INTERNATIONAL STANDARD

ISO
960
First edition
1988-08-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Polyamides (PA) — Determination of water content

Plastiques — Polyamides (PA) — Détermination de la teneur en eau

Reference number
ISO 960 : 1988 (E)

Foreword

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

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 960 was prepared by Technical Committee ISO/TC 61, *Plastics.*

It cancels and replaces ISO Recommendation R 960 : 1969, of which it constitutes a technical revision.

Printed in Switzerland

Plastics — Polyamides (PA) — Determination of water content

1 Scope

1.1 This International Standard specifies methods for the determination of the water content of polyamides and copolyamides as granules and finished articles. The methods are applicable for the determination of water contents down to 0,01 % (m/m). The water content is of importance in connection with the processing of PA, during which it should have a low value to prevent degradation, and for checking the moisture content of test specimens and finished articles.

1.2 Four alternative methods are specified in this International Standard.

Method A is an extraction method using anhydrous methanol and titration of the water by the Karl Fischer method. It is applicable to granules having a maximum size of 4 mm × 4 mm × 3 mm and can be used for all polyamides and copolyamides.

Method B is a method of extraction by melting under vacuum and titration of the water by the Karl Fischer method. It may be applied to granules or pieces of mouldings but not to fine powders (particle size less than 400 µm). For low water content (less than 0,1 % (m/m) and precise determination, it is necessary to make a correction for the water of polycondensation[1]. This method is not recommended when, during the determination, due to a change in molecular mass, additional water is formed or existing water is bound, except when appropriate correction can be made.

Method C is a method of extraction by dissolving in a mixture of 3-methylphenol and toluene and titration of the water by the Karl Fischer method. It yields results close to those obtained by method A. It has the advantage of being applicable to granules and powders of all sizes and to finished articles with little or no reduction in size.

NOTE — The three methods A, B and C differ in the procedure for isolating the water, which is determined in all three cases by the Karl Fischer method.

Method D is a manometric method. The water content is determined from the pressure increase after evaporation of the water under vacuum. This method cannot be used for polyamides polymerized with strong acids.

1.3 Method C is the reference method.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 3146 : 1985, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers.*

3 Method A — Extraction with anhydrous methanol

3.1 Principle

Extraction of a test portion with anhydrous methanol and determination of the extracted water by 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 % (m/m).

3.2.2 Karl Fischer reagent (see 6.2).

3.3 Apparatus

Ordinary laboratory apparatus and

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

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[1] See KLINE, G.M., Analytical Chemistry of Polymers. *Intersc. Pub.,* Part 1, p. 282 (1958).

3.3.2 Conical titration flasks, of capacity 150 ml, 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 Tubes, straight, containing calcium chloride, or other means of drying, with ground joints.

3.3.5 Means for heating the flasks (3.3.2), electrically, by hot air, or by any other suitable means.

3.3.6 Automatically filling pipettes, of capacity 50 ml (see figure 1).

3.3.7 Woolf bottles, with two tubes.

3.3.8 Calcium chloride tubes, curved or U-shaped.

3.3.9 Rubber bulb.

3.3.10 Pipette, of capacity 10 ml.

3.3.11 Desiccator, containing calcium chloride or another suitable drying agent.

3.3.12 Balance, accurate to 0,2 mg.

3.3.13 Apparatus for determining water content by the Karl Fischer method, in accordance with ISO 760.

3.4 Preparation of test sample

3.4.1 Granules

Place a representative test sample of approximately 100 g in a perfectly dry glass flask (3.3.1) and immediately close the flask with either a ground glass or a rubber stopper.

NOTE — It is desirable to predry the container in an oven and to cool it above a suitable water absorbent, for example silica gel.

3.4.2 Finished articles

Cut or saw the test sample into pieces of a few millimetres dimensions. Proceed quickly to minimize moisture absorption. Store the test sample as specified in 3.4.1.

3.5 Number of tests

Carry out two determinations for each sample. Use test portions of 10 to 15 g, depending on the estimated water content.

3.6 Procedure

3.6.1 Carefully dry the apparatus.

3.6.2 Weigh the test portion to the nearest 1 mg into a conical titration flask fitted with a ground glass stopper (3.3.2); let m be the mass, in grams, of this test portion. Measure 50 ml of anhydrous methanol (3.2.1) with an automatically filling pipette (3.3.6) and add it to 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.

3.6.3 Unstopper the flasks and quickly attach them to the reflux condensers (3.3.3) fitted on top with the calcium chloride tubes (3.3.4). Boil the contents of the conical flasks under reflux for 3 h and leave for 45 minutes to cool to room temperature. Separate the flasks from the condensers, quickly stopper them and place them in the desiccator.

3.6.4 Titrate the contents of each flask with the Karl Fischer reagent as indicated in clause 6.

4 Method B — Extraction by melting under vacuum

4.1 Principle

Melting of a test portion under vacuum at a temperature approximately 30 °C above the melting point of the resin and determination of the amount of water collected by the Karl Fischer method.

4.2 Reagents

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

4.2.1 Methanol, anhydrous, having a water content less than 0,1 % (m/m).

4.2.2 Karl Fischer reagent (see 6.2).

4.3 Apparatus

Ordinary laboratory apparatus, and

4.3.1 Vacuum melting unit, capable of maintaining a pressure of less than 500 Pa*). A four-directional unit, given as an example (see figure 2), comprises the following parts:

4.3.1.1 Heating block, made up of an insulated aluminium cylinder equipped with an electric heating collar having approximately 800 W power and 160 mm height. The temperature of the cyl-

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*) 500 Pa = 5 mbar ≈ 4 mmHg

inder is adjusted to approximately 30 °C above the melting point of the resin as determined by the method specified in ISO 3146, and maintained within ± 5 °C. This cylinder, which is 200 mm in height and 160 mm in diameter, has a central hole for insertion of a thermometer (or other means of measuring the temperature), and four holes 170 mm deep, 25 mm in diameter and spaced at regular intervals in relation to the vertical axis of the cylinder.

4.3.1.2 Four traps (P), in 6 mm × 8 mm U-shaped glass tubes in accordance with figure 2. These parts are inserted in a 1-litre Dewar flask containing a mixture of methanol or acetone and carbon dioxide ice (making it possible to obtain a temperature of approximately −78 °C).

4.3.1.3 Rubber vacuum tubing.

4.3.1.4 Two glass stopcocks (R_1) and (R_2), one for each direction.

4.3.1.5 Four heat-proof glass tubes (A), 22 mm in diameter, 220 mm in overall length (tube plus ground portion) and equipped with 24/40 ground-glass sockets, to contain the test tubes and to be inserted in the four holes provided in the cylinder.

4.3.1.6 Four heat-proof glass fittings (B), equipped with 24/40 ground-glass cones.

4.3.2 Four glass test tubes, 16 mm in diameter and 160 mm long, which contain the test portions.

4.3.3 Apparatus for producing a pressure less than 500 Pa in the traps.

4.3.4 Manometer, permitting the measurement of the pressure stated in 4.3.3.

4.3.5 Glass flasks, of capacity 250 ml, provided with ground glass or rubber stoppers.

4.3.6 Conical titration flasks, of capacity 150 ml, with standard ground necks and provided with ground glass stoppers.

4.3.7 Automatic burette, of capacity 10 ml.

4.3.8 Hot-air oven, capable of being maintained at a temperature of at least 100 °C.

4.3.9 Desiccators, capable of containing all of the glassware needed in the determination.

4.3.10 Balance, accurate to 0,2 mg.

4.3.11 Apparatus for determining water content by the Karl Fischer method, in accordance with ISO 760.

4.4 Preparation of test sample

4.4.1 Granules

Place a representative test sample of approximately 100 g in a perfectly dry glass flask (4.3.5) and close the flask immediately with either a ground glass stopper or a rubber stopper.

NOTE — It is desirable to predry the container in an oven and to cool it above a suitable water absorbent, for example silica gel.

4.4.2 Finished articles

Cut or saw the test sample into pieces of a few millimetres dimensions. Proceed quickly to minimize moisture absorption. Store the test sample as specified in 4.4.1.

4.5 Number of tests

Carry out two determinations for each sample. Use test portions of

- 10 g if the expected water content is less than 0,5 % (m/m);
- 2 g if the expected water content is greater than 0,5 % (m/m).

4.6 Procedure

4.6.1 Depending on the vacuum melting unit (4.3.1) available, one or several determinations may be carried out simultaneously. In the case of the apparatus described as an example in 4.3, follow the procedure indicated below. For any other apparatus, follow this procedure, taking the same general precautions.

4.6.2 Dry the glass accessories required for the determination in the oven (4.3.8) and place them in the desiccators (4.3.9) to cool.

4.6.3 Weigh the test portion, to the nearest 1 mg, into a weighed test tube (4.3.2); let m be the mass, in grams, of this test portion. Seal the test tube with a completely dry rubber stopper and replace it in the desiccator.

4.6.4 Place the trap (P) in the tubing and insert it in the Dewar flask. Evacuate the trap to a pressure of less than 500 Pa by attaching the vacuum apparatus (4.3.3) to the end of the tubing and opening stopcock (R_2), stopcock (R_1) being closed. Wait 5 min to allow the trap to reach a temperature of about −78 °C. Place the test tube, after removing its stopper, in a ground tube (A). Close the ground tube (A) with a glass fitting (B) and join to the trap by rubber vacuum tubing and stopcock (R_1). Open stopcock (R_1) and check that the pressure is less than 500 Pa by reading the manometer (4.3.4).

4.6.5 Place the ground tube for 30 min in the heating block, previously brought to the required temperature (about 30 °C above the melting point as determined by the method specified in ISO 3146), while the vacuum apparatus is continuously functioning. Close stopcocks (R_2) and (R_1) and shut off the vacuum apparatus. Remove the ground tube from the heating block and from the tubing. Remove the trap from the Dewar flask and leave it to regain ambient temperature while keeping the two stopcocks (R_1) and (R_2) closed. Carefully dry the outside of the trap and disconnect the vacuum tube.

4.6.6 Insert one of the ends of the trap in a titration flask (4.3.6) and add 10 ml of the anhydrous methanol (4.2.1) to the other end by means of the automatic burette (4.3.7). Evacuation shall be quickly carried out by continuously rinsing the water in the trap while taking care to scour all the sides of the tube with methanol. Remove the trap from the flask and stopper the flask immediately. Titrate the contents of the flask with the Karl Fischer reagent, as indicated in clause 6.

4.6.7 Carry out a blank test on a further 10 ml of anhydrous methanol (4.2.1).

5 Method C — Extraction by dissolution in a mixture of 3-methylphenol and toluene

5.1 Principle

Dissolution of a test portion in a boiling mixture of 3-methylphenol and toluene and determination of the water in the solution by the Karl Fischer method.

NOTE — Addition of toluene to the 3-methylphenol lowers the boiling point and prevents oxidation.

5.2 Reagents

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

5.2.1 3-methylphenol, anhydrous, water content less than 0,015 % (*m*/*m*).

5.2.2 Toluene, anhydrous, water content less than 0,005 % (*m*/*m*).

5.2.3 Methanol, anhydrous, water content less than 0,05 % (*m*/*m*).

5.2.4 Karl Fischer reagent (see 6.2).

5.3 Apparatus

Ordinary laboratory apparatus and

5.3.1 Glass flasks, of capacity 250 ml, provided with ground glass or rubber stoppers.

5.3.2 Conical titration flask, of capacity 250 ml, with standard ground neck and provided with a ground glass stopper.

5.3.3 Reflux condenser, with ground neck, capable of being fitted on the flask (5.3.2) and on to the tube (5.3.4).

5.3.4 Tube, containing silica gel, anhydrous calcium chloride, or other effective means of drying.

5.3.5 Means of heating, electrically, by hot air, or by any other suitable means.

5.3.6 Graduated burettes, of capacity 100 ml.

5.3.7 Pipette, of capacity 10 ml.

5.3.8 Balance, accurate to 0,2 mg.

5.3.9 Fragmented pumice stone.

5.3.10 Apparatus for determining water content by the Karl Fischer method, in accordance with ISO 760.

5.4 Preparation of test sample

Place a representative test sample of approximately 100 g in a perfectly dry glass flask (5.3.1). Close the flask immediately with either a ground glass or a rubber stopper.

NOTE — It is desirable to predry the flask in an oven and to cool it above a suitable water absorbent, for example silica gel.

5.5 Number of tests

Two determinations shall be carried out for each sample. Test portions of 2 to 10 g shall be used, depending on the estimated water content.

5.6 Procedure

5.6.1 Carefully dry the apparatus.

5.6.2 Weigh, to the nearest 1 mg, the test portion in a conical titration flask (5.3.2). Let m be the mass, in grams, of this test portion. Pour rapidly into the flask, using graduated burettes (5.3.6), 90 ml of anhydrous 3-methylphenol (5.2.1) and 60 ml of anhydrous toluene (5.2.2), and add the fragmented pumice stone (5.3.9). Connect the flask to the reflux condenser (5.3.3), previously rinsed with anhydrous methanol (5.2.3) and sealed off at the upper end against the humidity in the atmosphere by means of a tube containing, for example, calcium chloride. Heat the conical titration flask until the test portion is completely dissolved in the boiling solvent mixture; this requires 30 min to 2 h. Allow the apparatus to cool to ambient temperature and rinse the condenser at the top with 10 ml of anhydrous methanol (5.2.3), measured by means of the pipette (5.3.7). Disconnect the titration flask and seal immediately. Titrate the contents of the flask with the Karl Fischer reagent, as indicated in clause 6.

5.6.3 Carry out a blank test on volumes of 3-methylphenol, toluene and methanol equal to the volumes used in 5.6.2.

6 Determination of water content by the Karl Fischer method

6.1 Principle

Determination of the total quantity of water extracted by one of the methods A, B or C by titration with Karl Fischer reagent in accordance with ISO 760.

6.2 Reagent

Commercial Karl Fischer reagent, with an equivalence factor of approximately 5 mgH_2O/ml. Alternatively, prepare the reagent and check its equivalence factor as specified in ISO 760.

6.3 Determination

Carry out the titration with the test solutions and the blanks obtained in 3.6, 4.6 or 5.6.

6.4 Expression of results

6.4.1 The water content for each of the two determinations, expressed as a percentage by mass, is given by the following formula

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

where

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

V_2 is the volume, in milllitres, of Karl Fischer reagent used for the blank test;

T is the water equivalent of the Karl Fischer reagent expressed in grams of water per millilitre of reagent (see 6.2);

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

6.4.2 The two values for the water content shall not differ by more than 10 % relative or 0,02 % absolute, whichever is the greater. If they do, repeat the measurements until two consecutive values corresponding to these conditions are obtained and discard all unacceptable results.

6.4.3 The result shall be expressed as the average of these two determinations, rounded to the nearest 0,01 % (*m*/*m*).

7 Method D — Manometric method

7.1 Principle

Heating of a test portion to a specified temperature in a closed space under vacuum, thus ensuring complete evaporation of the water. Measurement of the resulting pressure increase, which is proportional to the water content. Calculation of the water content is by reference to a calibration factor, calculated by using a hydrate with a known water content, which loses its water under the conditions of the test.

7.2 Reagent

Sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$), of recognized analytical grade.

NOTE — Other hydrates that lose their water of crystallization under the conditions of test may be also used, for example barium chloride dihydrate ($BaCl_2.2H_2O$).

7.3 Apparatus

Ordinary laboratory apparatus and

7.3.1 Measuring apparatus

The apparatus shown diagrammatically in figure 3 is recommended. It 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 litre ± 0,05 litre and at least 1 litre 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). The tube (C) carries a connection to a vacuum pump 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.

NOTES

1 The use of an apparatus of a different design is allowed, provided that the repeatability requirements mentioned in 7.7.2 can be met.

2 Silicone oil is suitable for filling the manometer.

7.3.2 Heating device

An electric oven or any suitable device may be used to heat the sample tube to the specific temperature. The arrangement of the equipment should preferably be such as to allow easy installation and removal of the heating device.

7.4 Preparation of test sample

7.4.1 Granules

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

NOTE — It is desirable to predry the container in an oven and to cool it above a suitable water absorbent, for example silica gel.

7.4.2 Finished articles

Cut or saw the test sample into pieces of a few millimetres dimensions. Proceed quickly to minimize moisture absorption. Store the test sample as specified in 7.4.1.

7.5 Number of tests

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

NOTE — If the volumes of bulbs (A) and (B) are 0,5 litre ± 0,05 litre and at least 1 litre respectively, the recommended mass of the test portion can be taken from table 1.

Table 1

Expected water content, w % (m/m)	Mass of test portion, m g
$w > 1$	$0{,}5 > m > 0{,}2$
$1 > w > 0{,}5$	$1 > m > 0{,}5$
$0{,}5 > w > 0{,}2$	$2{,}5 > m > 1$
$0{,}2 > w > 0{,}1$	$4 > m > 2{,}5$
$0{,}1 > w$	$m > 5$

7.6 Procedure

7.6.1 Leakage check

Fix a dry, empty sample tube, which does not need to be heated during the check, to the apparatus. Turn stopcock (E) to connect the sample tube (M) with the vacuum pump, turn stopcock (F) to connect the tube (C) with the vacuum pump and turn stopcock (G) to connect bulbs (A) and (B) with the vacuum pump.

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 air-tightness during the determinations.

NOTE — When the oil in the manometer is replaced, evacuation of the apparatus for a few hours may be required for de-aeration of the new oil.

7.6.2 Determination

7.6.2.1 Quickly weigh into a dry sample tube, to the nearest 1 mg, a test portion as indicated in table 1 in 7.5 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. 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).

7.6.2.2 Heat the oven (7.3.2) to the test temperature. For PA6, PA66, PA69, PA610 and PA612, the test temperature shall be 195 °C ± 5 °C; for PA11 and PA12, it shall be 170 °C ± 5 °C.

7.6.2.3 Position the oven around the sample tube and heat the tube until the pressure difference indicated by the oil manometer remains constant to the nearest 1 mm for 5 min. Record the pressure difference, in millimetres, indicated by the oil manometer. Discontinue the heating of the sample tube, open stopcock (G) and break the vacuum in the sample tube by turning stopcock (E) to link the sample tube with the atmosphere.

When testing powder materials, open the stopcock for evacuation slowly. It is recommended that the test portion in the tube be covered by a thin layer of glass wool. Predry the glass wool in an oven, cool and store it above a suitable water absorbent, such as silica gel.

NOTE — For unknown samples, high moisture contents cannot be excluded. Therefore, the manometer should be frequently observed in the initial stage of the determination. If the pressure becomes too high, open stopcock (G) and repeat the test with a smaller test portion.

7.6.2.4 If the results differ by more than the value specified in 7.7.2, check for leaks (see 7.6.1) and carry out two further determinations.

7.6.3 Calibration

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

7.6.3.1 Weigh at least five test portions of sodium molybdate dihydrate, ranging in mass from about 30 to 40 mg, and place them in clean, dry sample tubes. Carry out the procedure specified in 7.6.2 at 200 °C ± 5 °C with each test portion of sodium molybdate dihydrate (7.2).

When a new batch of sodium molybdate dihydrate is used, check the water content by weighing, drying for 1 h at 200 °C and reweighing.

7.6.3.2 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 formula

$$\frac{m_1 w_1}{\Delta p}$$

where

m_1 is the mass, in grams, of the test portion of sodium molybdate dihydrate;

w_1 is the water content, in grams per gram, of sodium molybdate dihydrate;

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

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*) 100 Pa = 1 mbar ≈ 0,7 mmHg

If a hydrate other than sodium molybdate dihydrate is used for the calibration, adjust the mass of the test portions and the value of w_1 accordingly.

7.6.3.3 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.

7.7 Expression of results

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

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

where

f is the calibration factor, determined as described in 7.6.3;

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

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

7.7.2 The result shall be expressed as the average of two determinations, rounded to the nearest 0,01 % (m/m). The two individual results shall not differ by more than 0,02 % (m/m) in absolute value or by more than 10 % relatively, whichever is greater.

8 Test report

The test report shall include the following particulars:

a) reference to this International Standard;

b) complete identification of the test sample;

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

d) the individual values and the average value of the two determinations, to the nearest 0,01 % (m/m).

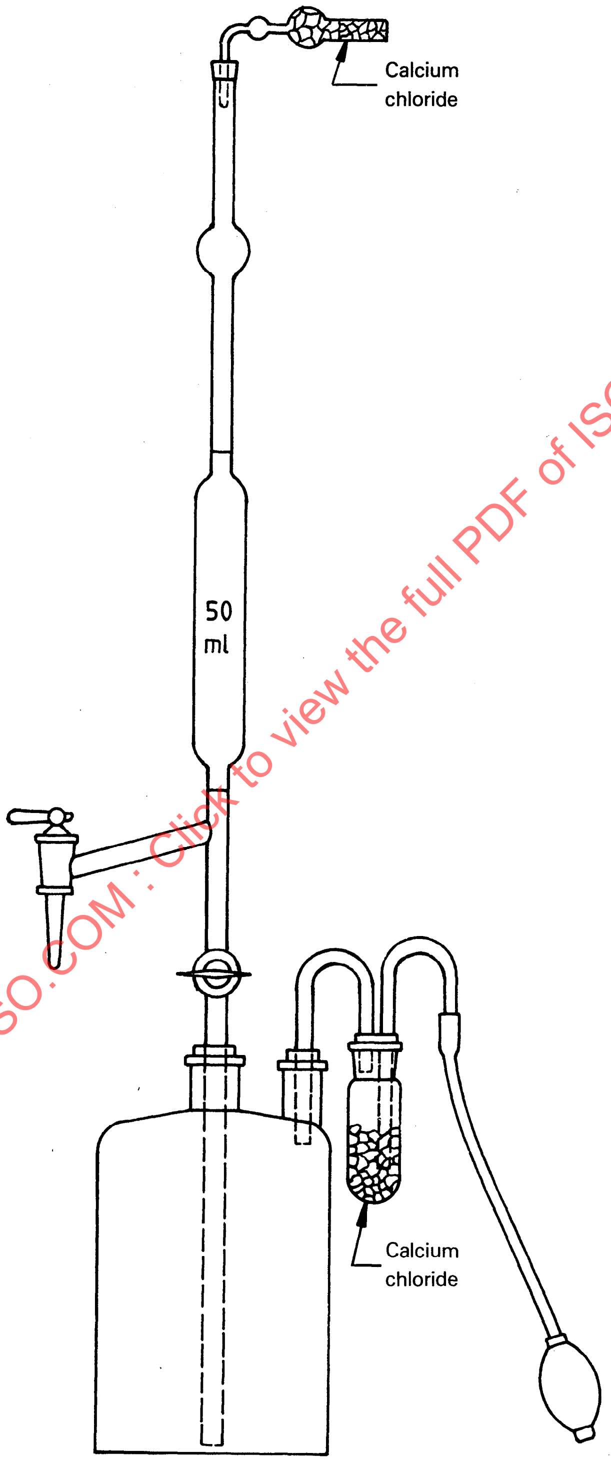


Figure 1 — Automatically filling pipette for method A (extraction with anhydrous methanol)

Dimensions in millimetres

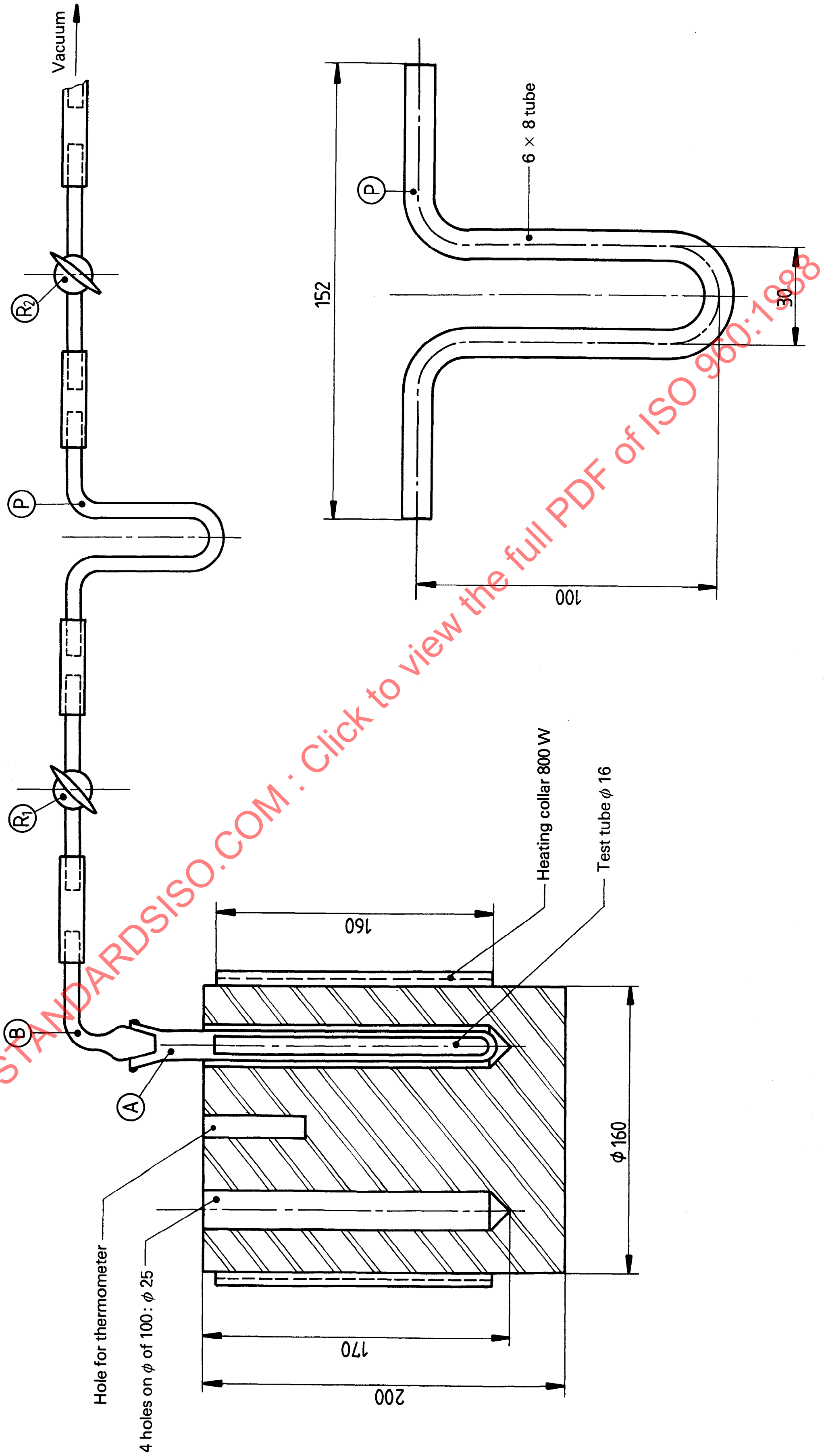
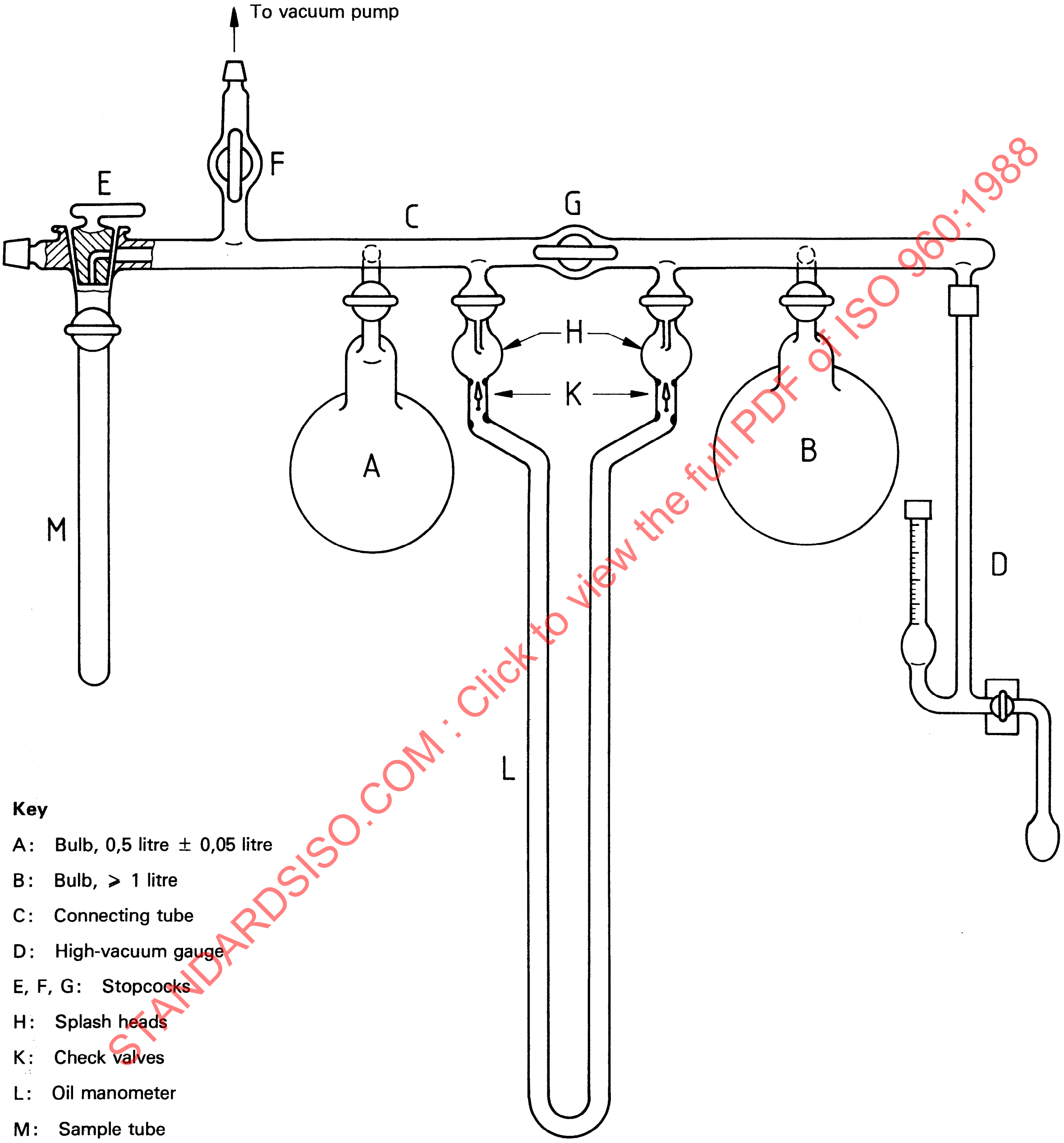


Figure 2 — Apparatus for determination of water content by method B (vacuum melting)



Key

A: Bulb, 0,5 litre ± 0,05 litre

B: Bulb, ⩾ 1 litre

C: Connecting tube

D: High-vacuum gauge

E, F, G: Stopcocks

H: Splash heads

K: Check valves

L: Oil manometer

M: Sample tube

Figure 3 — Apparatus for determination of water content by method D (manometric)