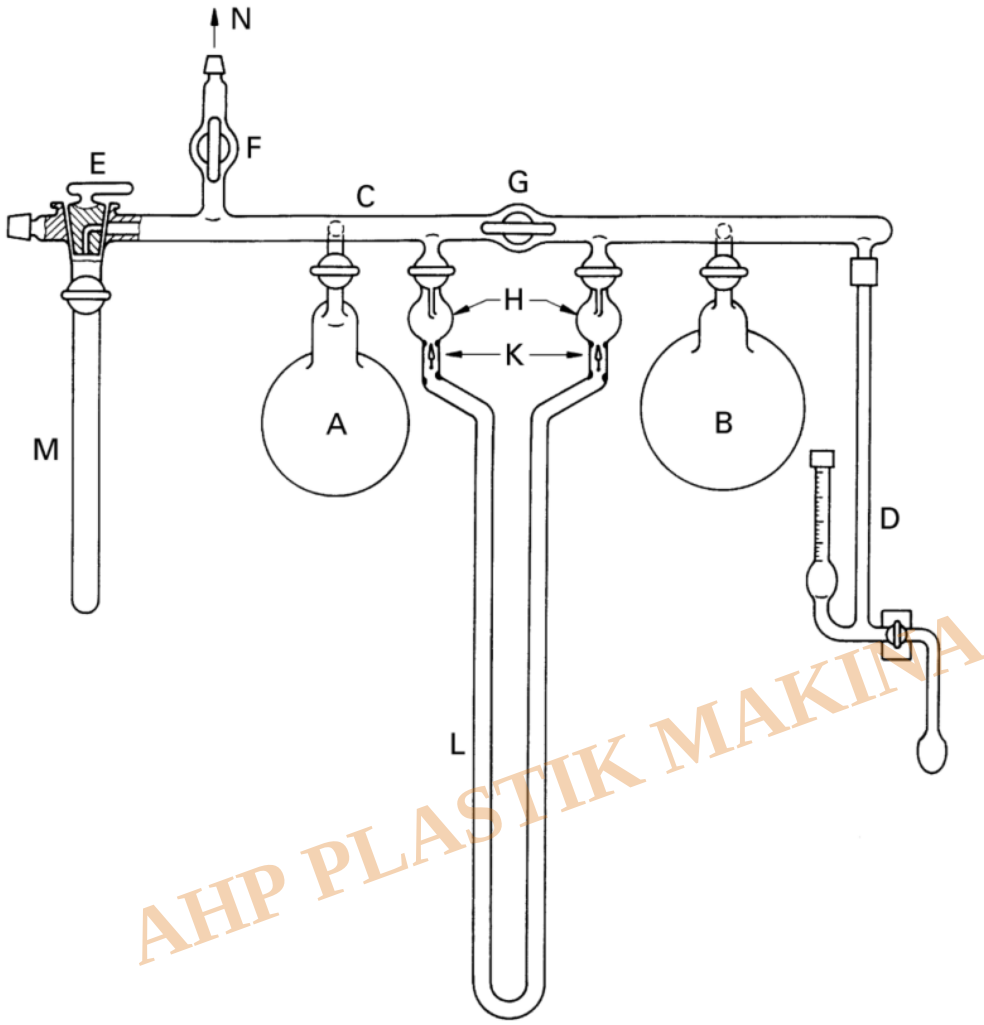
DIN EN ISO 15512 Plastics –Determination of Water Content / Method C

Description

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 (Na2MoO4?2H2O), 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 (BaCl2?2H2O).

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.

Category

1. Equipment for Standards
2. Standards