



SLOVENSKI STANDARD
SIST EN 1787:2022

01-junij-2022

Nadomešča:
SIST EN 1787:2001

Živila - Določevanje obsevanosti živil, ki vsebujejo celulozo, s spektroskopijo ESR

Foodstuff - Detection of irradiated foodstuff containing cellulose by ESR spectroscopy

Lebensmittel - ESR-spektroskopischer Nachweis von bestrahlten cellulosehaltigen
Lebensmitteln

Produits alimentaires - Détection par spectroscopie RPE d'aliments ionisés contenant de
la cellulose

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ICS:

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

EN 1787

NORME EUROPÉENNE

EUROPÄISCHE NORM

April 2022

ICS 67.050

Supersedes EN 1787:2000

English Version

Foodstuff - Detection of irradiated foodstuff containing cellulose by ESR spectroscopy

Produits alimentaires - Détection par spectroscopie
RPE d'aliments ionisés contenant de la cellulose

Lebensmittel - ESR-spektroskopischer Nachweis von
bestrahlten cellulosehaltigen Lebensmitteln

This European Standard was approved by CEN on 14 February 2022.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 1787:2022) 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of an identical text or by endorsement, at the latest by October 2022, and conflicting national standards shall be withdrawn at the latest by October 2022.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1787:2000.

The predecessor of 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 EU and EFTA countries, contributed jointly to the development of this protocol.

In comparison with the previous edition, the entire document was editorially revised according to current rules. Additionally, the following technical modifications have been made:

- a) the scope was supplemented by the information, that chemical bleaching of nuts in shells can lead to comparable signals;
- b) clause “Terms and definitions” was added;
- c) scientific language usage of clause “Principle” had been clarified;
- d) former 4.1, 4.2 and 4.7 were updated and a footnote added for the g-value calculation;
- e) clause “Procedure” was scientifically refined, its normative character (i.e. provisions set out) modified towards more exemplary/suggestive expressions of provision and aligned with EN 13708;
- f) subclause “General” was added to subclause “Sample preparation”;
- g) clause “Evaluation” was amended by supplementing two subclauses, 7.1 “G-value calculation” and 7.2 “Identification of irradiated samples”, in alignment with EN 13708, including implementing its former content into 7.2, whereas “6,0 mT” was refined to “6,05 mT ± 0,05 mT”;
- h) clause “Limitations” was extended;
- i) Figures A.5 and A.7 of irradiated but bleached nutshells from hazelnuts and walnuts and Figures A.6 and A.8 of irradiated (not bleached) nutshells from hazelnuts and walnuts were added;
- j) Annex B was extended by the matrix hazelnuts;
- k) the Bibliography was updated and extended by entry [23], [24] and [25].

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Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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1 Scope

This document specifies a method for the detection of foodstuff containing cellulose which have been treated with ionizing radiation, by analysing the electron spin resonance (ESR) spectrum, also called electron paramagnetic resonance (EPR) spectrum, of the foodstuff, see [1] to [13].

Interlaboratory studies have been successfully carried out with pistachio nut shells, [14] to [18], paprika powder [19] and [20] and fresh strawberries [21]. However, it has been shown that chemical bleaching of nuts in shells can lead to comparable signals. For further information, see Clause 8 on limitations.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 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 $m_s = +\frac{1}{2}$ and $m_s = -\frac{1}{2}$, 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 [13].

Radiation treatment produces specific radicals that can be mostly detected in solid and dry parts of the foodstuff. The intensity of the signal obtained increases with the concentration of the paramagnetic compounds and thus with the applied dose.

5 Apparatus and equipment

Usual laboratory apparatus and, in particular, the following:

- 5.1 **Commercially available X-Band ESR spectrometer** including magnet (electro or permanent), microwave bridge, console with field- controller and signal-channel, rectangular or cylindrical cavity¹.
- 5.2 **ESR tubes**, suitable for the ESR spectrometer used (e.g. Suprasil^{®2} quartz tubes).
- 5.3 **Balance**, accuracy of 1 mg (optional).
- 5.4 **Laboratory vacuum oven** or **freeze dryer**.
- 5.5 **Electric blender**.
- 5.6 **Filter paper**.
- 5.7 **Scalpel, pincers**.
- 5.8 **Water** complying with at least grade 3 of EN ISO 3696.

6 Procedure

6.1 Sample preparation

6.1.1 General

Do not grind the samples since grinding could either diminish the signal to noise ratio and also cause a change of the shape of the ESR spectrum or induce other ESR signals [25].

6.1.2 Shells and stones

Remove pieces of suitable size (about 50 mg to 100 mg, e.g. 3,0 mm to 3,5 mm in diameter) from the shells or stones of the foodstuff, e.g. using a scalpel or pincers. Drying [e.g. in a freeze-dryer or at approximately 40 °C in a laboratory vacuum oven (5.4)] is usually not necessary in the case of nutshells but recommended for pips and kernels of fruits.

6.1.3 Spices

For example, use about 150 mg to 200 mg of the spice sample. Drying (e.g. in a freeze-dryer or at approximately 40 °C in a laboratory vacuum oven (5.4)) is usually not necessary.

¹ g-value calculation unit including frequency counter magnetic field probe (magnetic resonance Teslameter) or any other built in g-value calculation unit.

² Suprasil[®] 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 of CEN or CENELEC of this product.

6.1.4 Strawberries

Strawberry samples should be measured immediately after receipt. Otherwise store the samples at approximately $-18\text{ }^{\circ}\text{C}$ until analysis.

For ESR measurement about 200 mg of seeds (achenes) of strawberries are needed. These can be gained usually from about 80 g of strawberries.

For separation of the small seeds from the main fruit body either peel off the skin (recommendation: in frozen state) or use the whole fruit (without stalks and leaves). Homogenize the strawberries in an electric blender (5.5). Add 500 ml of water to the fruit pulp and stir thoroughly. Allow the seeds to settle and decant most of the water together with the floating fruit pulp. Repeat this procedure once or twice to remove any remaining fruit pulp.

Place the seeds on filter paper to remove adhering water. Dry the seeds in a freeze dryer or at approximately $40\text{ }^{\circ}\text{C}$ in a laboratory vacuum oven (5.4) e.g. for 2 h.

Storing samples in the frozen state will not adversely affect the detection of treatment with radiation.

6.2 ESR Spectroscopy

6.2.1 Spectrometer settings

The parameters shown in Table 1 have been found to be successful in interlaboratory tests (see Clause 9). The values shown (Table 1) are given as examples and should be optimized per sample and ESR spectrometer as required.

Use a time constant and sweep rate (or sweep time) appropriate for an ESR signal with a peak to peak linewidth of approximately 0,8 mT.

Table 1 — Example for ESR spectrometer settings

Parameter	Setting
Microwave radiation:	Frequency 9,78 GHz ^a , power 0,4 mW (for e.g. pistachio nuts) to 0,8 mW (for e.g. paprika powder or strawberries) ^b .
Magnetic field:	348 mT centre field ^a , sweep width 20 mT.
Signal channel:	50 kHz or 100 kHz modulation frequency; 0,4 mT to 1,0 mT modulation amplitude; 100 ms to 200 ms time constant ^c , sweep rate 5 mT min ⁻¹ to 10 mT min ⁻¹ (sweep time 2,4 min to 1,2 min) or accumulation of 3 to 5 spectra at greater sweep rate and shorter time constant.
Gain:	Between approximately 10 ⁴ and 10 ⁶ .
Temperature:	Ambient temperature.
^a 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). ^b If saturation is suspected, the microwave power should be reduced, see [10]. ^c These values are for the specified sweep rate.	

EN 1787:2022 (E)**6.2.2 Analysis of sample**

Analyse the sample prepared as described in 6.1 in an ESR tube (5.2).

7 Evaluation**7.1 G-value calculation**

For calculating the g -value of the centre (i.e. zero point) of the ESR spectra (Figures A.1 to A.4, A.6 and A.8) it is necessary to measure the frequency ν (e.g. frequency counter) and the field B (e.g. gaussmeter) at this point.

A g -value of a signal, g_{signal} (g_S), is calculated using Formula (1):

$$g_S = \frac{71,448 \cdot \nu_{\text{ESR}}}{B} \quad (1)$$

where

ν_{ESR} is the microwave frequency, in Gigahertz (GHz);

B is the magnetic flux density (magnetic field setting of the spectrometer), in Millitesla (mT);
(10 Gauss = 10 G = 1 mT).

7.2 Identification of irradiated samples

A single central signal (C), with approximately $g_S = 2,004$ (Figure A.1 to A.4, A.6 and A.8). is observed in the ESR spectra of all foodstuff containing cellulose, including unirradiated samples. In the case of irradiated samples, the intensity of this signal is usually much greater and, a pair of lines occurs to the left (at lower magnetic field) and right (at higher magnetic field) of the central signal.

This pair of lines is due to cellulose radicals formed by the ionizing radiation. The spacing of this radiation-induced signal pair is $6,05 \text{ mT} \pm 0,05 \text{ mT}$ and is symptomatic of radiation treatment having taken place (see Figures A.2, A.4, A.6 and A.8).

In some types of foodstuff, broad lines of low intensity due to paramagnetic Mn^{2+} ions are observed in addition to the signals mentioned. However, their position in the magnetic field is different, and the spacing between two manganese lines being about 9,0 mT (coupling constant) differs from the spacing of the irradiation specific signals.

To check the sensitivity of the sample it might be helpful to irradiate a portion of the sample with an appropriate technologically relevant dose and examine it again.