

Foodstuffs — Detection of irradiated food containing fat — Gas chromatographic/ mass spectrometric analysis of 2-alkylcyclobutanones

The European Standard EN 1785:2003 has the status of a
British Standard

ICS 67.050

National foreword

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Foreword

This document (EN 1785:2003) has been prepared by Technical Committee CEN/TC 275, "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by February 2004, and conflicting national standards shall be withdrawn at the latest by February 2004.

This document supersedes EN 1785:1996.

This European Standard was elaborated on the basis of a protocol during a concerted action of the European Commission (DG XII C.5). Experts and laboratories from E.U. and EFTA countries, contributed jointly to the development of this protocol.

The predecessor of the present standard (EN 1785:1996) has been elaborated following a mandate of the European Commission.

Annex A is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: 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.

1 Scope

This European Standard specifies a method for the identification of irradiation treatment of food containing fat. It is based on the mass spectrometric (MS) detection of radiation-induced 2-alkylcyclobutanones after gas chromatographic (GC) separation [1] to [3].

The method has been successfully tested in interlaboratory trials on raw chicken, pork, liquid whole egg, salmon and Camembert [4] to [8].

Other studies demonstrate that the method is applicable to a wide range of foodstuffs [9] to [21].

2 Normative references

This European Standard incorporates by dated or undated references, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*.

3 Principle

During irradiation, the acyl-oxygen bond in triglycerides is cleaved and this reaction results in the formation of 2-alkylcyclobutanones containing the same number of carbon atoms as the parent fatty acid and the alkyl group is located in ring position 2. Thus, if the fatty acid composition is known, the 2-alkylcyclobutanones formed can be predicted.

The 2-alkylcyclobutanones which were analysed in interlaboratory studies were 2-dodecylcyclobutanone (DCB) and 2-tetradecylcyclobutanone (TCB) which are formed from palmitic and stearic acid, respectively, during irradiation. To date, there is no evidence that the 2-alkylcyclobutanones can be detected in unirradiated foods [4].

[9] to [21]. The 2-alkylcyclobutanones are extracted using n-hexane or n-pentane along with the fat. The extract is then fractionated using adsorption chromatography prior to separation using gas chromatography and detection with a mass spectrometer. Other 2-alkylcyclobutanones, e.g. 2 (tetradec-5'-enyl) cyclobutanone derived from oleic acid, have also been identified in irradiated foodstuffs [8], [15] to [20].

NOTE As an alternative procedure for extraction and purification of the 2-alkylcyclobutanones, supercritical fluid extraction (SFE), has been successfully employed [18], [21]. Argentation chromatography has been used effectively for the detection of foods irradiated at very low doses or containing irradiated ingredients [17]. Liquid chromatography (LC)-GC-MS coupling has been used successfully as an alternative procedure for purification and detection [14]. It should, however, be noted that these alternative procedures have not been validated by interlaboratory trials.

4 Reagents

4.1 General

All reagents and materials used shall be of recognized analytical grade the purity of which has to be tested regularly by the analysis of blank samples. Water shall be of at least grade 3 according to EN ISO 3696:1995.

4.2 n-Hexane ¹⁾

4.3 Sodium sulfate, anhydrous

4.4 Diethyl ether

4.5 Stock standard solutions

n-hexane or isooctane may be used to prepare solutions of 2-cyclohexylcyclohexanone (5 µg/ml), and 2-dodecylcyclobutanone²⁾ and 2-tetradecylcyclobutanone²⁾ (100 µg/ml). Store at -20 °C.

4.6 Working standard solutions

n-hexane or isooctane may be used to prepare solutions of 2-cyclohexylcyclohexanone (0,5 µg/ml) (internal standard), 2-dodecylcyclobutanone and 2-tetradecylcyclobutanone (10 µg/ml). Store at -20 °C.

4.7 Florisil^{®3)}, 150 µm to 250 µm (60 mesh to 100 mesh), pesticide residue analysis grade.

Before use, activate the adsorbent by heating at 550 °C for at least 5 h or overnight. Cool in a desiccator. Keep well sealed after cooling.

Prepare deactivated Florisil[®] by adding 20 parts of water to 100 parts of adsorbent (m/m). Approximately 30 g of activated Florisil[®] is required to prepare sufficient deactivated adsorbent for each column. Ensure that the deactivated Florisil[®] contains no lumps and that the powder flows freely. Leave to equilibrate overnight. Use within one week.

1) n-Hexane was the solvent used to validate the method. However, it is also possible to use n-pentane on health grounds provided it can be shown to lead to the same result.

2) Contact the National Standardization Organizations for the availability of reference standards

3) Florisil[®] is an example for a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of this product.