

---

**Polimerni materiali - Reciklirani polimerni materiali - Določevanje izbranih označevalcev spojin v hrani razreda recikliranega polietilenskega tereftalata (PET)**

Plastics - Recycled plastics - Determination of selected marker compounds in food grade recycled polyethylene terephthalate (PET)

Kunststoffe - Kunststoff-Rezyklate - Bestimmung von Markierungsstoffen in Polyethylenterephthalat (PET)-Rezyklaten für die Lebensmittelindustrie

Plastiques - Plastiques recyclés - Détermination de compositions de traceurs sélectionnés dans les poly(téréphtalate d'éthylène) PET recyclés de qualité alimentaire

<https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015>

**Ta slovenski standard je istoveten z: CEN/TS 16861:2015**

---

**ICS:**

13.030.50	Recikliranje	Recycling
83.080.20	Plastomeri	Thermoplastic materials

**SIST-TS CEN/TS 16861:2015****en,fr,de**

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST-TS CEN/TS 16861:2015](https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015)

<https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015>

TECHNICAL SPECIFICATION  
SPÉCIFICATION TECHNIQUE  
TECHNISCHE SPEZIFIKATION

**CEN/TS 16861**

June 2015

ICS 13.030.50; 83.080.20

English Version

**Plastics - Recycled plastics - Determination of selected marker compounds in food grade recycled polyethylene terephthalate (PET)**

Plastiques - Plastiques recyclés - Détermination de compositions de traceurs sélectionnés dans les poly(téréphtalate d'éthylène) (PET) recyclés de qualité alimentaire

Kunststoffe - Kunststoff-Rezyklate - Bestimmung von Markierungsstoffen in Polyethylenterephthalat (PET)-Rezyklaten für die Lebensmittelindustrie

This Technical Specification (CEN/TS) was approved by CEN on 19 April 2015 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.

CEN members are the national standards bodies of 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 United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

## Contents

Page

Introduction .....	5
1 Scope .....	6
2 Normative references .....	6
3 Terms and definitions .....	6
4 Symbols and abbreviations .....	8
5 Principle .....	8
6 Reagents .....	8
6.1 Carrier gases for gas chromatography .....	9
6.2 Solvent .....	9
6.3 Standard solutions .....	9
7 Apparatus .....	9
7.1 Cryogenic mill .....	9
7.2 Laboratory glassware .....	9
7.3 Headspace gas chromatograph-mass spectrometer .....	9
7.4 Capillary column .....	9
7.5 Refrigerator and freezer .....	9
7.6 Sieves .....	10
7.7 Analytical balance .....	10
8 Procedures .....	10
8.1 Introduction .....	10
8.2 Sample conservation .....	10
8.3 Grinding of the PET samples .....	10
8.3.1 General .....	10
8.3.2 Initial preparation .....	10
8.3.3 Cleaning of the cryogenic mill prior to use and in-between samples .....	10
8.3.4 Cryogenic milling of the samples .....	11
8.3.5 Sieving of ground samples .....	11
8.4 Preparation of standard solutions .....	11
8.4.1 General .....	11
8.4.2 Stock solution A .....	11
8.4.3 Stock solution B .....	12
8.4.4 Spiking solution .....	12
8.5 Blank determinations .....	13
8.6 Preparation of sample vials .....	13
8.7 Gas chromatographic analysis .....	13
8.7.1 General .....	13
8.7.2 Identification and quantification of analytes using the standard addition method .....	13
9 Interference .....	15
9.1 Interference during sampling and storage .....	15
9.2 Interference due to co-elution .....	15
10 Test report .....	15
Annex A (informative) Representative chromatograms .....	16
A.1 Total ion chromatogram of all six analytes .....	16
A.2 Chromatograms of individual analytes in the selected ion monitoring mode .....	16

<b>Annex B (informative) Example of instrument settings .....</b>	<b>18</b>
<b>B.1 General .....</b>	<b>18</b>
<b>B.2 Headspace sampler conditions .....</b>	<b>18</b>
<b>B.3 Gas chromatography - mass spectrometry (GC-MS) – conditions .....</b>	<b>19</b>
<b>B.4 Specific ions for selected ion mode (SIM) .....</b>	<b>19</b>
<b>Annex C (informative) Performance characteristics .....</b>	<b>20</b>
<b>C.1 General .....</b>	<b>20</b>
<b>C.2 Limit of detection.....</b>	<b>20</b>
<b>C.3 Limit of quantification .....</b>	<b>20</b>
<b>C.4 Precision .....</b>	<b>21</b>
<b>C.5 Accuracy .....</b>	<b>21</b>
<b>C.6 Linearity.....</b>	<b>22</b>
<b>C.7 Validation results.....</b>	<b>22</b>
<b>Bibliography.....</b>	<b>23</b>

## **iTeh STANDARD PREVIEW** **(standards.iteh.ai)**

[SIST-TS CEN/TS 16861:2015](https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015)

<https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015>

**CEN/TS 16861:2015 (E)****Foreword**

This document (CEN/TS 16861:2015) 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.

According to the CEN-CENELEC Internal Regulations, the national standards organizations 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.

**iTeh STANDARD PREVIEW  
(standards.iteh.ai)**

[SIST-TS CEN/TS 16861:2015](https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015)

<https://standards.iteh.ai/catalog/standards/sist/3989ea37-ed3d-4889-975d-1a74d6ea19e1/sist-ts-cen-ts-16861-2015>

## Introduction

In addition to drivers such as the recycling targets in the EU waste packaging Directive (94/62/EC) and the economic cost of landfill taxes, there is a strong demand for recycled food grade plastic products from packaging end-users who are concerned about their corporate image and promoting their environmental responsibilities.

To ensure that recycling systems and plants used for recycling plastics from post-consumer waste for food contact use are fit for purpose, the EU Commission published Regulation (EC) 282/2008 in 27th March 2008 (Recycled plastic materials and articles intended to come into contact with foods). Two of the main purposes of Regulation (EC) 282/2008 are to define the conditions under which a recycling process should be run and managed and how an application to EFSA (European Food Safety Authority) to have the process authorized can be made. Even when plastics materials and articles are produced using a recycling process which has been authorized by EFSA, it is essential that they comply with the applicable food contact regulations, such as the Plastics Regulation (EU) 10/2011.

It can take a long time for validation to approve new recycling processes, and the “Challenge” test to demonstrate the effectiveness of recycling processes, which is described in Regulation (EC) 282/2008 and ultimately required by EFSA, can be relatively expensive and time consuming. The analytical method presented in this Technical Specification represents a novel, cost effective and relatively quick quality assurance tool that would support new process development and assist organisations to conform to the EC regulations on recycled plastics. Also, because of its flexibility with respect to sample geometry, and small scale nature, the method can also be used in an ad hoc way to assess the quality of a wide range of recycled PET samples and products, for example flake, pellets, and products such as bottles and trays. The chemical compounds (called Marker compounds) for which the method is validated fall into two categories: those that are representative of the PET plastic (e.g. residual monomers), and the common flavour compound limonene.

This Technical Specification is intended to serve two main purposes:

- to provide an analytical method to enable recyclers and end users of recycled food grade poly(ethyleneterephthalate) (PET) to identify and quantify the level of specific chemical compounds. As such, it provides a means of providing a cost-effective, comparative assessment of its quality in terms of the presence and level of these chemical contaminants;
- to provide a template for the development of analytical methods for the analysis of specific “marker compounds” in other types of recycled food contact materials and articles, for example high density polyethylene (HDPE).

This Technical Specification is intended to complement, but not to replace in any way, the existing chemical analysis tests, such as the EFSA “Challenge” test, used to assess the efficiency of PET recycling processes for food grade products, or the EU overall and specific migration tests on food grade PET materials and articles using food products and/or food simulants. It is not intended to be used as a pass or fail type method, but to enable changes in the level of specific chemical compounds to be detected. This information could then be used in a number of ways, depending upon the exact nature of the samples analysed, such as contributing to the information needed to justify a re-examination of a recycling process using the EFSA “Challenge” test.

This Technical Specification is based on FP7 Project SupercleanQ.

**CEN/TS 16861:2015 (E)****1 Scope**

This Technical Specification specifies an analytical method for testing food grade, recycled polyethylene terephthalate (PET). This analytical method provides / is intended to be used as a quality control check. This test identifies and quantifies certain specified contaminants. Such contaminants are referred to as Marker Compounds.

The analytical method is applicable for use on PET samples and products at all stages in the recycling process and will therefore be useful to recycling companies producing commercial, recycled PET for food contact materials and articles, and the manufacturers of such articles.

This Technical Specification is without prejudice to any existing legislation.

NOTE Marker compounds are known to originate from two sources:

- from the PET material itself (i.e. residual monomers, degradation products or reaction/breakdown products);
- from food products that the PET has contacted during its “first use”.

**WARNING – The use of this Technical Specification might involve hazardous materials, operations and equipment.**

**Persons using this Technical Specification should be familiar with normal laboratory practise. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practises and to ensure compliance with any national regulatory conditions.**

**IMPORTANT – It is absolutely essential that tests conducted according to this Technical Specification be carried out by suitably trained staff.**

**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/TS 16011, *Plastics - Recycled plastics - Sample preparation*

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

**3 Terms and definitions**

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

**3.1****analyte**

substance to be determined

Note 1 to entry: In the context of this standard 'analyte' refers to the selected marker compounds (3.4) in the PET sample.

**3.2****diagnostic ion**

selected fragment ion, molecular ion or other characteristic ion from the mass spectrum of the target compound, chosen to provide good specificity and sufficient sensitivity



**3.3****laboratory sample**

sample or subsample(s) sent to or received by the laboratory

Note 1 to entry: In the context of this standard 'laboratory sample' refers to sample of recycled PET, in the form of flakes or pellets, or recycled PET food grade products intended to be used for the laboratory tests and from which the test sample (3.9) will be removed.

Note 2 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, sawing, coring, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

Note 3 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

**3.4****marker compound**

chemical compound that is a typical contaminant present in post-consumer food grade PET and is the type of species that an approved recycling process is expected to remove to a safe level

Note 1 to entry: The marker compound can originate from either the PET itself or the food products that it has been in contact with during service.

**3.5****recycled food grade PET**

recycled PET in the form of flake or pellets from an extruder that has been recycled from post-consumer food grade PET waste using a recycling process that has passed a "Challenge" test and meets the requirements of the EU Regulation (EC) 282/2008

Note 1 to entry: Flake dimensions are typically 2 mm to 5 mm.

**3.6****recycled food grade PET products**

PET products, such as bottles and trays, which are intended for food use and have been manufactured from recycled PET that meets the requirements of EU Regulation (EC) 282/2008

**3.7****selected ion mode, SIM**

measuring the intensity of selected diagnostic ions only

**3.8****standard solution**

solution of accurately known concentration, prepared using standard substances in one or several ways

Note 1 to entry: In the context of this standard a 'standards solution' is a solution containing a set of marker compounds (analytical standards) prepared at a known concentration.

**3.9****test sample**

sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

Note 1 to entry: In the context of this standard 'test sample' refers to aliquot of cryogenically ground and sieved laboratory sample (3.3) which will be analysed for the marker compounds.

## CEN/TS 16861:2015 (E)

## 4 Symbols and abbreviations

CAS	Chemical abstracts service
GC-MS	Gas chromatography with mass spectrometric detection
HDPE	High density polyethylene
LOD	Limit of detection
LOQ	Limit of quantification
m/z	Mass [m] to charge [z] ratio i.e. modulus of the quotient of the particle mass in u and the particle charge in units of electronic charge
<i>n</i>	Number
PDMS	Polydimethyl siloxane
PET	Polyethylene terephthalate
RMS	Root mean square
RSD	Relative standard deviation, in %
R <sup>2</sup>	The square of the Pearson product moment correlation coefficient through data points in known y's and known x's
S/N	Signal to noise ratio

## 5 Principle

## iTeh STANDARD PREVIEW

The recycled PET sample is cryogenically ground using a laboratory-scale mill and sieved to a prescribed particle size and then accurately weighed into a headspace vial and sealed. The vial is heated and the headspace analysed by GC-MS using a capillary column of low polarity. Concentrations of specific analytes (selected marker compounds) are quantified using a standard addition headspace method. The specific analytes are listed in Table 1.

Table 1 — Selected analytes – i.e. marker compounds

Marker compound	CAS-number
Acetaldehyde	75-07-0
2-Methyl-1,3-dioxolane	497-26-7
Ethanol	64-17-5
Ethyl acetate	141-78-6
Hexanal	66-25-1
Limonene	5989-27-5

## 6 Reagents

All reagents shall be of recognized analytical grade. Verify whether the reagents are applicable for this specific purpose and free of interfering compounds.

## 6.1 Carrier gases for gas chromatography

Carrier gas for gas chromatography shall be helium.

## 6.2 Solvent

Acetone for preparation of standard solutions (Propan-2-one, CAS 67-64-1).

## 6.3 Standard solutions

A mixed solution of the set of analytes listed in Table 1 shall be prepared at a known concentration of 4 mg/ml for each compound, in acetone (8.4).

## 7 Apparatus

### 7.1 Cryogenic mill

A cryogenically-cooled laboratory mill fitted with a 2 mm trapezoidal ring capable of operating at 16 000 r/min has been found suitable for this purpose.

### 7.2 Laboratory glassware

All glassware that comes into contact with the sample shall be free of the analytes and any interfering compounds. The following glassware will be used in practice:

- headspace vials fitted with inert septa;
- volumetric flasks;
- volumetric pipettes;
- inert septum-sealed glass vial – e.g. 7 ml capacity;
- microlitre syringes.

### 7.3 Headspace gas chromatograph-mass spectrometer

Gas chromatograph shall be equipped with:

- a capillary column (according to 7.4);
- a mass spectrometric detector;
- an automated headspace sampler.

### 7.4 Capillary column

A capillary column shall be appropriate for sufficient resolution of the analytes in Table 1.

**NOTE** An example of a capillary column which has been found to be satisfactory for this type of work is one that is 30 m long, has a diameter of 320  $\mu\text{m}$ , and a 5 % phenyl 95 % PDMS stationary phase with a film thickness of 3  $\mu\text{m}$ .

### 7.5 Refrigerator and freezer

A laboratory freezer shall be used capable of maintaining a temperature of  $-10\text{ }^{\circ}\text{C}$  to  $-20\text{ }^{\circ}\text{C}$ .