INTERNATIONAL STANDARD

ISO 15512

Second edition
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Plastics — Determination of water content

Plastiques — Dosage de l'eau

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

This second edition cancels and replaces the first edition (ISO 15512:1999), which has been technically revised.

Introduction

The inter-laboratory comparability of water content determinations is often low. Major causes for this are the sample packaging, sample handling and differences between equipment and settings. In order to be able to compare data between two laboratories, special care needs to be taken with sample packaging and sample handling. Samples should e.g. be packed in special glass containers or water barrier sealed bags. Sample handling should preferably be carried out in a dry nitrogen or air environment. To improve the repeatability and reproducibility, the procedure specified in this International Standard should be followed strictly.

The temperature settings for the vaporization method described in this International Standard are not specified in the standard. For the manometric method, a temperature of 200 °C is often used. However, for some condensation materials this might be too high and could e.g. cause generation of water due to a condensation reaction.

The heating temperature should be optimized concerning the material to be tested, the equipment in use and the practical circumstances. If the temperature is too low, the total amount of water in the material to be tested will not be evaporated completely, whereas too high temperatures cause water generation due to effects like degradation and condensation reactions.

In this International Standard, a procedure is included for optimization of the heating temperature in order to choose the correct temperature for the water content determination and to improve the inter-laboratory comparability.

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 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)*

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

Carefully dry the apparatus.

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.

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.

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

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.

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.

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

A test portion 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:

$$I_2 + SO_2 + H_2O \rightarrow 2HI + SO_3$$

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

$$2I^- \rightarrow I_2 + 2e^-$$

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, in accordance with the equipment manufacturer's instructions (for use when a titration cell with a diaphragm is being used).

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 (for use when a titration cell with a diaphragm is being used).

4.2.3 Universal reagent, containing iodide ions to generate iodine in the reaction mixture, prepared in accordance with the equipment manufacturer’s instructions (for use when a titration cell without a diaphragm is being used).

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

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

4.2.6 SICAPENT®[1] molecular sieve or **phosphorus pentoxide**, for use as a carrier-gas desiccant.

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

4.2.8 Nitrogen gas (N_2), containing less than 5 µg/g of water.

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1) SICAPENT® is a trade name of MERCK. It is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

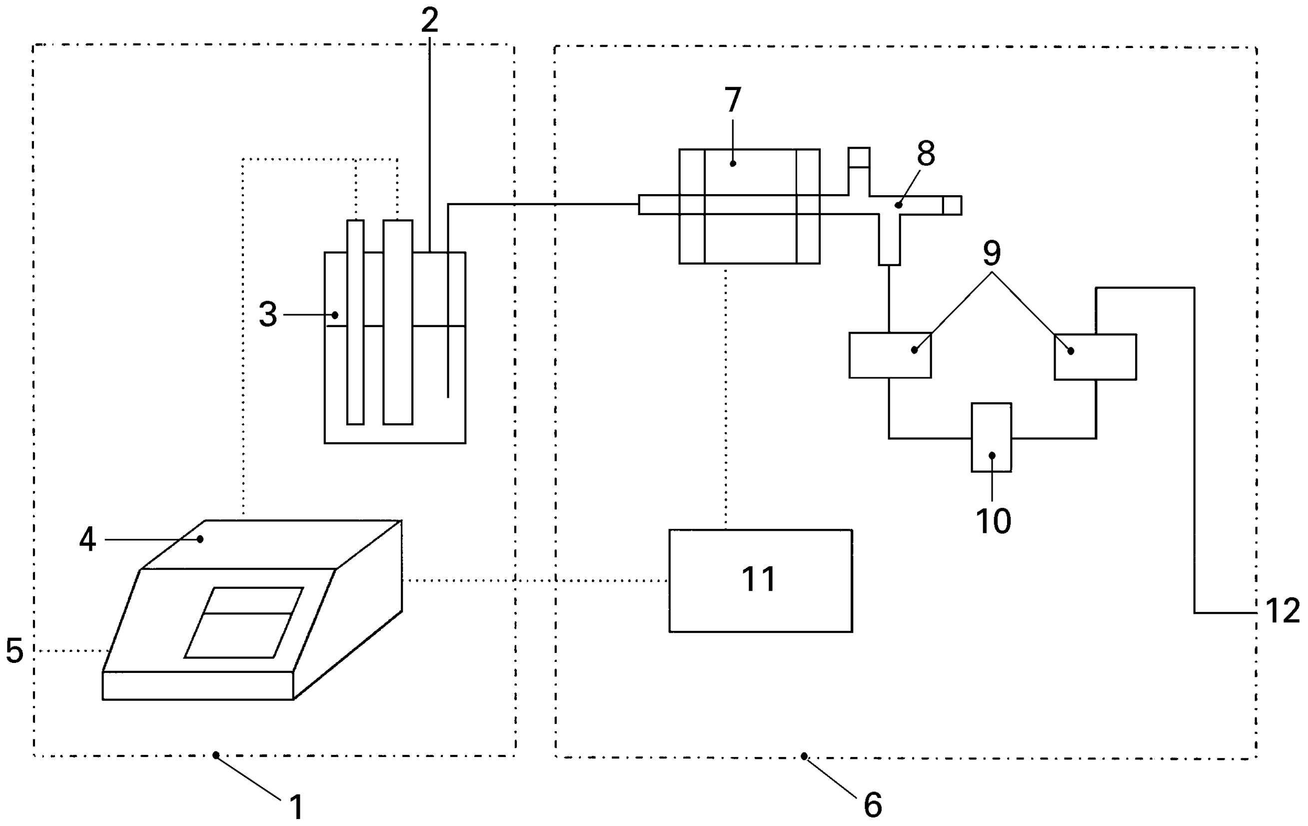
4.3 Apparatus

Ordinary laboratory apparatus and the following:

4.3.1 Coulometric Karl Fischer titrator, consisting of a control unit and a titration-cell assembly equipped with a generator cell with or without a diaphragm, dual platinum sensing electrodes and a magnetic stirrer (see Figure 1). The instrument is designed to coulometrically generate iodine that reacts stoichiometrically with the water present in the cell. The number of coulombs of electricity required to generate the reagent are converted to micrograms of water which is given as a direct digital readout.

A diaphragm-free cell is accurate enough for many applications. However, check with the equipment supplier to verify whether the particular applications for which this method is being used require a diaphragm. A cell with a diaphragm is recommended if the best possible accuracy is required.

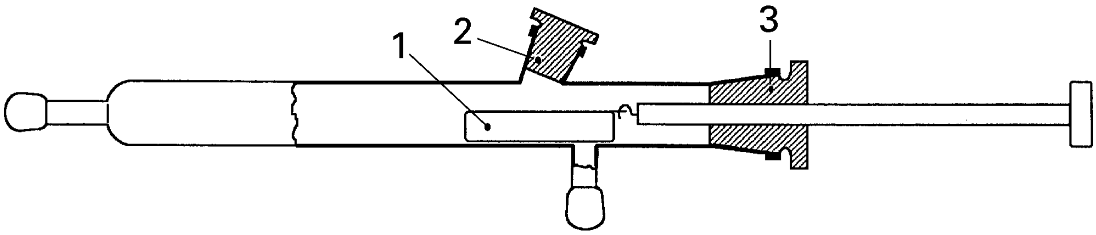
4.3.2 Water vaporizer, consisting of an oven capable of heating the test portion to at least 300 °C, a heating tube, a temperature control unit, a carrier-gas flowmeter and carrier-gas drying tubes containing desiccant (see Figures 1 and 2).



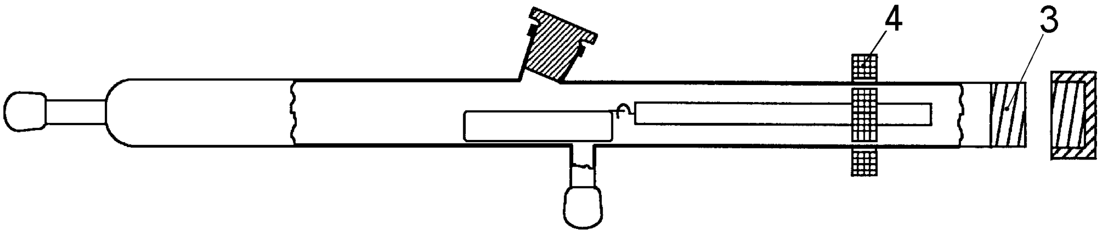
Key

1	coulometric Karl Fischer meter	7	oven
2	exhaust gas	8	heating tube
3	titration cell	9	water-absorption tubes (filled with desiccant, for example P_2O_5)
4	titration controller	10	flowmeter
5	power supply	11	temperature controller
6	water vaporizer	12	N_2 gas

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



a) **Push-bar type**



b) **Magnet type**

Key

1	sample boat	3	sample-boat inlet
2	sample inlet	4	magnet

Figure 2 — Heating tubes

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

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

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

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

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

Refer to Figure 1 for the following procedure.

Assemble the water vaporizer (4.3.2) and coulometric Karl Fischer titrator (4.3.1) as directed in the manufacturer's instructions. Fill the absorption tubes with desiccating agent (see 4.3.2) as directed in the manufacturer's instructions.

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 [or use the universal reagent (4.2.3) if a titration cell without a diaphragm is being used]. The surface of the cathode solution shall be below the level of the anode solution to prevent backflow contamination of the cathode solution.

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 (4.2.4).

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

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.

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 ± 250) µg.

4.5.3.2 To check if the whole system is functioning properly, carry out a determination with 50 mg of sodium tartrate dihydrate ($Na_2C_4H_4O_6 \cdot 2H_2O$) at 150 °C. Follow 4.5.3.1 to measure the quantity of water contained in the sodium tartrate dihydrate. The result should be (15,6 ± 0,5) %.

4.5.4 Determination

Heat the oven to the desired temperature and 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.

For the heating temperature, the user is referred to the material standard. As the heating temperature is also dependent on the equipment used and the practical circumstances, it is recommended that the heating temperature be optimized using the method described in Annex A. If the heating temperature is not mentioned in the material standard or if there is no material standard, then it is also recommended that the method specified in Annex A be used to determine the optimum heating temperature.

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

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 next paragraph). Appropriate sizes of test portion are given in Table 1.

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.

Table 1 — Test portion

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

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.

Verify that the instrument is in the READY status. Press the START button. Move the sample boat into the oven (see Figure 2). 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_{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 ($Na_2MoO_4 \cdot 2H_2O$), of recognized analytical grade.

Other hydrates losing their water of crystallization under the conditions of test may also be used, for example barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$).

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 ± 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 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.

The use of an apparatus of a different design is allowed, provided that the repeatability requirements mentioned in 5.5.4.6 can be met.

NOTE Silicone oil is suitable for filling the manometer.

5.3.2 Heating device, suitable for heating the sample tube to the specified temperature, e.g. an electric oven.

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

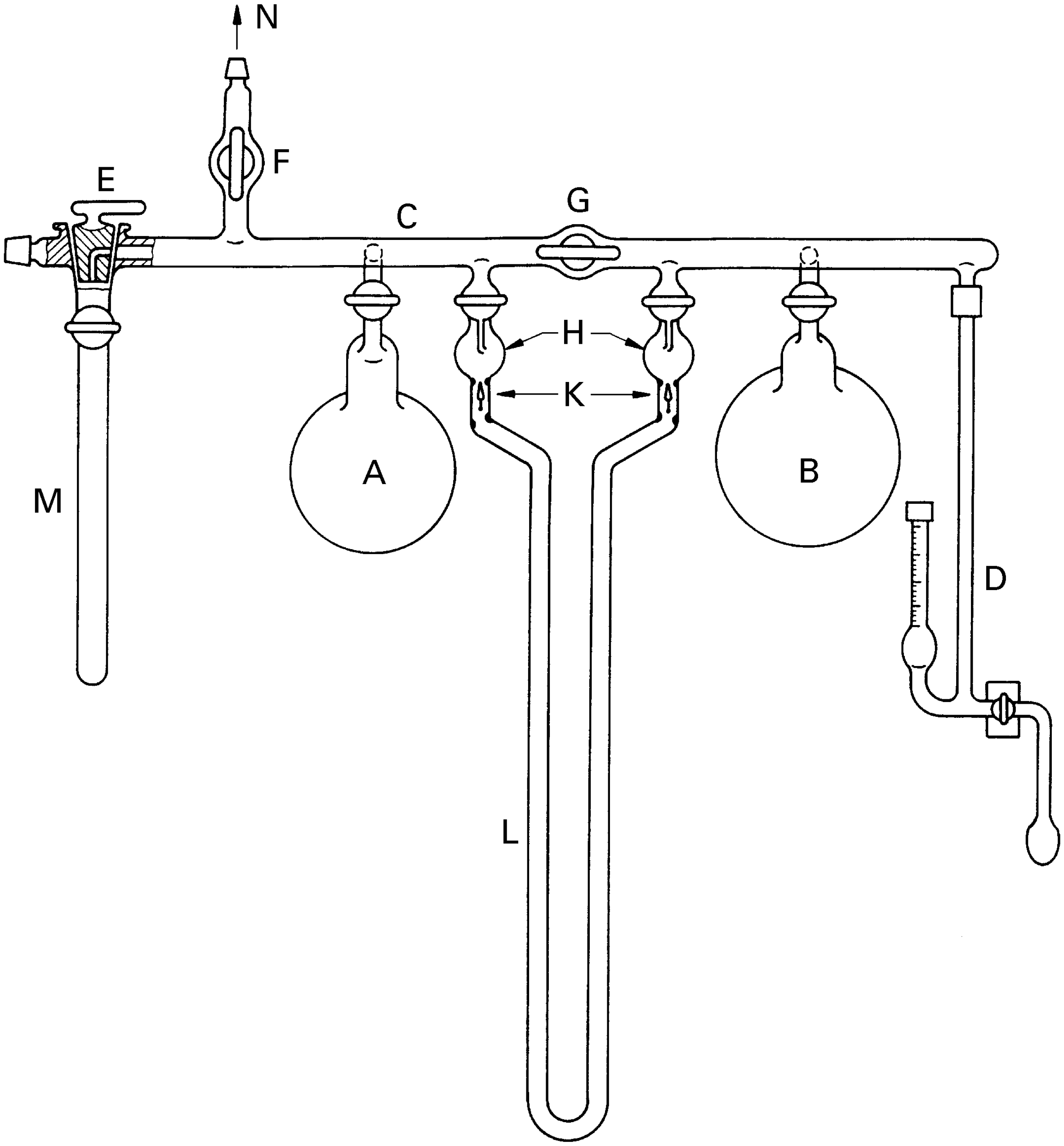
5.4 Preparation of sample

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 ± 0,05) l	E, F, G	stopcocks	M	sample tube
B	bulb, volume ⩾ 1 l	H	splash heads	N	to vacuum pump
C	connecting tube	K	check valves		
D	high-vacuum gauge	L	oil manometer		

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 ± 0,05) l and at least 1 l, respectively, the recommended test portion mass given in Table 2 can be taken.

Table 2 — Test portion

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

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 the temperature specified in the relevant material standard (see, however, 5.5.4.3), 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, 5.5.4.4).

5.5.4.3 A temperature of (200 ± 5) °C is often used for the manometric method. However, the temperature of 200 °C might be too high for some condensation materials. If the temperature is too low, the total amount of water in the material to be tested will not be evaporated, whereas too high temperatures cause water generation due to effects like degradation and condensation reactions. It is recommended that the heating temperature be optimized with regard to the material to be tested, the equipment in use and the practical circumstances using the method described in Annex A.

5.5.4.4 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.

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

Discontinue the heating of the sample tube, open stopcock G and break the vacuum in the sample tube by turning stopcock E.

Allow the sample tube to cool and weigh its contents to the nearest 1 mg.

5.5.4.6 If the results of the two determinations differ by more than 0,005 % by mass, check for leaks (see 5.5.3) and carry out two further determinations.

5.5.5 Calibration

Weigh at least five test portions of sodium molybdate dihydrate, about 30 mg to 40 mg each, and place them in clean, dry sample tubes.

Carry out the procedure specified in 5.5.4.2 to 5.5.4.5 with each portion of sodium molybdate dihydrate. The length of the heating period may be reduced from 50 min to 15 min.

Calculate the calibration factor f, corresponding to the mass of water, in grams, required to produce a pressure difference of 1 mm of oil, using the equation:

$$f = \frac{m_{\text{ref}} \times w_{\text{ref}}}{\Delta p}$$

where

m_{ref} is the mass, in grams, of the test portion of sodium molybdate dihydrate;

w_{ref} is the water content, in grams per gram, of the sodium molybdate dihydrate;

Δp is the pressure difference, in millimetres of oil, indicated by the manometer.

If a hydrate other than sodium molybdate dihydrate is used for the calibration, adapt the mass of the test portions and the value of w accordingly.

Calculate the factor f as the average of the values obtained with the different test portions. In this calculation, disregard results that differ by more than 5 % from the average of the other results.

When a new batch of sodium molybdate dihydrate is used, check its water content by first weighing a test portion, then drying it for 1 h at 200 °C and finally reweighing it.

Do not use water, as such, for the calibration since the amounts required would be too small for weighing with sufficient accuracy.

5.6 Expression of results

The water content w, expressed as a percentage by mass, is given by the formula

$$w = \frac{f \times \Delta p}{m} \times 100$$

where

f is the calibration factor, determined as described in 5.5.5;

Δp is the pressure difference, in millimetres of oil, indicated by the manometer;

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

5.7 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.

6 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the sample;

c) the method used (A, B or C);

d) the heating temperature;

e) the individual values and the average value of the two determinations, to the nearest 0,01 % by mass;

f) if method B was used, details of the equipment and equipment settings used;

g) the date of the test.

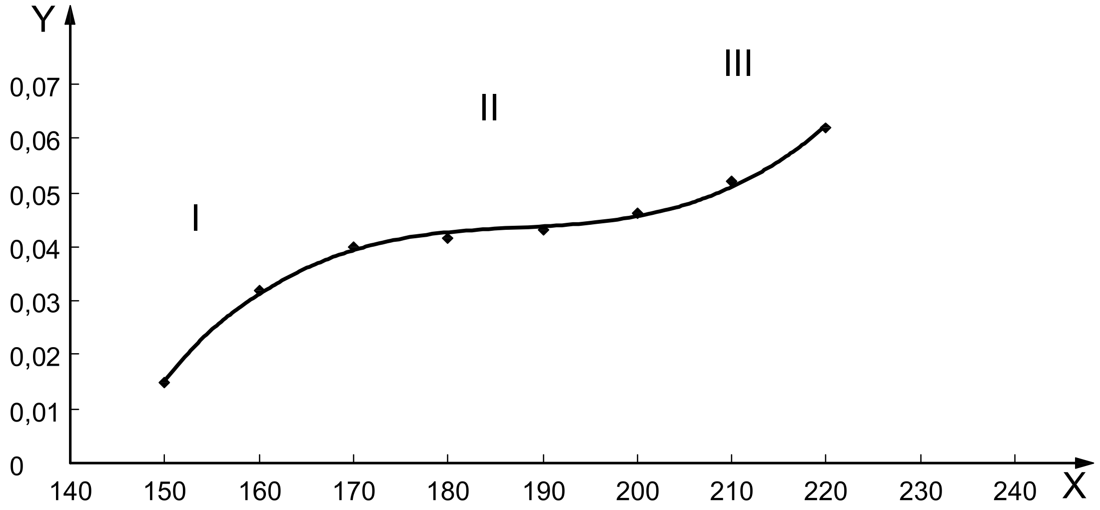
Annex A
(informative)

Selection of the optimum heating temperature for the water content determination

A.1 Procedure

Select the optimum heating temperature for the material to be tested by carrying out water-content determinations at several temperatures. The steps between the different temperatures need to be selected in such a way that a curve can be plotted as shown in Figure A.1. A temperature range from approximately 120 °C to 220 °C is recommended for determination of the optimum heating temperature in maximum steps of 20 °C.

Supplemental solution-viscosity experiments can be carried out to confirm either the absence of water generation or the occurrence of water-generating reactions.



Key

X temperature, °C

Y moisture content, %

I temperature too low

II optimum heating temperature

III temperature too high

Figure A.1 — Selection of optimum heating temperature

A.2 Interpretation of results

In region I, the water in the sample is not vaporized completely and increases in proportion to the temperature increase.

In region II, the water content approaches a (nearly) constant level. The temperature region in which the (nearly) constant water-content value is achieved is considered as the optimum heating temperature for the material under investigation and the practical experimental circumstances. The absence of water-generating reactions can be confirmed by solution-viscosity measurements on the material before and after the water determination (ISO 307, ISO 1628).

In region III, the water content appears to increase. The higher water content is caused by the generation of water due to effects like thermal degradation and condensation reactions at these higher temperatures.

Bibliography

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