International Standard 6188

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

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

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

Second edition — 1986-08-01

UDC 678.742.2'524-492.3 : 543.812 **Ref. No. ISO 6188-1986 (E)**

Descriptors : plastics, polyalkylene terephthalate, granular materials, chemical analysis, determination of content, water, test equipment.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6188 was prepared by Technical Committee ISO/TC 61, *Plastics.*

ISO 6188 was first published in 1980. This second edition cancels and replaces the first edition (ISO 6188-1980), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Plastics — Poly(alkylene 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) and poly(butylene 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 per cent to prevent degradation.

The method is not applicable to poly(ethylene terephthalate) and poly(butylene 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

Sodium molybdate, dihydrate ($Na_2MoO_4 \cdot 2H_2O$), of recognized analytical quality.

NOTE — Other hydrates which lose their water of crystallization under the conditions of test, such as barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$), may also be used.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Measuring apparatus

The use of a measuring apparatus as described below is recommended. The equipment is shown diagrammatically in the figure. 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 ± 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 6.2.2 can be met.

2 Silicone oil is suitable for filling the manometer.

4.2 Heating device

An electric oven or any other suitable device may 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.

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

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 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 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. (See note 1.)

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 100 Pa and close stopcocks (F) and (G). (See note 2.)

Position the heating device, previously heated to 200 ± 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 up to 1 mm for 5 min. (See note 3.)

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 0,01 g.

NOTES

1 Initially, the test portion is measured by its volume to minimize moisture uptake from the atmosphere. The weighing is carried out after the heating.

2 When testing powder materials, carry out the 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 and cool and store it above a suitable water absorbent, such as a blue silicagel.

3 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.

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) and carry out two further determinations.

6.3 Calibration

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

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

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 equation

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

where

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

w 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 correspondingly.

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.

NOTES

1 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.

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

* 100 Pa = 1 mbar ≈ 0,7 mmHg