



BSI Standards Publication

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

National foreword

This Published Document is the UK implementation of CEN/TS 16861:2015.

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.

© The British Standards Institution 2015.

Published by BSI Standards Limited 2015

ISBN 978 0 580 88025 4

ICS 13.030.50; 83.080.20

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 June 2015.

Amendments/corrigenda issued since publication

Date	Text affected
-------------	----------------------

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

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

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.

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.

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