SLOVENSKI STANDARD

SIST ISO 2596:1998

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Ģelezove rude ï Doloļevanje higroskopske vlage v vzorcih za analizo ï Gravimetrijska in Karl Fischerjeva metoda

Iron ores -- Determination of hygroscopic moisture in analytical samples -- Gravimetric and Karl Fischer methods

Minerais de fer -- Détermination de l'humidité hygroscopique dans les échantillons pour analyse -- Méthodes gravimétrique et selon Karl Fischer

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INTERNATIONAL STANDARD

ISO 2596

Fourth edition
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Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric and Karl Fischer methods

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

Reference number
ISO 2596:1994(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 2596 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 2, *Chemical analysis*.

This fourth edition cancels and replaces the third edition (ISO 2596:1984), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

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Introduction

In the analysis of iron ores, the reporting of the analytical value of each constituent on a dry sample basis can, in most cases, be achieved by using a predried sample. However, with certain types of ores, where the constituent being determined is above a certain concentration level, as specified in clause 1 of this International Standard, 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 and Karl Fischer methods

1 Scope

This International Standard specifies the following two methods for the determination of 0,05 % *(m/m)* to 6 % *(m/m)* of hygroscopic moisture content in test samples of natural or processed iron ores:

— Method 1 — Gravimetric method;

— Method 2 — Karl Fischer method.

Either method 1 or method 2 is used where the analytical value of the constituent to be calculated to a dry sample basis is higher than 10 % *(m/m)* in the following types of ores:

a) processed ores containing metallic iron (direct reduced iron);

b) natural or processed ores in which the sulfur content is higher than 0,2 % *(m/m)*;

c) natural or processed ores in which the content of combined water is higher than 2,5 % *(m/m)*.

The result from the determination of hygroscopic moisture using this International Standard is not reported as part of the analysis of an ore sample.

NOTES

1 Where the reportable hygroscopic moisture content of a commercial consignment of ores is required, the procedure in ISO 3087:1987, *Iron ores — Determination of moisture content of a consignment*, is used.

2 With natural or processed ores outside the field of application specified in a) or b) or c), a determination of a constituent at any level of concentration can be conducted using a predried test sample prepared as specified in ISO 7764:1985, *Iron ores — Preparation of predried test samples for 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 385-2:1984, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 760:1978, *Determination of water — Karl Fischer method (General method).*

ISO 3081:1986, *Iron ores — Increment sampling — Manual method.*

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Method 1 — Gravimetric method

3.1 Principle

Equilibration of the test sample with the laboratory atmosphere. Heating of a test portion at 105 °C ± 2 °C in a heated tube in a stream of dry nitrogen, and collection of the evolved moisture in an absorption tube containing a desiccant. Measurement of the corrected increase in mass of the absorption tube.

3.2 Reagents

3.2.1 Desiccant, anhydrous magnesium perchlorate [$Mg(ClO_4)_2$] of size 0,8 mm to 1,25 mm, or other suitable desiccant of equivalent drying efficiency.

It is essential that the same desiccant be used in both the drying tower and the absorption tubes, since the incoming nitrogen and the gas leaving the system have to be dried to exactly the same degree. The freshness of the desiccant in both the drying tower and the absorption tubes is important, and reliance should not be placed on self-indicating desiccants.

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

3.2.2 Silica gel.

3.2.3 Copper(II) sulfate pentahydrate ($CuSO_4.5H_2O$), free-flowing crystalline material, press-crushed if necessary under a pestle by hand, without grinding, to a size of approximately 1 mm.

3.2.4 Nitrogen, filtered, predried, oil-free, containing less than 10 µl of oxygen per litre at a pressure of approximately 35 kPa above atmospheric pressure.

3.3 Apparatus

A suitable apparatus for the determination is shown diagrammatically in figure 1.

3.3.1 Balance, capable of reading the mass load 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 °C ± 2 °C over a minimum tube length of 160 mm.

3.3.3 Glass drying tubes and connections, as shown diagrammatically in figure 2.

3.3.4 Drying towers, of capacity 250 ml, one filled with silica gel (3.2.2) and the other packed with desiccant (3.2.1), to dry the stream of nitrogen (3.2.4) entering the drying tubes.

3.3.5 Flowmeters, capable of measuring a flow rate within the range 100 cm^3/min to 200 cm^3/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, of a suitable design and able to contain sufficient desiccant (3.2.1) to remove the moisture completely from the stream of nitrogen (3.2.4).

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

3.3.7 Sample boats, of an inert and stable material such as glass, stainless steel or porcelain. Approximate dimensions are 100 mm × 20 mm × 10 mm, and the sample loading shall not exceed 1,5 mg/mm^2. Before use, boats should be dried at approximately 105 °C, then cooled and stored in a desiccator.

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

3.3.9 Flexible connections, for which neoprene elastomer tubing is suitable. Some types of silicone tubing have been found to be permeable. For the gas flow lines after the drying towers, the length of the flexible connections should be kept to a minimum, with such tubing being used essentially for only the connection of butt-jointed glass sections.

3.3.10 Flow control needle valves, placed on the outlet side of each flowmeter.

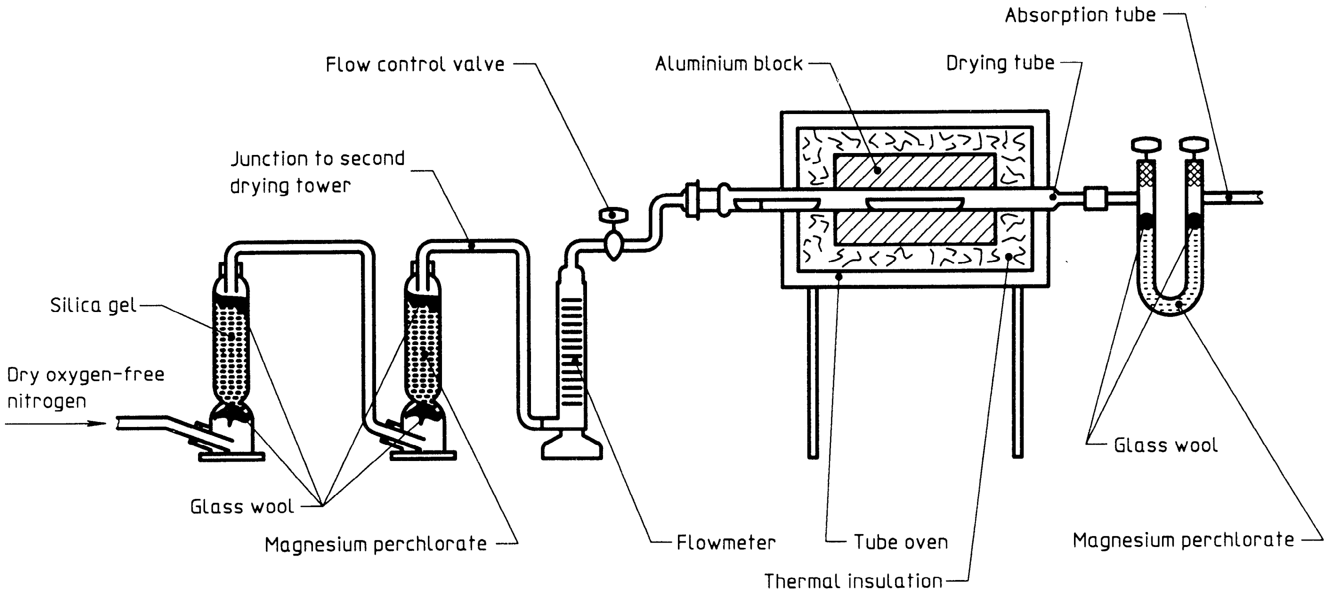

Figure 1 — Apparatus for the determination of hygroscopic moisture — Method 1 (Gravimetric method)

Dimensions in millimetres

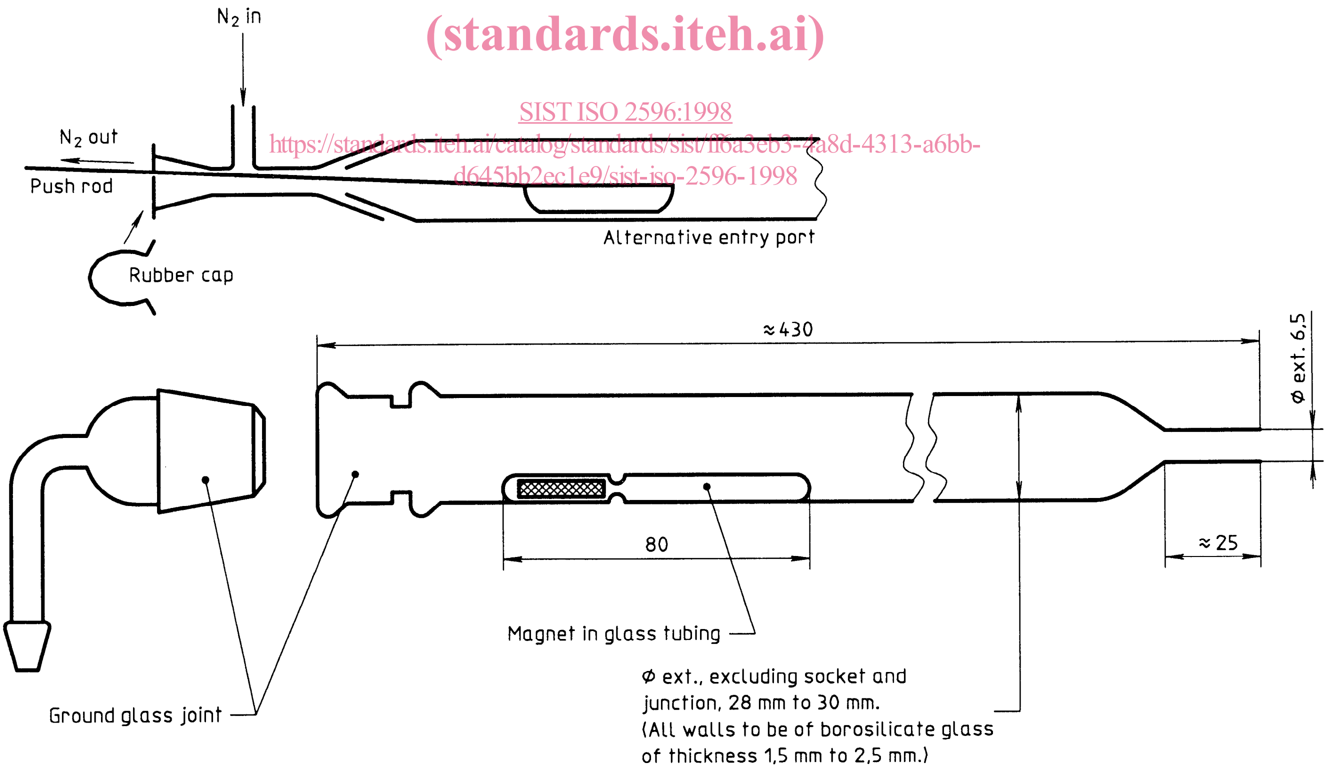

Figure 2 — Drying tube