flexible connections between the drying and absorption tubes.

### 3.3.10 Flexible connections

The selection of polymeric tubing shall be made by taking into consideration that some materials are permeable to moisture. Annealed copper/stainless steel tubing is preferable. Swagelock-type connectors and quick-release neoprene "O"-ring connector joints are recommended. On serviceable components that necessitate removal, quick-release neoprene "O"-ring connector joints shall be used. Glass ends should be sufficiently smoothed to minimize coupling-seal damage.

3.3.11 **Flow-control needle valve**, placed on the inlet of each flowmeter.

## 3.4 Sampling and samples

### 3.4.1 Laboratory sample

For analysis, use a laboratory sample of particle size less than 100 µm or less than 160 µm, which has been taken and prepared in accordance with ISO 3082.

### 3.4.2 Preparation of test sample

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container.

The test sample is brought into equilibrium with the laboratory atmosphere by exposure for at least 2 h on an inert tray at a layer density not greater than 0,1 g/cm<sup>2</sup>. The sample shall be thoroughly mixed immediately before the determination.

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## 3.5 Procedure

### 3.5.1 Apparatus conditioning

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#### 3.5.1.1 Conditioning of drying tube

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Bring the temperature of the drying tubes (3.3.3) to 105 °C ± 2 °C and maintain this temperature throughout steps 3.5.1 to 3.5.5.

Adjust the rate of flow of nitrogen (3.2.5) to provide a constant flow rate of 100 cm<sup>3</sup>/min to 200 cm<sup>3</sup>/min through the drying tube, and maintain this flow rate throughout steps 3.5.1.2 to 3.5.5.

Connect the outlet from each drying tube to the inlet of a guard tube. Open the taps of the guard tube, and pass nitrogen through the tube for a minimum of 15 min.

NOTE A preferred method for conditioning the drying tubes requires maintaining a constant gas flow rate of 50 cm<sup>3</sup>/min to 100 cm<sup>3</sup>/min (and a temperature of 105 °C ± 2 °C) during periods in which the instrument is idle.

#### 3.5.1.2 Conditioning of absorption tube

Adjust the rate of flow of nitrogen (3.2.5) to provide a constant flow rate of 100 cm<sup>3</sup>/min to 200 cm<sup>3</sup>/min. Connect a closed absorption tube (3.3.6) and guard tube (3.3.7) to an empty drying tube. Open the taps of the guard tube first, followed by the outlet tap of the absorption tube, and lastly the inlet valve. Pass nitrogen through the assembled apparatus for a minimum of 15 min.

NOTE Conditioning of the absorption tube is only necessary following the addition of a fresh charge of desiccant.

### 3.5.1.3 Weighing of absorption tube

Close the taps of the absorption tube, outlet first, and remove from the drying tube. Reconnect the opened guard tube to the drying tube. Wipe the absorption tube with a clean, dry cloth, free from loose fibres, and allow to stand for 20 min in the balance room.

To minimize the transfer of contaminants such as moisture and grease, conditioned absorption tubes and sample boats shall be handled using cotton gloves.

Open the taps of the absorption tube momentarily to equalize the pressure, and then weigh to the nearest 0,1 mg.

### 3.5.2 System checks

Reconnect the weighed absorption tube and guard tube, opening the taps on the absorption tube outlet first, and restore the nitrogen flow to the previous rate. After 2 h, follow the procedure exactly as described in 3.5.1.2.

If the absorbent efficiency of drying-tower magnesium perchlorate (3.3.4) and absorption-tube (3.3.6) desiccants are equal, no increase in absorption tube mass will occur during a system check. If an increase in absorbent tube mass of greater than 0,1 mg is observed, drying-tower desiccant quality, system leaks, and absorbent tube weighings should be checked as possible error sources.

### 3.5.3 Blank test

Reconnect the weighed absorption tube and guard tube, open the taps in accordance with 3.5.1.2, and restore the nitrogen flow to the previous rate.

Quickly remove the drying-tube end cap (3.3.3), and place an empty sample boat (3.3.8) in the entrance of the heat zone. Fit the end cap (and/or inlet connector), and using a pushrod (manual or magnetic), immediately move the boat to the centre of the oven and note the introduction time.

During the placement of the boat in the drying tube, as specified in 3.5.2 to 3.5.4, precautions should be taken, essentially involving a careful technique, to minimize entry of laboratory air (containing moisture) into the drying tube.

To minimize the transfer of contaminants such as moisture and grease, conditioned absorption tubes and sample boats should be handled using cotton gloves.

After 2 h, follow the procedure exactly as described in 3.5.1.3, recording the mass to the nearest 0,1 mg.

The increase in mass of the absorption tube for the blank test should be as low as possible and not more than 2 mg. Repeat the determination of the blank test after the analysis of the test sample, to ensure that the blank test value is essentially constant.

### 3.5.4 Check test

NOTE 1 The check test is required when first commissioning the complete apparatus and at other appropriate times, e.g. when changes in the equipment or operators have been made, and when a regular interval check on the condition of the absorption tubes is required.

When a satisfactory value for the blank test has been obtained, weigh 0,05 g to 0,2 g of copper(II) sulfate pentahydrate (3.2.4) to the nearest 0,2 mg, into the cooled sample boat used for the blank test. The mass taken should be such that its moisture content approximates the anticipated moisture content of the ore type being analysed.

Repeat the procedure in 3.5.2, using the boat containing the weighed copper(II) sulfate. The increase in mass of the absorption tube, corrected with the blank test value, should indicate a value of the water content of

copper(II) sulfate within the range of 28,5 % (mass fraction) to 29,2 % (mass fraction). If not, the cause should be determined.

NOTE 2 Alternatively, a calibrated micro-syringe (accuracy and reproducibility  $\pm 1$  %) may be used to introduce water directly into the heated zone of the drying tube through a septum.

**3.5.5 Determination**

When a satisfactory value for the blank test has been obtained (and similarly for the check test, if appropriate), weigh from the air-equilibrated sample (3.4.2), the required test portion for the determination of the constituent to be reported on a dry basis. Immediately weigh to the nearest 0,1 mg, in accordance with Table 1, the test portion for the determination of hygroscopic moisture.

**Table 1 — Mass of test portion — Method 1 (Gravimetric method)**

Hygroscopic moisture content % (mass fraction)	Mass of test portion g
0,05 to 2	2,0
2 to 4,5	1,0

Transfer the weighed sample portion into a conditioned sample boat (3.3.8), and distribute the material evenly. Immediately, repeat the procedure in 3.5.3, using the boat containing the test portion instead of an empty boat.

Sample loading should not exceed 0,5 g/cm<sup>2</sup>, for samples between 0,05 and 2,0 % moisture and 0,15 g/cm<sup>2</sup> for samples containing between 2,0 % and 4,5 % moisture.

NOTE Alternatively, the analytical sample can be weighed directly into a conditioned sample boat (3.3.8).

The weighing of analytical test samples shall be performed in parallel with hygroscopic moisture sampling and preparation operations; otherwise erroneous moisture corrections will result. The determination of hygroscopic moisture shall be performed whenever a constituent is reported to a dry basis.

The hygroscopic moisture values shall not be averaged, but shall be used individually to correct the corresponding constituent values.

**3.6 Expression of results**

**3.6.1 Calculation of hygroscopic moisture content**

The content of hygroscopic moisture (HM), on an air-dry basis, is calculated as a percentage by mass using the following equation:

$$HM = \frac{m_3 - m_4}{m_5} \times 100 \tag{2}$$

where

- $m_3$  is the increase in mass, in grams, of the absorption tube during the test;
- $m_4$  is the increase in mass, in grams, of the absorption tube during the blank test;
- $m_5$  is the mass, in grams, of the test portion.

As the hygroscopic moisture content of a test sample is specific to ambient measurement conditions, the result should be used for internal purposes only.

### 3.6.2 Hygroscopic moisture correction of analytical test portion mass

The sample mass for an analytical test sample shall be moisture corrected (as a percentage of the hygroscopic mass) using the following equation:

$$\text{MCM} = m_6 - \left( m_6 \times \frac{\text{HM}}{100} \right) \quad (3)$$

where

$m_6$  is the mass, in grams, of analytical test portion to be moisture corrected;

HM is the hygroscopic moisture content of the test portion, expressed as a percentage by mass;

MCM is the dried mass, in grams, of an analytical test portion.

## 4 Method 2 — Karl Fischer volumetric method

### 4.1 Principle

The hygroscopic moisture content of an environmentally equilibrated test portion is determined at  $105 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  in a drying tube, into which dry nitrogen is passed at a rate of 100 ml/min to 200 ml/min for 2 h. Evolved moisture (swept by gas) is collected in an aqueous absorbent solution where, upon completion of analysis, this solution is titrated (either automatically or under operator control) volumetrically.

### 4.2 Reagents

**4.2.1 Desiccant**, anhydrous magnesium perchlorate  $\text{Mg}(\text{ClO}_4)_2$  of size 0,80 mm to 1,25 mm, to ensure carrier-gas water-vapour pressures below  $5 \text{ } \mu\text{g H}_2\text{O/l}$ .

As measurement accuracy and precision are highly dependent on blank determinations, the stability of residual background moisture shall be controlled as a function of combining capacity.

The combining capacity ( $C_c$ ) is calculated as a percentage of desiccant and carrier-gas impurity masses using the following equation:

$$C_c = \frac{(m_1)L \cdot N}{m_2} \quad (4)$$

where

$m_1$  is the mass of residual carrier-gas moisture content, in centigrams per litre, where

$m_1 = 0,000 1 \text{ cg/l}$  for aluminium calcium silicate molecular sieves,

$0,000 2 \text{ cg/l}$  for silica gel desiccant;

$L$  is the number of litres of gas per bottle;

$N$  is the number of gas bottles consumed;

$m_2$  is the mass, in grams, of desiccant added to the drying tower.

For successful drying, the combining capacity should be limited to maximum 10 % (mass fraction).

**WARNING** Magnesium perchlorate is a powerful oxidant and cannot be allowed to be exposed to organic materials. When exhausted, it should not be discarded into waste bins, but should be washed down the sink.