

Foodstuffs — Detection of irradiated food containing fat — Gas chromatographic analysis of hydrocarbons

The European Standard EN 1784:2003 has the status of a
British Standard

ICS 67.050

National foreword

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Foreword

This document (EN 1784:2003) 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of an identical text or by endorsement, at the latest by February 2004, and conflicting national standards shall be withdrawn at the latest by February 2004.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 1784:1996.

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

The predecessor of the present standard (EN 1784:1996) has been elaborated following a mandate of the European Commission.

Annex A is normative. Annex B 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 which contains fat. It is based on the gas chromatographic (GC) detection of radiation-induced hydrocarbons (HC). The method has been successfully tested in interlaboratory trials on raw chicken, pork and beef [1] to [4] as well as on Camembert, avocado, papaya and mango [5], [6].

Other studies demonstrate that the method is applicable to a wide range of foodstuffs [7] to [28].

2 Normative references

This European Standard incorporates by dated or undated reference, 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 draft 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 chemical bonds are broken in primary and secondary reactions. In the fatty acid moieties of triglycerides breaks occur mainly in the α and β positions with respect to the carbonyl groups resulting in the respective C_{n-1} ¹⁾ and the $C_{n-2:1}$ ²⁾ HC. To predict these chief radiolytic products, the fatty acid composition of samples has to be known (see tables A.1 and A.2).

1) C_{n-1} : HC which has one carbon atom less than the parent fatty acid.

For detection of HC the fat is isolated from the sample by melting it out or by solvent extraction. The HC fraction is obtained by adsorption chromatography prior to separation using gas chromatography and detection with a flame ionization detector (FID) or a mass spectrometer (MS).

NOTE As alternative procedures for extraction and/or purification of hydrocarbons, miniaturized solid phase extraction (SPE) [18], [22], supercritical fluid extraction (SFE) [27] and argentation chromatography [22] have been successfully employed. Argentation chromatography [22] has been used effectively for identification of radiation treatment of avocados with 0,025 kGy, and for processed samples containing low amounts of irradiated ingredients. The method was also beneficial in reducing matrix effects e. g. with irradiated paprika powder. Liquid chromatography (LC)-GC-MS coupling has been used successfully as an alternative procedure for purification and detection [8], [17], [20]. 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 Sodium sulfate, anhydrous, calcined at 650 °C.

4.3 Florisil[®]³⁾, 150 µm to 250 µm (60 mesh to 100 mesh), deactivated by addition of water.

Heat at 550 °C for at least 5 h or overnight (5.10) and store it in a tightly stoppered container (5.22). If it is not used within the next 3 days, heat the Florisil[®] at 130 °C for at least 5 h (5.21) and allow to cool in a desiccator (5.11). Add 3 parts of water to 100 parts of the adsorbent (m/m) for deactivation. Shake this mixture for at least 20 min, and then store it in a stoppered container for at least 10 h to 12 h for equilibration. Use the deactivated adsorbent which is further stored in a stoppered container in the course of the next 3 days; after that reheat to 130 °C and follow the same procedure as described above.

2) C_{n-2:1}: HC which has two carbon atoms less than the parent fatty acid and an additional double bond in position 1.

3) Florisil[®] is an example of 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.

4.4 n-Pentane**4.5 n-Hexane** ⁴⁾**4.6 2-Propanol****4.7 Isooctane****4.8 Nitrogen**, for concentrating solutions.**4.9 Hydrogen, nitrogen or helium** as carrier gas.**4.10 HC standard solution** with concentrations of about 1 µg/ml to 4 µg/ml to be prepared by dissolving in n-pentane, n-hexane or isooctane:

1-dodecene (optional)	1-12:1
n-tridecane (optional)	13:0
1-tetradecene	1-14:1
n-pentadecane	15:0
n-hexadecane (optional)	16:0
1-hexadecene	1-16:1
1,7-hexadecadiene ⁵⁾	1,7-16:2
n-heptadecane	17:0
8-heptadecene ⁵⁾	8-17:1
n-octadecane (optional)	18:0
1-octadecene (optional)	1-18:1

and if available

1,7,10-hexadecatriene	1,7,10-16:3
6,9-heptadecadiene	6,9-17:2

4.11 n-Eicosane solution (internal standard) with a concentration of about 1 µg/ml to 4 µg/ml of solvent (n-pentane, n-hexane or isooctane).**5 Apparatus****5.1 General**

Usual laboratory equipment and, in particular, the following:

5.2 Electric blender and Homogenizer.**5.3 Centrifuge** with swing out rotor and suitable tubes, e.g. 100 ml, capable of producing a centrifugal force of at least 900 *g* at the outer end of the tubes.**5.4 Water bath**, capable of being maintained at 50 °C ± 5 °C.**5.5 Soxhlet apparatus**, with round bottomed flask of e.g. 250 ml and an extractor of e.g. 100 ml.

4) 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 results.

5) The National Standardization Organizations inform on the availability of 1,7-16:2 and 8-17:1.

5.6 Extraction thimbles, e. g. of cellulose, solvent washed for decontamination, or of glass fibre, heated to 470°C over night for decontamination, 25 mm × 100 mm.

5.7 Reflux apparatus, e.g. 250 ml flask with condenser.

5.8 Stoppered graduated cylinders, e.g. of 100 ml capacity.

5.9 Sealable glass tubes, e.g. of 10 ml capacity.

5.10 Muffle furnace, capable of being maintained at 550°C and 650 °C.

5.11 Desiccator

5.12 Chromatographic tube, made of glass, having a length of 200 mm to 300 mm and an internal diameter of 20 mm, fitted with a frit, a polytetrafluoroethylene (PTFE) stopcock and a ground glass joint at the top.

5.13 Graduated dropping funnel, e.g. of 100 ml capacity with pressure compensation.

5.14 Pear-shaped flask, e.g. of 100 ml capacity.

5.15 Graduated conical-bottom test tubes, e.g. of 10 ml capacity.

5.16 Volumetric flask or GC flask, e.g. of 1 ml capacity.

5.17 Rotary evaporator, with evaporation flask and a water bath capable of being controlled at 45 °C.

5.18 Apparatus for concentration of solutions under nitrogen.

5.19 Gas chromatograph (GC) equipped with flame ionization detector (FID) or mass spectrometer (MS).

5.20 Capillary column, with suitable performance characteristics, see Annex B.

5.21 Laboratory oven, capable of being maintained at 100° C to 130° C

5.22 Stoppered container

5.23 Round bottom flasks, e.g. of 250 ml.

5.24 Filter paper, solvent washed for decontamination

6 Sampling technique

When taking samples, give preference to those parts which have a high fat content (e. g. chicken skin). Keep the sample in a sealable glass tube or in fat-free metal foil. Foils having a wax coating or packing materials made of polyethylene should not be used.

7 Procedure

7.1 Reagent blank

Prepare a reagent blank for every analysis series.

The impurities which are encountered are mainly saturated HC, which have been detected in particular in Florisil[®], solvent, filter paper and extraction thimbles (for Soxhlet extraction). To remove impurities, wash filter paper and extraction thimbles of cellulose with solvent until no impurities can be detected. Solvent blank solutions should be concentrated before analysing them for contamination. Plastics materials should not be used for the analyses. Only thoroughly clean glassware should be used.