Procedure for determining radionuclides in plant samples (indicators) by gamma spectrometry

F-γ-SPEKT-PFLAN-01

Author:

A. Wiechen

Federal coordinating office for soil, vegetation, animal feed and food of vegetable or animal origin

(Leitstelle für Boden, Bewuchs, Futtermittel und Nahrungsmittel pflanzlicher und tierischer Herkunft)

ISSN 1865-8725 Version November 1998

Procedures manual for monitoring of radioactive substances in the environment and of external radiation
(Messanleitungen für die „Überwachung radioaktiver Stoffe in der Umwelt und externer Strahlung")

Procedure for determining radionuclides in plant samples (indicators) by gamma spectrometry*

1 Scope

The procedures described in the following are to be used for analysing plant matter (indicators) that has to be routinely monitored according to the Precautionary Radiation Protection Act and the Guideline for the Monitoring of Emissions and Immissions of Nuclear Installations.

2 Sampling

2.1 Grass (not animal feed)

The area to be sampled should be distant from potential obstacles (buildings, trees, etc.) by a minimum of twice the height of the obstacles. The sampling area needs to be at least 200 m², free of pollutants, and contain only a minor weed fraction. Only in exceptional cases is it permissible to reduce the minimum size to 100 m², i. e., if there are no more expansive areas available. This may apply, for example, in the case of a nuclear installation that lacks larger areas in the surroundings of the maximum immission point. To ensure that measuring results are comparable, grass cut in the months of May or June should be used as sample material.

It is expedient to demarcate several homogeneously distributed collecting spots by placing a V2A-steel or plastic frame of 70 cm x 70 cm (ca. 0,5 m²) on the ground, cutting the grass at a level of about 2 cm above the ground (e. g., with lawn shears or a similar tool, not a lawnmower), then combining the cuttings to a mixed sample of 10 kg to 15 kg that is transported in a plastic bag to the laboratory. If the sample amounts to less than 10 kg of fresh mass, further spots have to be sampled. Being precise with regard to the dimensions of the spots sampled and collecting all sample material without loss is necessary if the results of the subsequent analysis are to be related to units of area, e. g. m². Contaminating the sample with soil and roots must be avoided.

2.2 Leaves, needles

Solitary trees or small stands of trees are selected for sampling.

Leaves are stripped off from low branches in September or October prior to their changing colour in autumn. This task is easier when wearing leather working gloves and collecting the leaves in a bucket carried in a harness. Leaves from the previous year lying under the trees must not be collected. The sample size should be about 5 kg wet mass.

Conifer needles are sampled by cutting off this year's shoots (tips of shoots beyond the terminal branching) from low branches of (preferably) firs in October with the

---

* These procedures have been worked out in collaboration with Dr. H. Weller, LUFA Speyer and the VDLUFA (Verband der Landwirtschaftlichen Untersuchungs- und Forschungsanstalten), Expert group XI, Workgroup «Radioanalysis».

aid of garden clippers. It is not permissible to collect needles from previous years, e.g., from cavities in the tree or even from the ground under the tree. The sample size should amount to about 3 kg wet mass.

3 Analysis

3.1 Principle of the procedure

The sample material is usually desiccated at 105 ºC, milled, and measured by gamma spectrometry in this form. If only a gamma spectrometer with a low efficiency and/or relatively high background is available, it might be necessary to ash the samples at temperatures not exceeding 400 ºC and measure the resultant ash in order to attain the required detection limits.

3.2 Sample preparation

3.2.1 Grass

It is general practice to dry the samples at 105 ºC to constant weight, then crush them in a cross-beater mill fitted with a 1 mm-sieve or another device serving the same purpose, and thoroughly mix them until homogeneous.

3.2.2 Leaves, needles

Preparing the samples follows the principle described in section 3.2.1. The needle samples must be separated from their branches prior to crushing. Once desiccated, the needles readily fall off their branches so that they just have to be collected.

3.2.3 Ashing

Laboratories with access only to a semiconductor detector with a low efficiency and/or lead shielding with a high background, with ensuing excessive measuring periods to attain the required detection limits, may concentrate the samples prepared according to sections 3.2.1 and 3.2.2 by ashing and measuring the resultant ash. However, samples of needles should not be ashed, as deflagrations may readily occur with the risk of accidents!

For ashing, the homogenized sample material is filled into quartz dishes or similar vessels and ashed at a furnace temperature not exceeding 400 °C. To this end, the quartz dishes are placed in the furnace while it is still cold. The furnace air supply is minimized to carbonise the sample material and prevent it from igniting. The ventilation openings of the furnace are opened only after completion of this carbonisation process to obtain ash that is as light in colour as possible. After the ash has cooled down, it is crushed and homogenised in a tumbling or 3D mixer filled with ceramic or agate balls.

To avoid deposits of carbonisation products in the chimney with the attendant fire risk and to minimize environmental pollution it is advisable to use an ashing furnace with a catalytic afterburner for ashing larger volumes of biological materials. As particularly useful have proven those furnace designs in which the ashing chamber is directly linked by a generously dimensioned connection to a catalytic afterburner unit with a separate heater.

The yield of ash is recorded so that the results of the measuring process can be related to the dry mass (DM).

3.3 Radiochemical separation

No radiochemical separation is required.

4 Measuring the activity

Basic information on, and aids for, gamma spectrometry are contained in chapters IV.1.l through IV.1.3 of this procedures manual.

The gamma spectra are measured with a Ge-spectrometer (> 15 % efficiency relative to a 3" x 3" NaI(Tl)-detector for the 1,33 MeV-line of Co-60) in 1 l-Marinelli beakers or, in the case of ash, in screw-capped vessels of 50 cm³.

5 Calculation of the results

High-performance software for the analysis of gamma spectra is available from a number of software suppliers. Preference should be given to software that not only makes provision for calculating the specific activities of all major radionuclides, but also calculates decision thresholds and detection limits 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.

Results of specific activities of the nuclides or their detection limits are always, also in the case of ash, to be reported in $Bq \cdot kg^{-1}$ DM (dry mass).

6 Characteristic limits of the procedure

The characteristic limits for radionuclides in plant samples are determined by the efficiency of the gamma spectrometer used, the nuclear data of the radionuclides to be measured, and particularly by the radionuclide spectrum of the measured sample. The background spectrum of the measuring configuration is less important, because plants contain particularly large amounts of potassium (K-40). These K-40 concentrations vary substantially even in a given type of plant, and variations by a factor of 2 to 3 have been noted. The range of the detection limit for Co-60 varies accordingly.

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

As an indication for detection limits that can be readily achieved may serve a value of 0,46 $Bq \cdot kg^{-1}$ DM for Co-60 that was obtained by measuring a desiccated and milled sample of grass (400 g of desiccated plant matter in a 1 l-Marinelli beaker, detector: 25 % relative efficiency, measuring period: 12 hours).

7 Catalogue of chemicals and equipment

7.1 Chemicals

No chemicals are required.

7.2 Equipment

In addition to the measuring equipment listed in procedure F-γ-SPEKT-MILCH-01, a large drying cabinet (convection drying cabinet) and a cross-beater mill are required for sample preparation.