INTERNATIONAL STANDARD

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Plastics — Post-consumer poly(ethylene terephthalate) (PET) bottle recyclates —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Recyclats de bouteilles en poly(téréphtalate d'éthylène) (PET) post-consommation —

Partie 2: Préparation des éprouvettes et détermination des propriétés

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 12418-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

ISO 12418 consists of the following parts, under the general title *Plastics — Post-consumer poly(ethylene terephthalate) (PET) bottle recyclates*:

— *Part 1: Designation system and basis for specifications*

— *Part 2: Preparation of test specimens and determination of properties*

Plastics — Post-consumer poly(ethylene terephthalate) (PET) bottle recyclates —

Part 2:
Preparation of test specimens and determination of properties

1 Scope

This part of ISO 12418 specifies the test methods to be used in determining the properties of post-consumer poly(ethylene terephthalate) (PET) bottle recyclates.

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 472, *Plastics — Vocabulary*

ISO 1628-1, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

ISO 1628-5, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 5: Thermoplastic polyester (TP) homopolymers and copolymers*

ISO 15270, *Plastics — Guidelines for the recovery and recycling of plastics waste*

ISO 15512:2008, *Plastics — Determination of water content*

EN 15348:2007, *Plastics — Recycled plastics — Characterization of poly(ethylene terephthalate) (PET) recyclates*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 15270 apply.

4 Characterization of PET recyclates

4.1 Classification of characteristics

The characteristics of PET recyclates are classified into two types, given in Table 1, as follows:

— those which are required for all recyclates (i.e. mandatory characteristics);

— customer specifications (these are optional).

4.2 Test methods

The characteristics shall be determined using the test methods given in Table 1. A certificate of analysis, including the test results for each batch of recyclate, the sampling method and the number of replicates, shall be provided by the supplier to the purchaser on request.

NOTE The sampling method and the number of replicates are important in determining the quality of the material. Quality levels can vary within the batch and from batch to batch, depending on the source of the recyclate.

Table 1 — Characterization of PET recyclates

Characteristics	Unit	Test method	Notes
Mandatory items			
Form		Visual examination	Flakes, pellets or powder
Colour		Visual examination	Natural or coloured
Maximum flake size	mm		Determined by the size of the screen in the grinder
Maximum pellet size	mm		Determined by the die opening of the pelletizer and measured by ruler or callipers
Intrinsic viscosity (IV)	dl/g	ISO 1628-5 in combination with ISO 1628-1 (including its Amendment ISO 1628-1:2009/Amd 1)	Solvent shall be a mixture of phenol and 1,1,2,2-tetrachloroethane (6:4)
Contamination due to labels and other visible contaminants	mg/kg	Annex A	Only for flakes
Contamination due to poly(vinyl chloride)	mg/kg	Annex A	Only for flakes
Contamination due to poly-olefins, and including adhesive	mg/kg	Annex A	Only for flakes
Water content	%	ISO 15512:2008, method B	
Bulk density	kg/m^3	Annex B	
Optional items			
Melt volume-flow rate (MVR)	$cm^3/10$ min	Annex C	-
Alkalinity	pH	Annex D	Only for flakes
Filterability	10 MPa/h/cm^2	EN 15348:2007, Annex F	

Other tests may be carried out by agreement between the purchaser and the supplier. If they are, the results of such tests shall be reported.

Annex A
(normative)

Method for the determination of impurities in PET flakes

A.1 General

This annex specifies a method for the determination of the content of the following impurities in flakes of PET recyclates:

— pieces of label and other visible contaminants;

— poly (vinyl chloride) (PVC);

— polyolefins (POs), and including adhesive.

A.2 Principle

A weighed test portion of the recyclate is spread evenly over the bottom of a shallow tray.

The pieces of label and other visible contaminants are picked out using tweezers and weighed. The test portion, still in the tray, is then placed in a forced air circulation oven at 220 °C for 1 h.

During this heat treatment, the PVC in the test portion becomes discoloured and can be detected visually. The polyolefins in the test portion melt and turn yellow. The adhesive is also yellowed and might be indistinguishable from the polyolefins.

A.3 Apparatus

A.3.1 **Oven**, with forced air circulation, capable of being maintained at 220 °C.

A.3.2 **Technical balance**, capable of weighing to the nearest 0,1 g.

A.3.3 **Analytical balance**, capable of weighing to the nearest 0,000 1 g.

A.3.4 **Spatula**, made of wood or metal.

A.3.5 **Tweezers**.

A.3.6 **Tray**, made of aluminium or enamelled ironware, with a bottom of surface area not less than 0,05 m^2.

A.4 Procedure

Weigh out, to the nearest 0,1 g, approximately 100 g of flakes and record the mass (m_0). Spread the flakes evenly over the bottom of the metal tray.

Stir the flakes slowly in the tray with a spatula and pick out the pieces of label and other visible contaminants using tweezers. Continue this procedure for not less than 30 min. Weigh the pieces of label and other visible contaminants picked out (m_1), using an analytical balance.

Put the tray containing the remaining material into an oven, preheated to 220 °C, and leave it at that temperature for 1 h. Remove the tray, allow to cool and inspect the material for not less than 30 min, as follows:

a) Pick out the black carbonized particles (PVC). Weigh the particles picked out (m_2), using an analytical balance.

b) Pick out the yellowish particles (POs and adhesive). If they are stuck to PET flakes, separate them. Weigh the particles picked out (m_3), using an analytical balance.

A.5 Expression of results

A.5.1 The content of label and other visible contaminants, expressed in mg/kg, is given by the following formula:

$$\text{Content of label and other visible contaminants} = \frac{m_1 \times 10^6}{m_0} \quad \text{(A.1)}$$

where

m_1 is the mass of label and other visible contaminants, in g;

m_0 is the initial mass of the test portion, in g.

A.5.2 The PVC content, expressed in mg/kg, is given by the following formula:

$$\text{PVC content} = \frac{m_2 \times 10^6}{m_0} \quad \text{(A.2)}$$

where

m_2 is the mass of the discoloured (black) particles, in g;

m_0 is the initial mass of the test portion, in g.

A.5.3 The PO content (which will include any adhesive present), expressed in mg/kg, is given by the following formula:

$$\text{PO content} = \frac{m_3 \times 10^6}{m_0} \quad \text{(A.3)}$$

where

m_3 is the mass of the yellowish particles, in g;

m_0 is the initial mass of the test portion, in g.

A.6 Test report

The test report shall include the following information:

a) a reference to this part of ISO 12418 (ISO 12418-2);

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

c) the results of the test;

d) details of any additional operations carried out or changes made in the procedure;

e) the date of the test.

Annex B
(normative)

Method for the determination of the bulk density of PET flakes

B.1 Principle

A container of known volume is weighed and then filled with PET flakes up to the brim and reweighed. The bulk density is calculated from the container volume and the mass of the PET flakes.

B.2 Apparatus

B.2.1 **Container**, approximately 120 mm in diameter and approximately 150 mm in depth, with a smooth brim.

B.2.1 **Ruler**, to remove sample material protruding above the brim of the container.

B.2.1 **Balance**, capable of weighing to the nearest 0,1 g.

B.3 Procedure

Take a sufficient amount of sample to allow three determinations to be carried out.

Weigh the container (m_{c1}) to the nearest 0,1 g.

Using a suitable scoop, fill the container until it overflows. Tap the brim of the container three times and level the contents across the brim of the container using a ruler. Weigh the filled container (m_{c2}) to the nearest 0,1 g.

Repeat the above procedure twice to give three results.

B.4 Calculation

Calculate the bulk density from the following formula:

$$\rho_b = \frac{m_{c2} - m_{c1}}{V} \times 10^3$$

where

ρ_b is the bulk density, in kg/m^3;

m_{c1} is the mass of the container, in g;

m_{c2} is the mass of the test portion and container, in g;

V is the volume of the container, in cm^3.

B.5 Test report

The test report shall include the following information:

a) a reference to this part of ISO 12418 (ISO 12418-2);

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

c) the results of the test (the three individual results plus their mean);

d) details of any additional operations carried out or changes made in the procedure;

e) the date of the test.

Annex C
(informative)

Method for the determination of the melt volume-flow rate (MVR) of PET recyclates

C.1 General

This annex specifies a method for the determination of the melt volume-flow rate (MVR) of PET recyclates in accordance with ISO 1133-1 and ISO 1133-2.

C.2 Drying conditions

Dry a test sample of PET recyclate at 160 °C for 4^{+2}_{-0} h in a circulating-air oven, prior to preforming a compacted charge by compression.

NOTE Because PET is hygroscopic, its viscosity decreases rapidly due to hydrolysis if the material is allowed to absorb moisture. The test sample is therefore dried prior to testing in order to improve the comparability of test results.

C.3 Preforming for flakes

When flakes of PET recyclate are used, prepare a preformed compacted charge in accordance with Annex C of ISO 1133-1:2011, using the following conditions:

a) cylinder temperature: 240 °C to 242 °C;

b) mass of sample placed in cylinder: 6 g to 8 g;

c) load on piston: 1,5 kN;

d) loading time: 2 min under vacuum.

C.4 Size of die

A half size die of length 4,000 mm ± 0,025 mm and bore diameter 1,050 mm ± 0,005 mm (see ISO 1133-1 and ISO 1133-2) shall normally be used. If testing materials using this die gives a melt volume-flow rate of less than 5 $cm^3/10$ min, the standard die 8,000 mm ± 0,025 mm in length and having a bore of nominal diameter 2,095 mm should preferably be used.

C.5 Test temperature

A test temperature of 280 °C shall be used.

C.6 Size of test sample

A test sample of mass 6 g to 10 g shall be used.

C.7 Nominal load on piston

The nominal load on the piston shall be 5 kg.

C.8 Charging the cylinder

Introduce the dried test sample into the cylinder immediately after it has been taken out of the oven in order to avoid moisture regain. During the charging, compress the material with the packing rod using hand pressure. Ensure the charge is as free from air as possible. Complete the charging process in less than 30 s.

If the sample is in flake form, introduce the preformed compacted charge immediately after the preforming process, and complete the charging process in less than 30 s.

C.9 Preheating time

A preheating time of 5 min shall be used.

It is recommended that a die plug be inserted in the die exit so that it effectively prevents molten low-viscosity material from coming out through the die exit during the pre-heating period.

C.10 Measurements

Measure the time taken by the reference mark on the piston to cover a specified distance or measure the distance moved by the piston during a predetermined time.

The time between the end of charging the cylinder and the end of measurement should preferably not exceed 10 min to avoid degradation of the material during the test.

C.11 Cleaning the apparatus

The apparatus, including the cylinder, the piston and the die, shall be cleaned thoroughly after each determination. The presence of contaminants or their thermolysis products is a major problem as these might accelerate hydrolytic degradation of a hygroscopic material like PET.

C.12 Equation for converting MVR to IV

A very good correlation has been found between log(MVR) and IV. In the case of homopolymers, the following equation has been established on the basis of data from four laboratories:

$$\log(\mathrm{MVR}) = -2{,}400\,\mathrm{IV} + 2{,}735$$

Since such a conversion equation might depend on the type of comonomer, it is recommended that a specific equation be established for each type of PET. In the case of PET recyclate, it is necessary to establish a new conversion equation each time the source of the recyclate changes.

C.13 Test report

The test report shall include the following information:

a) a reference to this part of ISO 12418 (ISO 12418-2);

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

c) the result of the test;

d) details of any additional operations carried out or changes made in the procedure;

e) the date of the test.