International Standard 6188

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Plastics — Poly(ethylene terephthalate) granules — Determination of water content

Plastiques — Poly(éthylène téréphtalate) en granules — Détermination de la teneur en eau

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Foreword

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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.

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It has been approved by the member bodies of the following countries :

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New Zealand
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Plastics — Poly(ethylene terephthalate) granules — Determination of water content

1 Scope and field of application

This International Standard specifies a method for determination of the water content of poly(ethylene terephthalate) granules. It is applicable for the determination of water contents in the range 0,002 to 0,05 % (m/m).

Water content is of importance in connection with the processing of the material, during which it should be below a few hundredths of a percent to prevent degradation.

The method is not applicable to poly(ethylene terephthalate) samples containing volatile compounds, other than water, in amounts contributing significantly to the vapour pressure at room temperature. The error introduced by the small amount of acetaldehyde usually present in dried poly(ethylene terephthalate) granules is considered to be acceptable. Checks for the presence of larger amounts of volatile compounds shall be carried out periodically, for example by gas chromatography. Such checks are particularly required for new types or grades of material.

2 Principle

A test portion is heated to 200 ± 5 °C in a closed space under a high vacuum, thus ensuring complete evaporation of the water. The resulting pressure increase, to which the water content is proportional, is measured. The water content is calculated by reference to a calibration curve prepared using a hydrate with a known water content, such as sodium molybdate dihydrate, which loses its water under the conditions of the test.

3 Reagent

3.1 Sodium molybdate dihydrate ($Na_2MoO_4{\cdot}2H_2O$), analytical quality.

NOTE — Other hydrates which lose their water of crystallization under the conditions of test may also be used.

4 Apparatus

4.1 Measuring apparatus

The measuring apparatus, shown diagrammatically in the figure, is an all-glass system with vacuum-tight connections, preferably in the form of spherical joints.

Bulbs A and B shall have volumes of 0,5 ± 0,05 litre and at least 1 litre, respectively.

The bulbs shall be 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 shall carry a connection to a vacuum pump fitted with a stopcock F and shall be fitted with a stopcock G to separate the bulbs. On both sides of the stopcock G the tube shall be connected via splash heads H and check valves K to a U-tube oil manometer L, the legs of which shall have a length of at least 350 mm. The sample tube M shall be made of heat-resistant glass and shall have a volume below the stopcock E of approximately 50 ml. The sample tubes in a set shall not differ in volume by more than 5 ml.

4.2 Electric oven or oil bath

An electric oven or oil bath shall be used to heat the sample tube to 200 ± 5 °C.The arrangement of the equipment shall preferably be such as to allow easy installation and removal of the heating device.

5 Sampling

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

6 Procedure

6.1 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. Turn stopcock E to connect the sample tube to tube C and turn stopcock G to connect bulbs A and B.

Evacuate the system to a pressure of less than 70 Pa[1] and close stopcocks F and G.

After 1 h, check that the pressure is still less than 70 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 deaeration of the new oil.

6.2 Determination

6.2.1 Quickly pour a volume of the test sample corresponding to a mass of 12 to 18 g into a dry sample tube and fix the tube to the apparatus.

NOTE — Initially, the test portion is measured by its volume. The weighing is postponed until after the heating to minimize errors by moisture uptake from the atmosphere.

Turn stopcock E to connect the sample tube to tube C and turn stopcock G to connect bulbs A and B.

Evacuate the system to a pressure of less than 70 Pa and close stopcocks F and G.

Position the electric oven or the oil bath, previously heated to 200 ± 5 °C, around the sample tube and heat the tube at this temperature for 50 min.

NOTE — 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, read the pressure difference indicated by the oil manometer 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 0,01 g.

6.2.2 Carry out two determinations on each sample. If the results differ by more than 0,005 % (m/m), check for leaks (see 6.1), carry out two further determinations and report all four results.

6.3 Calibration

Weigh at least five test portions of sodium molybdate dihydrate, ranging in mass from about 10 to 70 mg, and place them in small, dry test tubes.

Carry out the procedure specified in 6.2.1 with each amount of sodium molybdate dihydrate. The length of the heating period may be reduced from 50 to 15 min.

Using the method of least squares, calculate the calibration factor f, corresponding to the mass of water, in milligrams, required to produce an indicated pressure difference of 100 mm of oil.

NOTES

1 When a new batch of sodium molybdate dihydrate is used, check the water content by weighing, drying for 1 h at 190 °C and re-weighing.

2 Water, as such, shall not be used for the calibration since the amounts required would be too small for weighing with sufficient accuracy.

7 Expression of results

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

$$\frac{0{,}001 \times \Delta p \times f}{m}$$

where

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

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

f is the calibration factor, determined as described in 6.3.

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) the individual test results and the average value of duplicate determinations to the nearest 0,001 % (m/m).

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[1] 70 Pa = 0,7 mbar ≈ 0,5 mmHg