

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

The European Standard EN 1784 : 1996 has the status of a
British Standard

ICS 67.020

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The preparation of this British Standard was entrusted to Technical Panel AW/-/3, Food analysis - Horizontal methods, upon which the following bodies were represented:

- Association of Public Analysts
- Food and Drink Federation
- Institute of Food Science and Technology
- Laboratory of the Government Chemist
- Ministry of Agriculture Fisheries and Food
- Royal Society of Chemistry

This British Standard, having been prepared under the direction of the Consumer Products and Services Sector Board, was published under the authority of the Standards Board and comes into effect on 15 September 1997

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The following BSI references relate to the work on this standard:
Committee reference AW/-/3
Draft for comment 95/500042 DC

ISBN 0 580 27431 4

Amendments issued since publication

Amd. No.	Date	Text affected

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National foreword

This British Standard has been prepared under the direction of Technical Panel AW/-/3 and is the English language version of EN 1784 : 1996 *Foodstuffs — Detection of irradiated food containing fat — Gas chromatographic analysis of hydrocarbons*, published by the European Committee for Standardization (CEN). EN 1784 was produced as a result of international discussions in which the United Kingdom took an active part.

Cross-reference

Publication referred to	Corresponding British Standard
EN ISO 3696 : 1995	BS EN ISO 3696 : 1995 <i>Water for analytical laboratory use. Specification and test methods</i>

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 16, an inside back cover and a back cover.

ICS 67.020

Descriptors: Foodstuffs, irradiated foodstuffs, ionizing radiation, food analysis, detection of irradiation treatment, fat containing foodstuffs, gas chromatography

English version

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

Produits alimentaires — Détection d'aliments
ionisés contenant des lipides — Analyse par
chromatographie en phase gazeuse des
hydrocarbures

Lebensmittel — Nachweis von bestrahlten
fetthaltigen Lebensmitteln —
Gaschromatographische Untersuchung auf
Kohlenwasserstoffe

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

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Foreword

This European Standard has been prepared by CEN/TC 275, Food analysis — Horizontal methods, of which the Secretariat is held by DIN.

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

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 June 1997, and conflicting national standards shall be withdrawn at the latest by June 1997.

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

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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 inter-laboratory tests on raw chicken, pork and beef [1] to [4] as well as on Camembert, avocado, papaya and mango [5], [6].

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 European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3696 : 1995 *Water for analytical laboratory use — Specification and test methods*

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 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).

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) [7] to [12].

The HC may alternatively be detected using liquid chromatography-GC (LC-GC) coupling [13] to [15].

4 Reagents

4.1 General

All reagents and materials used shall be of recognized analytical grade, of which the purity has to be tested regularly by analysis of blank samples, and 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 and store in a tightly stoppered container. If it is not used within the next 3 days, heat the Florisil[®] at 130 °C for at least 5 h and allow to cool in a desiccator (**5.10**), before 3 parts of water are added to 100 parts of the adsorbent (*m/m*) for deactivation. Shake this mixture for at least 20 min, and then store 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 again deactivate by addition of water as described above.

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:

1-dodecene (optional):	1-12:1;
<i>n</i> -tridecane (optional):	13:0;
1-tetradecene:	1-14:1;
<i>n</i> -pentadecane:	15:0;
<i>n</i> -hexadecane (optional):	16:0;
1-hexadecene:	1-16:1;
1,7-hexadecadiene ⁵⁾	1,7-16:2;
<i>n</i> -heptadecane:	17:0;
8-heptadecene ⁵⁾ :	8-17:1;
<i>n</i> -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;

in the solvent (*n*-pentane, *n*-hexane or isooctane), as standards.

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).

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

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⁾ *n*-Hexane is 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.