

## Foodstuffs - Detection of irradiated food containing cellulose Method by ESR spectroscopy

### 1 Scope

This European Standard specifies a method for the detection of foods containing cellulose which have been treated with ionizing radiation, by analysing the electron spin resonance (ESR) spectrum, also called electron paramagnetic resonance (EPR) spectrum, of the food, see [1] to [13].

Interlaboratory studies have been successfully carried out with pistachio nut shells, [14] to [18], paprika powder, [19] and [20] and fresh strawberries [21].

### 2 Principle

ESR spectroscopy detects paramagnetic centres (e.g. radicals). They are either due to irradiation or to other compounds present. An intense external magnetic field produces a difference between the energy levels of the electron spins  $m_S = +\frac{1}{2}$  and  $m_S = -\frac{1}{2}$ , 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 further information, see [1] to [13].

Radiation treatment produces radicals which can be detected in solid and dry parts of the food. The intensity of the signal obtained increases with the concentration of the paramagnetic compounds and thus with the applied dose.

### 3 Limitations

Detection limits and stability are influenced by the crystalline cellulose content and the moisture content of the samples. Positive identification of the cellulose radicals is evidence of irradiation but the absence of this signal does not constitute evidence that the sample is unirradiated.

Detection of irradiated pistachio nuts has been validated for doses of 2 kGy and above and stability is not expected to present limitations for detection of irradiation for at least one year after treatment.

Detection of irradiated paprika powder has been validated for doses of 5 kGy and above. Stability of cellulose radicals in paprika powder is largely dependent on storage conditions, (especially humidity), and may be shorter than the shelf-life of the products.

Detection of irradiated fresh strawberries has been validated for doses of 1,5 kGy and above. Detection of irradiated berries has been analysed for doses of 0,5 kGy and above. Detection is typically limited to about the first 3 weeks after treatment. Stability of cellulose radicals in berries depends on storage conditions and may be shorter than the shelf-life of the products.

### 4 Validation

This European Standard is based on two interlaboratory tests with pistachio nut shells [14] to [18], one interlaboratory test with paprika powder [19], [20] and one with fresh strawberries [21].

In an interlaboratory test carried out by the Community Bureau of Reference (BCR) [17], [18], 21 laboratories identified coded samples of pistachio shells 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 <sup>1)</sup>	No of false positive <sup>2)</sup>
Pistachio shells	84	15	2
<sup>1)</sup> False negatives are irradiated samples identified as unirradiated. <sup>2)</sup> False positives are unirradiated samples identified as irradiated.			

After improvement of the first protocol, in an interlaboratory test carried out by the German Federal Health Office (Bundesgesundheitsamt, BGA) [16], 17 laboratories identified coded samples of pistachio shells which were either unirradiated or irradiated to about 4 kGy or 6 kGy (see table 2).

**Table 2: Interlaboratory data**

Product	No of samples	No of false negative <sup>1)</sup>	No of false positive <sup>2)</sup>
Pistachio shells	68	0	1
<sup>1)</sup> False negatives are irradiated samples identified as unirradiated. <sup>2)</sup> False positives are unirradiated samples identified as irradiated.			

In an interlaboratory test carried out by the BGA [19], [20] 20 laboratories identified coded samples of paprika powder which were either unirradiated or irradiated to about 5 kGy or 10 kGy (see table 3).

**Table 3: Interlaboratory data**

Product	No of samples	No of false negative <sup>1)</sup>	No of false positive <sup>2)</sup>
Paprika powder	160	0	1
<sup>1)</sup> False negatives are irradiated samples identified as unirradiated. <sup>2)</sup> False positives are unirradiated samples identified as irradiated.			

In an interlaboratory test carried out by the German Federal Institute for Health Protection of Consumers and Veterinary Medicine (BgVV) [21], 23 Laboratories identified coded samples of fresh strawberries which were either unirradiated or irradiated to about 1,5 kGy or 3 kGy (see table 4).

**Table 3: Interlaboratory data**

Product	No of samples	No of false negatives	No of false positives	No of inconclusive results
Strawberries	184	7	0	2
<sup>1)</sup> False negatives are irradiated samples identified as unirradiated. <sup>2)</sup> False positives are unirradiated samples identified as irradiated.				