BS EN 15348:2014

BSI Standards Publication

Plastics — Recycled plastics — Characterization of poly(ethylene terephthalate) (PET) recyclates

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National foreword

This British Standard is the UK implementation of EN 15348:2014. It supersedes BS EN 15348:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/89, Plastics recycling.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

ISBN 978 0 580 81506 5

ICS 13.030.50; 83.080.20

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2014.

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15348

November 2014

ICS 13.030.50; 83.080.20

Supersedes EN 15348:2007

English Version

Plastics - Recycled plastics - Characterization of poly(ethylene terephthalate) (PET) recyclates

Plastiques - Plastiques recyclés - Caractérisation des recyclats de poly(éthylène téréphtalate) (PET)

Kunststoffe - Kunststoff-Rezyklate - Charakterisierung von Polyethylenterephthalat (PET)-Rezyklaten

This European Standard was approved by CEN on 20 September 2014.

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Contents

Page

Foreword

This document (EN 15348:2014) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15348:2007.

EN 15348:2014 includes the following significant technical changes with respect to EN 15348:2007:

a) the deletion of former informative Annex B and replacement by a reference to EN ISO 1133-2 in Table 1;

b) the use of a porcelain crucible instead of a crucible of platinum in B.5.

This European Standard is one part of a series of CEN publications on Plastics Recycling which is structured as follows:

— EN 15342, *Plastics — Recycled Plastics — Characterization of polystyrene (PS) recyclates*

— EN 15343, *Plastics — Recycled Plastics — Plastics recycling traceability and assessment of conformity and recycled content*

— EN 15344, *Plastics — Recycled Plastics — Characterization of Polyethylene (PE) recyclates*

— EN 15345, *Plastics — Recycled Plastics — Characterization of Polypropylene (PP) recyclates*

— EN 15346, *Plastics — Recycled plastics — Characterization of poly(vinyl chloride) (PVC) recyclates*

— EN 15347, *Plastics — Recycled Plastics — Characterization of plastics wastes*

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

— CEN/TR 15353, *Plastics — Recycled plastics — Guidelines for the development of standards for recycled plastics*

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Introduction

Recycling of plastics waste is one type of material recovery process intended to save resources (virgin raw materials, water, and energy), while minimizing harmful emissions into air, water and soil as well as any impacts on human health. The environmental impact of recycling has to be assessed over the whole life cycle of the recycling system (from the waste generation point to the disposal of final residues). To ensure that recycling constitutes the best environmental option for treating the available waste, some prerequisites should preferably be met:

— recycling scheme being contemplated should generate lower environmental impacts than alternative recovery options;

— existing or potential market outlets should be identified that will secure a sustainable industrial recycling operation;

— collection and sorting schemes should be properly designed to deliver recyclable plastics waste fractions fitting reasonably well with the available recycling technologies and with the (changing) needs of the identified market outlets, preferably at minimum costs to society.

This European Standard has been produced in accordance with the guidance produced by CEN on Environmental Aspects and in accordance with CEN/TR 15353.

NOTE CEN/TR 15353 considers the general environmental aspects which are specific to the recycling process.

It is often impossible to trace back each individual product at the end user stage and to check whether the product has been used correctly through its life. Consequently products are out of industrial control for a period of time. It is possible that during this period contamination with other materials might occur that could affect the product's suitability for recycling into the intended application.

1 Scope

This European Standard defines a method of specifying delivery conditions for poly(ethylene terephthalate) (PET) recyclates.

It gives the most important characteristics and associated test methods for assessing PET recyclates intended to be used for the production of semi-finished/finished products. It is intended for use by the supplier and purchaser of such materials, to assist them in agreeing on specifications.

This European Standard is applicable without prejudice to any existing legislation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TR 15353, *Plastics — Recycled plastics — Guidelines for the development of standards for recycled plastics*

EN ISO 472, *Plastics — Vocabulary (ISO 472)*

EN ISO 1133-2, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 2: Method for materials sensitive to time-temperature history and/or moisture (ISO 1133-2)*

EN ISO 11664-4, *Colorimetry — Part 4: CIE 1976 L*a*b* Colour space (ISO 11664-4)*

EN ISO 15512, *Plastics — Determination of water content (ISO 15512)*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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 3534-2, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN ISO 472 and CEN/TR 15353 and the following apply.

3.1
sieve retention
percentage-of the recycled test sample retained on a sieve at the end of the test

Note 1 to entry: The sieve retention is expressed in percent (mass fraction).

3.2
container retention
percentage of recycled matter retained in the container at the bottom of a stack of sieves, or under a single sieve, at the end of the test compared to the mass of the sample

3.3
average particulate dimension
single value of size, representing the dominant particle size for the whole test sample

4 Characterization of PET recyclates

A single batch is the quantity of recyclate that has homogenous characteristics within the specified tolerances.

The characteristics of PET recyclates, which shall be met for every batch (see ISO 3534-2) of PET recyclate are shown in Table 1, and are divided into two types:

— required characteristics needed to characterize PET recyclates in general and required for all recyclates;

— optional characteristics needed to characterize PET recyclates according to customer specifications and applications.

These characteristics shall be assessed by using the test methods given in Table 1 and detailed below.

A Certificate of Analysis, providing the test results for each batch of recyclate shall be provided by the supplier to the purchaser upon request.

The supplier shall provide the necessary information about the material composition of the recyclates, as specified by the purchaser or required by the applicable regulations.

Table 1 — Characterization of PET recyclates

Characteristics	Unit	Test method	Comments
Required			
Shape		Visual	Flakes, pellets
Maximum particle size	mm		Given by the size of the screen of the grinder
Fine particle content	%	Annex A	Value in percentage of the particles passing the sieve of 1 mm.
Colour		Visual inspection	Unicolour, tranparent, mixed
Water content	%	Annex B or EN ISO 15512	
PVC content	mg/kg	Annex C	
Polyolefin content	mg/kg	Annex C	
Optional			
Determination Melt Mass-Flow Rate (MFR)		EN ISO 1133-2	
Intrinsic viscosity (I.V.)	dl/g	ISO 1628-5	
Alkalinity	pH	Annex D	
Filterability	MPa/(h · cm²)	Annex E	
Other residual content	mg/kg		Analysis according to the appropriate method e.g. FTIR, XRF, DSC…
Colour	L,a,b values	Colourimeter according to EN ISO 11664-4	Injection molded disks from Flakes, pellets
Other tests may be carried out by agreement between the purchaser and the supplier and results reported.			

5 Quality assurance

In order that the purchaser of the recyclate can have confidence in the quality of the product, the supplier shall maintain records of the quality control carried out, including incoming materials, processes and finished products.

NOTE A quality management system certified to EN ISO 9001 may be a suitable guarantee of consistent recyclate quality but not the recycled content.

The specification and the standard deviation or range of values within and between batches of material shall be agreed between the supplier and the purchaser.

Where a statement of recycled content, or the previous history of the material, is requested, documentary evidence shall be provided. These records should be available to the purchaser on request.

Where a recyclate has been produced via a melt process, the supplier may choose to state the level of filtration applied during that process. This will determine the maximum size of any non-melting contaminants present in the recyclate. The statement of filtration level shall include details of the filter. Recyclates that have not passed through a melt process cannot be quantified in the same way, and the supplier may state this.

Annex A
(normative)

Determination of size and distribution of PET-R flakes by sieving

A.1 General

This procedure specifies a method for determining the dimensional distribution (size) of PET-R flakes by measuring the quantities retained in a range of sieves having meshes of various sizes.

The results are expressed in terms of quantity retained on the various sieves or as the mean particulate dimension for the whole sample tested (That which has the greatest frequency).

A.2 Principles

A determined sample is filtered through a single sieve, or stack of sieves of various mesh opening sizes, helped by mechanical or manual vibrations. When several sieves are selected to form a stack, the sieves are assembled in ascending order of the mesh opening sizes so that the largest mesh opening is at the top.

A.3 Apparatus

A.3.1 **Weighing scale**: With a precision of ± 0,1 g.

A.3.2 **Sieve**: The nominal diameter of 200 mm, in accordance with ISO 565 supplemented by a lid and by a receiving container. Sieves are made of ring wire.

The sieves used have the following square mesh opening sizes of: 1 mm; 2 mm; 3,15 mm; 4 mm; 6,30 mm; 8 mm and 12,5 mm.

A.3.3 **Mechanical sieve shaker**

A.4 Procedure

A.4.1 Examine the sieve, or the sieves, for the damage to the mesh or any deformation of the matrix of the meshes. Replace all defective meshes.

A.4.2 Weigh the sieve, or each individual sieve, with a margin of 0,1 g.

A.4.3 Weigh the container to the nearest 0,1 g.

A.4.4 Assemble the sieve, or the sieves, and the container, one on top of the other. By assembling a stack of sieves, you ensure that they are gathered in the ascending order of the mesh opening sizes so than the largest opening is at the top.

A.4.5 Weigh 100 g to 300 g with an accuracy of ± 0,1 g of flakes sample to be tested.

A.4.6 Transfer the sample to the uncovered sieve, by avoiding overflow.

A.4.7 Cover the sieve, or the stack of sieves by the lid and secure them in the mechanical device vibrator for sieving.

A.4.8 Set the timer of the vibration device to 12 min.

A.4.9 After the period of shaking, carefully separate the sieve, or the sieves, starting at the top, and weigh each sieve and the container with their contents.

A.5 Number of determinations

Make two determinations for each test sample.

A.6 Determination and expression of the results

A.6.1 The target is to calculate the average mass in grams of flakes maintained in a sieve of a given mesh opening and also the average of retentions ion the container. For each sieve and the container, calculate the average mass of maintained flakes for the two determinations as follows:

$$(m_1 - m_2) = m_3 \text{ and } (m_4 - m_5) = m_6 \quad \text{(A.1)}$$

$$(m_3 + m_6) / 2 = m_r \quad \text{(A.2)}$$

where

m_1 and m_4 are the respective masses of each sieve, or the container, with their contents of retained flakes, for the first and second determinations;

m_2 and m_5 are the respective masses of each sieve, or the empty container;

m_3 and m_6 are the respective masses, of retained flakes on each sieve, or the container, for the first and second determinations;

m_r is the average flakes mass retained on each sieve, or the container.

A.6.2 Calculation of the percentage of the test sample maintained on each sieve or in the container

$$R = m_r \times 100 / m_s \quad \text{(A.3)}$$

where

R is % of flakes maintained on sieve or in the container;

m_s is the average mass in grams of the two test samples taken;

m_r is the average flakes mass retained on each sieve, or the container.

A.7 Test report

The test report should contain the following particular conditions:

a) reference to this European Standard (EN 15348:2014);

b) reference of the batch of PET flakes to test;

c) average mass in grams of the sample used for the test;

d) values expressed as a mass and a percentage flakes retained for each sieve and the container;

e) date of the test.

Annex B
(normative)

Gravimetric method for the determination of residual humidity (water content)

B.1 General

Annex B defines a method for the determination of the water content in the flakes of poly(ethylene terephthalate) (PET) of compounds coming from the recycling of post-consume PET.

B.2 Principle

A given quantity of the sample is introduced into a forced air circulation oven at 150 °C. The water present evaporates and the humidity content is determined by the mass loss for the sample under test.

B.3 Apparatus

B.3.1 **Balance,** accurate to 0,000 1 g.

B.3.2 **Porcelain crucible**, from 80 ml to 90 ml.

B.3.3 **Desiccator.**

B.3.4 **Oven**, with forced air circulation and a maximum temperature of 250 °C.

B.4 Procedure

Weigh 50 g of the sample in the porcelain crucible previously dried and calibrated to constant mass and record the mass of the test portion and of the tare. Introduce the crucible with the test portion into the oven, previously heated to 150 °C and let it stay for 4 h at this temperature. Remove the crucible from the oven, let it cool in the desiccator for 30 min. and weigh again with a balance. By subtracting the mass of the tare, the mass of the dry test portion is obtained. The difference between the initial and the final mass of the test portion is to be related to the initial mass.

B.5 Expression of results

The water content expressed as a percentage, is given by Formula (B.1):

$$\frac{\left(m_0 - m_1\right) \times 100}{m_0} \tag{B.1}$$

where

m_1 is the mass of test portion, after drying in the oven, in g;

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

B.6 Test report

The test report shall include the following information:

a) reference to this European Standard (EN 15348:2014);

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

c) results of the tests;

d) mass of test portion;

e) details of additional operations or changes of the procedure, if any.

Annex C
(normative)

Rapid method for the determination of residual impurities

C.1 General

Annex C defines a method for the determination of impurities content in the flakes of PET-R:

— PVC;

— Polyolefin.

C.2 Principle

A known quantity of the sample is put into a forced air circulation oven at 220 °C.

During this treatment the contamination undergoes degradation phenomena, changing of colour, so making possible their visual separation from the remaining sample.

During this thermal treatment

— PVC is discoloured and can be visually detected in the sample,

— polyolefin melt and colouration which change partially can be visually detected in the sample.

C.3 Apparatus

C.3.1 Oven, with forced air circulation and a maximum temperature of 250 °C.

C.3.2 Technical balance accurate to 0,1 g.

C.3.3 Analytical balance, accurate to 0,000 1 g.

C.4 Procedure

Weigh exactly 1 000 g of flakes with an accuracy of 0,1 g and record the exact mass. Put them evenly in an aluminium (or anyway metallic) container.

Put the container into an oven, preheated to 220 °C, and leave it at that temperature for 1 h. After the necessary time for the cooling the sample, inspect it accurately.

— Separate the black carbonized particles. The separated particles shall be weighed (m_1) with the analytical balance and related to the mass of the tested sample.

— Polyolefin coming from caps are easily recognizable (shape, thickness), separate the coloured partially decomposed and melt particles, which appear as a yellowish colour. If stuck to PET flakes separate them. The separated particles shall be weighed (m_2) with the analytical balance and related to the mass of the rested sample.

C.5 Expression of results

C.5.1 Total PVC content, expressed in mg/kg, is given by Formula (C.1):

$$\frac{m_1 \times 10^6}{m_0} \quad \text{(C.1)}$$

where

m_1 is the mass of the discoloured particle, in g;

m_0 is the initial mass of the sample, in g;

C.5.2 Polyolefin content, expressed in mg/kg, is given by Formula (C.2):

$$\frac{m_2 \times 10^6}{m_0} \quad \text{(C.2)}$$

where

m_2 is the mass of polyolefin particles, in g;

m_0 is the initial mass of the sample, in g;

C.6 Test report

The test report shall include the following information:

a) reference to this European Standard (EN 15348:2014);

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

c) result of the test;

d) details of additional operations or changes of the procedure, if any.

Annex D
(informative)

Potentiometric method for the determination of the residual alkalinity

D.1 General

Annex D defines a method for the determination of the residual alkalinity in PET coming from the recycling process of post-consumer PET.

D.2 Principle

A given quantity of the sample is mixed, by stirring, with a measured volume of distilled water. The residual soda present on the surface of the PET produces an increase of the pH of the water, which can be measured through a combined glass electrode.

D.3 Apparatus

D.3.1 **Magnetic stirrer**, or different stirrer.

D.3.2 **Technical balance,** accurate to 0,1 g.

D.3.3 **Beaker**, of 1 000 ml.

D.3.4 **Calibrated cylinder**, of 500 ml.

D.3.5 **pH-meter** with combined glass electrode.

D.4 Reagents

Distilled water.

D.5 Procedure

Carry out the pH-meter calibration with buffered solutions with a known pH. Weigh exactly 100 g of the sample in a 1 000 ml beaker and add 500 ml of distilled water, whose pH was previously determined and is between 6 and 8. Stir for 10 min, ensuring that all flakes are completely immersed in the water.

After stopping the stirring and decanting the solution, the test electrode is immersed and the pH, when stabilized, is recorded.

D.6 Expression of results

The result of the analysis is reported as the pH of the solution, by comparison with the initial pH of the distilled water.

D.7 Test report

The test report shall include the following information:

a) reference to this European Standard (EN 15348:2014);

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

c) result of the test;

d) details of additional operations or changes of the procedure, if any.

Annex E
(informative)

Method for the determination of infusible impurities by filtration

E.1 General

Annex E defines a method for the determination of filtration of PET compounds coming from the recovery after use, to evaluate the amount of the contamination of infusible materials (such as aluminium, paper, carbonized PVC, etc.).

The method may be applied to all samples of PET of virgin polymer or its compounds. The method is not applicable to polymers which pyrolize with the formation of carbonized residues in the test conditions.

E.2 Principle

The increase of pressure observed during the extrusion of a melted polymer through a filter of appropriate dimensions is a function of the quantity of solid particles present in the polymer. If the test conditions are held strictly constant, this pressure increase gives a direct measurement of the purity of the material under test.

E.3 Apparatus

Single-screw extruder, with extrusion temperature control, equipped with a gear feeding pump, temperature and pressure recording before the filter, and a metallic net filter, mesh opening of 35 µm.

E.4 Procedure

Set up the net filter with 35 µm meshes into the extrusion head. According to the type of the available extruder, set the feeding pump at an optimum revolutions per minute for the operation of the extruder itself. Preset the temperatures of the extruder and of the filtration head such as to have a polymer temperature inside the head of (290 ± 1) °C. Start the extrusion of the polymer and record the pressure before the filter, when steady extrusion conditions have been reached. Record at given time intervals the pressure before the filter, during a test period of about 2 h to 3 h.

E.5 Expression of results

The result is expressed in the terms of the pressure increase, on the basis of Formula (E.1):

$$\Delta P = (P - P_0) \times Q_p / M_p \times A \qquad \text{(E.1)}$$

where

P is the pressure, in bars, after the time t;

P_0 is the initial pressure, in bars;

Q_P is the flow of the melted polymer;

M_P is the extruded polymer mass in the time t, in kg;

A is the filter area in cm^2.

E.6 Test report

The test report shall include the following information:

a) reference to this European Standard (EN 15348:2014);

b) type of the used extruder;

c) flow rate of the tested polymer;

d) filtering area;

e) all information necessary for the identification of the tested sample;

f) results of the test;

g) details of additional operations or changes of the procedure, if any.

Bibliography

[1] UNI 10667-1, *Plastic raw-secondary materials — Part 1: General*

[2] UNI 10667-7, *Plastic raw-secondary materials — Polyethylenterephtalate flakes for the production of fibres, from the recycling of post consumer liquid containers — Part 7: Requirements and test methods*

[3] UNI 10667-8, *Recycled plastics materials — Polyethylenterephtalate flakes for blow moulding, from the recycling of post- consumer liquid containers — Part 8: Requirements and test methods*

[4] UNI 10667-9, *Plastic raw-secondary materials — Polyethylenterephtalate flakes for the production of sheets and sheetings, from the recycling of post-consumer liquid containers — Part 9: Requirements and test methods*

[5] EN ISO 9001, *Quality management systems — Requirements (ISO 9001)*

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