BRITISH STANDARD

BS EN 1788:2001

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

The European Standard EN 1788:2001 has the status of a British Standard

ICS 67.050

National foreword

This British Standard is the official English language version of EN 1788:2001. It supersedes BS EN 1788:1997 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/-/3, Food analysis — Horizontal methods, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

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English version

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

Produits alimentaires - Détection par thermoluminescence d'aliments ionisés dont peuvent être extraits des minéraux silicatés

Lebensmittel - Thermolumineszenzverfahren zum Nachweis von bestrahlten Lebensmitteln, von denen Silikatmineralien isoliert werden können

This European Standard was approved by CEN on 18 August 2001.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

This European Standard replaces EN 1788:1996.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

This document was elaborated on the basis of a protocol developed following a concerted action supported by the Commission of European Union (XII C.5). Experts and laboratories from E.U. and EFTA countries, contributed jointly to the development of this protocol.

This predecessor of this document has been prepared under a mandate given to CEN by the Commission of the European Communities and the European Free Trade Association.

WARNING : The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

The annex A is normative ; the annexes B and C are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696).*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
thermoluminescence (TL)
light emission which occurs on heating a solid material in addition to black body radiation, due to the thermally stimulated release of trapped charge carriers

3.2
TL intensity
amount of light detected per unit temperature interval at a given heating rate. The integrated TL intensity over a stated temperature interval is measured in photon counts or coulombs.

3.3
glow curve
variation of TL intensity with temperature. The integral of the glow curve is expressed in counts or coulombs depending on the apparatus used.

3.4
Glow 1
glow curve recorded from the minerals of the prepared sample

3.5
Glow 2
glow curve recorded from the minerals of the prepared sample after measurement of Glow 1 and a subsequent exposure to a fixed known dose of radiation for the purpose of normalization

3.6
TL glow ratio
ratio of integrated TL intensities of Glow 1 to Glow 2, evaluated over a stated temperature interval

3.7
Minimum Detectable integrated TL- intensity Level (MDL)
full process blank level (Glow 1) plus three standard deviations over a stated temperature interval (full process blank levels should be measured in parallel with sample extractions using portions of the same stock solutions and following the procedure at all steps) defines the MDL, which should be consistent with freedom from luminescent contamination of discs, glassware and reagents (see Annex A)

3.8

background of the TL reader

integrated TL intensity measured without sample disc over the whole temperature range studied

4 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.

5 Reagents

5.1 General

Only use reagents of recognized analytical grade. Water shall be of at least grade 3 in accordance with EN ISO 3696. All reagents shall be kept free from particulate contamination throughout the procedure.

5.2 Sodium polytungstate $Na_6[H_2W_{12}O_{40}]$ x H_2O solution with a density of 2 g/ml. The solution may be recovered and purified for re-use [2].

5.3 Hydrochloric acid, substance concentration $c(HCl)$ = 1 mol/l and/or 4 mol/l to 6 mol/l (for special cases)

5.4 Ammonium hydroxide solution, e.g. $c(NH_4OH)$ = 1 mol/l

5.5 Acetone

5.6 Nitrogen gas, oxygen free, for flushing the TL heating chamber

5.7 Silicone spray (optional)

5.8 Ethanol

6 Apparatus

6.1 General

All laboratory surfaces and glassware should be carefully cleaned. Use usual laboratory apparatus and, in particular, the following:

6.2 TL reader

with glow curve recording facility and data evaluation; heating rate: about 6 °C/s; maximum temperature required: at least 350 °C; equipped with a suitable photomultiplier tube, e.g. a bi-alkali photocathode photomultiplier tube, in combination with filters to reject black body radiation. Acceptable filter combinations are Corning 7/59® + Schott KG 1® filters or Schott BG 39®[1] , or equivalent.

6.3 Stainless steel discs

or shallow cups having a diameter to suit the TL reader (usually about 9 mm up to 10 mm) and a thickness of 0,25 mm up to 0,5 mm.

6.4 Radiation source

capable of irradiating discs or cups with isolated minerals at a defined radiation dose before measurement of Glow 2. In the interlaboratory tests on herbs, spices and their mixtures [1] to [3], shellfish [4] to [6], fresh and dehydrated fruits and vegetables [7] to [9], various sources delivering ^{60}Co-y-rays have been employed at a fixed radiation dose of 1 kGy. In the interlaboratory test on potatoes [10] ^{60}Co-y-rays at a fixed dose of only 250 Gy have been used.

NOTE 1 Other fixed doses can be suitable.

NOTE 2 Alternatives to ^{60}Co-y-rays can be used, provided they have been found satisfactory.

6.5 Ultrasonic bath

capable of fitting several beakers of 150 ml. For larger sample volumes, e.g. with dehydrated fruits and vegetables or larger fruits or potatoes, an ultrasonic bath, capable of fitting several beakers of 1000 ml may be advantageous.

6.6 Nylon disposable sieves, comprised of e.g.:

6.6.1 Mini-sieve set (e.g. diameter 50 mm), consisting of two rings between which the nylon sieve cloth is clamped.

6.6.2 Nylon sieve cloth, with pore sizes of 125 µm and 250 µm.

6.7 Centrifuge

supplied with a swing-out rotor and suitable glass tubes, e.g. of 10 ml to 15 ml capacity with pointed bottom; providing a centrifugal acceleration of about 1000 *g* at the outer end of the tubes.

6.8 Vortex for centrifuge tubes (optional)

6.9 Vacuum pump (optional)

6.10 Laboratory oven, set to (50 ± 5) °C.

6.11 Reflux apparatus

6.12 Scalpel and forceps

7 Sampling technique

Whenever possible, the sample should be taken from a light-protected position in the food consignment, since the TL intensity decreases on exposure to light.

Before analysis, samples should be protected against light exposure and be stored in the dark. Avoid exposure of samples to temperatures in excess of 100 °C, since heating reduces TL intensity.

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1) Corning 7/59®, Schott KG 1® and Schott BG 39® are examples of suitable products available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of these products.

8 Procedure

8.1 General

Several procedures for mineral separation may be used, e.g. handpicking, rinsing by water, density separation and/or hydrolysis. The procedures for mineral isolation described in 8.2 have been found to be satisfactory. For any other procedure, it has to be ascertained that the mineral isolation procedure does not affect the qualitative classification.

The isolated silicate minerals should be free of organic material. The presence of organic matter could induce spurious (non-radiation induced) luminescence, or in extreme cases could obscure TL. Samples with organic residues are blackened by the TL measurement process.

Furthermore, during isolation the minerals should be protected against light exposure, i.e. not exposed to strong light or unnecessarily kept exposed to light, to prevent optical bleaching. It may be favourable to use subdued light conditions. Some authors prefer to work under safelight conditions. However, the interlaboratory tests with herbs, spices, their mixtures, shrimps [1] to [5], fresh and dehydrated fruits and vegetables [7], [9] and potatoes [10] demonstrate that protection from strong light under usual laboratory conditions may prove satisfactory.

The quantity of silicate minerals required for TL analysis, is approximately 0,1 mg to 5 mg. The minimum quantity required depends on the outcome of Glow 2. The lowest acceptable integrated TL intensity for Glow 2, shall be at least 10 times the MDL, see also 8.4.6.

8.2 Isolation of silicate minerals from food

8.2.1 Preconcentration step of minerals

8.2.1.1 Herbs, spices and their mixtures

Preconcentration of minerals by wet sieving is recommended for most samples using the following procedure. In some cases (depending on the particular sample) this preconcentration procedure can be omitted. In this case place 0,5 g to 1 g of the sample in a suitable centrifuge tube (6.7) and proceed immediately to the density separation step (as described in 8.2.2).

For the preconcentration, suspend 3 g to 20 g of the sample (depending on the degree of mineral contamination) in a 100 ml to 150 ml glass beaker with 50 ml to 100 ml water added.

Treat the sample in the beaker with ultrasound (6.5) for about 5 min to loosen the adhering minerals.

Sieve the sample in portions through a 250 µm nylon mesh (for coarse samples like aromatic herbs) or through a 125 µm nylon mesh (6.6) (for fine samples like ground spices) into a large beaker (e.g. 500 ml to 1000 ml), rinsing the minerals through with water each time e.g. using a strong jet of water from a wash bottle. Discard the constituents retained by the sieve cloth. Use a fresh nylon sieve cloth for each sample. Allow to settle for about 5 min.

Decant most of the water from the large beaker together with as much organic material as possible, leaving the minerals in only a few millilitres of water. If there are still fairly large amounts of organic material left, add water to a depth of 1 cm to 2 cm, swirl, wait for about 5 s to 10 s to let the minerals settle again and then decant again. Repeat this step until only small amounts of organic material are left together with the minerals.

Transfer the mineral fraction to a centrifuge tube (6.7), e.g. using a Pasteur pipette.

Centrifuge for 1 min at 1000 *g*. Alternatively allow sedimentation for 5 min. Decant off or remove the water by suction, leaving the mineral fraction behind.

8.2.1.2 Shellfish

NOTE Minerals can be associated with various parts of shellfish including the intestines. Intestinally entrained minerals are preferred for analysis. Intestines are found as a 1 mm to 2 mm broad dark tube on the convex side of shrimps and prawns, and in the interior of molluscs.

Cut the skin carefully using a scalpel and remove the intestine by means of forceps. Transfer intestines of several animals to a Petri dish and cut with a scalpel. After adding some drops of water, separate minerals from intestinal membranes. Transfer minerals e.g. using a Pasteur pipette to a suitable, e. g. 10 ml to 15 ml centrifuge tube. Alternatively, place the sliced intestinal tracts into a beaker (e.g. 150 ml), treat with ultrasound for about 15 min and sieve the sample in portions through a 250 µm nylon mesh into a large beaker (e.g. 500 ml to 1000 ml), rinsing the minerals through with water each time e.g. using a strong jet of water from a wash bottle. Discard the residual sample from the sieve. Use a fresh nylon sieve cloth for each sample. Allow to settle for about 5 min to 10 min.

Decant most of the water from the large beaker, leaving the minerals in only a few millilitres of water. Transfer the mineral fraction to a centrifuge tube (6.7), e.g. using a Pasteur pipette.

Centrifuge for about 1 min at 1000 *g*. Alternatively allow sedimentation for 5 min. Decant off or remove the water by suction, leaving the mineral fraction behind.

A further alternative to separate minerals from shellfish is hydrolysis. A procedure for acid hydrolysis which has been tested in an interlaboratory trial is described in 8.2.3.

8.2.1.3 Fresh fruits and vegetables including potatoes

If enough minerals can be isolated just by collection or washing, put them in a centrifuge tube (6.7) and proceed immediately to the density separation step (as described in 8.2.2). Otherwise, preconcentration of minerals by wet sieving is recommended using the following procedure.

Place the sample (one or more fruits, vegetables or potatoes, depending on the degree of mineral contamination) into one or more glass beakers, e.g. of 1000 ml, and add enough water to cover the sample.

Treat the sample in the beaker with ultrasound for about 5 min to loosen the adhering minerals. Remove fruits, vegetables or potatoes, rinsing with water as needed to recover the minerals and proceed as described in 8.2.1.1, paragraph 5 and following. It is recommended to sieve the minerals through a 250 µm nylon mesh to remove very coarse mineral grains or organic parts.

8.2.1.4 Dehydrated fruits and vegetables

Preconcentration of minerals by wet sieving is recommended for most samples using the following procedure.

Place 50 g to 200 g of the sample (depending on the degree of mineral contamination) into one or more glass beakers, e.g. of 1000 ml, and add enough water to cover the sample.

Treat the sample in the beaker with ultrasound for about 5 min (to loosen the adhering minerals) and proceed as described in 8.2.1.1, paragraph 4 and following.

To increase recovery of minerals from dehydrated fruits and vegetables, the settling time in the beaker can be extended to 10 min. During this first preconcentration step, use many rinsing steps to remove organic material.

8.2.2 Density separation step to free the minerals from organic material

Add 5 ml of sodium polytungstate solution (5.2) to the mineral fraction in the centrifuge tube (6.7). Shake vigorously (Vortex) and agitate in an ultrasonic bath for about 3 min (e.g. in case of 8.2.1.3) or 5 min to 15 min (e.g. in case of 8.2.1.1).

Centrifuge for 2 min at 1000 *g*. Silicate minerals (density 2,5 g/ml to 2,7 g/ml) will sediment whereas organic components will float.

Carefully overlay the polytungstate solution with water to facilitate removal of the organic material. Extract the upper water layer and the organic material either by decantation or vacuum suction, leaving the minerals behind in the lower polytungstate layer. If necessary, clean the centrifuge tube side by wiping with a small moist tissue. If not all organic material is removed, overlay the polytungstate solution with water again and repeat extraction. Alternatively, decant off the polytungstate solution and the organic material, and if necessary, clean the centrifuge tube side by wiping with a small moist tissue.

In the case of dehydrated fruits and vegetables, some samples, e.g. apples, form a gel in the polytungstate layer. Instead of immediately extracting the upper water layer, it is recommended to leave the centrifuge tube overnight (room temperature, stoppered tube) and extract the water layer the next day. Add an additional 2 ml to 3 ml of polytungstate solution, shake vigorously (Vortex) and agitate with ultrasound (for about 5 min to 15 min). Centrifuge for 2 min at 1000 *g*, overlay the polytungstate solution with water, and leave the tube overnight. Although part of the gel now still may be in the upper part of the polytungstate layer, it can be more easily extracted, leaving the mineral fraction at the bottom.

Extract the sodium polytungstate layer, being careful to leave the mineral fraction behind. If too much organic material is still present, put further sodium polytungstate solution and repeat the procedure. The sodium polytungstate solution can be collected and purified for re-use [2].

Wash the minerals twice to remove the tungstate residues by filling the centrifuge tube with water, allow the minerals to settle or centrifuge briefly at 1000 *g* and remove the water.

To dissolve carbonates adhering to the silicate minerals, add 1 ml to 2 ml of hydrochloric acid $c(\mathrm{HCl}) = 1$ mol/l (5.3), agitate, and leave for 10 min. If necessary, increase the amount and/or concentration of hydrochloric acid. Especially for potatoes, it is recommended to add at least 5 ml of hydrochloric acid of $c(\mathrm{HCl}) = 4$ mol/l.

Neutralize the acid using the ammonium hydroxide solution (5.4), fill up the centrifuge tube with water, allow the minerals to settle or centrifuge briefly. Remove the supernatant and wash the mineral residue twice with water.

To displace the residual water, add about 3 ml of acetone (5.5) and agitate. If the acetone becomes turbid, remove it and add fresh acetone.

8.2.3 Acid hydrolysis silicate extraction for shellfish

NOTE It has been shown in an interlaboratory study on shellfish [6] that this technique produced higher yields and lower background luminescence of the unirradiated samples compared with physical density separation.

Either the whole sample or intestinal tract removed from the whole sample with a scalpel and forceps, can be used in this process.

Place 10 g to 20 g of the whole sample or 10 mg to 20 mg of the intestinal tract in a round bottomed flask containing 200 ml of 6 mol/l hydrochloric acid for the whole sample or 20 ml for the intestinal tract sample. Reflux the whole sample for 2 h to 3 h. For intestines, heating to 50 °C for 15 min to 30 min is sufficient. During digestion the solution changes from colourless to a clear brown solution.

After cooling, add slowly 400 ml (40 ml for the intestines) of water. Leave the solution for 15 min to allow the minerals to settle.

Decant off the solution carefully to leave minerals at the bottom of the flask or if preferred remove the hydrochloric acid by rotary evaporation to leave remaining minerals.

Carefully transfer the minerals to a centrifuge tube where they are washed thoroughly twice with water. Rinse the minerals with acetone to remove any traces of water (as in 8.2.2).

8.3 Fixing the minerals on discs for TL measurement

Carefully clean stainless steel discs (6.3), e. g. by rinsing in water, ultrasonic agitation, two to three washings in acetone, a second ultrasonic treatment, drying in an oven, and storage under dust free conditions. (The cleaning procedure may be checked as described in annex A.)

Transfer the isolated minerals in acetone to a disc using a Pasteur pipette. After suction of the mineral suspension into the pipette, the minerals will immediately sediment to the outlet of the pipette and can then be easily transferred dropwise (allow the acetone to evaporate in between) in an adequate amount to the disc. Store the discs overnight at 50 °C in a laboratory oven (6.10).

As an alternative to dropping minerals on the disc, transfer the mineral suspension in acetone to one or a set of clean flat bottomed tubes each containing a clean stainless steel disc. Place these tubes upright in a laboratory oven at 50 °C overnight. The acetone will dry off, leaving a deposit of minerals adhering to the discs.

The deposit of minerals can be fixed on the disc by using silicone spray (5.7).

8.4 Thermoluminescence (TL) measurements

8.4.1 General

For comparison of different analyses, identical measuring conditions should be assured. Measure the background of the TL reader (3.8) regularly and ensure that it remains at the same level. Clean the optical filter and the heating plate (planchet) regularly with ethanol.

To reduce spurious TL, flush the TL heating chamber with nitrogen (5.6), at a constant flow rate during each measurement.

8.4.2 Measurement conditions

The following instrument settings have been found satisfactory.

Initial temperature:	70 °C
Heating rate:	6 °C/s
Final temperature:	350 °C to 500 °C

8.4.3 Measurement of Glow 1

Place the disc with the mineral deposit (as prepared in 8.3) on the heating plate of the TL reader (6.2), and glow it under the specified conditions.

8.4.4 Irradiation for the purpose of normalization

After measurement of Glow 1, irradiate the discs with the mineral deposit with a defined radiation dose using the radiation source (6.4).

With herbs and spices and their mixtures [1] to [3], and shellfish [4] to [6], fresh and dehydrated fruits and vegetables [7] to [9], which are irradiated in commercial practice for decontamination purposes with radiation doses near or above 1 kGy, the interlaboratory tests have shown that a fixed radiation dose of about 1 kGy with a ^{60}Co-y-source proves satisfactory. The commercial irradiation of potatoes for sprout inhibition is carried out with radiation doses in the range of 50 Gy to 150 Gy. The interlaboratory test with potatoes [10] has shown that a dose of 250 Gy with ^{60}Co-y-rays proves satisfactory for potatoes as a normalizing dose. It shall be noted that the TL limits (8.4.7) and classification criteria (see clause 9) depend on the radiation dose used for normalization.

NOTE Some studies indicate that suitable alternatives to ^{60}Co-y-rays may be used, see e.g. [2], [7] and [23].

The applied radiation dose for normalization should be controlled by adequate dosimetry.

The discs should be packed in a manner which protects them from loss of material, exposure to light or cross contamination. It is essential that the minerals on the discs to be irradiated, and subsequently measured for Glow 2, are identical with the minerals measured during Glow 1. If significant loss of minerals occurs, the discs should be rejected. This may be checked by visual inspection or by weighing of the discs.

After irradiation of the discs, store them overnight at 50 °C in the laboratory oven (6.10) before recording Glow 2.

8.4.5 Measurement of Glow 2

Measure Glow 2 under the same conditions as Glow 1 (8.4.2).

8.4.6 Estimation of MDL

Measure full process blank levels in parallel with sample extractions using portions of the same stock solutions and following the procedure at all stages. Calculate the MDL in accordance with 3.7. Contamination will be indicated by higher blank levels [31].

8.4.7 TL limits for Glow 2

For samples with a Glow 2 lower than 10 times the MDL, evaluated over a stated temperature interval, no assessment can be made of whether irradiation treatment of the food product has occurred.

If the TL of Glow 2 approximates the counting saturation limit, reject the sample and repeat the analysis using a smaller amount of minerals. Alternatively, a restrictive aperture or a neutral density filter to reduce count rate may be effective (both for Glow 1 and Glow 2).

9 Evaluation

The identification of irradiated foods and food ingredients by TL analysis depends on the value of the TL glow ratio (3.6) and the shapes of the glow curves.

The recommended temperature interval for evaluation of the TL glow ratio is approximately in the range of 150 °C to 250 °C. The absolute temperature scale can be determined in the TL reader using calibrated thermocouples. Alternatively, temperature intervals may be defined by evaluating the glow curve of a well characterized phosphor like feldspar or lithium fluoride (see annex B). A temperature interval comprising ± 10 °C up to ± 40 °C within the recommended range may be chosen. The peak temperature is a function of post-irradiation delay and storage temperature, and therefore usually differs between Glow 1 and Glow 2 for a given sample (see annex C). Calculate the integral of Glow 1 and Glow 2 over the recommended temperature interval (see 8.4.7), and the TL glow ratio (3.6). TL glow ratios from irradiated samples are typically greater than 0,1 whereas those from unirradiated samples are usually below 0,1.

In addition to the TL glow ratio, interpretation of the shape of the glow curves is needed to decide whether the sample has been irradiated or not. Usually, Glow 1 curves of irradiated foodstuffs exhibit a maximum between 150 °C and 250 °C, whereas low level natural radioactivity causes TL signals in the deep traps above 300 °C (Examples of glow curves are given in annex C).

NOTE It should be recognized that in cases where only part of the food has been irradiated, e.g. spice blends with one or more irradiated ingredients, the TL glow ratio can decrease below 0,1 whereas the shape of Glow 1 clearly indicates irradiation treatment.

10 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.

11 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].

12 Test report

The test report shall contain at least the following:

a) information necessary for identification of the sample;

b) a reference to this European Standard;

c) date of sampling and sampling procedure (if known);

d) date of receipt;

e) date of test;

f) the result;

g) any particular points observed in the course of the test;

h) any operations not specified in the method or regarded as optional which might have affected the results.

Annex A
(normative)

Estimation of blank levels

It should be ascertained that stainless steel discs, glassware and reagents are free from particulate contamination. As a general control, a full process blank is carried through the procedure. Integrated TL intensity of the first glow of the blank plus three standard deviations defines the MDL.

As a further control, the cleanliness of the employed stainless steel discs may be checked by irradiation of the cleaned discs with e.g. a radiation dose of 1 kGy, and recording of integrated TL intensities. Discs with TL levels more than three standard deviations above the mean background of the TL reader (3.8) indicate surface contamination.

If the discs are not completely clean, diverging results depending on the kind of contamination may be obtained. (Most frequently, only unirradiated dust grains will cause contamination, leading to false negative results, since the normalization procedure contains a radiation step, by which the contaminating dust on the discs will be excited and contribute to Glow 2.)

If the full process blank shows integrated TL intensities greater than three standard deviations above the mean clean disc blank level, contamination of glassware or reagents is indicated and has to be checked and eliminated.

Annex B
(informative)

Practical example for defining temperature intervals of the TL heating unit

Calibration of the absolute temperature scale of the TL heating unit can be performed applying calibrated thermocouples.

Alternatively, practical temperature ranges can be defined by a well-characterized phosphor like feldspar or lithium fluoride. An example using lithium fluoride (LiF, TLD-100)®2), which is a frequently used phosphor in radiation dosimetry, is shown in figure B.1.

LiF (TLD-100)® pellets or chips (LiF powder should not be used due to the risk of contamination) are irradiated with a radiation dose of about 0,5 Gy with ^{60}Co-y-rays. At least 10 pellets or chips are used to counteract variability in between individual LiF pellets. The glow curve is measured under conditions used for minerals.

The positions of the peaks V (= PV) and VI of the LiF glow curve (see figure B.1) on the temperature axis are measured and the temperature difference IS between the two values (using the mean values of at least 10 measurements) is calculated. The temperature interval I, extending from (PV-IS) to PV, is recommended for evaluation.

This temperature interval has been proven satisfactory for discrimination between irradiated and unirradiated samples in interlaboratory tests with herbs, spices, their mixtures [2], [3], shrimps [4] and potatoes [10], but also in other studies [7], [22], [23], [25], [38], [40], [41], [45], [46].

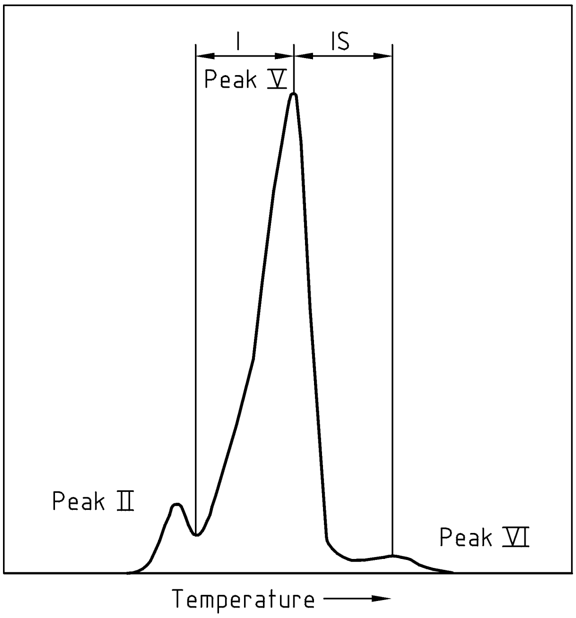

Figure B.1 — Glow curve of an irradiated LiF chip

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2) TLD-100® is an example of a product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of these products.

Annex C
(informative)

Examples for TL glow curves using various readers

Glow curves with logarithmic y scales

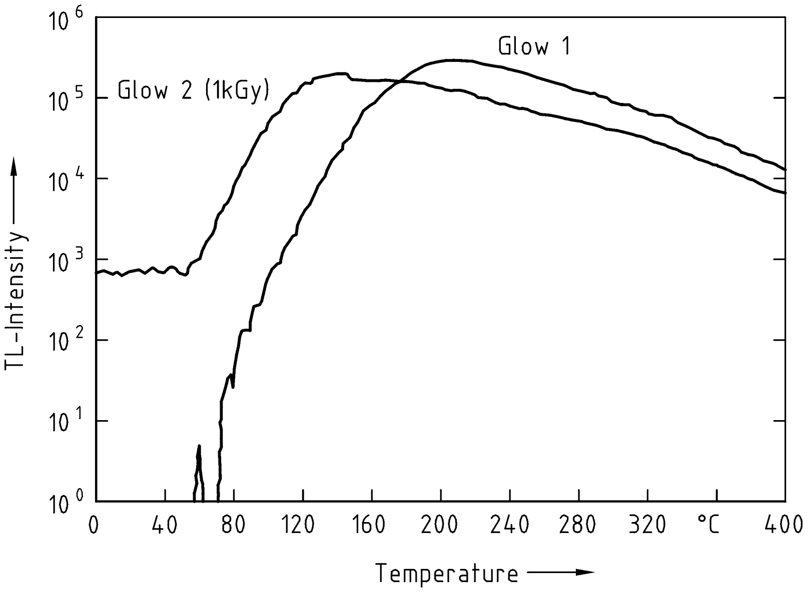

Figure C.1 — TL glow curves from an irradiated sample

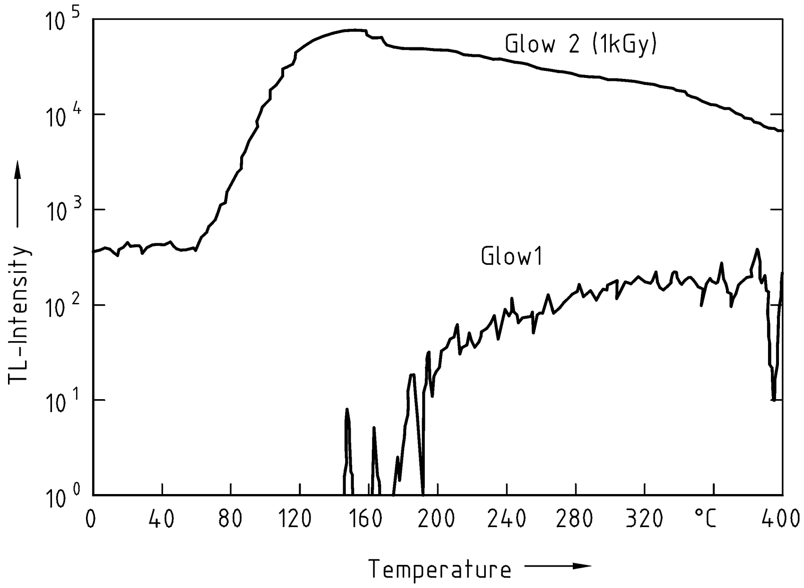

Figure C.2 — TL glow curves from an unirradiated sample

Glow curve with linear y scales

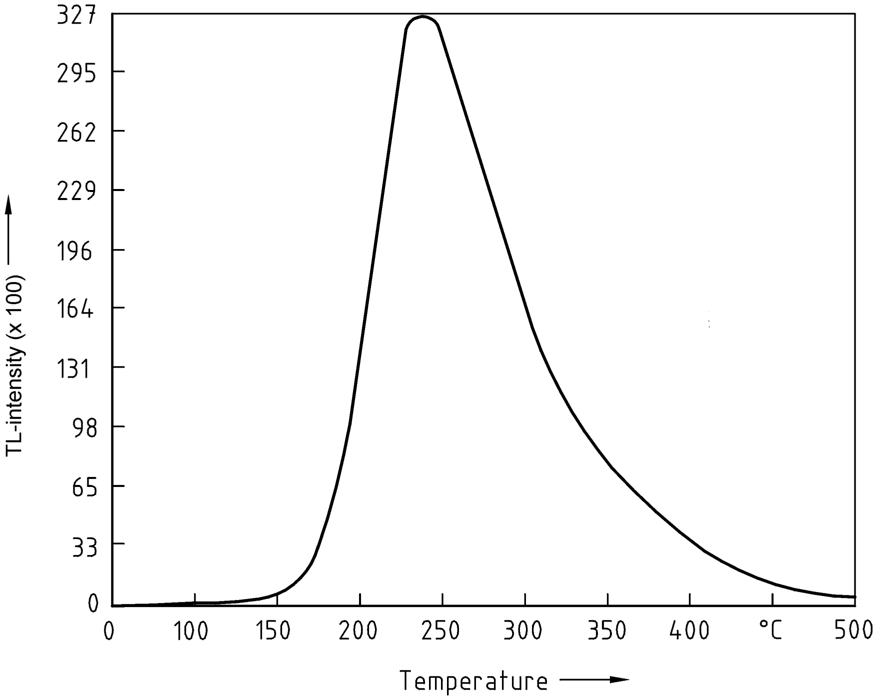

Figure C.3 — TL glow curve from an irradiated sample

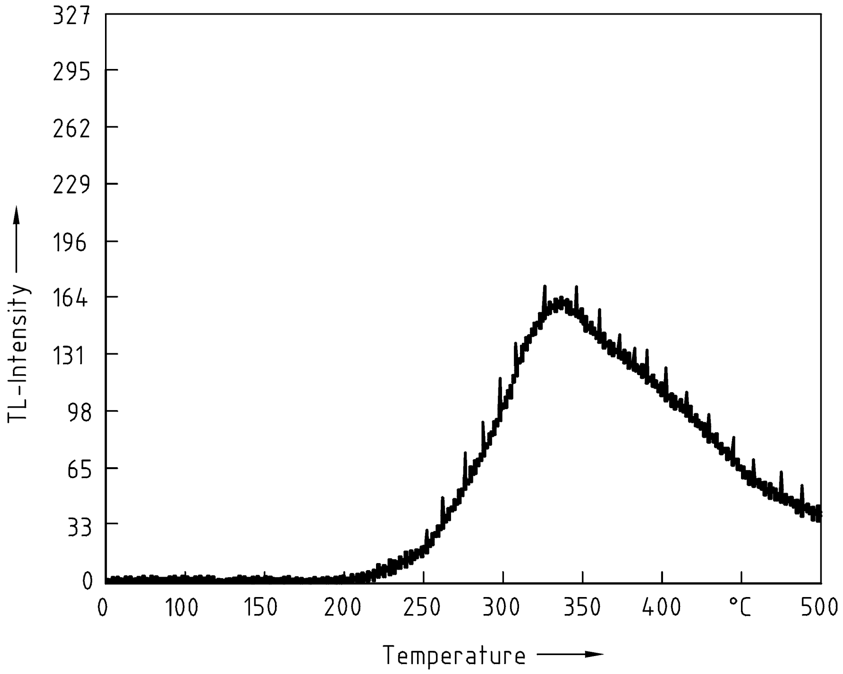

Figure C.4 — TL glow curve from an unirradiated sample

(It should be noted that the y scales of figure C.3 and figure C.4 differ by a factor of 100)

Bibliography

[1] Sanderson, D.C.W., Schreiber, G.A., Carmichael, L.A.: A European Trial of TL Detection of Irradiated Herbs and Spices, 1991, SURRC Report to BCR.

[2] Schreiber, G.A., Wagner, U., Leffke, A., Helle, N., Ammon, J., Buchholtz, H.-V., Delincée, H., Estendorfer, S., Fuchs, K., von Grabowski, H.-U., Kruspe, W., Mainczyk, K., Münz, H., Nootenboom, H., Schleich, C., Vreden, N., Wiezorek, C., and Bögl, K.W.: Thermoluminescence analysis to detect irradiated spices, herbs and spice- and herbs mixtures - an intercomparison study. Bericht des Instituts für Sozialmedizin und Epidemiologie des Bundesgesundheitsamtes, 1993, SozEp-Heft 2/1993, German Federal Health Office (Bundesgesundheitsamt, Berlin).

[3] Schreiber, G.A., Helle, N., and Bögl, K.W.: An inter-laboratory trial on the identification of irradiated spices, herbs and spice-herbs mixtures by thermoluminescence analysis. JAOAC, 1995, **78**, 88-93.

[4] Schreiber, G.A., Mager, M., Ammon, J., Brunner, J., Buchholtz, H.-V., Butz, B., Delincée, H., Fienitz, B., Frohmuth, G., Hammerton, K., Jahr, D., Kispéter, J., Klein, H., Kruspe, W., Kühn, T., Mainczyk, K., Meyer, H., Münz, H., Nootenboom, H., Pfordt, J., Pinnioja, S., Roberts, P.B., Sanderson, D.C.W., Schleich, C., Vreden, N., Zachäus, U., Zehnder, H.J., and Bögl, K. W.: An interlaboratory study on the detection of irradiated shrimps by thermoluminescence analysis. A report of the Federal Institute for Health Protection of Consumers and Veterinary Medicine. BgVV-Heft 3/1995 (Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin, Berlin).

[5] Schreiber, G.A., Helle, N., Schulzki, G., Linke, B., Spiegelberg, A., Mager, M., and Bögl, K.W.: Interlaboratory tests to identify irradiation treatment of various foodstuffs via gas chromatographic detection of hydrocarbons, ESR spectroscopy and TL analysis. Detection Methods for Irradiated Foods - Current Status. Edited by: McMurray C.H., Stewart, E. M., Gray, R., and Pearce, J., Royal Society of Chemistry, Cambridge, UK, 1996, 98-107.

[6] Sanderson, D.C.W., Carmichael, L., Naylor, J.D. and Fisk, S.: An international collaborative blind trial of thermoluminescence detection of irradiated shellfish,1997, SURRC report to MAFF.

[7] Schreiber, G.A., Wagner, U., Helle, N., Ammon, J., Buchholtz, H.-V., Delincée, H., Estendorfer, S., von Grabowski, H.-U., Kruspe, W., Mainczyk, K., Münz, H., Schleich, C., Vreden, N., Wiezorek., C., and Bögl, K.W.: Thermoluminescence analysis to detect irradiated fruit and vegetables -an intercomparison study. Bericht des Instituts für Sozialmedizin und Epidemiologie des Bundesgesundheitsamtes. SozEp-Heft 3/1993, German Federal Health Office (Bundesgesundheitsamt, Berlin).

[8] Sanderson, D.C.W., Carmichael, L., Naylor, J.D. and Fisk, S.: An international collaborative blind trial of thermoluminescence detection of irradiated fruits and vegetables, 1997, SURRC report to MAFF.

[9] Marchioni, E., Anklam, E., Chabane, S., Delincée, H., Douifi, L., Hungerbühler, H., Pelleau, Y., Pinnioja, S., Raffi, J., Sanderson, D., Wagner, U.: Detection by Thermoluminescence of an Irradiation Treatment of Five Species of Dehydrated Fruit and Vegetables. Report on a CTCPA/AIFLD International Interlaboratory Study. Edited by Marchioni, E., Delincée, H. Berichte der Bundesforschungsanstalt für Ernährung, 1999, BFE-R—99-02 (Bundesforschungsanstalt für Ernährung, Karlsruhe).

[10] Schreiber, G.A., Wagner, U., Ammon, J., Brunner, J., Butz, B., Carmichael, L.A., Delincée, H., Eisen, S., Fienitz, B., Hammerton, K., Helle, N., Jahr, D., Kispéter, J., Klein, H., Kruspe, W., Kühn, T., Mainczyk, K., Meyer, H., Münz, H., Nootenboom, H., Pfordt, J., Sanderson, D.C.W., Schleich, C., Vreden, N., Zachäus, U., Zoost, C., Bögl, K.W.: An Interlaboratory Study on the Identification of Irradiated Potatoes and on the Estimation of Applied Doses by Thermoluminescence Analysis. Report of the Federal Institute for Health Protection of Consumers and Veterinary Medicine, 1997, BgVV-Hefte 13/1997 (Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin, Berlin).

[11] Sanderson, D.C., Slater, C., and Cairns K.J.: Detection of irradiated food. Nature, 1989, **340**, 23-24.

[12] Sanderson, D.C.W., Slater, C., and Cairns, K.J.: Thermoluminescence of foods: origins and implications for detecting irradiation. Radiat. Phys. Chem. 1989, **34**, 915-924.

[13] Sanderson, D.C.W.: Luminescence detection of irradiated foods. Food Irradiation and the Chemist, edited by Johnston, D.E. and Stevenson, M.H.: Royal Society of Chemistry, Cambridge, UK, 1990, 25-56.

[14] Heide, L., Guggenberger, R., and Bögl, K.W.: Application of thermoluminescence measurements to detect irradiated strawberries. J. Agric. Food Chem., 1990, **38**, 2160-2163.

[15] Autio, T., and Pinnioja, S.: Identification of irradiated foods by the thermoluminescence of mineral contamination. Z. Lebensm. Unters. Forsch., 1990, **191**, 177-180.

[16] Göksu, H.Y., Regulla, D.F., Hietel, B., and Popp, G.: Thermoluminescent dust for identification of irradiated spices. Rad. Protect. Dosimetry, 1990, **34**, 319-322.

[17] Delincée, H.: Detection methods for irradiated food. Proceedings Symp. "Irradiation for the food sector", Saint-Hyacinthe (Québec) Canada, 13 May, 1992, Agriculture Canada, 24-60.

[18] Sanderson, D.C., Carmichael, L.A., Clark, P.A., and Clark, R.J.: Development of luminescence tests to identify irradiated food. Final report on project N 1701. Thermoluminescence of irradiated fruits and vegetables, 1992, SURRC report to MAFF.

[19] Autio, T., and Pinnioja, S.: Identification of irradiated foods by thermoluminescence of the contaminating minerals.: Recent advances on detection of irradiated food. Edited by Leonardi, M., Raffi, J.J., Belliardo, J.-J. BCR-Information. 1993, Luxembourg: Commission of the European Communities, 1993, (Report EUR/14315/en), 183-191.

[20] Pinnioja, S., Autio, T., Niemi, E., and Pensala, O.: Import control of irradiated foods by the thermoluminescence method. Z. Lebensm. Untersuch. Forsch., 1993, **196**, 111-115.

[21] Pinnioja, S.: Suitability of the thermoluminescence method for detection of irradiated foods. Radiat. Phys. Chem., 1993, **42**, 397-400.

[22] Schreiber, G.A., Hoffmann, A., Helle, N., and Bögl, K.W.: Methods for routine control of irradiated food: determination of the irradiation status of shellfish by thermoluminescence analysis. Radiat. Phys. Chem., 1994, **43**, 533-544.

[23] Schreiber, G.A., Ziegelmann, B., Quitzsch, G., Helle, N., and Bögl, K.W.: Luminescence techniques to identify the treatment of foods by ionizing radiation. Food Structure, 1993, **12**, 385-396.

[24] MAFF: Detection of Irradiated Herbs and Spices. Ministry of Agriculture, Fisheries and Foods, London: MAFF validated methods for the Analysis of foodstuffs, 1992, V 27, 16 p.,- also published as MAFF: Detection of Irradiated Herbs and Spices: Scottish Universities Research and Reactor Centre Procedure for Thermoluminescence Detection of Irradiated Herbs and Spices using renormalized separated minerals, J. Assoc. Publ. Analysts, 1993, **29**, 187-200.

[25] Untersuchung von Lebensmitteln: Nachweis einer Strahlenbehandlung (ionisierende Strahlung) von - Gewürzen und Gewürzmischungen (L 53.00-2), - Frischobst (L 29.00-3) und - Shrimps (L 12.01.02-1) durch Messung der Thermolumineszenz. (Identification of irradition treatment (ionizing radiation) of spices and spice mixtures [L 53.00-2], fresh fruit [L 29.00-3] and shrimps [L 12.01.02-1] by thermoluminescence analysis). In: Amtliche Sammlung von Untersuchungsverfahren nach § 35 LMBG; Verfahren zur Probenahme und Untersuchung von Lebensmitteln, Tabakerzeugnissen, kosmetischen Mitteln und Bedarfsgegenständen/BgVV (In: Official Collection of methods under article 35 of the German Federal Food Act; Methods of sampling and analysis of foods, tobacco products, cosmetics and commodity goods/BgVV). Loseblattausgabe, Stand Nov. 1994 Bd. 1 (loose leaf edition, as of 1994-11 Vol. 1) Berlin, Köln: Beuth Verlag GmbH.

[26] Raffi, J., Delincée, H., Marchioni, E., Hasselmann, C., Sjöberg, A.-M., Leonardi, M., Kent, N., Bögl, K.W., Schreiber, G.A., Stevenson, H., and Meier, W.: Concerted Action of the Community Bureau of Reference on Methods of Identification on Irradiated Foods. BCR-Information. 1994, Luxembourg: Commission of the European Communities, 1994, (Report EUR/15261/en).

[27] Sanderson, D.C.W., Carmichael, L. A., Ni Riain, S., Naylor, J., and Spencer, J. Q.: Luminescence studies to identify irradiated food. Food Sci. Technol. Today, 1994, **8**, 93-96.

[28] Sillano, O., Román, A., Deza, A., Rubio, T., and Espinoza, J.: Application of thermoluminescence measurements to detect low dose gamma-irradiated table grapes. Radiat. Phys. Chem., 1994, **43**, 585-588.

[29] Calderón, T., Rendell, H.M., Beneitez, P., Townsend, P.D., Millán, A., and Wood, R.: Thermoluminescence spectra of inorganic dust from irradiated herbs and spices. J. Food Sci., 1994, **59**, 1070-1071.

[30] Raffi, J., Fakirian, A. and Lesgards, G.: Comparison between electron spin resonance and thermoluminescence in view of identification of irradiated aromatic herbs. Ann. Fals. Exp. Chim., 1994, **87**, 125-134.

[31] Sanderson, D.C.W., Carmichael, L.A., and Naylor, J.D.: Recent advances in thermoluminescence and photostimulated luminescence detection methods for irradiated foods. Detection Methods for Irradiated Foods - Current Status. Edited by McMurray, C.H., Stewart, E.M., Gray, R., and Pearce, J., Royal Society of Chemistry, Cambridge, UK, 1996, 124-138.

[32] Hammerton, K.M. and Banos, C.: Detection of irradiated spices by thermoluminescence analysis. Detection Methods for Irradiated Foods - Current Status. Edited by McMurray, C.H., Stewart, E.M., Gray R., and Pearce J., Royal Society of Chemistry, Cambridge, UK, 1996, 168-177.

[33] Roberts, P.B. and Hammerton, K.M.: Thermoluminescence detection of irradiated herbs and spices: An Australasian trial. Detection Methods for Irradiated Foods - Current Status. Edited by C.H. McMurray, E.M. Stewart, R. Gray, and J. Pearce, Royal Society of Chemistry, Cambridge, UK, 1996, 178-181.

[34] Kawamura, Y., Murayama, M., Uchiyama, S., and Saito, Y.: Comparison of thermoluminescence detection methods for irradiated spices. Detection Methods for Irradiated Foods - Current Status. Edited by C.H. McMurray, E.M. Stewart, R. Gray, and J. Pearce, Royal Society of Chemistry, Cambridge, UK, 1996, 149-157.

[35] Sanderson, D.C.W., Carmichael, L.A., Spencer, J.Q., and Naylor, J.D.: Luminescence detection of shellfish. Detection Methods for Irradiated Foods - Current Status. Edited by C.H. McMurray, E.M. Stewart, R. Gray, and J. Pearce, Royal Society of Chemistry, Cambridge, UK, 1996, 139-148.

[36] Calderón, T., Correcher, V., Millán, A., Beneitez, P., Rendell, H.M., Larsson, M., Townsend, P.D., and Wood, R.A.: New data on thermoluminescence of inorganic dust from herbs and spices. J. Phys. D: Appl. Phys., 1995, **28**, 415-423.

[37] Beneitez, P., Correcher, V., Millán, A., and Calderón, T.: Thermoluminescence analysis for testing the irradiation of spices. J. Radioanalyt. Nucl. Chem., 1994, **185**, 401-410.

[38] Khan, H.M. and Delincée, H.: Detection of irradiation treatment of dates using thermoluminescence of mineral contaminants. Radiat. Phys. Chem., 1995, **46**, 717-720.

[39] Pinnioja, S. and Pajo, L.: Thermoluminescence of minerals useful for identification of irradiated seafood. Radiat. Phys. Chem., 1995, **46**, 753-756.

[40] Polónia, I., Esteves, M.P., Andrade, M.E., and Empis, J.: Identification of irradiated peppers by electron spin resonance, thermoluminescence and viscosity. Radiat. Phys. Chem., 1995, **46**, 757-760.

[41] Khan, H.M.and Delincée, H.: Detection of radiation treatment of spices and herbs of Asian origin using thermoluminescence of mineral contaminants. Appl. Radiat. Isot., 1995, **46**, 1071-1075.

[42] Sanderson, D.C.W., Carmichael, L.A., and Naylor, J.D.: Photostimulated luminescence and thermoluminescence techniques for the detection of irradiated food. Food Sci. Technol. Today, 1995, **9**, 150-154.

[43] Lesgards, G., Fakirian, A., and Raffi, J.: Thermoluminescence identification of irradiated foodstuffs: LARQUA research. Detection Methods for Irradiated Foods -Current Status. Edited by C.H. McMurray, E.M. Stewart, R. Gray, and J. Pearce, Royal Society of Chemistry, Cambridge, UK, 1996, 158-167.

[44] Leffke, A., Helle, N., Linke, B., Bögl, K.W., and Schreiber, G.A.: Studies on detection of irradiated citrus fruit and grains: Germination and some other techniques. Recent advances on detection of irradiated food. Edited by Leonardi, M., Raffi, J.J., and Belliardo, J.-J. BCR-Information, 1993, Luxembourg: Commission of the European Communities, 1993, (Report EUR/14315/en), 111-121.

[45] Khan, H.M., Bhatti, I.A., and Delincée, H.: Identification of irradiated pulses by thermoluminescence of the contaminating minerals. Radiat. Phys. Chem. 1998, **52**, 145- 149.

[46] Villavicencio, A.L.C.H., Mancini-Filho, J., and Delincée, H.: Application of different techniques to identify the effects of irradiation on Brazilian beans after six months storage. Radiat. Phys. Chem.1998, **52**, 161-166.

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