Procedure for determining higher concentrations of radionuclides in milk by gamma spectrometry

F-γ-SPEKT-MILCH-02

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Procedures manual for monitoring of radioactive substances in the environment and of external radiation
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1 Scope

The procedure described in the following is to be applied when elevated activities may have been deposited as a result of tests of nuclear weapons or an accident at a nuclear installation leading to a high contamination of milk. The gamma spectrometric measurements are intended to rapidly provide an overview of the type, severity and extent of the contamination of milk.

2 Sampling

Collecting and processing samples with elevated activity concentrations require improved caution to avoid contamination of laboratories and measuring instruments.

The recommended sample size is about 2 l of milk. Other than that, the sampling procedures described in procedure F-γ-SPEKT-MILCH-01 apply analogously.

3 Analysis

3.1 Principle of the procedure

The samples of milk are measured in their original liquid form with a Ge-gamma spectrometer in a 1 l-Marinelli beaker or, better, in a vessel with a tightly closing screw-cap.

3.2 Sample preparation

In the case of elevated activities, samples must not be transferred into the measuring vessels in the laboratory or room where the measuring process is to be carried out. During transfer, contaminating the measuring vessels on their outside surfaces must be strictly avoided. Measuring vessels must not be reused.

No further preparation of samples is required.

3.3 Radiochemical separation

No radiochemical separation is required.

4 Measuring the activity

Basic information on, and aids for, gamma spectrometry are provided in chapters IV.1.1 through IV.1.3 of this procedures manual.

The gamma spectra are measured using a Ge-spectrometer (> 15 % efficiency relative to a 3" x 3" NaI(Tl)-detector for the 1,33 MeV-line of Co-60). Liquid milk is measured in 1 l-Marinelli beakers or screw-capped vessels.

Measuring periods are to be adapted to the activity of the sample.

With regard to matters such as calibrating the gamma spectrometer, the problem of summation losses, which may reach significant values when large-volume detectors are used, as well as self-absorption issues in samples, detailed information is provided in section 4 of procedure F-γ-SPEKT-MILCH-01.

5 Calculation of the results

High-performance software for the analysis of gamma spectra and calculation of activity concentrations of radionuclides is available from a number of software suppliers. Preference should be given to software that makes provision for calculating decision thresholds and detection limits for all major radionuclides according to chapter IV.5 of this procedures manual (see also section 6) and employs the decision threshold as a criterion in the search algorithms to decide whether or not a line is distinct from the background.

In the case of elevated contamination levels it is essential that the laboratory be equipped with fully automated analysis facilities for gamma spectra, as large numbers of samples need to be processed rapidly. Programming and using so-called macros may be an effective means for automating analyses up to printing out the measurement results; this tool is available in most software products.

Nuclide activity concentrations or their detection limits are always to be reported in $Bq \cdot l^{-1}$ of milk.

6 Characteristic limits of the procedure

The characteristic limits of gamma spectrometry analyses of milk samples are determined by the efficiency of the detector used, the nuclear-physical data of the radionuclides to be measured and by the K-40 content of the sample to be measured. The background spectrum of the measuring configuration is less important when the K-40 content of the sample is large. In the case of more strongly contaminated samples, detection limits will also depend to a significant extent on the type and activity of the artificial radionuclides present.

Detection limits are calculated according to equation (4.32a) of chapter IV.5, section 4.5 of this procedures manual. If the algorithms for calculating the detection limits of the software used do not correspond to the equation in chapter IV.5, corrections may have to be applied subsequently.

Examples of how to calculate characteristic limits in gamma spectrometry are also provided in chapter IV.5, sections 6.4 and 6.5. These examples may be applied to the present case analogously.

Attainable detection limits are shown in the following table. The values were obtained by measuring liquid milk (1 l of milk in a Marinelli beaker) on a detector with 25 % relative efficiency, which like its shielding is a low-level model, applying measuring periods of 0,5 hours. The milk that was analysed for determining detection limits contained no significant activities of nuclides other than natural K-40 at an activity concentration of 50 $Bq \cdot l^{-1}$. The tabulated values for the detection limits of the reference radionuclide Co-60 and radionuclides that may be present in milk are reported in $Bq \cdot l^{-1}$.

Detection limits in milk

Radionuclide	Detection limit in $Bq \cdot l^{-1}$
Co-60	0,98
I-131	0,69
Te-132	0,64
I-132	0,73
I-133	0,69
Cs-134	0,69
Cs-136	0,73
Cs-137	0,83
Ba-140	2,55
La-140	0,77

Detection limits will be significantly higher in cases of elevated contaminations.

7 Catalogue of chemicals and equipment

7.1 Chemicals

Other than sodium azide for the preserving of samples, no other chemicals are required.

7.2 Equipment

- Marinelli beakers or screw-capped vessels for gamma spectrometric measuring;
- Ge- or Ge(Li)-semiconductor detector (> 15 % relative efficiency, full width at half maximum < 2,1 keV at 1,33 MeV) with pre-amplifier and high-voltage power supply unit;
- Main amplifier (NIM module);
- Analogue-digital converter (NIM module);
- Multi-channel analyser of the conventional type or with a corresponding external data processing unit, with at least 4096 channels (NIM module);
- Rack with power supply for NIM modules;
- PC or workstation with software for the analysis of gamma spectra.