BRITISH STANDARD

BS ISO
2596:2006

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

ICS 73.060.10

BSi
British Standards

National foreword

This British Standard reproduces verbatim ISO 2596:2006 and implements it as the UK national standard. It supersedes BS ISO 2596:1994 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international publications referred to in this document may be found in the *BSI Catalogue* under the section entitled "International Standards Correspondence Index", or by using the "Search" facility of the *BSI Electronic Catalogue* or of British Standards Online.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, the ISO title page, pages ii to vi, pages 1 to 32, an inside back cover and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 June 2006

ISBN 0 580 48834 9

Amendments issued since publication

Amd. No.	Date	Comments

INTERNATIONAL STANDARD

ISO 2596

Fifth edition
2006-05-01

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

Reference number
ISO 2596:2006(E)

Contents

Page

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 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.

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.

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 or 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 °C ± 2 °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 $Mg(ClO_4)_2$ of size 0,80 mm to 1,25 mm, to ensure carrier-gas water-vapour pressures below 5 µg H_2O/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:

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

where

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

m_1 = 0,000 1 cg/l for aluminium calcium silicate molecular sieves,

0,000 2 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 °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 °C for 4 h.

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

3.2.5 Nitrogen, filtered, predried, oil-free, containing less than 10 µl 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 °C ± 2 °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 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, 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 × 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 × 20 mm × 10 mm. Before use, boats should be dried at approximately 105 °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^2. The sample shall be thoroughly mixed immediately before the determination.

3.5 Procedure

3.5.1 Apparatus conditioning

3.5.1.1 Conditioning of drying tube

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^3/min to 200 cm^3/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^3/min to 100 cm^3/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^3/min to 200 cm^3/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 ± 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^2, for samples between 0,05 and 2,0 % moisture and 0,15 g/cm^2 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:

$$\mathrm{HM} = \frac{m_3 - m_4}{m_5} \times 100 \qquad (2)$$

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:

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

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 °C ± 2 °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 $Mg(ClO_4)_2$ of size 0,80 mm to 1,25 mm, to ensure carrier-gas water-vapour pressures below 5 µg H_2O/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:

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

where

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

m_1 = 0,000 1 cg/l for aluminium calcium silicate molecular sieves,

0,000 2 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.

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

Before use, sieves shall be dried by heating to 400 °C for 4 h.

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

4.2.3 Silica gel desiccant, blue self-indicating.

Before use, gel shall be dried at 105 °C for 4 h.

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

4.2.5 Nitrogen, filtered, predried, oil-free, containing less than 10 µl of oxygen per litre at a pressure of approximately 35 kPa to 50 kPa above atmospheric pressure.

4.2.6 Methanol (CH_3OH) anhydrous, Karl Fischer grade of water content less than 0,005 %.

4.2.7 Ethylene glycol ($CH_2OH \cdot CH_2OH$), anhydrous Karl Fischer grade

4.2.8 Karl Fischer composite (one component) titrant, of 1,0 mg to 3,0 mg H_2O/ml titration capacity.

This reagent is commercially available or can be prepared as specified in ISO 760. Standardize this solution using one of the following standard reagents:

a) a standard water/methanol solution;

b) water added by means of a microsyringe;

Transfer an appropriate quantity of one of the reagents a) or b) into the absorption cell (by means of a microsyringe through the septum) containing absorbent solution that has been titrated to the end-point, then follow the titration procedure described in 4.5.2.

The factor (F), in milligrams of water per millilitre of Karl Fischer solution, is calculated from this titration.

4.3 Apparatus

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

4.3.1 Oven, preferably of the aluminium metal-block type, capable of accommodating one, but preferably several, drying tubes (4.3.2) and of maintaining a temperature within the range 105 °C ± 2 °C over a minimum tube length of 160 mm.

4.3.2 Borosiliate glass drying tubes and connections, fitted with Viton “O”-ring seal pushrod cap assemblies.

NOTE A suitable apparatus is shown diagrammatically in Annex B.

4.3.3 Drying towers, of 250 ml capacity, one filled with molecular sieves (4.2.2) or silica gel (4.2.3) and the other packed with magnesium perchlorate desiccant (4.2.1), to dry the stream of nitrogen (4.2.5) 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.

4.3.4 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.

4.3.5 Sample boats, of an inert and stable material, such as glass, stainless steel or glazed porcelain. Approximate dimensions are 100 mm × 20 mm × 10 mm. Before use, boats should be dried at approximately 105 °C, and then cooled to ambient temperature in a desiccator. Boats shall be stored in a desiccator prior to use.

4.3.6 Filter discs, of sintered metal, sintered glass or similar, inserted between the drying tubes and the inlets to the absorption cells.

4.3.7 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.

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

4.3.9 Titration cells, preferably brown glass vessels of capacity 50 ml to 75 ml possessing drain valves. The entries for the platinum electrodes should preferably be near the cell walls, whilst the burette tip should preferably be centred above the stirrer to ensure rapid distribution of the added reagent.

NOTE 1 A suitable titration cell is shown diagrammatically in Annex D.

Burettes, detection electrode and gas inlets and outlets shall be impermeable to water vapour (ground-glass joints are preferred).

NOTE 2 If a standard water/methanol solution is not to be used for the calibration, only one burette entry to the absorption cell will be required.

4.3.10 Guard tubes, of a suitable design and able to contain sufficient desiccant (4.2.1) to prevent back diffusion of moisture (into the titration cell) and emission of methanol and hydrocarbons (from the titration vessel).

4.3.11 Platinum electrodes, either as a pair or as a dual platinum electrode.

4.3.12 Magnetic stirrers and rotators, of chemically inert material and variable speed.

4.3.13 Amperometric titrator, suitable for Karl Fischer volumetric analysis, equipped with an ammeter (0,50 µA) or an equivalent means for the electrometric indication of the end-point.

4.3.14 Burettes, of suitable capacity (25 ml to 50 ml), conforming to class A specifications (or equivalent), preferably possessing a smoke-glass reservoir bottle.

The burette should be vented solely by an anhydrous atmosphere of suitable capacity. Fillers should not be pressurized through the lower inlet of the burette.

4.4 Sampling and samples

4.4.1 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.

4.4.2 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 approximate equilibrium with the laboratory atmosphere by exposure for at least 2 h on an inert tray at a layer density not greater than 1,0 mg/mm^2.

The sample shall be thoroughly mixed immediately before the determination.

4.5 Procedure

4.5.1 Conditioning of drying tube

Bring the temperature of the drying tubes (4.3.2) to 105 °C ± 2 °C and maintain this temperature throughout steps 4.5.3 to 4.5.5.

Adjust the rate of flow of nitrogen (4.2.5) to provide a constant flow rate of 100 cm^3/min to 200 cm^3/min through drying tubes and maintain this flow rate throughout steps 4.5.2 to 4.5.6.

Connect the outlet from each drying tube to the inlet of a guard tube (4.3.10). 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^3/min to 100 cm^3/min (and temperature of 105 °C ± 2 °C) during periods in which the instrument is idle.

4.5.2 Preparation of titration unit

Remove the rubber septum from the absorption cell and transfer 20 ml of ethylene glycol (4.2.7) and 20 ml of methanol (4.2.6), or equivalent commercially available absorption solutions, into the titration cell.

Where automated titrators are used, instrument operators should ensure that the manufacturer's instructions are followed with respect to reagent capacities.

NOTE Commercially available Karl Fischer two-component systems (absorption solvent and titrants) may be used as an alternative to ethylene glycol/methanol absorption and composite Karl Fischer titrant solutions.

Switch on the electrometric titrator (4.3.13) and magnetic stirrer (4.3.12), adjusting the speed of the latter to ensure adequate mixing. Maintain a constant stirring speed during the titration and between the successive titrations of the blank test, standard, and test sample.

4.5.3 Titration

Add Karl Fischer solution (4.2.8) slowly to the absorption cell by burette (4.3.14). The approaching end-point will be indicated by a rapid increase in current due to the presence of free iodine from the excess Karl Fischer solution. At this point, a current is selected for the end-point (30 µA to 40 µA). The titration is continued until this current can be maintained for a period of 20 s. The absorbent solution shall be titrated to this end-point immediately prior to the commencement of all tests and calibrations.

The manufacturer's recommendations regarding titration procedures should be followed if commercially purchased equipment and reagents are used.

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

4.5.4 Blank test

Restore the gas flow to a conditioned titration cell (4.3.9) and adjust the nitrogen flow to a rate of 100 ml/min to 200 ml/min.

Quickly remove the drying-tube end cap (4.3.2), and place an empty sample boat (4.3.5) in the entrance of the heated 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 boat introduction.

During the placement of the boat in the drying tube, as specified in 4.5.2 to this subclause, precautions should be taken, essentially involving a careful technique, to prevent entry of moisture from extraneous sources into the drying tube.

After 2 h, disconnect the outlet of the drying tube from the absorption cell, and connect an opened guard tube to the tube oven outlet. Follow the procedure exactly as described in 4.5.2.

Background-corrected blank tests 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.

4.5.5 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 performance of the equipment is required.

When a satisfactory value for the blank test has been obtained, weigh 0,05 g to 0,20 g of copper(II) sulfate pentahydrate (4.2.4), to the nearest 0,2 mg, into the cooled sample boat used for the blank test. The mass taken should relate to the approximate anticipated maximum moisture content of the ore type being analysed.

Repeat the procedure in 4.5.3 using the boat containing the weighed copper(II) sulfate. The indicated water content, corrected with the blank test value, should fall within the range 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.

4.5.6 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 (4.4.2), 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 2, the test portion for the determination of hygroscopic moisture.

Table 2 — Mass of test portion — Method 2 (Karl Fischer volumetric method)

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

Transfer the weighed sample portion into a conditioned sample boat (4.3.5), and distribute the material evenly. Immediately, repeat the procedure in 4.5.3, using the boat containing the test portion instead of an empty boat, and record the volume of Karl Fischer titrant consumed.

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

NOTE Alternatively, the analytical sample may be weighed directly into a conditioned sample boat (4.3.5).

The weighing of analytical test samples shall be performed in parallel to 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.

4.6 Expression of results

4.6.1 Calculation of hygroscopic moisture content

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

$$\mathrm{HM} = \frac{(V_1 - V_2) \times F}{m_3 \times 1\,000} \times 100 \qquad (5)$$

where

V_1 is the volume, in millilitres, of Karl Fischer solution (4.2.8) consumed in the titration of the test portion (see 4.5.5);

V_2 is the volume, in millilitres, of Karl Fischer solution (4.2.8) consumed in the titration of the blank test (4.5.3);

F is the factor, in milligrams of water per millilitre of Karl Fischer solution, as determined in 4.2.8;

m_3 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.

4.6.2 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:

$$\mathrm{MCM} = m_4 - \left(m_4 \times \frac{\mathrm{HM}}{100} \right) \qquad (6)$$

where

m_4 is the mass, in grams, of analytical test portion to be moisture corrected;

HM is the hygroscopic moisture content of a portion;

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

5 Method 3 — Karl Fischer coulometric method

5.1 Principle

The hygroscopic moisture content of an environmentally equilibrated test portion is determined at 105 °C ± 2 °C in 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 titrated coulometrically using automated Karl Fischer equipment, and the percentage moisture content is determined.

5.2 Reagents

5.2.1 Desiccant, anhydrous magnesium perchlorate $Mg(ClO_4)_2$ of size 0,80 mm to 1,25 mm, to ensure carrier-gas water-vapour pressures below 5 µg H_2O/l.

As measurement accuracy and precisions 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:

$$C_c = \frac{(m_1)L \cdot N}{m_2} \tag{7}$$

where

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

m_1 = 0,000 1 cg/l for aluminium calcium silicate molecular sieves,

0,000 2 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.

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.

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

Before use, sieves shall be dried by heating to 400 °C for 4 h.

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

5.2.3 Silica gel desiccant, blue self-indicating.

Before use, the gel shall be dried by heating at 105 °C for 4 h.

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

5.2.5 Nitrogen, filtered, predried, oil-free, containing less than 10 µl of oxygen per litre at a pressure of approximately 35 kPa to 50 kPa above atmospheric pressure.

5.2.6 Methanol (CH_3OH) anhydrous, Karl Fischer grade of water content $< 0{,}005$ %, and non-volatile matter less than 0,001 %.

5.2.7 Karl Fischer anode reagent, suitable for use with drying ovens, of titration capacity 10 mg H_2O/ml.

5.2.8 Karl Fischer cathode reagent, of titration capacity 60 mg H_2O/ml.

5.3 Apparatus

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

5.3.1 Oven, preferably of the aluminium metal block type, capable of accommodating one, but preferably several, drying tubes (5.3.2) and of maintaining a temperature within the range 105 °C ± 2 °C over a minimum tube length of 160 mm.

5.3.2 Borosilicate glass drying tubes and connections, fitted with Viton "O"-ring seal pushrod cap assembly.

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

5.3.3 Drying towers, of capacity 250 ml, one filled with molecular sieves (5.2.2) or silica gel (5.2.3) and the other packed with magnesium perchlorate desiccant (5.2.1), to dry the stream of nitrogen (5.2.5) entering the drying tubes.

Molecular sieves (5.2.2) and silica gel (5.2.3) drying towers shall be repacked with freshly dried desiccants fortnightly.

5.3.4 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.

5.3.5 Sample boats, of an inert and stable material, such as glass, stainless steel or glazed porcelain. Approximate dimensions are 100 mm × 20 mm × 10 mm. Before use, boats should be dried at approximately 105 °C, and then cooled to ambient temperature in a desiccator. Boats shall be stored in a desiccator prior to use.

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

5.3.7 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.

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

5.3.9 Titration cells, preferably brown glass vessels. The entries for the platinum electrodes should preferably be near the cell walls, whilst the generator electrode should preferably be centred above the stirrer to ensure rapid distribution of the added reagent.

NOTE A suitable titration cell is shown diagrammatically in Annex E.

5.3.10 Guard tubes, of capacity 250 ml, filled with molecular sieves (5.2.2) to prevent back diffusion of moisture (into the titration cell) and emission of methanol and chlorinated hydrocarbons (from the titration vessel).

5.3.11 Platinum electrodes, either as a pair or as a dual platinum electrode.

5.3.12 Magnetic stirrers and rotators, of chemically inert material and variable speed.

5.3.13 Coulometric titrator, suitable for Karl Fischer analysis, or an equivalent means for the electrometric indication of the end-point.

5.3.14 Data logger, suitably interfaced for the acquisition of titration time, instantaneous titration rate and integrated moisture content at sampling intervals of 30 s to 90 s.

5.4 Sampling and samples

5.4.1 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.

5.4.2 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 approximate 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^2.

The sample shall be thoroughly mixed immediately before the determination.

5.5 Procedure

5.5.1 Preparation of titration unit

Anodic and cathodic compartments shall be filled in accordance with the manufacturer's recommendations using appropriate reagents, ensuring that the solute level of the catholyte is lower than the anolyte solute level (to prevent diffusion into anodic compartments).

As volatilization of anodic methanol will occur during analysis, the addition of anhydrous methanol (5.2.6) shall be performed whenever anodic solution levels drop below cathodic levels.

To ensure that the coulometric cell is maintained in a dry state, the titrator shall be left on.

NOTE Due to the nature of the coulometric titration process, a persistent instrument background (visible as titrated moisture) will be present at all times.

5.5.2 Conditioning of drying tube

Bring the temperature of the drying tube (5.3.2) to 105 °C ± 2 °C and maintain this temperature throughout steps 5.5.2 to 5.5.5.

Adjust the rate of flow of nitrogen (5.2.5) to provide a constant flow rate of 100 cm^3/min to 200 cm^3/min through the drying tube and maintain this flow rate throughout steps 5.5.2 to 5.5.5.

Connect the outlet from each drying tube to the inlet of a guard tube (5.3.10). Open the taps of the guard tube, and pass nitrogen through the tube for a minimum of 4 h.

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

5.5.3 Blank test

Restore the gas flow to the coulometric unit and adjust the nitrogen flow to a rate of 100 ml/min to 200 ml/min, start the data logger and titrator, and allow the background to stabilize (< 0,001 µg/s) for a minimum of 5 min.

NOTE As the combined backgrounds (instrument and nitrogen) are required for corrections, it is imperative that this remains constant throughout the total duration of the determination.

Quickly remove the drying tube end cap (5.3.2), and place an empty sample boat (5.3.5) in the entrance of the heated 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 this subclause to 5.5.5, 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, switch off the titrator and data logger, disconnect the outlet of the drying tube from the titrator unit, and connect an opened guard tube to the oven. Where necessary, add anhydrous methanol (5.2.6) to the anodic compartment, and insert the bung.

The background-corrected blank is calculated, in grams, using the following equation:

$$\text{Blank} = \mathrm{H_2O}_{T\mathrm{B}=2} - \mathrm{H_2O}_{T=0} - \left(D_T \times t\right) \tag{8}$$

where

$\mathrm{H_2O}_{T=0}$ is the titrated moisture content, in grams, at sample introduction (T = 0);

$\mathrm{H_2O}_{T\mathrm{B}=2}$ is the total titrated moisture content, in grams, after 2 h (7 200 s);

D_T is the background titration rate, in grams per second (at T = 0);

t is the titration time, in seconds (= 7 200 s).

Background-corrected blank tests 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.

5.5.4 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 performance of the equipment 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 (5.2.4) 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 should approximate the anticipated moisture content of the ore type being analysed.

Repeat procedure 5.5.3 using the boat containing the weighed copper(II) sulfate. The indicated water content, corrected with the blank test value, should fall within the range 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 ± 1 %) may be used to introduce water directly into the heated zone of the drying tube through a septum.

5.5.5 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 (5.4.2), 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 3, the test portion for the determination of hygroscopic moisture.

Table 3 — Mass of test portion — Method 3 (Karl Fischer coulometric method)

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

Transfer the weighed sample portion into a conditioned sample boat (5.3.5), and distribute the material evenly. Immediately repeat the procedure in 5.5.3, using the boat containing the test portion instead of an empty boat.

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

NOTE Alternatively, the analytical sample may be weighed directly into a conditioned sample boat (5.3.5).

The weighing of analytical test samples shall be performed in parallel to 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.

5.6 Expression of results

5.6.1 Calculation of hygroscopic moisture content

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

$$\mathrm{HM} = \frac{\mathrm{H_2O}_{T=0} - \mathrm{H_2O}_{T=2} - B - \left(D_T \times t\right)}{m_3} \times 100 \qquad (9)$$

where

$\mathrm{H_2O}_{T=0}$ is the titrated moisture content, in grams, at sample introduction (T = 0);

$\mathrm{H_2O}_{T=2}$ is the titrated moisture content, in grams, after 2 h (7 200 s);

B is the background-corrected blank (boat blank) content, in grams;

D_T is the background titration rate, in grams per second at (T = 0);

t is the titration time, in seconds (= 7 200)

m_3 is the mass, in grams, of the test portion.

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

5.6.2 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:

$$\mathrm{MCM} = m_4 - \left(m_4 \times \frac{\mathrm{HM}}{100} \right) \tag{10}$$

where

m_4 is the mass, in grams, of analytical test portion to be moisture corrected;

HM is the hygroscopic moisture content of a test portion;

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

6 Method 4 — Mass-loss method

6.1 Principle

Using a convection drying oven, the hygroscopic moisture content of an environmentally equilibrated test portion is determined using a specialized weighing chamber. Contained within its chamber, a test sample is heated to 105 °C ± 2 °C for 2 h, under a stream of dry nitrogen (at a rate of 100 ml/min to 200 ml/min), and the percentage moisture content is determined via mass loss.

6.2 Reagents

6.2.1 Desiccant, anhydrous magnesium perchlorate $Mg(ClO_4)_2$ of size 0,80 mm to 1,25 mm, to ensure carrier-gas water-vapour pressures below 5 µg H_2O/l.

As measurement accuracy and precisions 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:

$$C_c = \frac{(m_1)L \cdot N}{m_2} \tag{11}$$

where

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

m_1 = 0,000 1 cg/l for aluminium calcium silicate molecular sieves,

0,000 2 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.

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.

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

Before use, sieves shall be regenerated by heating to 400°C for 4 h.

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

6.2.3 Silica gel desiccant, blue self-indicating.

Before use, the gel shall be dried by heating at 105 °C for 4 h.

6.2.4 Nitrogen, filtered, predried, oil-free, containing less than 10 µl of oxygen per litre at a pressure of approximately 35 kPa to 50 kPa above atmospheric pressure.

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

6.3 Apparatus

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

6.3.1 Balance, capable of reading the mass of the weighing container to 0,1 mg.

6.3.2 Oven, capable of accommodating several weighing chambers (6.3.8) and of maintaining a temperature within the range 105 °C ± 2 °C.

6.3.3 Drying towers, of capacity 250 ml, one filled with molecular sieves (6.2.2) or silica gel (6.2.3) and the other packed with magnesium perchlorate desiccant (6.2.1), to dry the stream of nitrogen (6.2.3) entering the weighing chambers.

Molecular sieves (6.2.2) and silica gel (6.2.3) drying towers shall be repacked with freshly dried desiccants fortnightly.

6.3.4 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.

6.3.5 Guard tubes, of a suitable design, containing magnesium perchlorate desiccant (6.2.1) to prevent back diffusion of moisture into weighing chambers.

6.3.6 Filter discs, of sintered metal, sintered glass or similar, inserted between the drying towers and weighing chamber.

6.3.7 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.

6.3.8 Weighing chamber, of an inert and stable material (titanium is preferred) of less than 50 g mass and approximately 8 ml volume.

NOTE A suitable apparatus is shown diagrammatically in Annex G.

6.3.9 Flow-control needle valve, placed prior to each flowmeter.

NOTE Commercially available "Parcher" equipment, as shown in Annex H (available from Initiative Scientific Products), may be used as an alternative to the described mass-loss apparatus.

6.4 Sampling and samples

6.4.1 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.

6.4.2 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^2. The sample shall be thoroughly mixed immediately before the determination.

6.5 Procedure

6.5.1 Apparatus conditioning

Bring the temperature of the oven (6.3.2) to 105 °C ± 2 °C and maintain this temperature throughout steps 6.5.1.1 to 6.5.3.

Adjust the rate of flow of nitrogen (6.2.4) to provide a constant flow rate of 100 cm^3/min to 200 cm^3/min through desiccants and gas transfer tubes for a minimum of 4 h before determinations.

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

If a manifold arrangement is to be used to accommodate multiple samples, appropriate restrictors shall be inserted between manifold branches to control individual weighing-chamber gas flows to 100 cm^3/min to 200 cm^3/min.

6.5.1.1 Conditioning of weighing chamber

Insert a weighing chamber in the oven, and restore the nitrogen flow through the chamber and note the introduction time.

After 2 h, close the taps of the weighing chamber (whilst under a dry nitrogen atmosphere), outlet first, and remove the chamber from the oven. Wipe the weighing chamber with a clean dry cloth free from loose fibres, and allow to cool to ambient balance-room temperature, and then weigh to the nearest 0,1 mg.

To minimize the transfer of contaminants such as moisture and grease, conditioned weighing chambers shall be handled using cotton gloves.

NOTE Do not pressure equilibrate the weighing chamber prior to weighing.

The mass change of the weighing chamber should be as low as possible and no more than 0,5 mg.

6.5.2 Check test

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 weighing-chamber seals have been replaced.

When a satisfactory value for an empty weighing chamber has been obtained, weigh 0,05 g to 0,2 g of copper(II) sulfate pentahydrate (6.2.5) to the nearest 0,2 mg, into a conditioned weighing chamber. 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 6.5.1.1, using a weighing chamber containing the weighed copper(II) sulfate. The loss in mass of the weighing chamber 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.

6.5.3 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 (6.4.2), 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 4, the test portion for the determination of hygroscopic moisture.

Table 4 — Mass of test portion — Method 4 (Mass loss method)

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

Transfer the weighed sample portion into a conditioned weighing chamber (6.3.8), and distribute the material evenly. Immediately, repeat the procedure in 6.5.1.1 using the chamber containing the test portion instead of an empty chamber.

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

NOTE Alternatively, the analytical sample may be weighed directly into a conditioned weighing chamber (6.5.1.1).

The weighing of analytical test samples shall be performed in parallel to 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.

6.6 Expression of results

6.6.1 Calculation of hygroscopic moisture content

The content of hygroscopic moisture (HM) is calculated as a percentage by mass using the following equation:

$$\mathrm{HM} = \frac{m_3 + m_4 - m_5}{m_4} \times 100 \qquad (12)$$

where

m_3 is the in mass, in grams, of the conditioned (blank) weighing chamber;

m_4 is the mass, in grams, of the test portion used for moisture analysis;

m_5 is the mass, in grams, of the dried test portion and weighing chamber.

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

6.6.2 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:

$$\mathrm{MCM} = m_6 - \left(m_6 \times \frac{\mathrm{HM}}{100} \right) \qquad (13)$$

where

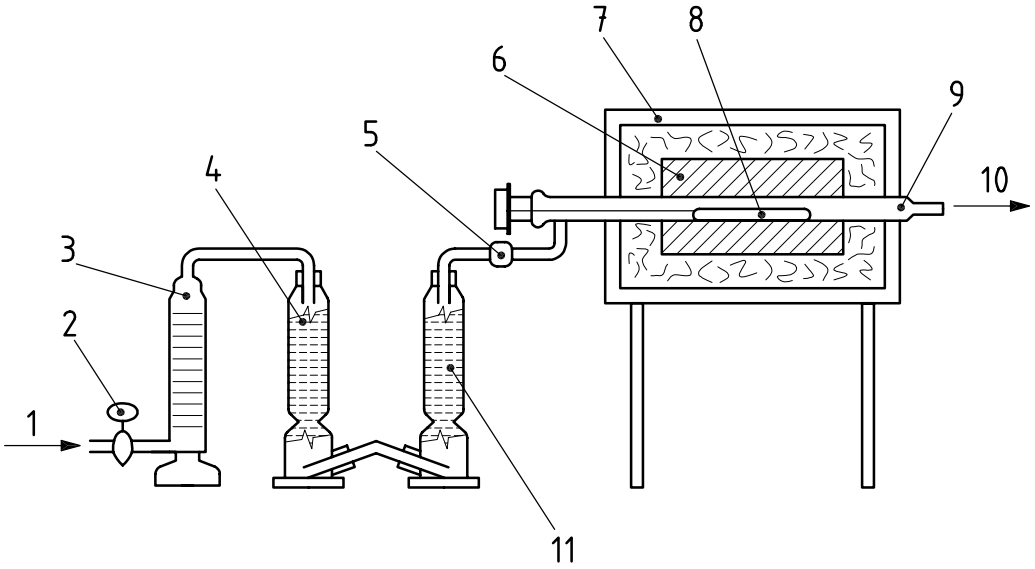
m_6 is the mass, in grams, of an analytical test portion to be moisture corrected;

HM is the hygroscopic moisture content of a test portion;

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

Annex A
(informative)

Gravimetric and Karl Fischer apparatus



Key

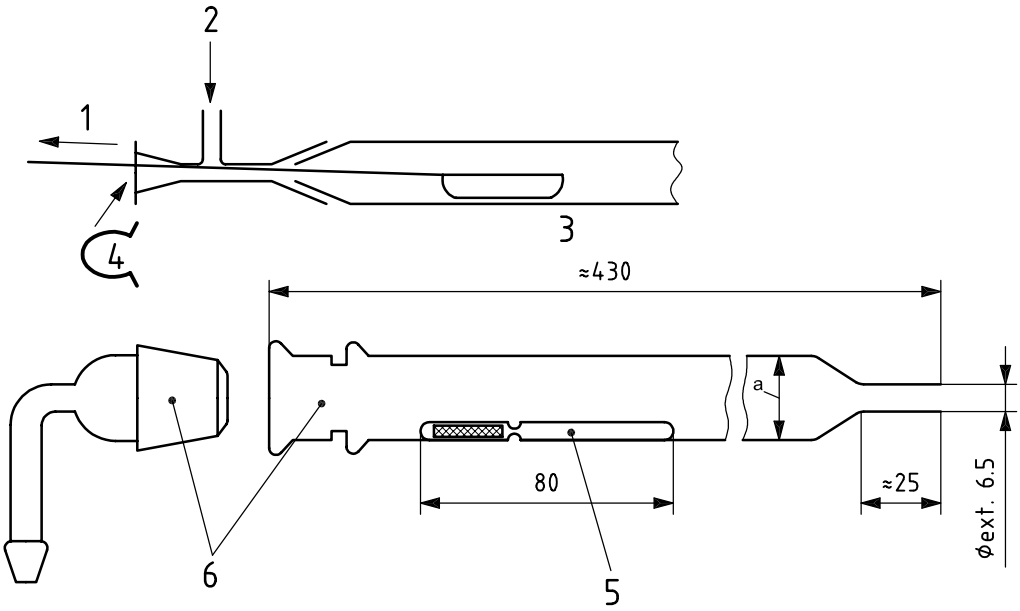
1 nitrogen inlet
2 needle valve
3 flowmeter
4 molecular sieves
5 frit filter
6 aluminium block
7 tube oven
8 sample boat
9 drying tube
10 outlet to Karl Fischer titrator or absorption tube
11 magnesium perchlorate

Figure A.1

Annex B
(informative)

Drying tube

Dimensions in millimetres



Key

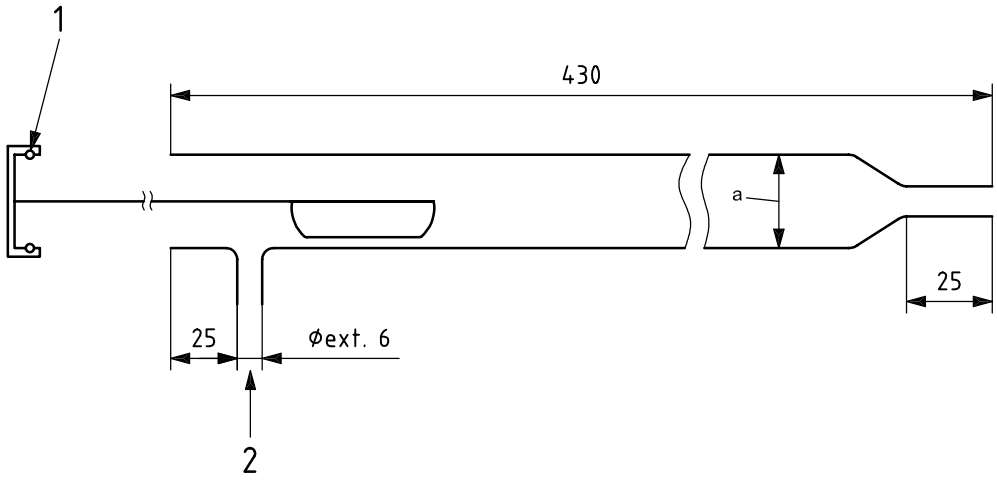
1 N_2 out, push rod
2 N_2 in
3 alternative entry port
4 rubber cap
5 magnet in glass tubing
6 ground-glass joint

[a] ∅ ext., excluding socket and junction, 28 mm to 30 mm. (All walls to be of borosilicate glass of thickness 1,5 mm to 2,5 mm.)

a)

Figure B.1 — Drying tube — Alternative port entry (*continued*)

Dimensions in millimetres



Key

1 "O"-ring seal drying-tube cap assembly

2 N_2 inlet

[a] ∅ ext., excluding socket and junction, 28 mm to 30 mm. (All walls to be of borosilicate glass of thickness 1,5 mm to 2,5 mm.)

b)

Figure B.1 — Drying tube — Alternative port entry

Annex C
(informative)

Titanium absorption tube

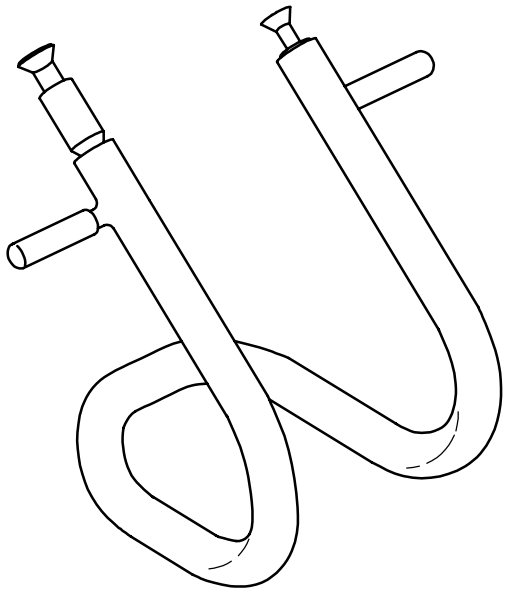
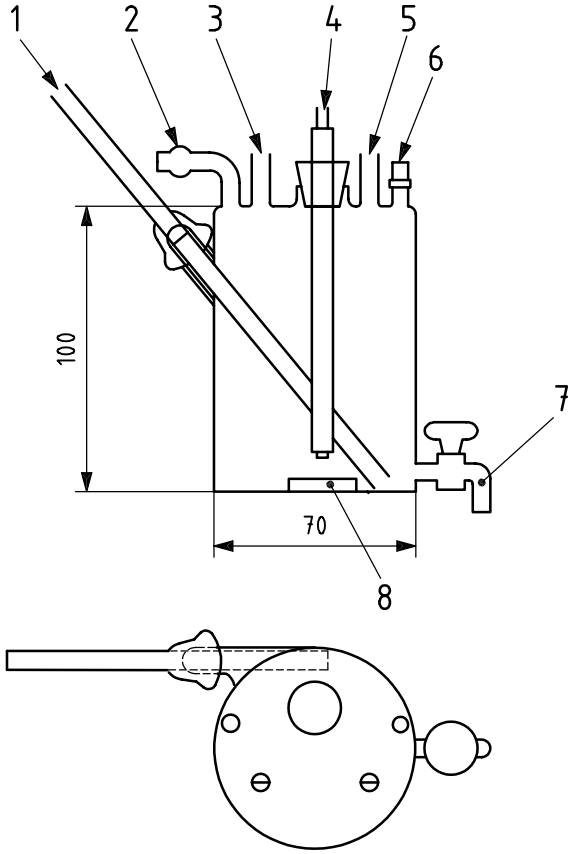


Figure C.1

Annex D
(informative)

Volumetric titration cell

Dimensions in millimetres



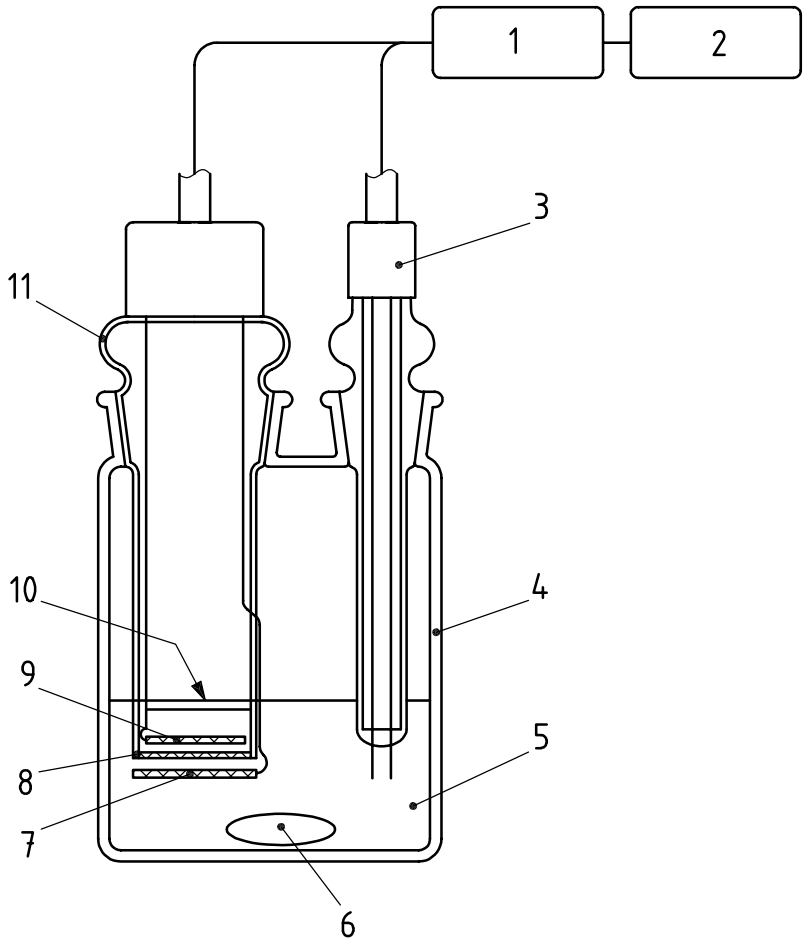
Key

1 gas inlet
2 gas outlet
3 Karl Fischer burette entry
4 dual platinum electrode
5 water/methanol burette entry
6 rubber septum entry for calibration using water
7 tap outlet
8 rotator

Figure D.1

Annex E
(informative)

Coulometric titration cell



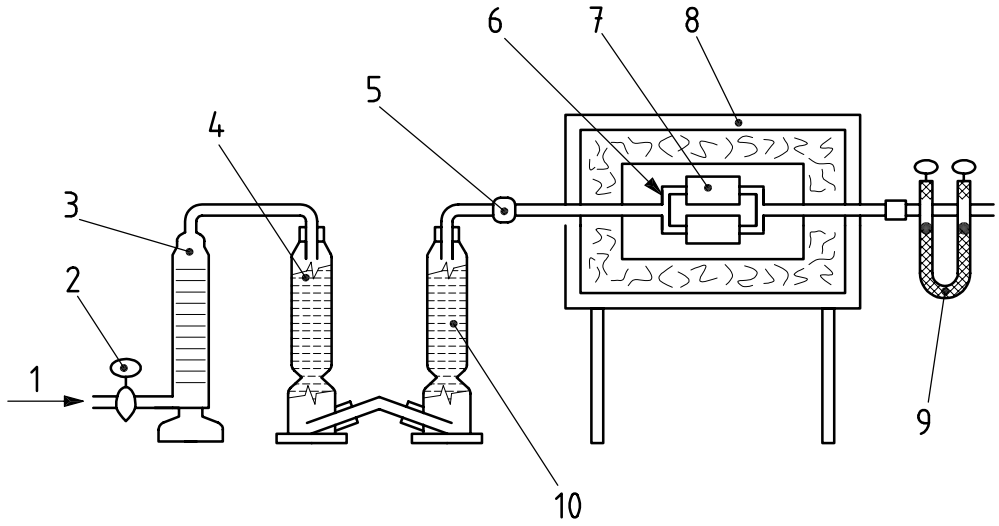
Key

1	detection/control unit	8	membrane
2	micro-processor	9	cathode
3	detecting electrode	10	catholyte
4	titration cell	11	iodine generator
5	anolyte		
6	strirring rotor		
7	anode		

Figure E.1

Annex F
(informative)

Modified weighing chamber apparatus



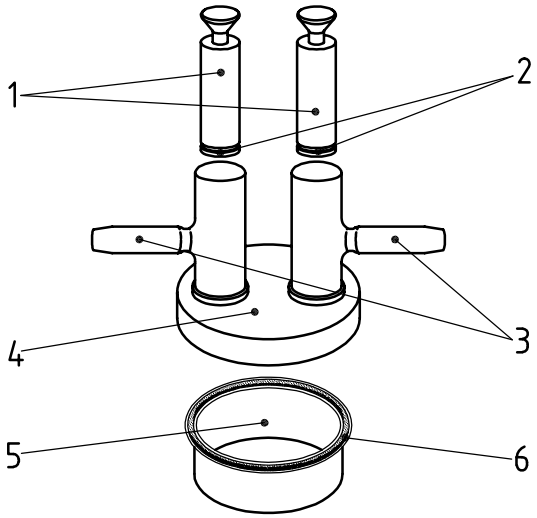
Key

1 nitrogen inlet
2 needle valve
3 flowmeter
4 molecular sieves
5 frit filter
6 manifold
7 weighing chamber
8 oven
9 protector tube
10 magnesium perchlorate

Figure F.1

Annex G
(informative)

Modified weighing chamber



Key

1 slider valves
2 Viton valve seals
3 venting ports
4 cap assembly
5 sample-holder body
6 sample-holder seal (Viton)

Figure G.1

Annex H
(informative)

Parcher equipment

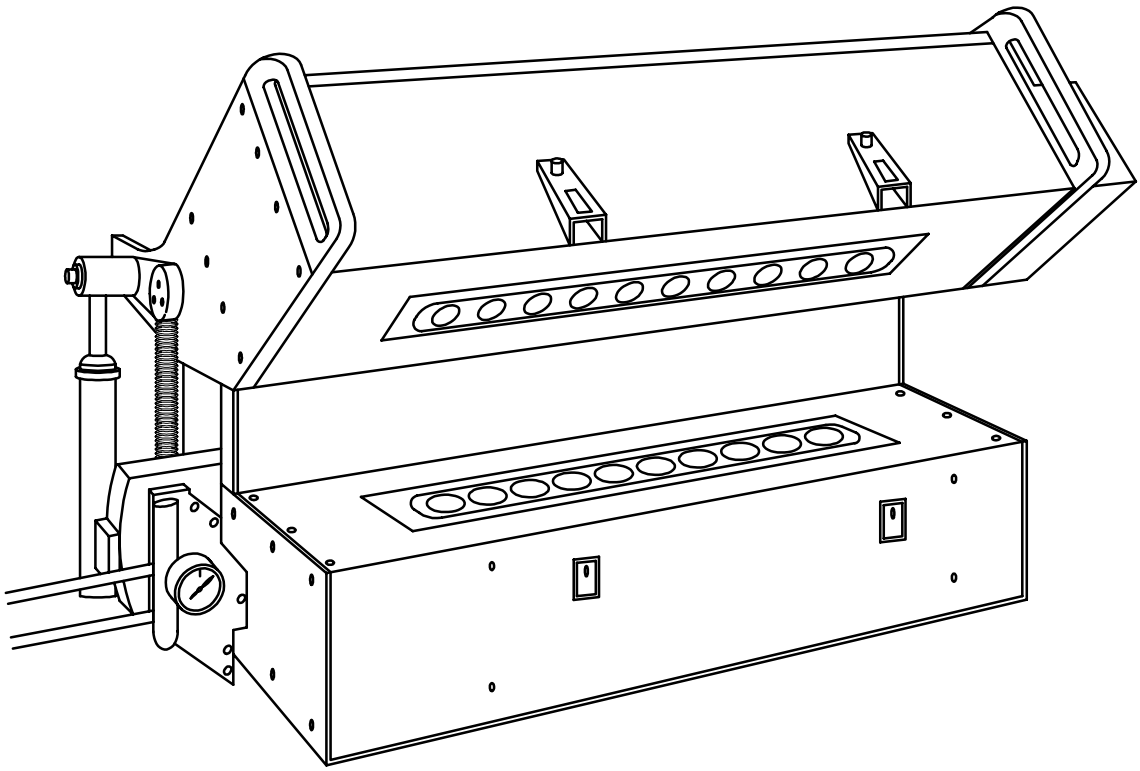


Figure H.1

Bibliography

[1] ISO 3087, *Iron ores — Determination of moisture content of a lot*

[2] ISO 3696, *Water for analytical laboratory use — Specification and test methods*

[3] ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

blank

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsi-global.com/bsonline.

Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL