EN 1784 – English version

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

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

2 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 ß 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).

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].

3 Limitations

Saturated HC are frequently present both as contaminants and as naturally occuring compounds in food. Therefore, they are not used in isolation for identification of an irradiated sample.

Detection of irradiated raw meat and Camembert has been validated for doses of about 0,5 kGy and above covering the majority of commercial applications.

Detection of irradiated fresh avocado, papaya and mango has been validated for doses of approximately 0,3 kGy and above.

The concentration of HC derived from fatty acids which are of low concentration in the particular fat will be low and might be below the detection limit in the case of low radiation doses. Particularly in fruit the applied doses might be lower than the doses used in the interlaboratory test (see clause 10, table 3).

Detection limit is not influenced by usual applied commercial storage time, see [3], [5].

4 Validation

The method has been tested in four interlaboratory tests:

In an interlaboratory test carried out by the Community Bureau of Reference (BCR), 4 laboratories quantified HC in 15 chicken samples irradiated with about 5 kGy and in 3 unirradiated samples about 2 weeks and 6 to 8 weeks after irradiation, respectively. Radiation-induced HC were detected in all irradiated samples [1].

In a second interlaboratory test carried out by BCR, 8 laboratories quantified HC in 15 coded samples of chicken meat which were either unirradiated or given doses of approximately 0,5 kGy, 3,0 kGy or 5,0 kGy, 1 and 6 months after irradiation, respectively [2] (see table 1).

---

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.

Table 1: Interlaboratory data

Time after irradiation	No. of samples	No. of false negatives 1)	No. of false positives 2)
1 month	119	7	2
6 months	120	8	0

1) The false negatives were all associated with samples given approximately 0,5 kGy. False negatives are irradiated samples identified as unirradiated.

2) The false positives were due to misinterpretation of the data. False positives are unirradiated samples identified as irradiated.

In an interlaboratory test carried out by the German Federal Health Office (Bundesgesundheitsamt, BGA), 17 laboratories identified coded chicken, pork and beef samples which were unirradiated or irradiated with 0,8 kGy, 2,8 kGy or 7 kGy (mean doses) three and six months after irradiation, respectively [3], [4] (see table 2).

Table 2: Interlaboratory data

Species	Time after irradiation	No. of samples	No. of false negatives 1)	No. of false positives 2)
Chicken	3 months	160	0	0
Chicken	6 months	126	1	0
Pork	3 months	153	1	3
Pork	6 months	140	1	4
Beef	3 months	149	2	2
Beef	6 months	136	1	0

1) The false negatives were associated with samples given approximately 0,6 kGy to 0,8 kGy (except one sample which received approximately 2,8 kGy).

2) The false positives were due to either contaminations, mix up of samples or misinterpretation of data.

In a second interlaboratory test carried out by the BGA/BgVV (Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin, German Federal Institute for Consumer protection and veterinary medicine), 22 laboratories identified coded Camembert samples which were either unirradiated or irradiated with a dose of approximately 0,5 kGy or 1 kGy and coded avocado, papaya and mango samples either unirradiated or irradiated with doses of approximately 0,3 kGy, 0,5 kGy or 1 kGy [5], [6] (see table 3).

Table 3: Interlaboratory data

Product	No. of samples	No. of false negative 1)	No. of false positives 2)
Camembert	126	1	0
Avocado	103	1	0
Papaya	104	0	0
Mango	98	5	1

1) Out of seven false negatives, four came from one laboratory. The remaining three false negatives were reported from three different laboratories and referred to mango samples irradiated with approximately 0,3 kGy.

2) The false positive was due to a mix up with a sample irradiated with about 1 kGy.