SLOVENSKI STANDARD
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Nadomeġļa:
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Živila - Detekcija obsevane hrane z uporabo fotostimulativne luminescence

Foodstuffs - Detection of irradiated food using photostimulated luminescence

Lebensmittel - Nachweis von bestrahlten Lebensmitteln mit Photostimulierter Lumineszenz

Produits alimentaires - Détection d'aliments ionisés par luminescence photostimulée

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67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products

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Foodstuffs - Detection of irradiated food using photostimulated luminescence

Produits alimentaires - Détection d'aliments ionisés par luminescence photostimulée

Lebensmittel - Nachweis von bestrahlten Lebensmitteln mit Photostimulierter Lumineszenz

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Foreword

This document (EN 13751:2009) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

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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 [1]. From other studies it may be concluded that the method is applicable to a large variety of foods [2], [3], [4].

2 Terms and definitions

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

2.1
photostimulated luminescence
PSL
radiation specific phenomenon resulting from energy stored by trapped charge carriers

NOTE Release of this stored energy by optical stimulation can result in a detectable luminescence signal.

2.2
PSL intensity
amount of light detected during photostimulation, in photon count rate

2.3
screening PSL
initial PSL
PSL intensity recorded from the sample as received or following preparation

2.4
calibrated PSL
PSL intensity recorded from the test sample following irradiation to a known dose, after initial PSL measurement

2.5
thresholds
values of PSL intensity used for classification. In screening mode, two thresholds, a lower threshold (T_1) and an upper threshold (T_2) are used to classify the sample

2.6
negative PSL result
PSL intensity below the lower threshold (less than T_1)

2.7
intermediate PSL result
PSL intensity between the upper and the lower threshold (greater than or equal to T_1, less than or equal to T_2)

2.8
positive PSL result
PSL intensity above the upper threshold (greater than T_2)

2.9
dark count
photon count rate from the photomultiplier with an empty chamber in the absence of stimulation

2.10
light count
photon count rate with a reference light source (e.g. ^{14}C loaded scintillant, or equivalent) in the sample chamber

2.11
empty chamber run
PSL intensity measured from an empty sample chamber to ensure absence of contamination of the chamber

3 Principle

3.1 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 [5], [6], [7]. It has subsequently been shown that the same spectra can be obtained from whole herb and spice samples and other foods using photostimulation [2], [8], [9]. 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).

3.2 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, e.g. [3], [4], [8].

3.3 Calibrated PSL

For calibration, the sample is exposed to a defined radiation dose after the initial PSL measurement, and then re-measured. 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.

4 Reagents

4.1 Aerosol silicone grease, e.g. Electrolube SC0200H[1)]

4.2 Water, deionized

5 Apparatus

5.1 PSL system, e.g. SURRC PPSL Irradiated food screening system[1)] [10], [11], [12], [13],

comprising sample chamber, stimulation source, pulsed stimulation and synchronised photon counting system. For instrumental set-up, see 7.4.

NOTE For the interlaboratory tests, the SURRC PPSL system has been used.

5.2 Disposable Petri-dishes

NOTE For the interlaboratory tests, 5 cm Petri-dishes have been used.

5.3 Radiation source,

capable of irradiating samples with a defined radiation dose before measurement of calibrated PSL. In the interlaboratory tests on shellfish and herbs, spices and their mixtures [1], sources delivering ^{60}Co-rays have been employed at a fixed radiation dose of 1 kGy.

Alternative sources may be used providing they have been found satisfactory.

NOTE Other fixed doses can be suitable.

5.4 ^{14}C-Source (optional)

5.5 Laminar flow cabinet (optional)

5.6 Air duster (optional)

6 Sampling technique

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

Before analysis, samples should be protected against light exposure. Store them in the dark.

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1) Electrolube SC0200H and Scottish Universities Research and Reactor Center Pulsed Photostimulated Luminescence (SURRC PPSL) are examples of products available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement of CEN of these products. Equivalent products may be used if they can be shown to lead to equivalent results.

7 Procedure

7.1 General

All dispensing and handling of samples should be carried out under subdued lighting whenever possible. Samples are dispensed into disposable Petri-dishes and introduced to the system.

Samples should be handled with care to avoid cross-contamination during dispensing. It is recommended that samples are dispensed individually, under a laminar flow cabinet (5.5), and fresh tissue is placed on the bench for each sample. The Petri-dish should be covered with a lid to reduce the possibility of contamination.

7.2 Preparation of herb, spice and seasoning samples

Samples are dispensed into clean Petri-dishes, in duplicate. If these test samples lead to inconsistent classifications, a further four aliquots shall be dispensed and classification based on the highest two results. Some samples can require a minimum of preparation; e.g. vanilla pods may need to be cut to fit the dish and wrappings should be removed.

Samples can either be dispensed in a thick layer within the Petri-dish or in a thin layer, applied to a dish already sprayed with silicone grease (4.1) to fix the sample. Thicker layer samples are less likely to be affected by bleaching; subsurface minerals can be exposed by gentle agitation.

NOTE Thin layer samples can also be dispensed into planchets or other shallow containers suitable for irradiation with ^{90}Sr or other sources. If a gamma source is used for calibration either dispensing method is suitable.

7.3 Preparation of shellfish

7.3.1 General

PSL analysis can be conducted using whole samples including shell, shelled whole samples and dissected intestines or minerals extracted by flushing with water (4.2).

If enough sample material is available, it is recommended that samples be divided into at least six portions, i.e. six Petri-dishes.

7.3.2 Whole samples

Whole samples including shell can be placed as received in the Petri-dish. In some cases it can be necessary to cut the shellfish to fit the Petri-dish. If the intestinal tract is visible, it is preferable to place this uppermost.

7.3.3 Shelled whole samples

Shelled whole samples can be placed whole in the Petri-dish, again with the intestinal tract facing upwards, using as many individual shellfish as will fit in the Petri-dish.

7.3.4 Shellfish intestines

Shellfish intestines can be found as a thin dark tube on the convex side of prawns or shrimps, and in the interiors of molluscs. Using a scalpel, slice the flesh open and with tweezers remove the intestinal tract. Repeat this technique on several samples of shellfish (recommended: 6 intestines per Petri-dish).

7.4 Instrumental Set-Up

This section describes the set-up of the SURRC PPSL system, as an example.