



# SLOVENSKI STANDARD

## SIST-TS CEN/TS 16011:2014

01-maj-2014

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### Polimerni materiali - Reciklirani polimerni materiali - Priprava vzorcev

Plastics - Recycled plastics - Sample preparation

Kunststoffe - Kunststoff-Rezyklate - Probenvorbereitung

Plastiques - Plastiques recyclés - Préparation des échantillons

Ta slovenski standard je istoveten z: CEN/TS 16011:2013

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#### **ICS:**

13.030.50	Recikliranje	Recycling
83.080.01	Polimerni materiali na splošno	Plastics in general

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TECHNICAL SPECIFICATION  
SPÉCIFICATION TECHNIQUE  
TECHNISCHE SPEZIFIKATION

**CEN/TS 16011**

January 2013

ICS 13.030.50; 83.080.01

English Version

**Plastics - Recycled plastics - Sample preparation**

Plastiques - Plastiques recyclés - Préparation des  
échantillons

Kunststoffe - Kunststoff-Rezyklate - Probenvorbereitung

This Technical Specification (CEN/TS) was approved by CEN on 22 October 2012 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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## Foreword

This document (CEN/TS 16011:2013) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

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 Technical Specification is one part of a series of CEN publications on Plastics Recycling that 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 — Characterisation of Polyethylene (PE) recyclates*
- EN 15345, *Plastics — Recycled Plastics — Characterisation of Polypropylene (PP) recyclates*
- EN 15346, *Plastics — Recycled plastics — Characterisation of poly(vinyl chloride) (PVC) recyclates*
- EN 15347, *Plastics — Recycled Plastics — Characterisation of plastics wastes*
- EN 15348, *Plastics — Recycled plastic — Characterization of poly(ethylene terephthalate) (PET) recyclates*
- CEN/TR 15353, *Plastics — Recycled Plastics — Guidelines for the development of standards for recycled plastics*
- CEN/TS 16010, *Plastics — Recycled plastics — Sampling procedures for testing plastics waste and recyclates*

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## Introduction

Recycling of plastics waste is one type of material recovery process intended to save resources (virgin raw materials, water, energy), while minimising harmful emissions into air, water and soil as well as their impacts on human health. The environmental impact of recycling should 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:

- the 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;
- the 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 for society.

This Technical Specification has been produced in accordance with the guidance produced by CEN on Environmental Aspects and in accordance with CEN/TR 15353, *Plastics — Recycled Plastics — Guidelines for the development of standards for recycled plastics*.

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

The purpose of this Technical Specification is to define the procedures to be followed to prepare samples taken in accordance with CEN/TS 16010 in readiness for testing various material characteristics as set out in other relevant standards for recycled plastics (EN 15342 to EN 15348).

It is not the intention of this Technical Specification to develop new sample preparation methods.

## 1 Scope

This Technical Specification specifies the preparation of samples of recycled plastics prior to testing and takes account of the specifics of the material.

Sample preparation should avoid any process that causes 'de-mixing' of the sample. Following preparation, the sample should remain representative.

The behaviour of contaminants should be carefully analysed and observed to ensure this is homogeneous.

This Technical Specification does not address any legal or product safety issues.

## 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 15353:2007, *Plastics — Recycled plastics — Guidelines for the development of standards for recycled plastics*

CEN/TS 16010, *Plastics — Recycled plastics — Sampling procedures for testing plastics waste and recyclates*

EN ISO 472:2001, *Plastics — Vocabulary* (ISO 472:1999)

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement*

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling*

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ISO 11648-1, *Statistical aspects of sampling from bulk materials — Part 1: General principles*

ISO 11648-2:2001, *Statistical aspects of sampling from bulk materials — Part 2: Sampling of particulate materials*

ISO 13909-7, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*

ISO 13909-8, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*

## 3 Terms and definitions

For the purposes of this document, the terms, definitions and abbreviated terms given in EN ISO 472:2001, in CEN/TR 15353:2007 apply.

## 4 Symbols and abbreviations

$C_v$  coefficient of variation

## 5 General requirements

### 5.1 Implementation

Successful implementation of this Technical Specification depends on effective communication between the data user, the sampler and test laboratory. The aim is to close the information chain of the whole processes around the sampling in combination with the laboratory analysis.

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NOTE The data user could be the supplier, purchaser or a third party.

**5.2 Instructions**

The appropriate instructions for sampling, sample preparation and testing shall be reviewed by all those involved in the sampling and testing process. If there are no instructions, the test laboratory should contact the data user.

**5.3 Storage**

Samples should be collected in appropriate containers for the tests required. The storage conditions shall ensure that the sample characteristics do not change.

**5.4 Documentation**

The whole sampling and sub-sampling process shall be documented with all relevant facts recorded. It is also useful to record the apparatus used and their calibration details.

**5.5 Sample preparation rooms**

All sample preparations shall be done in rooms or at places which do not influence the samples, or where the influence is negligible. All these areas shall be free from contamination, be easily decontaminated and be vented to control dust and remove fumes. It is good laboratory practice to avoid unnecessary flame, heat and fume.

**5.6 Volatiles**

If volatiles and non-volatiles are to be determined, then precautions shall be taken to ensure that the volatiles are not able to escape during the sampling and sample preparation process.

**6 Sampling**

Take the sample in accordance with CEN/TS 16010.

**7 Methods****7.1 General**

The general principles of sample preparation are described in Annex A and ISO 11648-2.

**7.2 Determination of the minimum laboratory sample**

The determination of the minimum laboratory sample is dependent on the characteristics and properties to be tested. It should be noted that the minimum mass of the laboratory analysis sample increases with the maximum grain size or mass of the individual particle of the sample.

In Table 1 the relationship is given between the maximum particle size and sample mass to achieve a different fundamental error with 15 %, 10 % and 5 %. The determination of this minimum value is dependent on the maximum mass/particle size of the material being analysed, the required measurement tolerance and the statistical probability value with which the result is correlated. If no information is available, the minimum value of the laboratory sample should be determined by Table 1. Note the different coefficients of variation  $C_v$ .

Samples for the determination of moisture should be a minimum of 1 kg, see 7.5.2.



Table 1 — Minimum value of the laboratory sample

Required minimum mass of the laboratory sample g	Maximum allowable particle size <sup>a</sup> cm $C_v < 15 \%$	Maximum allowable particle size <sup>a</sup> cm $C_v < 10 \%$	Maximum allowable particle size <sup>a</sup> cm $C_v < 5 \%$	Sieve size (ISO 565)
0,1	0,05	0,04	0,03	500 $\mu\text{m}$
1	0,10	0,08	0,06	1,0 mm
2	0,13	0,10	0,08	1,4 mm
5	0,17	0,13	0,10	1,7 mm
10	0,21	0,16	0,13	2,0 mm
30	0,31	0,24	0,19	2,8 mm
50	0,37	0,28	0,22	3,35 mm
100	0,46	0,35	0,28	4,0 mm

<sup>a</sup> The particle size is dependent on the maximum linear dimension of the particle. It is the determined effective size or size distribution of particles, mostly done by manual or automatic sieves.

### 7.3 Homogenisation of samples

Suitable equipment for sample homogenisation and division are the riffle splitter or the sectorial splitter.

### 7.4 Sample preparation before mechanical testing

The preparation of samples before mechanical testing is dependent on the physical form of the material. Usually the aim is to produce a moulded part prior to mechanical testing. For flakes and agglomerates then additional granulation (size reduction) might be required before moulding. If the sample is in the form of a compound and information is available on the melt filtration used, then additional granulation might be unnecessary. See Table 2.

Table 2 — Sample preparation before mechanical testing

Powder	Flake	Agglomerate	Compound	Moulded part
				shredding
	granulation (optional)	granulation (optional)	granulation (optional)	granulation (optional)
moulding	moulding	moulding	moulding	moulding
testing	testing	testing	testing	testing

### 7.5 Preparation of samples for the determination of moisture

#### 7.5.1 Sample Type

The sample to be tested shall be either:

- a sample collected exclusively for the determination of moisture; or
- a sample on which determinations of moisture and other quality characteristics are required.

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## 7.5.2 Mass

The mass of each moisture sample shall not be less than 1 kg. Recommended masses of samples at various nominal maximum particle sizes are given in Table 3 (taken from ISO 11648-2:2001). Samples of nominal maximum particle size greater than 22,4 mm may be subjected to a two-stage drying procedure, as described in Annex A.4.1 b).

**Table 3 — Example of recommended minimum mass of sample for the determination of moisture**

Nominal maximum particle size of material	Maximum layer thickness	Minimum mass	Maximum allowable difference between subsequent weightings of dried samples	Accuracy of weighing	Minimum drying time
mm	mm	kg	g	g	h
63,0	70	110	110	10	16
45,0	50	40	40	4	12
31,5	35	14	14	1	8
22,4	25	5	5	0,5	6
16,0	20	2	2	0,2	4
11,2	13	1	1	0,1	4

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## 7.5.3 Precautions against loss of moisture

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One of the main difficulties in determining moisture is that of minimising changes in the moisture content of the sample when preparing the moisture sample. Take every precaution to minimise changes in moisture content due to unsuitable containers and by evaporation during handling, particularly if the bulk material is extremely wet. Keep all moisture samples in sealed containers in a cool place and out of direct sunlight before and after preparation as well as during any interval between stages of sample preparation.

Take care to minimise changes in moisture content during particle size reduction, by using equipment in which there is no appreciable heating, and by reducing the amount of air passing through the mill to a minimum.

Take care to minimise changes in moisture content when carrying out sample division; carry out such operations as quickly as possible. In some circumstances, it might be necessary to carry out moisture determinations on each increment, to minimise moisture changes (see Annex B).

Where moisture samples are to be retained for any length of time; for example for more than five to seven days in the case of umpire and shipping samples, place them into moisture-impervious plastic bags which are sealed so as to minimise free air space. Then store them in an airtight container.

## 7.5.4 Samples for determination of moisture

Moisture in bulk material may be determined by heating a sample of material at 105 °C in air until constant mass is achieved.

Preparation of the sample may include preliminary air-drying (see A.3) if the material is visibly wet.

## 7.6 Preparation of samples for chemical analysis

### 7.6.1 General

The procedure for preparation of the chemical analysis sample is described in 7.5.2 and shown schematically in Annex B. The moisture sample may be used as the chemical analysis sample after drying, provided it is sufficiently representative.

### 7.6.2 First stage in the preparation

If necessary, the sample may be dried in accordance with A.3. Pass the whole sample through the size reduction equipment (see A.4), mix it, then divide it (see A.6) to decrease the mass to the value appropriate to the nominal maximum particle size of the size reduced bulk material (see A.2).

### 7.6.3 Further stages in the preparation

In the further stages of preparation, reduce the particle size of the portion retained from the first stage to the required nominal maximum particle size for analysis (see A.4), then divide it to obtain as many samples as contractually required, each having a recommended mass of no less than 50 g.

Then place the chemical analysis sample in a sealed container and label it with all necessary identification details.

## 8 Precision of sample preparation

Check the precision of sample preparation periodically and whenever a new scheme is brought into operation.

Test procedures for the determination of precision in sample preparation are given in ISO 3085 and ISO 13909-7 and these procedures may be applied to other bulk materials.

## 9 Bias in sample preparation

### 9.1 General

Check the bias in sample preparation periodically and whenever a new scheme is brought into operation.

Procedures for checking bias in sample preparation are given in ISO 3086 and ISO 13909-8 and these procedures may be applied to other bulk materials.

Contamination is often a major source of bias during sample preparation. Contamination might be attributable to preparation equipment or cross-contamination.

### 9.2 Cross-contamination

Potential causes of cross-contamination include the following:

- a) **Dust:** Dust settling on open samples can be a possible cause of contamination. Minimise dust settling by ensuring as much work as possible is carried out using a dust extraction system. If possible, any dust escaping from size reduction equipment should be extracted downwards and to the rear. Keep all ducting, machinery and floors clean.
- b) **Sample:** To prevent cross-contamination, clean all equipment used between samples, ensuring that the sample has been removed first.