

English version

**Materials and articles in contact with foodstuffs - Plastics -
Polymeric additives - Test method for the determination of the
mass fraction of a polymeric additive that lies below 1000
Daltons**

Werkstoffe und Gegenstände in Kontakt mit
Lebensmitteln — Kunststoffe — Polymere Zusatzstoffe —
Prüfverfahren zur Bestimmung des Masseanteils eines
polymeren Zusatzstoffes unter 1000 Dalton

This Technical Specification (CEN/TS) was approved by CEN on 17th March 2003 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.

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Foreword

This document (CEN/TS 14577:2003) has been prepared by Technical Committee CEN/TC 194, "Utensils in contact with food", the secretariat of which is held by BSI.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

For relationship with EU Directive(s), see informative annex ZA, which is an integral part of this document.

Annex A is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Specification: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

Some additives used to make plastics intended to come into contact with foodstuffs are known as 'polymeric additives' [1]. They are added to plastics in order to achieve a technical effect but they do not in isolation have the physical properties required of usable plastics materials and articles. In this respect they differ from the high polymers that are used to make plastics. These polymeric additives do not generally have a single molecular weight but they have a molecular weight distribution as a result of synthesis. They may also show further complexity with different end-group chemistry (because of different initiation and termination processes), different repeat units (because of two or more monomers used to make random or block copolymers) and different chain shapes (because of the formation of linear, branched and cyclic structures within the chains).

These additives may migrate into foodstuffs placed in contact with the plastics and so their safety must be evaluated. Only the fraction of the additive with molecular mass below 1000 Daltons (D) is regarded as being toxicologically relevant and so a distinction has been made by the Scientific Committee for Food (SCF) between polymeric additives with a weight averaged molecular mass (MW) below 1000 D and those with MW above 1000 D [2]. For those polymeric additives with a MW above 1000 D, only a reduced set of data may be required to allow an evaluation of their safety.

The requirements of the SCF and the practical implications have been described and discussed in a Commission document [3]. In deciding which toxicological information is needed, the SCF considers the data available on the monomers used to make the polymeric additive, the size of the additive fraction with MW below 1000 D, and the formulation level of the additive used in the plastic [2]. It is possible that a polymeric additive considered by the SCF is given approval to be used in plastics intended for food contact, but with specifications including a limit on the mass fraction of the additive below MW 1000. For these reasons, for petitions and for testing for compliance, a method of test is required to determine the mass fraction of a polymeric additive that falls below the MW 1000 cut-off set by the SCF.

1 Scope

This Technical Specification specifies a test method to determine the mass fraction of a polymeric additive that falls below a MW of 1000. The method is applicable to polymeric additives *per se* - i.e. supplied in their usable form, before formulation into a plastic. The method is also applicable to polymeric additives extracted from finished plastic materials and articles, provided that precautions are taken to ensure the absence of any interference from any other co-extractives derived from the plastic.

2 Principle

The polymeric additive is fractionated using size exclusion chromatography (SEC) and the mass fraction below MW 1000 D is determined either gravimetrically, off-line, or by using chromatographic detectors such as refractive index, infra-red, ultra-violet, or light-scattering detectors, on-line.

The chromatogram is calibrated for molecular weight either by using calibration standards, where these substances are available, or by using mass-selective detectors such as mass spectroscopy. Under ideal conditions where there is no interaction with the stationary phase, in the SEC separation of oligo- or polymeric multicomponent systems the components are separated with respect to their hydrodynamic volumes. Consequently, retention times (elution volumes) indicate the molecular size and can be converted into molecular weight by use of calibration standards run concurrently or by using molecular weight sensitive detectors (on-line or off-line).

The polymeric additive submitted to the test should be the same as that used to formulate the finished plastic material or article. If the additive is not available as such, then it may be extracted from the finished plastic and then submitted to this test procedure provided that care is taken to ensure that no significant interference occurs from any other co-extractives from the plastic.

NOTE For the separation of polar substances in aqueous systems, SEC is sometimes referred to as gel filtration chromatography (GFC). For the separation of organic substances in non-aqueous systems it is sometimes called gel permeation chromatography (GPC). The term and abbreviation SEC is used here to describe all these types of chromatography.

3 Reagents

3.1 General

All reagents shall be of recognised analytical quality, unless otherwise specified.

3.2 Molecular weight standards

3.2.1 General

A variety of molecular weight standards are available commercially. Some are available in pure form, and others as mixtures with two or more species present. Since the aim of MW calibration is to set the 1000 D cut-off point in the SEC chromatogram, it is preferably to have one MW standard close to 1000 D with two others each side (i.e. above and below 1000 D) so that the cut-off point can be determined by interpolation.

3.2.2 For polar polymeric additives

Examples of polar polymeric additives include polyethylene glycols (PEGs), lignosulfonates, polyacrylamides and polyacrylates. PEG oligomers are commonly used for SEC calibration in both aqueous and polar organic solvents. PEG calibrants are available commercially from the dimer (MW 106) upwards. Polyethylene oxide (PEO) standards are also available commercially.