EN 1786 - English version

Foodstuffs - Detection of irradiated food containing bone - Method by ESR spectroscopy

1 Scope

This European Standard specifies a method for the detection of meat containing bone and fish containing bone which have been treated with ionizing radiation, by analysing the electron spin resonance (ESR) spectrum, also called electron paramagnetic resonance (EPR) spectrum, of the bones, see [1] to [11].

Interlaboratory studies have been successfully carried out with beef bones, trout bones and chicken bones, see [12] to [18]. Since the radiation induced ESR signal is attributed to hydroxyapatite (see 7.1), which is the principal component of bones, it is expected that the application of the method can be extended to all meat and fish species containing bones. These expectations are consistent with laboratory experience (see Annex B).

The detection limit depends on the state of mineralization of the bones which is usually lower for small species (see clause 8).

2 Principle

ESR spectroscopy detects paramagnetic centres (e.g. radicals). An intense external magnetic field produces a difference between the energy levels of the electron spins m_S=+ ½ and m_S= - ½, leading to resonance absorption of an applied microwave beam in the spectrometer. ESR spectra are conventionally displayed as the first derivative of the absorption with respect to the applied magnetic field.

The field and frequency values depend on the experimental arrangements (sample size and sample holder), while their ratio (i.e. g value) is an intrinsic characteristic of the paramagnetic centre and its local coordination. For an identification of irradiated samples it may be helpful to measure the g values of the ESR signals. For further information, see [1] to [12].

Radiation treatment produces radicals which can be quite stable in solid and dry components (e.g. bones) of the food, and can be detected. The intensity of the signal obtained increases with the concentration of the paramagnetic compounds and thus with the applied dose.

3 Limitations

Detection of irradiated bone samples is typically possible above a dose of approximately 0,5 kGy, covering the majority of commercial applications.

Detection limits and stability are influenced by the degrees of mineralization and crystallinity of hydroxyapatite in the sample.

In general, the bones of larger animals and species are highly mineralized with low minimum detectable doses. However, variations within individual animals and species have been noted, see [19] to [21].

In case of meat bones the results of this detection method are not significantly influenced by heating of the sample (e.g. boiling in water). Detection of irradiation treatment is not significantly influenced by storage times of up to 12 months. For poorly mineralized fish bones it has been noted that non radiation induced signals are strongly enhanced if the temperatures recommended (6.1) for drying are exceeded and may interfere with the radiation specific signals.

4 Validation

This European Standard is based on interlaboratory tests with meat bones and fish bones [12] to [18].

In an interlaboratory test carried out by the Community Bureau of Reference (BCR) [13], [16], 21 laboratories identified coded samples of beef bones and trout bones which were either unirradiated or irradiated to about 2 kGy, 4 kGy or 7 kGy (see table 1).

Table 1: Interlaboratory data

Product	No of samples	No of false negative[1)]	No of false positive[2)]
Beef bone	84	0	0
Trout bone	84	5[3)]	0

[1)] False negatives are irradiated samples identified as unirradiated.
[2)] False positives are unirradiated samples identified as irradiated.
[3)] The five false negatives were due to misinterpretation of the spectra.

In an interlaboratory test carried out by the German Federal Health Office (Bundesgesundheitsamt, BGA) [17], 18 laboratories identified coded samples of chicken and trout bones which were either unirradiated or irradiated to about 2 kGy, 4 kGy or 6 kGy (see table 2).

Table 2: Interlaboratory data

Product	No of samples	No of false negative[1)]	No of false positive[2)]
Chicken bone	108	0	0
Trout bones	108	0	2[3)]

[1)] False negatives are irradiated samples identified as unirradiated.
[2)] False positives are unirradiated samples identified as irradiated.
[3)] The two false positives were due to misinterpretation of the spectra.