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**Plastics — Determination of water content**

*Plastiques — Dosage de l'eau*

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Reference number  
ISO 15512:1999(E)

## Contents

1 Scope .....	1
2 Normative references .....	1
3 Method A — Extraction with anhydrous methanol .....	2
4 Method B — Water vaporization.....	4
5 Method C — Manometric method.....	8
6 Test report .....	12

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 15512 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

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# Plastics — Determination of water content

## 1 Scope

1.1 This International Standard specifies methods for the determination of the water content of plastics in the form of granules and finished articles. These methods do not test for water absorption (kinetics and equilibrium) of plastics as measured by ISO 62. The methods are suitable for the determination of water content as low as the following levels:

- Method A 0,1 % or better;
- Method B 0,01 % or better;
- Method C 0,01 % or better.

Water content is an important parameter for processing materials, and should remain below the level specified in the appropriate material standard.

1.2 Three alternative methods are specified in this International Standard.

- a) **Method A** is an extraction method using anhydrous methanol followed by a Karl-Fischer titration of the extracted water. It can be used for all plastics and is applicable to granules having a maximum size of 4 mm × 4 mm × 3 mm.
- b) **Method B** is a vaporization method using heated, dry air or nitrogen gas to evaporate the water, followed by a Karl-Fischer titration of the collected water. It can be used for all plastics and is applicable to granules smaller than 4 mm × 4 mm × 3 mm.
- c) **Method C** is a manometric method. The water content is determined from the increase in pressure which results when the water is evaporated under a vacuum. This method is not applicable to plastic samples containing volatile compounds, other than water, in amounts contributing significantly to the vapour pressure at room temperature. Checks for the presence of large amounts of volatile compounds should be carried out periodically, for example by gas chromatography. Such checks are particularly required for new types or grades of material.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 62, *Plastics — Determination of water absorption*.

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

### 3 Method A — Extraction with anhydrous methanol

#### 3.1 Principle

A test portion is extracted with anhydrous methanol and the extracted water determined by titration using the Karl Fischer method.

#### 3.2 Reagents

During the analysis, use only reagents of recognized analytical grade.

**3.2.1 Methanol**, anhydrous, having a water content less than 0,1 % by mass.

**3.2.2 Karl Fischer reagent**, with an equivalence factor of approximately 3 mg/ml to 5 mg/ml of water. If the reagent is prepared, check its equivalence factor as specified in ISO 760.

#### 3.3 Apparatus

Ordinary laboratory apparatus and the following:

**3.3.1 Glass flasks**, 250 ml capacity, provided with ground-glass or rubber stoppers.

**3.3.2 Conical titration flasks**, 150 ml capacity, with standard ground necks and provided with ground-glass stoppers.

**3.3.3 Reflux condensers**, with ground neck capable of being fitted on to the flasks (3.3.2) and on to the tubes (3.3.4).

**3.3.4 Water-absorption tubes with ground joints**, containing calcium chloride or other drying agent.

**3.3.5 Electrical or hot-air heaters**, for the flasks (3.3.2).

**3.3.6 Pipettes**, 50 ml capacity. (Automatic filling pipettes are acceptable.)

**3.3.7 Woulfe bottles**, with two tubes.

**3.3.8 Curved or U-shaped water-absorption tubes**, filled with calcium chloride.

**3.3.9 Rubber pipette filler**.

**3.3.10 Pipette**, 10 ml capacity.

**3.3.11 Desiccator**, containing calcium chloride.

**3.3.12 Analytical balance**, accurate to 0,2 mg.

**3.3.13 Karl Fischer apparatus**, for determining water content in accordance with ISO 760.

#### 3.4 Preparation of test sample

##### 3.4.1 Granules

Take a representative sample of approximately 100 g. Put the sample into a pre-dried glass flask (3.3.1) and immediately close it with a stopper.

NOTE It is desirable to pre-dry the container in an oven, then cool it over a suitable water absorbent, for instance silica gel.

##### 3.4.2 Finished articles

Cut or saw the sample into pieces of appropriate size, i.e. having a maximum size of 4 mm × 4 mm × 3 mm. Proceed quickly to minimize moisture absorption.

## 3.5 Procedure

### 3.5.1 Precautions

Due to the low quantities of water measured, maximum care shall be exercised at all times to avoid contaminating the sample with water from the sample container, the atmosphere or transfer equipment. Hygroscopic resin samples shall be protected from the atmosphere.

### 3.5.2 Preparation of test portions

Conduct the test on two test portions from the same sample. Use test portions containing 10 mg to 20 mg of water based on the estimated water content of the sample.

### 3.5.3 Determination

**3.5.3.1** Carefully dry the apparatus.

**3.5.3.2** Weigh each test portion to the nearest 1 mg into a conical titration flask (3.3.2) fitted with a ground-glass stopper. Pipette 50 ml (3.3.6) of anhydrous methanol (3.2.1) into the conical flask containing the test portion. At the same time, add 50 ml of anhydrous methanol to another conical flask for a blank test. Stopper the flasks. Keep the stoppered flasks in the desiccator (3.3.11) pending continuation of the test.

**3.5.3.3** Unstopper the flasks and quickly attach them to reflux condensers (3.3.3) fitted with calcium chloride tubes (3.3.4). Reflux the contents of the conical flasks for 3 h, then leave them for 45 min to cool to room temperature. Separate the flasks from the condensers, quickly stopper them and place them in the desiccator.

**3.5.3.4** Use the Karl Fischer apparatus (3.3.13) to titrate the contents of each flask with Karl Fischer reagent (3.2.2).

## 3.6 Expression of results

**3.6.1** The water content  $w$ , expressed as a percentage by mass, for each of the two determinations is given by the following formula:

$$w = \frac{(V_1 - V_2) T}{m} \times 100$$

where:

$V_1$  is the volume, expressed in millilitres, of Karl Fischer reagent used for the determination;

$V_2$  is the volume, expressed in millilitres, of Karl Fischer reagent used for the blank test;

$T$  is the water equivalent, expressed in grams of water per millilitre of reagent, of Karl Fischer reagent;

$m$  is the mass, in grams, of the test portion.

**3.6.2** The two values for the water content shall not differ by more than 10 % relative or 0,02 % absolute, whichever is the greater. If the difference is greater, repeat the measurements until acceptable consecutive values are obtained and discard all unacceptable results.

**3.6.3** The result is expressed as the average of these two determinations, rounded to the nearest 0,01 % by mass.

## 3.7 Precision

The precision of this test method is not known because interlaboratory data are not available. If and when interlaboratory data are obtained, a precision statement will be added at a subsequent revision.

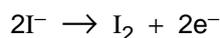
## 4 Method B — Water vaporization

### 4.1 Principle

The sample is weighed then placed in an oven. The water contained in the test portion is vaporized and carried to the titration cell by a dry nitrogen carrier gas. The water is then titrated using the coulometric Karl Fischer method. This method is based on the reduction of iodine by sulfur dioxide in the presence of water to form sulfur trioxide and hydroiodic acid as follows:



Unlike the conventional Karl Fisher reagents that include iodine, the coulometric technique generates the iodine electrolytically from iodide



with 10,71 C of generating current corresponding to 1 mg of water in accordance with Faraday's law.

### 4.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

**4.2.1 Anode solution**, containing iodide ions (to generate iodine in the reaction mixture), pyridine (or another base such as dipyrldylpropane, alkylaminopyridine, imidazole or diethanolamine), sulfur dioxide and methanol (or another suitable organic solvent), prepared in accordance with the equipment manufacturer's instructions.

**4.2.2 Cathode solution**, containing a suitable salt in methanol (or another suitable organic solvent), prepared in accordance with the equipment manufacturer's instructions.

NOTE For the anode (4.2.1) and cathode solutions (4.2.2), hydranal Karl Fischer reagents are recommended, as these reagents do not contain pyridine, are non-toxic, are very stable, and have no offensive odour.

**4.2.3 Neutralization solution**, consisting of approximately 4 mg/ml of water in propylene carbonate, ethylene glycol monomethyl ether, 2-methoxyethanol or methyl cellosolve.

**4.2.4 Silica gel**, in the form of granules of approximately 2 mm diameter, for use as a desiccant.

**4.2.5 SICAPENT<sup>®</sup>1)** molecular sieve or phosphorus pentoxide, for use as a carrier-gas desiccant.

**4.2.6 Grease**, containing little or no water and having low water absorptivity, for lubricating the ground-glass joints to maintain the airtightness of the system.

**4.2.7 Nitrogen gas (N<sub>2</sub>)**, containing less than 5 µg/g of water.

### 4.3 Apparatus

Ordinary laboratory apparatus and the following:

**4.3.1 Coulometric Karl Fischer titrator**, consisting of a control unit, a titration-cell assembly (see Figure 1) (equipped with a generator cell, a cathode solution cell, dual platinum sensing electrodes and a magnetic stirrer). The instrument is designed to coulometrically generate iodine that reacts stoichiometrically with the water present in the cell. The coulombs of electricity required to generate the reagent are converted to micrograms of water and is given as a direct digital readout.

**4.3.2 Water vaporizer**, consisting of an oven capable of heating the sample to at least 300 °C, a heating tube (see Figure 2), a temperature control unit, a carrier-gas flowmeter and carrier-gas drying tubes containing desiccant.

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1) SICAPENT<sup>®</sup> is a trade name of MERCK.

4.3.3 **Microsyringe**, capacity 10 µl, calibrated.

4.3.4 **Sample boat**: aluminium foil may be used to hand fashion a disposable boat large enough to contain the sample and small enough to fit into the heating tube.

4.4 **Preparation of sample**

4.4.1 The test material may be in any form, such as granules, moulding powder, fabricated shapes or moulded items.

4.4.2 Cut fabricated shapes and moulded items to a suitable size. Granules shall be smaller than 4 mm × 4 mm × 3 mm.

4.4.3 Take a representative sample of ≤ 10 g. Due to the small size of the sample, care shall be taken to ensure that it is in fact representative.

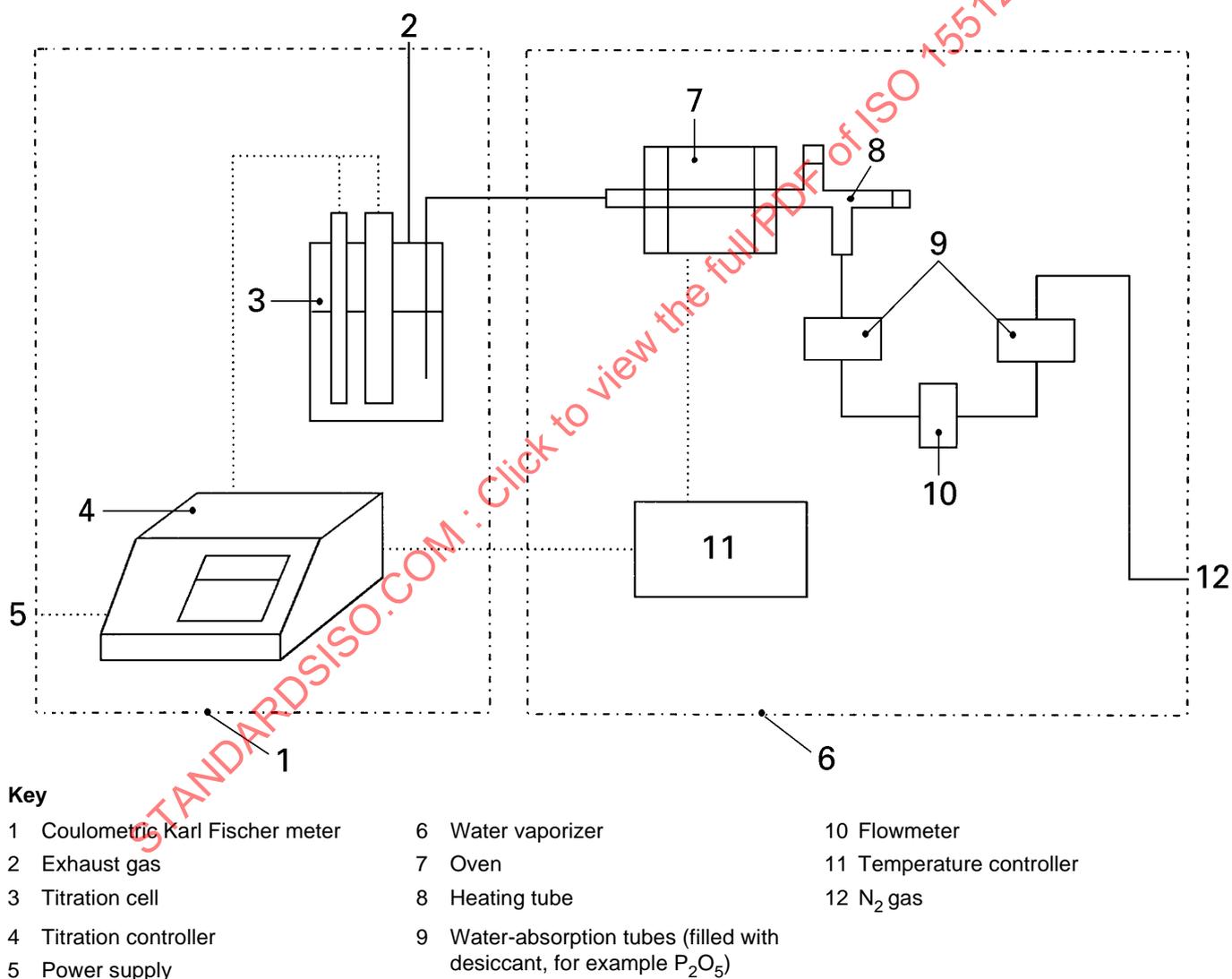
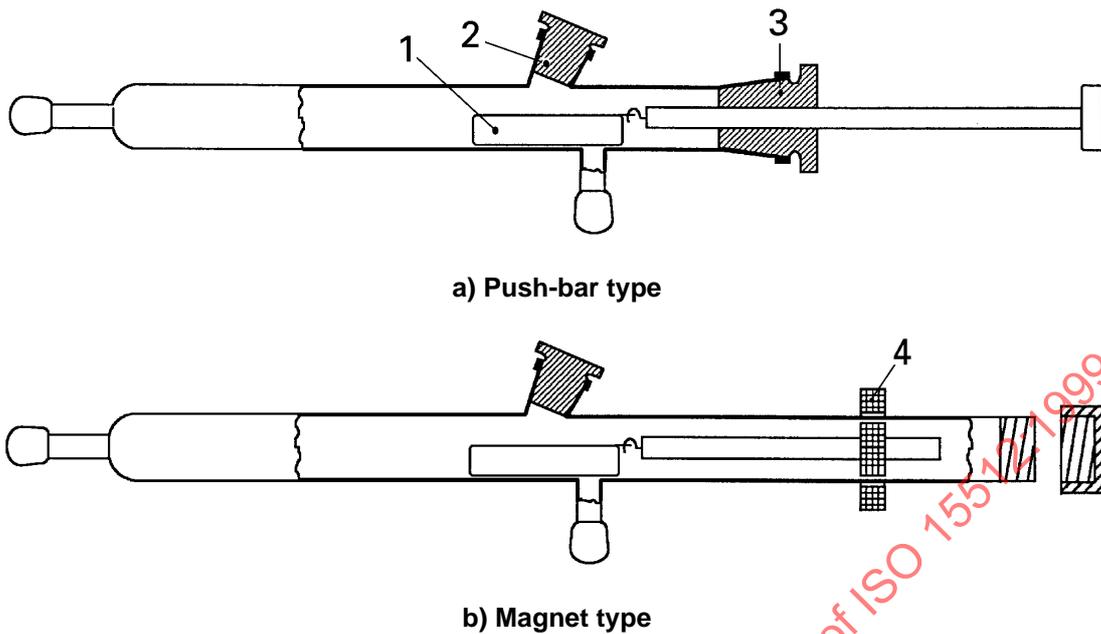


Figure 1 — Flow chart of system used for the determination of water content using method B

**Key**

- |                |                     |
|----------------|---------------------|
| 1 Sample boat  | 3 Sample-boat inlet |
| 2 Sample inlet | 4 Magnet            |

**Figure 2 — Heating tubes****4.5 Procedure****4.5.1 Precautions**

Due to the low quantities of water measured, maximum care shall be exercised at all times to avoid contaminating the sample with water from the sample container, the atmosphere or transfer equipment. Hygroscopic resin samples shall be protected from the atmosphere.

**4.5.2 Preparation of apparatus**

**4.5.2.1** Refer to Figure 1 for the following procedure.

**4.5.2.2** Assemble the water vaporizer (4.3.2) and coulometric Karl Fischer titrator (4.3.1) as directed in the manufacturer's instructions.

**4.5.2.3** Fill the absorption tubes with desiccating agent (see 4.3.2) as directed in the manufacturer's instructions.

**4.5.2.4** Pour approximately 200 ml (adjust for the container size) of the anode solution (4.2.1) into the generator cell and 10 ml of the cathode solution (4.2.2) into the cathode solution cell (see 4.3.1). The surface of the cathode solution shall be below the level of the anode solution to prevent backflow contamination of the cathode solution.

**4.5.2.5** Turn the cell power switch on. If the cell potential shows a negative value, indicating that the anode solution contains an excess of iodine, add 50 µl to 200 µl of the neutralization solution (4.2.3).

**4.5.2.6** Disconnect the tube connecting the vaporizer unit to the titration cell. Set the carrier-gas flow to (200 to 300) ml/min and heat the oven to the desired temperature to remove any residual water from the vaporizer (4.3.2).

**4.5.2.7** Lift the titration cell and agitate the solution by gently swirling the cell to remove any residual water from the walls. Stir the solution for a minute in the TITRATION mode to dry and stabilize the inner atmosphere.

**4.5.2.8** Re-connect the tube from the vaporizer unit to the titration cell. Keep the carrier-gas flow on during the whole titration. The instrument is now ready for sample analysis.

### 4.5.3 Equipment check

**4.5.3.1** To check if the coulometric Karl Fisher titrator is functioning properly, carry out a determination with a known amount of water as follows: With the analyser stabilized and in the READY position, press the start switch and carefully inject 5 µl of water into the cell using a 10 µl syringe (4.3.3). When the END light comes on, record the reading. This reading should be  $(5\ 000 \pm 250)$  µg.

**4.5.3.2** To check if the whole system is functioning properly, carry out a determination with 50 mg of sodium tartarate dihydrate ( $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$ ) at 150 °C. Follow 4.5.3.1 to measure the quantity of water contained in the sodium tartarate dihydrate. The result should be  $(15,6 \pm 0,5)$  %.

### 4.5.4 Determination

**4.5.4.1** Place the sample boat (4.3.4) in the heating tube and push it into the oven to dry and also to backpurge any residual water contained in the sample-boat inlet.

**4.5.4.2** After a few minutes, move the sample boat to the sample inlet port and allow it to cool.

**4.5.4.3** Weigh a test portion of the sample (see 4.4) directly into the sample boat (which will have to be removed from the heating tube for this purpose) or on to a piece of aluminium foil (see note). Appropriate sizes of test portion are given in Table 1.

NOTE If the sample boat is made of glass, or another material which is not designed to be discarded after use, a piece of aluminium foil may be wrapped around the test portion to prevent the test portion from sticking to the boat when it melts. Wrapping the test portion in this way will also prevent it from scattering during introduction into the sample boat.

If the test portion was weighed directly into the sample boat, place the sample boat back in the heating tube as quickly as possible. If the test portion was weighed on to a piece of aluminium foil, wrap it and introduce it, again as quickly as possible, into the sample boat either through the sample inlet or the sample-boat inlet.

Table 1 — Test portion

Expected water content $w$ % by mass	Mass of test portion $m$ g
$w > 1$	$0,2 > m \geq 0,1$
$1 \geq w > 0,5$	$0,4 > m \geq 0,2$
$0,5 \geq w > 0,1$	$1 > m \geq 0,4$
$0,1 \geq w$	$m \geq 1$

**4.5.4.4** Verify that the instrument is in the READY status. Press the START key. Move the sample boat into the oven (see Figure 4). Allow the titration to proceed until the END light comes on, then record the instrument reading in micrograms.

### 4.6 Expression of results

Calculate the water content in the test portion  $w$ , expressed as a percentage by mass, as follows:

$$w = \frac{m_{\text{water}}}{m_{\text{test portion}}} \times 10^{-4}$$

where

$m_{\text{water}}$  is the mass, expressed in micrograms, of water found in the test portion;

$m_{\text{test portion}}$  is the mass, expressed in grams, of the test portion.

## 4.7 Precision

The precision of this test method is not known because interlaboratory data are not available. If and when interlaboratory data are obtained, a precision statement will be added at a subsequent revision.

## 5 Method C — Manometric method

### 5.1 Principle

A test portion is heated to a specified temperature in a closed container under vacuum, thus ensuring complete evaporation of the water. The resulting pressure increase, which is proportional to the water content, is measured. The water content in the sample is calculated using a calibration factor. The calibration factor is obtained by determining the water loss from a hydrate of known water content subjected to the same conditions as the test portion.

### 5.2 Reagent

#### 5.2.1 Sodium molybdate dihydrate ( $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ ), of recognized analytical grade.

NOTE Other hydrates losing their water of crystallization under the conditions of test may be also used, for example barium chloride dihydrate ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ).

### 5.3 Apparatus

Ordinary laboratory apparatus and the following:

#### 5.3.1 Pressure-measurement apparatus: the apparatus shown diagrammatically in Figure 3 is recommended.

The apparatus shown is an all-glass system with vacuum-tight connections, preferably in the form of spherical joints. Bulbs A and B have volumes of  $(0,5 \pm 0,05)$  l and at least 1 l, respectively. The bulbs are connected to a tube (C), which is connected at one end to a high-vacuum gauge (D), and at the other end to a sample tube adapter fitted with a stopcock (E). Tube C carries a connection to a vacuum pump (N) fitted with a stopcock (F) and is fitted with a stopcock (G) to separate the bulbs. On both sides of the stopcock (G) the tube is connected via splash heads (H) and check valves (K) to a U-tube oil manometer (L), the legs of which have a length of at least 350 mm. The sample tube (M) shall be made of heat-resistant glass. The sample tubes in a set shall not differ in volume by more than 5 ml.

NOTE 1 The use of an apparatus of a different design is allowed, provided that the repeatability requirements mentioned in 5.5.4.2 can be met.

NOTE 2 Silicone oil is suitable for filling the manometer.

#### 5.3.2 Heating device, such as an electric oven, suitable for heating the sample tube to the specified temperature.

The arrangement of the equipment should preferably be such as to allow easy installation and removal of the heating device.

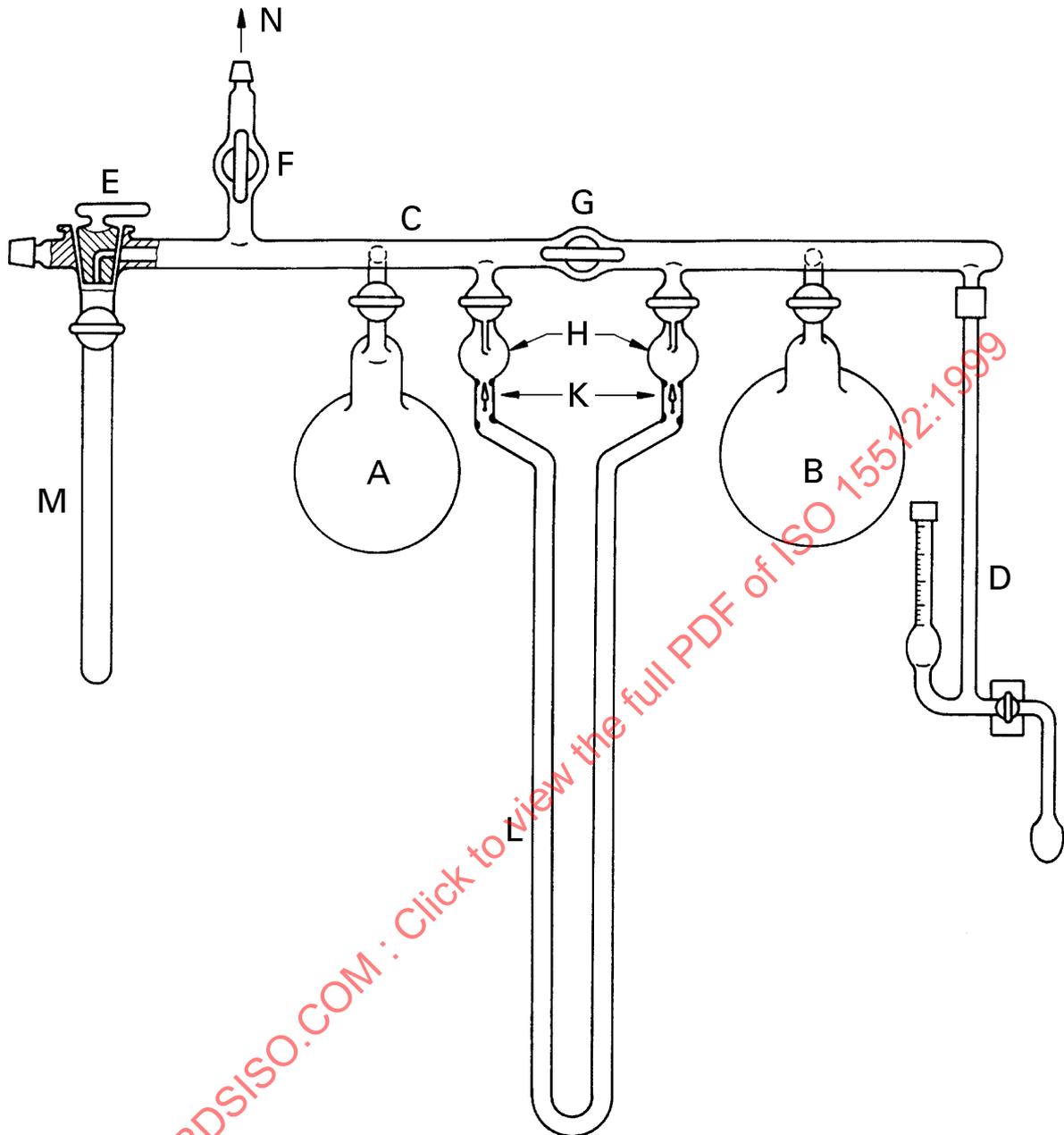
### 5.4 Preparation of sample

#### 5.4.1 Granules

Quickly fill a pre-dried container with a representative sample of the test material and immediately close it to minimize moisture uptake from the atmosphere.

#### 5.4.2 Finished articles

Cut or saw the sample into pieces measuring a few millimetres. Proceed quickly to minimize moisture absorption. Store the sample as specified in 5.4.1.

**Key**A Bulb, volume  $(0,5 \pm 0,05)$  lB Bulb, volume  $\geq 1$  l

C Connecting tube

D High-vacuum gauge

E, F, G Stopcocks

H Splash heads

K Check valves

L Oil manometer

M Sample tube

N To vacuum pump

**Figure 3 — Apparatus for the determination of water content using method C (manometric)**

## 5.5 Procedure

### 5.5.1 Precautions

Due to the low quantities of water measured, maximum care shall be exercised at all times to avoid contaminating the sample with water from the sample container, the atmosphere or transfer equipment. Hygroscopic resin samples shall be protected from the atmosphere.

### 5.5.2 Preparation of test portions

Carry out two determinations. Choose a mass of test portion to obtain a pressure difference of at least 50 mm.

If the volumes of bulbs A and B are  $(0,5 \pm 0,05)$  l and at least 1 l, respectively, the recommended mass of the test portion can be taken from Table 2.

Table 2 — Test portion

Expected water content $w$ % by mass	Mass of test portion $m$ g
$w > 1$	$0,5 > m \geq 0,2$
$1 \geq w > 0,5$	$1 > m \geq 0,5$
$0,5 \geq w > 0,2$	$2,5 > m \geq 1$
$0,2 \geq w > 0,1$	$5 > m \geq 2,5$
$0,1 \geq w$	$m \geq 5$

### 5.5.3 Leakage check

Check the apparatus for leakage as follows: Fix a dry, empty sample tube, which does not need to be heated during the check, to the apparatus. Open stopcocks E, F and G.

Evacuate the system to a pressure of less than 100 Pa and close stopcocks F and G. After 1 h, check that the pressure is still less than 100 Pa and that the pressure difference indicated by the manometer is less than 2 mm of oil. If these requirements are not met, check for leaks and repeat the test.

Carry out checks as frequently as necessary to ensure airtightness during determinations.

NOTE When the oil in the manometer is replaced, it may be necessary to put the apparatus under vacuum for a few hours to remove any contamination from the new oil.

### 5.5.4 Determination

**5.5.4.1** Quickly weigh a test portion (see Table 2), to the nearest 1 mg, into a dry sample tube and fix the tube to the apparatus. Open stopcocks E and G. Turn stopcock F to connect the system with the vacuum pump and open the vacuum manometer (D). Evacuate the system to a pressure of less than 100 Pa. Turn stopcock F to disconnect the apparatus from the vacuum pump. Close stopcock G.

**5.5.4.2** Position the heating device, previously heated to  $(200 \pm 5)$  °C, around the sample tube and heat the tube at this temperature for 50 min, or until the pressure difference indicated by the oil manometer remains constant to within 1 mm for 5 min. (See, however, next paragraph.)

For unknown samples, high moisture contents cannot be excluded. Therefore, constantly observe the manometer for the first 10 min of the test and, if the pressure becomes too high, open stopcock G and repeat the test with a smaller test portion.

After 50 min, or when the pressure difference remains constant, read the pressure difference to the nearest millimetre.