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

iTeh STANDARD PREVIEW

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

[ISO 2596:1994](#)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

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 $105\text{ °C} \pm 2\text{ °C}$ 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 [$\text{Mg}(\text{ClO}_4)_2$] 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 ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), free-flowing crystalline material, press-crushed if necessary under a pestle by hand, without grinding, to a size of approximately 1 mm.

3.2.4 Nitrogen, filtered, predried, oil-free, containing less than $10\ \mu\text{l}$ 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 $105\text{ °C} \pm 2\text{ °C}$ 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 $100\text{ cm}^3/\text{min}$ 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, 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 $100\text{ mm} \times 20\text{ mm} \times 10\text{ mm}$, and the sample loading shall not exceed $1,5\text{ mg}/\text{mm}^2$. 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.

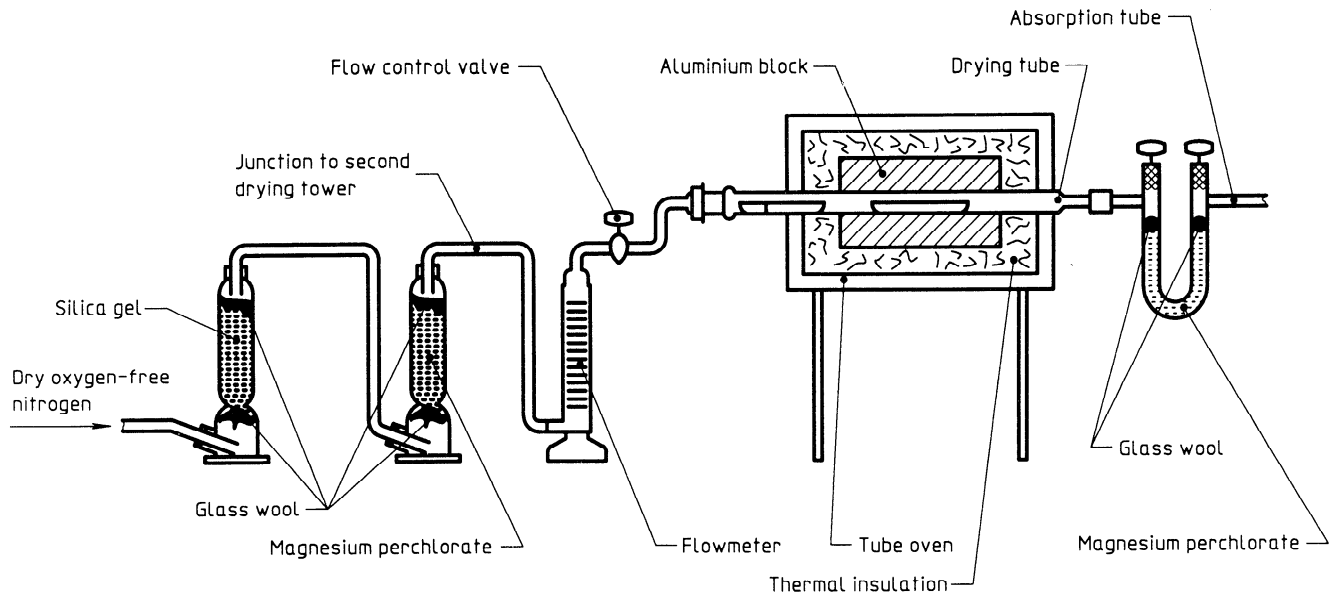


Figure 1 — Apparatus for the determination of hygroscopic moisture — Method 1 (Gravimetric method)

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Dimensions in millimetres

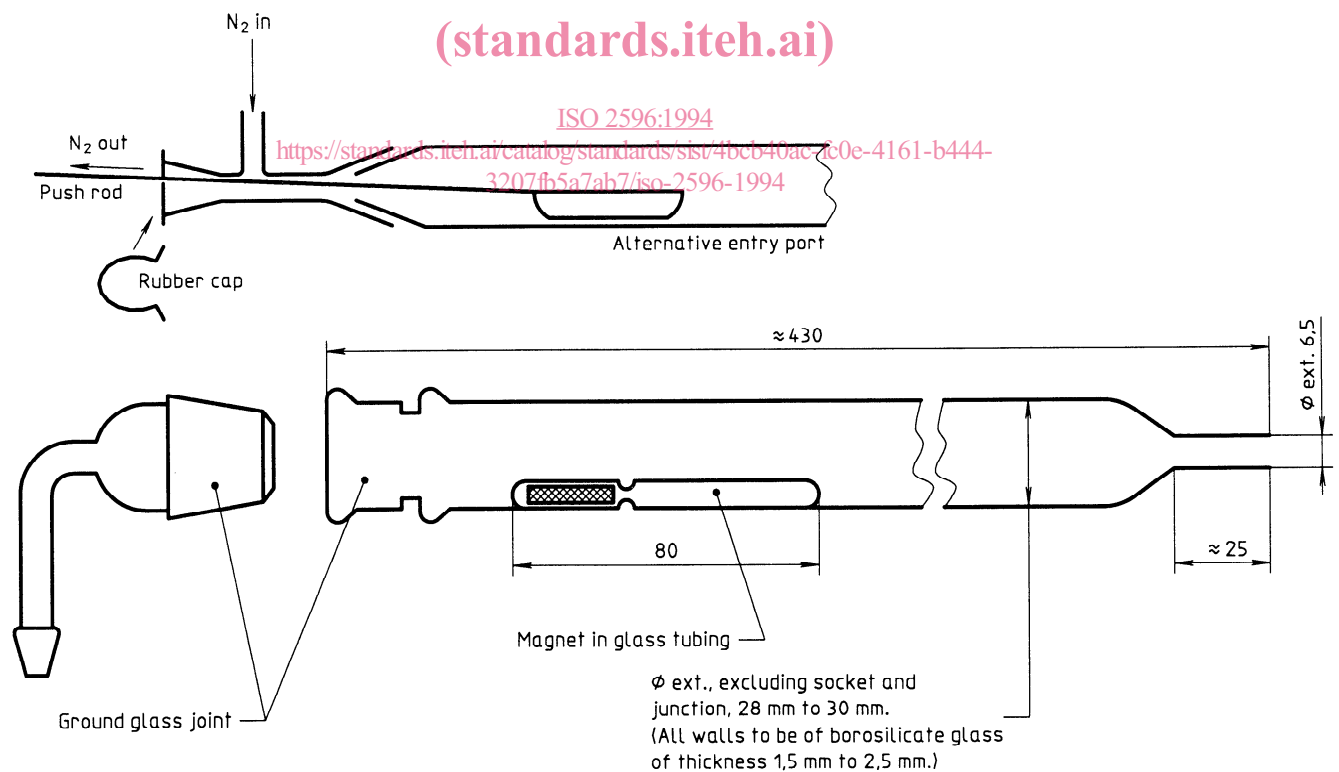


Figure 2 — Drying tube

3.4 Sampling and samples

3.4.1 Laboratory sample

For analysis, use a laboratory sample of particle size $< 160 \mu\text{m}$ which has been taken in accordance with ISO 3081 or ISO 3082, and prepared in accordance with ISO 3082 or ISO 3083.

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 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 mg/mm^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 absorption tube

Bring the temperature of the drying tubes (3.3.3) to $105 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ and maintain this temperature throughout steps 3.5.1 to 3.5.4.

Adjust the rate of flow of nitrogen (3.2.4) to provide a constant flow rate of $150 \text{ cm}^3/\text{min}$ to $200 \text{ cm}^3/\text{min}$ through the drying tubes. Connect a closed absorption tube (3.3.6) to an empty drying tube and check for leaks in the system (zero flow). Open the taps of the absorption tube, readjust the flow rate if necessary, and pass nitrogen through the drying tube for 15 min.

NOTE 3 The conditioning of the absorption tube is necessary only before the first determination of the day.

3.5.1.2 Weighing of absorption tube

Close the taps of the absorption tube, closing the outlet tap first, and stop the nitrogen flow. Disconnect the absorption tube and seal the outlet of 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. Open the taps of the absorption tube momentarily to equalize the pressure, and then weigh to the nearest 0,1 mg.

3.5.2 Blank test

Reconnect the weighed absorption tube, restore the nitrogen flow to the previous rate, check for leaks in the system and open the taps on the absorption tube.

Quickly remove the inlet connector to the drying tube (3.3.3), place an empty sample boat (3.3.7) in the drying tube at the entrance to the heated zone, and insert the magnetic push rod. Immediately replace the inlet connector and move the boat to the centre of the oven, using a magnet.

NOTES

4 While the sample boat is being placed in the drying tube as specified in 3.5.2 to 3.5.4, precautions should be taken, essentially involving careful technique, to prevent entry of moisture from extraneous sources into the drying tube.

5 Alternatively, a narrow-holed inlet connector and external push rod may be used, provided that the rod is withdrawn and the inlet closed off as quickly as possible.

After 2 h, follow the procedure exactly as described in 3.5.1.2.

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.

3.5.3 Check test

NOTE 6 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 0,1 g to 0,2 g of copper(II) sulfate pentahydrate (3.2.3), to the nearest 0,000 2 g, 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 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 water content of copper(II) sulfate within the range 28,5 % (m/m) to 29,2 % (m/m). If not, the cause should be determined.

3.5.4 Determination

When a satisfactory value for the blank test has been obtained (and similarly for the check test, if appropri-

ate), weigh, from the equilibrated test sample (3.4.2), the required test portion for the determination of the constituent to be reported on a dry basis. Reserve this test portion in the vessel specified. 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 % (m/m)	Mass of test portion g
0,05 to 2	2,0
2 to 6	1,0

Transfer the test portion to a dried sample boat (3.3.7), distributing the material evenly. Without delay, repeat the procedure in 3.5.2, using the boat containing the test portion instead of an empty boat, and record the total increase in mass of the absorption tube.

Repeat the determination of hygroscopic moisture whenever the determination of the constituent to be reported on a dry basis is repeated, preferably with a change of operator but otherwise at a different time. The hygroscopic moisture values shall not be averaged but shall be used individually to correct the corresponding constituent value.

3.6 Expression of results

3.6.1 Calculation of hygroscopic moisture content

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

$$A = \frac{m_1 - m_2}{m_3} \times 100 \quad \dots (1)$$

where

m_1 is the increase in mass, in grams, of the absorption tube during the test;

m_2 is the increase in mass, in grams, of the absorption tube during the blank test;

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

3.6.2 Calculation of the final result

The result is calculated to four decimal places and rounded off to the second decimal place as follows:

- where the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged;
- where the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or where the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;
- where the figure in the third decimal place is 5 and the figure 0 is in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

For internal reporting purposes, the percent hygroscopic moisture shall be reported to the nearest 0,05 % (m/m). For this calculation, double the final result which has been rounded off to two decimal places, round it off to the first decimal place using the procedure described above, and divide the result by 2.

3.7 Test report (for internal laboratory use only)

The test report shall include the following information:

- name and address of the testing laboratory;
- date of issue of the test report;
- reference to this International Standard;
- details necessary for the identification of the sample;
- result of the analysis;
- reference number of the result;
- any characteristics noticed during the determination, and any operations not specified in this International Standard, which may have had an influence on the results.

4 Method 2 — Karl Fischer method

4.1 Principle

Equilibration of the test sample with the laboratory atmosphere. Heating of a test portion at $105 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ in a heated tube in a stream of dry nitrogen, and collection of the evolved moisture in ethylene glycol. Measurement of the moisture con-

tent by titration with Karl Fischer solution using electrometric detection of the end-point.

4.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water that complies with grade 3 of ISO 3696.

4.2.1 Desiccant, anhydrous magnesium perchlorate [$\text{Mg}(\text{ClO}_4)_2$] of size 0,8 mm to 1,25 mm, or other suitable desiccant of equivalent drying efficiency.

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.

4.2.2 Silica gel

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

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

4.2.5 Ethylene glycol ($\text{CH}_2\text{OH} \cdot \text{CH}_2\text{OH}$), anhydrous.

4.2.6 Karl Fischer solution, 2,5 mg $\text{H}_2\text{O}/\text{ml}$ to 3,0 mg $\text{H}_2\text{O}/\text{ml}$.

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 standard water/methanol solution;
- water added by means of a microsyringe;
- citric acid monohydrate [$\text{C}(\text{OH})(\text{COOH})(\text{CH}_2\text{COOH})_2 \cdot \text{H}_2\text{O}$];
- disodium tartrate dihydrate [$(\text{CHOH} \cdot \text{COONa})_2 \cdot 2\text{H}_2\text{O}$].

Transfer an appropriate quantity of one of the reagents a), b), c) or d) into the absorption cell containing ethylene glycol 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.

Add water by means of a microsyringe through the rubber septum. For the addition of citric acid or disodium tartrate dihydrate, remove the rubber septum and, with nitrogen flowing through the apparatus, add the reagent, then immediately replace the septum. Add standard water/methanol solution using a burette.

4.3 Apparatus

A suitable apparatus for the determination is shown diagrammatically in figure 3.

Ordinary laboratory apparatus, including one-mark pipettes complying with the specifications of ISO 648, and

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 $105\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$ over a minimum tube length of 160 mm.

4.3.2 Glass drying tubes and connections, as shown diagrammatically in figure 4.

4.3.3 Drying towers, of capacity 250 ml, one filled with silica gel (4.2.2) and the other packed with desiccant (4.2.1), to dry the stream of nitrogen entering the drying tubes.

4.3.4 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.

4.3.5 Sample boats, of an inert and stable material such as glass, stainless steel or porcelain. Approximate dimensions are $100\text{ mm} \times 20\text{ mm} \times 10\text{ mm}$, and the sample loading shall not exceed $1,5\text{ mg}/\text{mm}^2$. Before use, boats should be dried at approximately $110\text{ }^\circ\text{C}$, then cooled and stored in a desiccator.

4.3.6 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.

4.3.7 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.

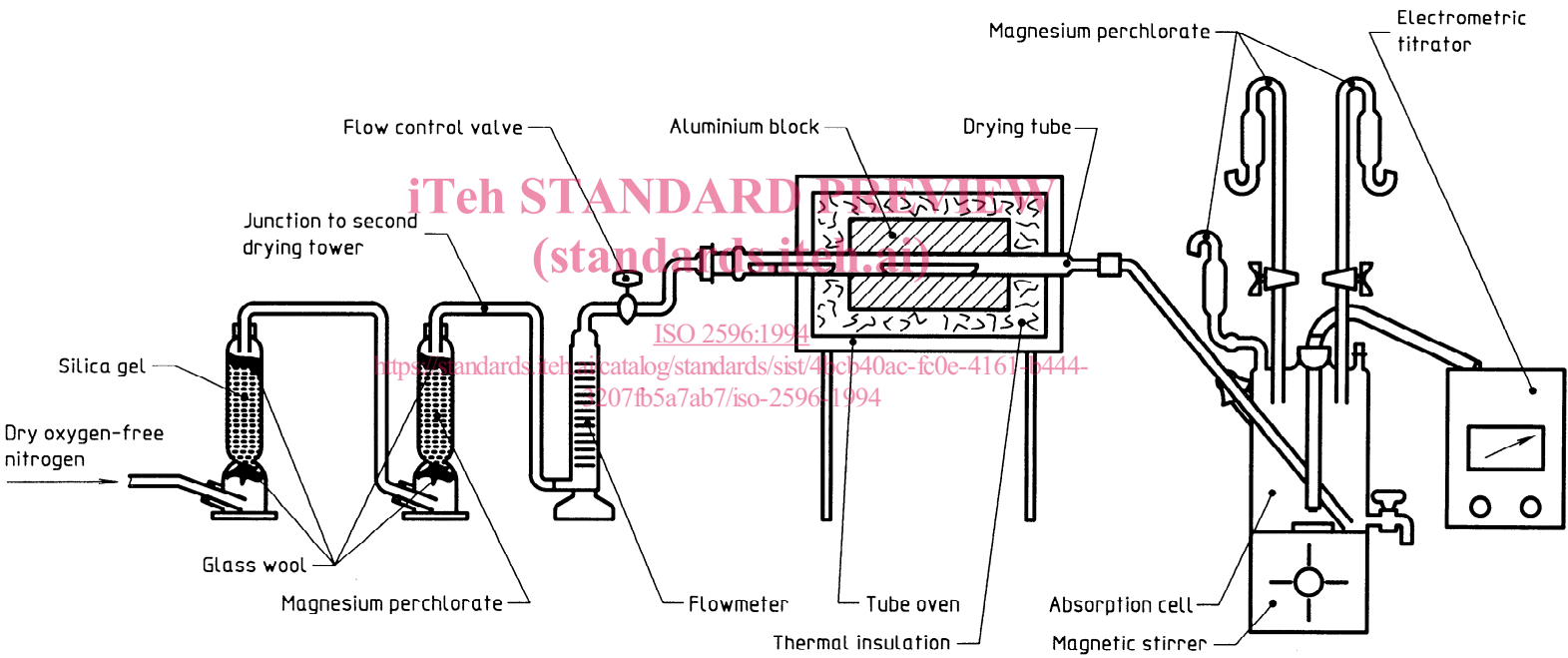


Figure 3 — Apparatus for the determination of hygroscopic moisture — Method 2 (Karl Fischer method)