

Foodstuffs — Detection of irradiated food containing crystalline sugar by ESR spectroscopy

1 Scope

This European Standard specifies a method for the detection of foods containing crystalline sugars 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 [7].

Interlaboratory studies have been successfully carried out on dried figs, dried mangoes, dried papayas and raisins [1] to [3].

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 magnetic field and microwave 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 co-ordination. For further information, see [1] to [7].

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

While the general formation processes of radiation-induced radicals are known, identification of the specific radicals responsible for individual signals has not yet been achieved. Nevertheless, the association between radiation treatment and the signals illustrated in clause 5 and figures A.2 and A.4 has been demonstrated in a number of studies [1] to [7].

Multicomponent ESR spectra prove prior irradiation but the absence of the specific spectrum does not constitute evidence that the sample is unirradiated. Different mono- or disaccharides may dominate in the sample producing different ESR spectra after irradiation. Moreover, if no sugar crystals are present in the sample, irradiation will not produce specific ESR signals.

Detection of irradiated dried figs, dried mangoes, dried papayas and raisins has been validated. The lower limit of detection mainly depends on the crystallinity of the sugar in the sample. Detection of irradiation treatment is not significantly influenced by storage of at least several months.

The applicability of this method depends on the presence of sufficient quantities of crystalline sugar in the sample at all stages of handling between irradiation and testing. Confirmation of sensitivity to radiation can be achieved, where necessary, by irradiating a portion of the sample and re-testing. It is important that dried fruits have not been re-hydrated prior to testing.

4 Validation

This European Standard is based on two interlaboratory tests, one with dried papayas and raisins, [1], [2], and one with dried mangoes and dried figs [3].

In an interlaboratory test carried out by the Community Bureau of Reference (BCR) [1], [2], 21 laboratories identified coded samples of dried papayas and raisins which were either unirradiated or irradiated to about 0,5 kGy, 1 kGy, 2 kGy, 4 kGy or 7 kGy (see Table 1).

Table 1 – Interlaboratory data

Product	No of samples	No of false negatives ^a	No of false positives ^b
Raisins	126	7 ^c	1
Dried papayas	126	2 ^d	0
^a	False negatives are irradiated samples identified as unirradiated.		
^b	False positives are unirradiated samples identified as irradiated.		
^c	Obtained from the 19 samples irradiated at 0,5 kGy.		
^d	Obtained from the 21 samples irradiated at 0,5 kGy		

In another interlaboratory test carried out by the German Federal Institute for Health Protection and Veterinary Medicine (BgVV) [3], 17 laboratories identified coded samples of dried mangoes and dried figs which were either unirradiated or irradiated to about 1 kGy, 3 kGy or 5 kGy (see Table 2)

Table 2 – Interlaboratory data

Product	No of samples	No of false negatives ^a	No of false positives ^b
Dried mangoes	184	0	0
Dried figs	184	2	0
^a	False negatives are irradiated samples identified as unirradiated.		
^b	False positives are unirradiated samples identified as irradiated.		