Sampling methods for pasture, soil and deposition for radioactivity emergency preparedness in the Nordic countries

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The aim of this work was to compare sampling techniques for pasture, soil and deposition, planned for radioactivity surveillance in emergency situations in the Nordic countries. The basis of the survey was a questionnaire, sent to radiation protection authorities and laboratories. Sampling of pasture is performed with a cutting height between 1 and 5 cm above the ground from an area of about 1 m². The sampling plots are usually randomly positioned. Soil samples, 3 to 20 cores in various patterns, are generally taken by a corer of varying diameter. For deposition sampling, precipitation collectors of different sizes are used. When comparing results, the differences between laboratories should be borne in mind so that proper corrections can be made. It is, however, important to consider that, especially in an emergency situation, the use of standardised methods may worsen the results if these methods are not part of the daily work.

Introduction

In the event of a nuclear accident, which has caused fallout of radioactive material on the ground, suitable sampling and analysis methods are of great importance to obtain data for decisions made by different authorities (e.g. grazing restrictions). The measurement data are also important when calculating the further transport of the radioisotopes in the ecosystems and for assessment of the radiation dose to man.

Since several different organisations and authorities may be involved in sampling and

measurements it is important that the results are comparable and not influenced by differences in the sampling procedure. To study the homogeneity of sampling and analysis methods planned for radioactivity surveillance in emergency situations in the Nordic countries a questionnaire (Appendix 1) was sent out to 13 laboratories and radiation protection authorities in the five Nordic countries. These are not the only laboratories active in sampling in the case of an emergency situation, but merely represent a sample. This work is based on the response from 7 of the laboratories (presented in Appendix 2),

representing all of the five Nordic countries.

The sampling methods included in this study are sampling of pasture, soil and deposition. The methods presented are also restricted to techniques for emergency preparedness and monitoring, and sampling techniques for scientific studies of special processes are thus not included. Also included in the comparison are methods for sample preparation for measurement, type of measurement and sample storage (since the main purpose of the measurements is to obtain rapid results, mainly sampling for gamma spectroscopic measurements are surveyed). A similar study, covering more sample types, was reported in 1985 (Taipale 1985) and many of the sampling methods then used are still valid.

Results and discussion

Sampling of pasture

Table 1 shows a summary of sampling methods for vegetation (pasture). The cutting height of the grass varies between 1 and 5 cm above the ground and the sampled areas are usually about 1 m². The positioning of the sampling plots on the investigated area (field) is usually at random and the number of plots taken over the area varies between the laboratories.

The analysis is mostly made with high-resolution gamma spectrometry, but also with NaI(Tl)-detectors, on fresh and/or dried samples. In some cases also ashed samples are analysed. Both fresh weight and dry weight are determined and the results are given in Bq kg⁻¹ fresh weight, Bq kg⁻¹ dry weight and Bq m⁻². The samples are usually stored dried, frozen or ashed.

The methods for sampling of pasture are also discussed in IAEA-TECDOC-1092 (IAEA 1999). In this report the recommendations are that at least 1 kg of vegetation is sampled from an area of 1 m² or more and that the grass is cut 2 cm above the ground. The procedures in use in the Nordic countries (among the laboratories participating in this study) agree in most cases with these recommendations. The difference in cutting height between 1 and 5 cm may, however, have a great influence on the results. In

Sweden 5 cm is a standard cutting height, recommended by the Swedish Radiation Protection Authority, which is aimed to give comparable results in Sweden.

Soil sampling

A summary of sampling methods for soil is shown in Table 2. Soil samples are generally taken by different models of corers (varying diameter), down to a depth of 5 to 50 cm. The number of cores taken at the sampling site ranges from 3 to 20 and the cores are usually sliced. The activity in the top soil is in some cases determined in the first slice, with thickness in the range 2–5 cm. Eventually, the activity in the top soil is determined using a number of shallow cores, in addition to deeper profiles used to determine the depth distribution. Several sampling patterns are used and also the habit of pooling the samples differs between the laboratories.

The analysis is made with HPGe- or Ge(Li)-detectors on fresh or dried samples, depending on the expected radionuclides present. A method where a complete soil core is scanned vertically by a collimated Ge-detector to determine the depth distribution is also used. In these measurements the soil core is placed on a rotating turntable and moved vertically in 5 mm steps during the measurement (Finck and De Geer 1992). Both fresh weight and dry weight are registered for comparison with field-gamma spectrometry and determination of the soil humidity. The results are given in Bq kg⁻¹ fresh weight, Bq kg⁻¹ dry weight or Bq m⁻².

In the event of fresh fallout only the upper soil (5 cm) generally needs to be sampled. There may, however, be of interest to sample larger depths for further studies of the migration of the fallout and these larger depths are therefore included in Table 2. The method for soil sampling used in Sweden (as recommended by the Swedish Radiation Protection Authority) is based on Isaksson and Finck (2002). Taking soil samples with a corer may result in contamination of deeper soil layers by radionuclides from upper layers and surface soil. This cross contamination effect was studied by Isaksson and Erlandsson

Table 1. Summary of methods for pasture sampling and analysis.

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN ¹⁾ , Norway	NRPA, Norway	Sweden ²⁾
Criteria for sampling area	Grass height 15–20 cm preferred	Flat open area, uniform in growth height, not in the vicinity of roads or ditches	Open and undisturbed area, not closer to dusty roads than 100 m and not close to ditches	Samples of soil and vegetation should be taken at the same site	Samples of soil and vegetation should be taken at the same site	Pre-chosen farms
Area size	One or more square metres	25 x 25 cm², 40 x 40 cm² or other sizes	One or more square metres	1 m² or at significant growth 0.25 m² or collected from the top soil core (area defined by the core)	$1 m^2$	$3\times1~\text{m}^2$ or $6\times0.5~\text{m}^2$
Area definition tool	Ruler, measuring tape	Frames of different sizes	50×50 cm wooden frame			Outlined by 56.4 cm rod, connected to a central pole
Sample distribution	Only one area sampled	Grid	Vegetation from 4 randomly selected plots within a few meters distance combined. More plots combined in areas with poor vegetation			Three samples diagonally distributed over a field or six sampling plots in W-shaped pattern over the field. Pooled
Cutting height	At ground level, ~ 1 cm	1–2 cm or more	2 cm above the soil	2 cm above the soil	2 cm above the soil	5 cm above ground
Cutting tool	Garden scissors	Garden scissors	Garden scissors	Scissors	Scissors	Cutter equipped with a device to keep the distance constant at 5 cm above ground
Sample preparation	Weighing, measurements on both fresh and ashed samples	Weighing, homogenising, drying, ashing	Drying, grinding, homogenising, weighing	Drying to constant weight at 105 °C. Homogenised with mills and sieved (1 mm mesh size)	Drying to constant weight at 105 °C. Homogenised with scissors/food processor and sieved (2 mm mesh size)	Drying, chopping
Detector	Ge	HPGe	HPGe	Nal or HPGe	Nal or HPGe	HPGe or Ge
Measurement geometry	Fresh samples: 1 Marinelli. Ashed samples cylindrical container	560 ml Marinelli, 30 ml and 110 ml cylindrical beakers	1000 ml Marinelli beakers or 200 ml cylindrical beakers (Ø 72 mm)	Beakers varying from 25 to 250 ml volume	28 ml, 105 ml, 215 ml or 250 ml beakers	Not specified
Sample storage	Plastic bag, Marinelli and cylindrical container (finally)	Cold, dried, frozen or ashed	Cylindrical plastic beakers at room temperature	Frozen or dried		Frozen or dried
Reporting	Bq kg ⁻¹ (fresh, dry), Bq m ⁻²	Bq kg ⁻¹ (fresh, dry), Bq m ⁻²	Bq kg ⁻¹ (fresh, dry), Bq m ⁻²	Bq m ⁻² or Bq kg ⁻¹ (fresh, dry)	Bq m ⁻² or Bq kg ⁻¹ (fresh, dry)	Bq kg ⁻¹ (fresh, dry), Bq m ⁻²

1) AUN is an abbreviation of Agricultural University of Norway. 2) The method described for Sweden is the official method decided by the Swedish Radiation Protection Authority (SSI).

Table 2. Summary of methods for soil sampling and analysis.

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	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN¹), Norway	NRPA, Norway	Sweden ²⁾
Criteria for sampling area	Flat, open areas with undisturbed soil, avoiding shadowing by trees, bushes or other objects	Flat, open areas with undisturbed soil, avoiding shadowing by trees, bushes or other objects	Open and undisturbed area, not closer to dusty roads than 100 m and not close to ditches	Samples of soil and vegetation should be taken at the same site	Samples of soil and vegetation should be taken at the same site	Flat, open areas with undisturbed soil, avoiding shadowing by trees, bushes or other objects
Area size	$4 \times 4 \text{ m}^2$. For fresh deposition e.g. $10 \times 10 \text{ cm}^2$	Varying size, e.g. 30 ×30 m²	Old method: $50 \times 50 \text{ cm}^2$ New method: Straight line (e.g. 100 m)	From an area of 0.25 m² 0.5 cm depth is scraped off	1 m² (if frozen ground, 0.5 cm depth is scraped off from an area of 0.25 m²)	Varying size
Sampling tool	Soil corer with inner diameter 6.5 cm	Steel augers (Ø 70 mm or 106 mm). For surface layers frame (e.g. 20 × 20 cm²)	Old method: Soil corers with inside diameter of 5.0 cm New method: Soil corer with inside diameter 17 mm and capable of taking 30 cm long cores	Soil corer with 5–15 cm diameter (5–20 cm depth)	Soil corer with 5–15 cm diameter (usually 10 cm)	Soil corer with inner diameter 5–15 cm
Sample distribution	13 samples at each sampling site	4 to 12 separate samples	Old method: 3 samples in a triangle geometry. New method: Composite sample of about 20 cores taken in a straight line at regular intervals	4 samples taken from each quadrant within a 1-m² square	4 samples taken from each quadrant in a 1-m² square	3 samples in a triangle with side length 30 cm
Sampling depth	50 cm. For fresh deposition 5 cm	5 to 15 cm	5–30 cm	5 cm (10–20 cm for mobility and migration studies using seq extraction)	5 cm (10 cm for migration studies)	10 cm
Subdivision of soil	Sliced in layers, varying from 3 to 10 cm and pooled	0-2, 2-5, 5-10, 10-15 cm	0–5 cm, 5–10 cm, 10–15 cm etc.	Sliced in 1 or 2 cm layers	Sliced in 1 (or 2) cm layers	0–2, 2–5, 5–10 cm
Sample preparation	Drying, sieving, mixing and weighing	Weighing, drying, sifting (2 mm). Without drying if rapid results are needed. Acid digestion, acid extraction or buffered acidic solution extraction for radiochemical separations	Drying, homogenisation and sieving (2 mm)	Stones and roots are removed. Drying at room temperature (mobility), 105 °C (dry weight) and 450 °C (LO)), homogenized in a mill and sieved through a mesh sieve	Stones and big roots are removed. Drying to constant weight at 105 °C. Homogenisation with pestle and mortar	Weighing, drying and homogenising, depending on the method used

Detector	Ge	HPGe	HPGe	HPGe	HPGe	HPGe
Measurement geometry	200 ml cylindrical beaker	560 ml Marinelli, cylindrical beakers (30 ml, Ø 42 mm and 110 ml, Ø 74 mm)	200 ml cylindrical beaker	25– 250 ml beakers	28 ml, 105 ml or 250 ml beakers	Not specified
Sample storage	Dry samples stored	For short periods refrigerator and for longer time periods dried or frozen	Cylindrical beakers at room temperature	Dried or frozen		Not specified
Reporting	Bq kg⁻¹ fresh weight, Bq kg⁻¹ dry weight and Bq m⁻²	Bq kg ⁻¹ fresh weight, Bq kg ⁻¹ dry weight (without stones) and Bq m ⁻²	Bq kg ⁻¹ d.w. (and/or f.w.) and Bq m ⁻² decay corrected to time of sampling, or some other reference time if needed	Bq kg⁻¹ dry weight and Bq m²²	Bq kg⁻¹ dry weight and Bq m⁻²	Bq kg ⁻¹ fresh weight, Bq kg ⁻¹ dry weight and Bq m ⁻² , decay-corrected to sampling

¹⁾ AUN is an abbreviation of Agricultural University of Norway.
²⁾ The method described for Sweden is the official method decided by the Swedish Radiation Protection Authority (SSI)

(1995) and was found to be negligible when a corer of 80 mm diameter was used. Other types of corers may, however, cause cross contamination and the disturbance of the soil ought to be investigated for each type of corer.

Deposition sampling

Table 3 shows a summary of sampling methods for deposition. Precipitation collectors of a wide variety of sizes are used in sampling to determine the activity concentration in the precipitation. The amount of dry deposition may also be determined by wiping the precipitation collector. The activity measurements are made either directly on a water sample (if the activity concentration is sufficiently high) or by analysing ion exchange resins (often both anion and cation exchange resins, together with filter papers, are used to capture different radioisotopes in solution and in particulate material). High-resolution gamma spectrometry is used and the results are given in Bq 1⁻¹ and/or Bq m⁻². In some cases hot spots are investigated using autoradiography on a surface soil layer.

Conclusions

In the three types of sampling studied in this work: pasture, soil and deposition the methods used in the Nordic countries differ to some extent. When comparing results from different laboratories these differences should therefore be borne in mind so that proper corrections can be made. It is, however, also important that each laboratory is familiar with their sampling and analysis methods, especially in an emergency situation. Many laboratories take samples for use in research and have a well functioning procedure for this kind of sampling and measurements. The use of standardised methods in a fallout situation may therefore worsen the results if these methods are not previously implemented in the daily work.

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Table 3. Summary of methods for deposition sampling and analysis.

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN ¹⁾ , Norway	NRPA, Norway	NPL ²⁾ , Sweden
Method for wet deposition sampling	10 m² stainless steel collector with an ion-exchange column mounted at the outlet	1) Stainless steel collector (area 0.07 m²) with light heating inside the funnel in winter. 2) Stainless steel sampler (0.5 m²) with light heating inside the funnel in winter. Samples gathered into plastic cans	Most of the samplers have a collecting area of 0.02 m² (Icelandic Meteorological Office)	GM-tubes or Nal detector at the ground or 1 m above ground	Wet+dry deposition measured by analysing soil samples with HPGe detector	Aluminium precipitation collector (4 m²). Precipitation poured through ion-exchange resins
Method for dry deposition sampling	High-volume air sampler	The collecting surfaces are wiped using distilled water	High volume air sampler	A 1 cm layer within a small, well-defined area is scraped off by a spoon	Continuous air filter analyses by HPGe at 5 locations	Aluminium precipitation collector (4 m²) wiped using a mild detergent.
Sample preparation	Drying of ion- exchange resins	Addition of carriers, concentrating by evaporation	In a case of emergency, where higher levels are suspected, samples would be measured without pre-treatment	Dried at room temperature		Oven-dried ion- exchange resins and filter paper
Detector	Ge	HPGe	HPGe	Autoradiography, HPGe, SEM XRMA®	HPGe	HPGe
Measurement geometry	Cylindrical boxes	30 ml cylindrical geometry or Marinelli geometry if measured without any concentrating	1000 ml Marinelli beaker or 200 ml cylindrical beaker (Ø 72 mm)	Cylindrical geometry		Plastic cans (60 ml or 180 ml)
Sample storage	No special routines	Adding HNO ₃ to the sample cans, or as dried or ashed in room temperature		Dried		Room temperature
Reporting	Bq m³ and Bq m²	Bq m-² per sampling period	Bq/litre and Bq m ⁻²			Bq m⁻²

¹⁾ AUN is an abbreviation of Agricultural University of Norway.
²⁾ NPL is an abbreviation for Nuclear Physics at Lund.
³⁾ Scanning Electron Microscopy X-Ray MicroAnalysis

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Appendix 1. Questionnaire.

Pasture sampling

How is the sampling area chosen?

What is the size of the area usually sampled?

What kind of tool is used to define the area?

How are the sampled areas distributed on a sampling site?

At what height is the vegetation cut? (If the cutting height varies for different types of vegetation, please indicate this).

What kind of tool is used for cutting?

How are the samples prepared for measurement (weighing, drying, chemical pre-treatment, etc.)?

What kinds of detectors are used for the activity determination?

What measurement geometry is used?

How are the samples stored before and after measurement?

How are the results reported?

Soil sampling

How is the sampling area chosen?

What kind of tool is used for the soil sampling?

What is the size of the area usually sampled?

How are the samples distributed on a sampling site and how many samples are taken?

What is the depth sampled in an emergency situation?

Do you subdivide the soil? How?

How are the samples prepared for measurement (weighing, drying, chemical pre-treatment, etc.)?

What kinds of detectors are used for the activity determination?

What measurement geometry is used?

How are the samples stored before and after measurement?

How are the results reported?

Deposition sampling

Please describe the method you use for wet deposition sampling (size of precipitation collectors, ion exchange systems, etc.)?

Please describe the method you use for dry deposition sampling?

How are the samples prepared for measurement (weighing, drying, chemical pre-treatment, etc.)?

What kinds of detectors are used for the activity determination?

What measurement geometry is used?

How are the samples stored before and after measurement?

How are the results reported?

Appendix 2. Participating laboratories and contact persons.

Risø National Laboratory, Denmark (Sven P. Nielsen, Christian Lange Fogh, Risø, NUK-114, P.O. Box 49, DK-4000 Roskilde, Denmark)

Finnish Centre for Radiation and Nuclear Safety (STUK), Finland (Aino Rantavaara, Ritva Saxén, Eila Kostiainen, STUK, P.O. Box 14, FIN-00881 Helsinki, Finland)

Icelandic Radiation Protection Institute (Geislavarnir ríkisins), Iceland (Sigurður Emil Pálsson, Geislavarnir ríkisins, Rauðarárstíg 10, IS-150 Reykjavík, Iceland)

Laboratory for Analytical Chemistry, Agricultural University of Norway, Norway (Brit Salbu, NLH, P.O. Box 5026, N-1432 Ås, Norway)

Norwegian Radiation Protection Authority (NRPA), Norway (Astrid Liland, Statens strålevern, P.O. Box 55, N-1332 Østerås, Norway)

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