EN 1788 - English version

Foodstuffs — Thermoluminescence detection of irradiated food from which silicate minerals can be isolated

1 Scope

This European Standard specifies a method for the detection of irradiation treatment of food and/or food ingredients by thermoluminescence analysis of contaminating silicate minerals. This method is applicable to those foodstuffs from which a sufficient amount of silicate minerals can be isolated.

The method has been successfully tested in interlaboratory tests with herbs and spices as well as their mixtures [1] to [3], shellfish including shrimps and prawns [4] to [6], both fresh and dehydrated fruits and vegetables [7] to [9], and potatoes [10]. Other studies [11] to [46] demonstrate that the method is applicable to a large variety of foodstuffs.

2 Principle

Silicate minerals contaminating foodstuffs store energy by charge trapping processes as a result of exposure to ionizing radiation. Releasing such energy, by controlled heating of isolated silicate minerals, gives rise to measurable TL glow curves.

Silicate minerals are therefore isolated from the foodstuffs, mostly by a density separation step. In order not to obscure the TL, the isolated silicate minerals should be as free of organic constituents as possible. A first glow of the separated mineral extracts is recorded (Glow 1). Since various amounts and/or types of minerals (quartz, feldspar etc.) exhibit very variable integrated TL intensities after irradiation, a second TL glow (Glow 2) of the same sample after exposure to a fixed dose of radiation is necessary to normalize the TL response.

The TL glow ratio, thus obtained, is used to indicate radiation treatment of the food, since the population of irradiated samples on principle yields higher TL glow ratios than that of unirradiated samples. Glow shape parameters offer additional evidence for identifying irradiated foods. This method of TL analysis relies solely on the silicate minerals which can be separated from various foods and is not principally influenced by the kind of food product.

3 Limitations

This method of TL analysis can, in principle, be applied to detect irradiation of any food from which silicate minerals can be isolated. Detection limits and stability of the method depend on the quantities and types of minerals recovered from individual samples, and the glow temperature intervals selected for analysis. Minerals from unirradiated samples show a residual geologically derived TL signal with maximum intensity at glow curve temperatures above 300 °C, and minor components in the 200 °C to 300 °C region which can influence detection limits. The stability of TL signals is strongly influenced by glow curve temperature and is greater for higher temperatures. For temperatures from 200 °C to 250 °C TL signals are stable for many years.

The method has been validated with samples which have either been wholly irradiated or unirradiated. In cases where irradiated and unirradiated products are mixed or blended, the outcome of the analysis depends on the relative sensitivities of the irradiated and unirradiated components.

Detection of irradiated herbs, spices and their mixtures has been validated for doses of approximately 6 kGy, and above, and timescales up to nine months covering the range of commercial applications [1] to [3]. Other studies [11] to [13], [15] to [17], [19], [20], [21], [23] to [27], [29] to [34], [36], [37], [40] to [43] have shown that the method may be applied to doses above 1 kGy, and timescales up to several years.

Detection of irradiated shellfish, has been validated for the range of 0,5 kGy to 2,5 kGy and for time scales covering the shelf life of commercial applications [4] to [6]. Other studies [19] to [23], [27], [31], [35], [39], [42] have shown the applicability of TL analysis to shellfish.

Detection of irradiated fresh and dehydrated fruits and vegetables has been validated for doses of about 1 kGy for fresh fruits and vegetables [7], [8] and a radiation dose of about 8 kGy for dehydrated fruits and vegetables [9]. Other studies [11] to [14], [17] to [21], [23], [25], [27], [28], [31], [38], [42] have shown the applicability of TL analysis to fruits and vegetables.

In some cases problems may arise due to a limited amount of silicate minerals present on the samples. In one interlaboratory test [8], participating laboratories were only able to obtain valid results on 97% of strawberries, 82% of avocados, 48% of mushrooms, 83% of papayas and 95% of mangoes, due to a restricted amount of sample and consequently minerals. In practice, larger sample volumes will mostly overcome these problems.

A similar problem occurred in another interlaboratory test on fresh fruits and vegetables [7] and in one on dehydrated fruits and vegetables [9]. In the latter study, particularly, the apple samples showed very low mineral contents. Sufficiently materials could only be obtained from 75% of the samples.

It should be recognized that irradiation of fresh fruits and vegetables for disinfestation purposes occur at lower dose levels than those used in the present interlaboratory tests [7], [8]. In this special case a similar procedure as for potatoes [10] can be adapted (see 8.4.4).

Detection of irradiated potatoes has been validated for radiation doses as low as about 50 Gy about four months after irradiation [10]. Other studies [7], [11], [13], [15], [18], [23], have shown the applicability of TL analysis to potatoes. One of these studies [15] has shown that detection of irradiated potatoes is possible during the whole shelf life.

As mineral debris occur ubiquitously on all foodstuffs which have been exposed to wind and soil, all kinds of agricultural products may be evaluated by TL. In addition to the above mentioned produce, also bulbs such as onion and garlic [18], [23], cereals [44] and pulses [45], [46] have been tested by TL.

4 Validation

The procedure as described in this European Standard is based on interlaboratory studies with herbs, spices, their mixtures [1] to [3], shellfish [4] to [6], fresh and dehydrated fruits and vegetables [7] to [9], and potatoes [10] as well as on studies with other food items [11] to [46].

In the case of herbs and spices, the method was tested in a small preliminary intercomparison organized by the Community Bureau of Reference (BCR) with six participating laboratories, each of which analysed 12 irradiated and unirradiated herbs and spices [1].

In another, larger interlaboratory test organized by the former German Federal Health Office (Bundesgesundheitsamt, BGA, successor institute: Federal Institute for Health Protection of Consumers and Veterinary Medicine, BgVV), 14 participants tested 18 different herbs and spices or mixtures three and/or nine months after irradiation with a dose of about 6 kGy or 11 kGy respectively. Of a total of 317 samples examined, 99,1% were correctly identified. Only three irradiated samples were classified as unirradiated. None of the unirradiated samples were classified as irradiated [2], [3].

In an interlaboratory test organized by the German BgVV, 23 participating laboratories analyzed coded shrimp samples, namely Vietnam Cat Tiger and China Reds, which were either unirradiated or irradiated with doses of 1 kGy or 2 kGy. Of a total of 125 samples examined, 123 samples were identified correctly. Two samples irradiated with 1 kGy were identified as unirradiated using a fixed threshold value of 0,50 for the TL glow ratio. If the glow curve shape had been considered additionally to the TL ratio, all samples would have been identified correctly [4], [5].

In an interlaboratory test with shellfish organized on behalf of the British Ministry of Agriculture Fisheries and Food (MAFF), seven participating laboratories analyzed five species, namely prawns (Norway lobsters), black tiger prawns, brown shrimps, mussels and king scallops. The coded samples were either unirradiated or irradiated with doses of 0,5 kGy or 2,5 kGy. From a total of 105 samples, adequate amounts of silicate minerals could be isolated from 103 samples, and these 103 samples were all identified correctly [6].

In an interlaboratory test with fruits and vegetables organized on behalf of the British MAFF, nine participating laboratories analyzed five types of fruits and vegetables, namely strawberries, avocados, mushrooms, papayas and mangoes. These were presented for blind analysis in three conditions: unirradiated, irradiated to 1 kGy, and irradiated to 1 kGy and optically bleached. From a total of 405 samples, valid results were obtained from 327 samples, all of which were identified correctly. The remaining 78 samples did not yield adequate amounts of silicate minerals [8].

In an interlaboratory test with dehydrated fruits and vegetables organized by the French Centre Technique de la Conservation des Produits Agricoles (CTCPA), eight participating laboratories analyzed five kinds of dehydrated fruits and vegetables, namely apple cubes, sliced carrots, leeks and onions, and powdered asparagus. The coded samples were either unirradiated or irradiated with a dose of about 8 kGy and analyzed six months after irradiation. From a total of 240 samples, adequate amounts of silicate minerals could be isolated from 220 samples. Participants were asked to apply fixed thresholds to the TL glow ratio; considering the sample as irradiated at a value higher than 0,5 and unirradiated at a value lower than 0,1, whereas samples with TL glow ratios between 0,1 and 0,5 were considered as in doubt. 202 samples of the 220 samples were identified correctly, two unirradiated samples were deemed irradiated, (probably due to miscoding), and 16 samples were classified as in doubt or yielded inconsistent results [9].

In an interlaboratory test organized by the German BgVV, 22 participating laboratories analyzed coded potato samples which were either unirradiated or irradiated with doses of about 50 Gy, 160 Gy or 310 Gy. Applying the identification criteria according to clause 9, 216 samples out of a total number of 220 samples were identified correctly. Two samples had to be excluded due to inconsistent results, whereas one unirradiated sample was identified as irradiated and one irradiated sample was identified as unirradiated [10].