Foodstuffs — Detection of irradiated food using photostimulated luminescence

1 Scope

This European Standard specifies a method for the detection of irradiated foods using photostimulated luminescence (PSL). The technique described here comprises an initial measurement of PSL intensity which may be used for screening purposes, and a calibration method to determine the PSL sensitivity to assist classification. It is necessary to confirm a positive screening result using calibrated PSL or another standardised (e.g. EN 1784 to EN 1788) or validated method.

The method has been successfully tested in interlaboratory trials using shellfish and herbs, spices and seasonings. From other studies it may be concluded that the method is applicable to a large variety of foods.

2 Principle

2.1 General

Mineral debris, typically silicates or bioinorganic materials such as calcite which originate from shells or exoskeletons, or hydroxyapatite from bones or teeth, can be found on most foods. These materials store energy in charge carriers trapped at structural, interstitial or impurity sites, when exposed to ionising radiation. Excitation spectroscopy has shown that optical stimulation of minerals releases charge carriers. It has subsequently been shown that the same spectra can be obtained from whole herb and spice samples and other foods using photostimulation. PSL measurements do not destroy the sample, therefore whole samples, or other mixtures of organic and inorganic material, can be measured repeatedly. PSL signals, however, decrease if the same sample is measured repeatedly.

The methodology comprises screening (initial) PSL measurements to establish the status of the sample (see 2.3) and an optional second measurement following a calibration radiation dose to determine the PSL sensitivity of the sample (see 2.4).

2.2 Screening PSL

For screening (see 2.3) the signal levels are compared with two thresholds (see 2.5). The majority of irradiated samples produce a strong signal above the upper threshold level. Signals below the lower threshold suggest that the sample has not been irradiated. Signal levels between the two thresholds, intermediate signals, show that further investigations are necessary. The use of thresholds produces an effective screening method which can also be backed up by calibration, by TL as described in EN 1788 or another validated method.

2.3 Calibrated PSL

For calibration, the sample is exposed to a defined radiation dose after the initial PSL measurement, and then remeasured. Irradiated samples show only a small increase in PSL after this radiation exposure, whereas unirradiated samples usually show a substantial increase in PSL signal after irradiation.

3 Limitations

The PSL method may, in principle, be applied to detect irradiation of any food which contains mineral debris. PSL sensitivity of a sample depends on the quantities and types of minerals within the individual sample. Signals below the lower threshold (T1) are generally associated with unirradiated material, but can derive from low sensitivity irradiated materials. Calibration can help to distinguish these cases. Samples with low sensitivity (negative or intermediate signals after calibration) should be investigated further by TL analysis or another validated or standardized method.

In general, calibrated PSL measurements are recommended for shellfish with low mineral contents and "clean" spices (e.g. nutmeg, ground white and black pepper) to avoid false negative results.

Optimum results are obtained from unblended products. Compound foods e.g. curry powders, and blends may contain debris with a range of PSL sensitivities, in which case calibrated PSL may provide ambiguous results.

The presence of salt in a product may dominate the PSL intensity to an extent which masks signals from any remaining irradiated ingredients. Hydration of the product followed by re-measurement can both identify and rectify this situation.

4 Validation

In the case of shellfish, the method was tested in a small intercomparison organized by SURRC on behalf of the Ministery of Agriculture, Fisheries and Food (MAFF) with 5 participating laboratories, each of which analysed 10 irradiated and 5 unirradiated blind samples from 5 warm and cold water species. The 10 irradiated samples consisted of one of each species irradiated to each of 2 doses (0,5 kGy and 2,5 kGy). Participants were asked to measure 6 aliquots of whole samples and 6 of intestines for 60 s, and in each case to use the two highest results to make qualitative screening decisions relative to thresholds of T1 = 1 000 counts/60 s and T2 = 4 000 counts/60 s. On this basis all 75 samples were correctly classified (see Table 1). Calibrated PSL measurements were subsequently performed disregarding low sensitivity aliquots. Identical qualitative results were obtained by both screening and calibrated measurements.

In another larger interlaboratory test organized by SURRC on behalf of MAFF, 9 participants tested 40 varieties of herbs, spices and seasonings, and 4 blends presented blind either in unirradiated form or irradiated with a maximum dose of 10 kGy. Thresholds of T1 = 700 counts/60 s and T2 = 5 000 counts/60 s and measurement times of 60 s were used.

662 screening measurements were reported from the samples (345 from irradiated and 317 from unirradiated samples), leading to 577 qualitative classifications based on negative or positive instrumental readings. The irradiation status of 569 (98,6 % of positive or negative outcomes) samples was correctly identified. Eight (1,4 % either false positive or false negative) were incorrect and attributed to operator error. Out of 662 samples examined in the screening study, 85 samples (12,8 %), produced intermediate signals (24 of the 345 irradiated samples, and 61 of the 317 unirradiated samples). These samples required further investigations (see Table 1).

Calibrated measurements were returned from 400 samples (201 irradiated and 199 unirradiated) of which 345 samples were correctly classified. From the 400 samples, 55 determinations (13,8 %) had produced intermediate screening results. After calibration 33 positive results were recorded, confirming the sensitivity to irradiation. This permitted classification of these samples as unirradiated, thus correctly resolving 60 % of the intermediate cases. The remaining 22 intermediate samples (5,5 % of the 400 samples examined here) produced intermediate or negative response to irradiation, and therefore required resolution by another validated or standardized method, such as EN 1788.

The study included four examples of blended mixtures of irradiated spices at 1 %, 5 % and 10 % concentrations in unirradiated spices of matched sensitivity. In this study all blends were correctly identified as containing irradiated material; however, it is recognised that the general problem of detecting minor irradiated components includes variable sensitivity mixtures for which detection performance may be more limited.

Table 1 — PSL screening results from interlaboratory trials of shellfish, herbs, spices, seasonings and blends

	Irradiated				**Unirradiated**			
	Correctly identified		False negative		Correctly identified		False positive	
Shellfish[a]	100	(10 0 %)	0	(0 %)	50	(100 %)	0	(0 %)
Herbs, spices and seasonings and blends[b]	320 344	(93 %)[c] (99,7 %)[d]	1	(0,3 %)[c]	249 10	(78,5 %)[c] (97,8 %)[d]	7	(2,2 %)[c]

[a] These results refer to a total of 75 blind samples, analysed independently both using whole samples, and as intestinal material. Two results per sample were reported, which were in agreement in all cases.

[b] PSL screening for a total of 662 blind samples of herbs, spices, seasonings and blends.

[c] These figures refer to initial PSL screening results in the positive (irradiated) and negative (unirradiated) bands, and show the numbers and proportions of the 577 positive or negative results which could be correctly classified on the basis of screening results alone.

[d] In this tabulation screening results are considered to be targeted simply to select samples for further investigation based on positive or intermediate results. The selection of an irradiated sample for such investigation is considered as a correct identification in these figures.