Procedure for determining radionuclides in foodstuffs at elevated levels of contamination by gamma spectrometry

E-γ-SPEKT-LEBM-02

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Procedures manual for monitoring of radioactive substances in the environment and of external radiation
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Procedure for determining radionuclides in foodstuffs at elevated levels of contamination by gamma spectrometry

1 Scope

The procedure described in the following is to be applied for the analysis of foodstuffs (except milk and milk products, fish and fish products) that have been affected by an elevated degree of contamination through the deposition of increased activities following a nuclear weapons test or accident at a nuclear installation.

The gamma spectrometric analyses are meant to quickly obtain an overview of the type, degree and extent of the contamination of affected foodstuffs.

2 Sampling

2.1 General

In the case of an event, the type of samples, extent of sampling, and selection of sampling locations are determined by the existing monitoring programmes that are based upon the Precautionary Radiation Protection Act.

Particular safety measures need to be taken during sampling and the subsequent processing of these samples in the case of elevated activity concentrations, both to prevent the dispersal of radioactive substances and the contamination of laboratories and measuring equipment.

The notes on sampling and sample selection specified in section 2 of the procedure E-γ-SPEKT-LEBM-01 apply here analogously.

For controlling elevated emissions of radionuclides, the sample material should be stored in well-sealed containers already during sampling (e. g., polyethylene vessels with screw caps or polyethylene bags).

2.2 Principles of sample selection at elevated contamination levels

The aim of sampling is to obtain an overview of the degree of contamination. The selection of samples should therefore be random from a given set. By no means should a pre-selection be made with the aid of a contamination monitor.

3 Analysis

3.1 Principle of the procedure

The samples are analysed directly with a Ge-gamma spectrometer in a suitable measurement geometry.

3.2 Sample preparation

The principle, "only material ready for consumption is to be analysed" should also prevail in the case of more severely contaminated foodstuffs. The directives for the processing of samples provided in procedure E-γ-SPEKT-LEBM-01 apply analogously.

It is recommended that a dedicated room be used for the preparation of more severely contaminated samples. The workflow has to be organised in such a manner that the possibility of cross-contamination is excluded. Samples with higher activity concentrations must never be filled into measuring containers in the rooms that are used for the actual measurements. When transferring samples from one container to another, care must be taken not to contaminate the outside of the sample measurement vessels. The sample measurement vessels should additionally be sealed in PE-bags. The sample measurement vessels and PE-bags must not be reused. Workstations should be covered with PE-foil to simplify their decontamination.

Tools and workstations need to be monitored with a contamination monitor. The directives for handling open radioactive substances of the Radiation Protection Ordinance must be complied with.

3.3 Radiochemical separation

No radiochemical separation is required for the gamma spectrometric procedure outlined here.

4 Measuring the activity

Fundamental aspects of, and support for, gamma spectrometry are outlined in chapters IV.1.1 through IV.1.3 of this procedures manual.

Gamma spectra are measured with a Ge-spectrometer (> 15 % relative efficiency compared to a 3" x 3" NaI(Tl)-detector for the 1,33 MeV-line of Co-60). Samples are preferably measured in flat-bottomed, cylindrical vessels with screw caps (e.g., PE wide-necked) with a volume of 0,5 litre or 1 litre.

The sample vessel is placed on, or in front of, the detector. If sample masses vary, the impact of the filling height on the efficiency needs to be known. The measurement periods need to be adapted to the activity concentrations of the samples.

In the case of elevated immissions of radionuclides, it is absolutely crucial that the measuring room is free of any contamination.

In order to prevent the introduction of radioactivity to the measuring room, it is recommended to change clothing and shoes or wear a laboratory coat and overshoes.

Faulty readings must be avoided by scheduling quality-control measurements for background effect more frequently than during routine operations.

To prevent contamination of the detector caps, it is recommended to cover the cap with polyethylene foil (ca. 0,1 mm thick) and fix it in place with adhesive tape. In

the case of an external contamination of the foil by liquids or dust from a leak in the sample vessel, such contamination can be easily removed by replacing the foil. Even acidic radionuclide solutions will not very rapidly diffuse through this foil. If in spite of all precautions the detector cap (aluminium) becomes contaminated, it is suggested to decontaminate as follows: After removing the protective foil from the detector, its surface or neck is first wiped with absorbent paper soaked with water, then with a solution of hydrochloric acid (1 $mol \cdot l^{-1}$), and finally with an aqueous solution of tetra-sodium salt of ethylene diamine tetra-acetic acid (EDTA) 0,1 %.

The use of aqueous EDTA solution is always recommended when so-called corrosion nuclides, like, e.g., zinc, manganese, iron, cobalt etc., have contaminated the surface of a detector. The procedure is finally completed by cleaning the detector with water and acetone. The success of the decontamination procedure has to be verified by measuring the background spectrum.

5 Calculation of the results

High-performance software for PCs for the analysis of gamma spectra and for determining the activity of specific nuclides is available from a range of providers. Preference should be given to those programmes that make provision for the calculation of the decision threshold and detection limits of all major radionuclides according to chapter IV.5 of this procedures manual (see also chapter 6) and use the decision threshold in their search algorithms as the key criterion for deciding whether or not a line is distinct from the background.

In the case of an elevated degree of contamination it is essential that the laboratory be set up for a fully automated analysis of gamma spectra, because there will be large numbers of samples to be processed within short periods.

Measuring results of specific activity above the detection limit and the detection limits themselves always have to be stated in $Bq \cdot kg^{-1}$ wet mass (WM).

6 Characteristic limits of the procedure

The characteristic limits of the gamma spectrometry of more strongly contaminated foodstuffs are determined by the detector efficiency, the nuclear properties of the radionuclides in question, but especially by the radionuclide content of the sample to be measured. In this case, the background spectrum of the measurement configuration is insignificant.

Characteristic limits are calculated according to chapter IV.5 of this procedures manual (section 4.5, equation (4.32a)). If the algorithms of the analysis software used for the calculation of detection limits are not based upon the equation in chapter IV.5, it is necessary to a posteriori correct these results in an additional step. Examples for calculating the detection limits in gamma spectrometry can be found in chapter IV.5, sections 6.4 and 6.5. In the present case, these examples can be followed analogously.

The values obtained from a sample of beef are given in the following table and can serve as a guideline:

Detection limits in a sample of beef (Bq·kg^{-1} WM)

Radionuclide	Mass: Geometry: *E* (keV)	0,5 kg 1 l PE bottle	0,5 kg 1 l Marinelli
Co-60	1332,5	4,4	1,0
1-131	364,5	5,1	1,8
Te-132	228,2	4,5	1,9
1-132	667,7	4,9	1,6
1-133	529,9	4,6	1,6
Cs-134	604,7	4,0	1,3
Cs-137	661,7	4,9	1,7
Ba-140	537,4	18,5	5,8
La-140	1596,5	3,4	1,5

Measuring conditions: direct measurement of beef samples [0,5 kg WM in 1 litre PE bottles with screw caps (PE bottles) and 1 litre Marinelli beakers, respectively]; detector: Ge(Li) with 20 % relative efficiency; shielding: with 3 cm lead, steel, aluminium and Perspex each (from the outside to the inside); measurement period: 0,5 h.

7 Catalogue of chemicals and equipment

7.1 Chemicals

— Homogenisation agent (e. g., TWEEN 80);
— Acetone, techn.;
— EDTA, tetra-sodium salt of ethylene diamine tetra-acetic acid;
— Absorbent paper;
— PE-bags.

7.2 Equipment

— Household kitchen-type food processor;
— Household kitchen-type blender;
— GE- or Ge(Li)-semiconductor detector (> 15 % relative efficiency, half-width < 2,1 keV at 1,33 MeV) with pre-amplifier and high-voltage power supply unit;
— Main amplifier;
— Analogue-to-digital converter;
— Multi-channel analyser of the conventional type or corresponding external storage capacity with at least 4096 channels;
— PC with software suitable for the analysis of gamma spectra.