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Livsmedel – Analys av bestrålade livsmedel innehållande kristallint socker – ESR-spektroskopi

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

ICS 67.050

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English version

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

Produits alimentaires - Détection par spectroscopie de Résonance Paramagnétique Electronique d'aliments ionisés contenant des sucres cristallisés

Lebensmittel - ESR-spektroskopischer Nachweis von bestrahlten Lebensmitteln, die kristallinen Zucker enthalten

This European Standard was approved by CEN on 20 August 2001.

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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 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of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.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 European Standard was elaborated on the basis of a protocol developed following a concerted action supported by the Commission of European Union (XII C.). Experts and laboratories from E.U. and EFTA countries, contributed jointly to the development of this protocol.

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 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 = + ½ and m_s = - ½, 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 Apparatus and equipment

Usual laboratory apparatus and, in particular, the following:

3.1 Commercially available X-Band ESR spectrometer including magnet, microwave bridge, console with field-controller and signal-channel, rectangular or cylindrical cavity

3.2 G-value measurement unit including frequency counter, magnetic field probe (nuclear magnetic resonance (NMR) Gaussmeter), or any other built-in g-value measurement unit.

3.3 ESR tubes, of internal diameter about 4,0 mm (e.g. Suprasil®1) quartz tubes)

3.4 Balance, accurate to the nearest 1 mg (optional)

3.5 Laboratory vacuum oven, or freeze dryer

3.6 Scalpel

4 Procedure

4.1 Sample preparation

Prepare suitable pieces (50 mg to 100 mg) of the fruit, e.g. using a scalpel.

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1) Suprasil® is an example of a suitable product available commercially. This information is only given for the convenience of users of this International Standard and does not constitute an endorsement by CEN of this product.

NOTE Various parts of the fruits can contain different quantities of crystalline sugars. It can be advantageous to take the test sample from the outer parts of the fruits.

Transfer a test portion directly into the ESR tube (3.3) and start the measurement.

Difficulties in tuning the spectrometer cavity can be experienced if the sample is insufficiently dry. In this case either reduce the sample quantity or dry it further. Samples should be dried in a laboratory vacuum oven at approximately 40 °C under reduced pressure or in a freeze-dryer.

WARNING: Excessive heating can reduce the signal.

4.2 ESR Spectroscopy

4.2.1 Spectrometer settings

Use a time constant and sweep rate appropriate for ESR signals with linewidths of 0,2 mT to 0,4 mT. For example, the following ESR spectrometer settings have been found to be satisfactory:

Microwave radiation: 9,78 GHz[2)], power 5 mW

Magnetic field: 348 mT centre field[2)], sweep width 10,0 mT to 20,0 mT;

Signal channel: 50 kHz or 100 kHz modulation frequency,
0,15 mT to 0,4 mT modulation amplitude;
100 ms to 200 ms time constant[3)]
sweep rate 5 mT min^{-1} to 10 mT min^{-1}
or accumulation of 3 to 5 spectra at greater sweep rate and shorter time constant;

Gain: between 10^4 and 10^6;

Temperature: ambient temperature.

4.2.2 Analysis of sample

Analyse the sample prepared as described in 4.1 in an ESR tube (3.3).

5 Evaluation

5.1 Identification of irradiated samples

5.1.1 General

Irradiated food containing crystalline sugar show typical multicomponent ESR spectra reflecting the presence of radiation-induced radicals in the sample. Dried fruits often contain sugar particles in crystalline form, and therefore the appearance of a typical multicomponent ESR spectrum (see annex A) indicates radiation treatment. Due to different mono- and disaccharides and due to the changes in saccharide composition various ESR spectrum types can occur.

Other irradiated sugar-containing foodstuffs reveal ESR spectra which have similar structures. Since the overall spectrum structure depends on the radical composition and on the crystallinity of the mono- and disaccharides present in the sample, variations in the spectrum characteristics occur.

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2) These values are for the specified microwave frequency and magnetic field; if the frequency is higher (lower) the magnetic field strength will be higher (lower).

3) These values are for the specified sweep rate.