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Sampling methods

A survey of methods in use in the Nordic countries

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Abstract

This report is a survey of sampling techniques currently in use for radioactivity measurements in the Nordic countries, but restricted to sampling techniques for pasture, soil and deposition in emergency situations. It is found that the participating laboratories apply similar sampling procedures for pasture, including cutting height and size of sampled areas. Soil samples are generally taken by some sort of corer of different diameter. The number of cores taken varies, different sampling patterns are used, and pooling of the samples is done by some of the laboratories. The analysis of pasture and of soil is made with NaI-detectors or by high-resolution gamma spectrometry on fresh or dried samples. Precipitation collectors of a range of sizes are used to determine the activity concentration in precipitation and of dry deposited radionuclides. The analysis is made with high-resolution gamma spectrometry, either directly on a water sample or on ion exchange resins.

Keywords

Radionuclides, sampling, emergency preparedness, pasture, soil, deposition

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Sampling methods –
A survey of methods in use in the Nordic countries

(NKS/BOK-1.1: Laboratory measurements and quality assurance
a sub-project within the NKS/BOK-1 project)

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Contents

1. Executive summary

The aim of this work is to compile information on the sampling techniques currently in use for radioactivity measurements in the Nordic countries. It is restricted to sampling techniques for emergency preparedness, and monitoring and sampling for scientific studies of special processes are thus not included. This survey has also been restricted to the sampling of pasture, soil and deposition.

This report is based on contributions from some of the radiation protection authorities and research departments in the Nordic countries, which have responded to a questionnaire about sampling methods, sampling equipment, sample preparation, methods of measurement, sample storage and reporting of results. All of the five Nordic countries are represented in the study.

The participating laboratories apply similar sampling procedures for pasture. The cutting height of the grass varies between 1 and 5 cm above the ground and the sampled areas are usually about 1 m^2. The positioning of the sampling plots on the investigated area (field) is usually at random but the number of plots on the area varies. The analysis is made with NaI or high-resolution gamma spectrometry on fresh and/or dried samples, depending on the fallout situation.

Soil samples are generally taken by some sort of corer of different diameter, down to a depth of 5 to 50 cm. The number of cores taken at the sampling site varies from 3 to 20 and the cores are usually sliced. The activity in the upper part of the soil is often determined in the first slice, with thickness in the range 2 – 5 cm. Different sampling patterns are used and pooling of the samples is done by some of the laboratories. The analysis is made with high-resolution gamma spectrometry on fresh or dried samples, depending on the expected radionuclides present. Both fresh weight and dry weight are registered for comparison with field-gamma spectrometry and determination of soil water content.

For deposition sampling precipitation collectors of a range of sizes are used to determine the activity concentration in the precipitation. The precipitation collector may also be wiped to determine dry deposited radionuclides. The analysis is made with high-resolution gamma spectrometry, either directly on a water sample or by measuring ion exchange resins.

2. Introduction

As stated in *Planer for NKS-programmet 1998-2001* (1999), the objectives of the BOK-1.1 sub-project *Laboratory measurements and quality assurance* are "to establish quality assurance and quality control of laboratory measurements; to perform intercomparison of sampling techniques and γ-spectrum analysis software; and to improve co-operation concerning laboratory procedures, and work on accreditation" That is, to assist participating laboratories to improve and demonstrate the quality of their measurements, taking into consideration the needs of emergency preparedness and radioecology.

In the first part of the project the following work tasks are being dealt with:
Sampling methods - A survey of methods in use in the Nordic Countries
Intercomparisons on radioactivity in environmental samples
Effects of the introduction of a new standard, ISO/IEC DIS 17025, on the process of accreditation of laboratories

The present work constitutes the first task, where the aim is to compile information on the sampling techniques currently in use for radioactivity measurements in the Nordic countries. It is restricted to techniques for emergency preparedness and monitoring, and sampling techniques for scientific studies of special processes are thus not included. At present the project concentrates on the sampling of:
pasture
soil
deposition

A preliminary report was presented at a BOK-1.1 seminar in Skagen, Denmark, August 23, 1999.

3. Contributors and questionnaire

The collection of sampling methods given in this report is based on contributions from some of the radiation protection authorities and research departments in the Nordic countries, which have responded to a questionnaire (Appendix A). Some information about the sampling of soil is also based on a questionnaire sent out as a part of the NKS EKO-3.2.1 subproject in the former project period (1994-1997). The participating authorities and laboratories are:
Risø National Laboratory, Denmark
Finnish Centre for Radiation and Nuclear Safety (STUK), Finland
Icelandic Radiation Protection Institute (Geislavarnir), Iceland
Agricultural University of Norway (AUN), Norway
Norwegian Radiation Protection Authority (NRPA), Norway
Department of Nuclear Physics at Lund University (NPL), Sweden
Swedish Radiation Protection Institute (SSI), Sweden

4. Sampling methods used at different laboratories

4.1. Sampling of pasture

Risø, Denmark

There are no specific rules for site selection, but to facilitate sampling a grass height of 15 – 20 cm height is preferred. Usually 1 m^2 is sampled, defined by a folding ruler or measuring tape, but if the deposition is low several square metres may be used. There are no rules concerning the distribution of the sampled areas at the sampling site. The grass is cut with garden scissors near the ground, at a height of about 1 cm above ground.

The fresh weight is determined and the fresh grass is analysed in a 1 l Marinelli beaker with Ge gamma spectrometry. The grass samples are dried and ashed. Strontium-90 is determined on ashed samples, which are also measured with Ge detectors in cylindrical containers. The results are given in Bq/kg fresh weight, Bq/kg dry weight and Bq/m^2. Samples are first stored in plastic bags, then in Marinelli beakers and finally in the cylindrical container.

STUK, Finland

The chosen sampling site should be flat, open areas with a uniform vegetation height and not situated next to roads or ditches. The size of the area represented by the sampling could vary, e.g. 30×30 m^2 and the areas are defined by frames (25×25 cm^2, 40×40 cm^2 or other sizes) placed in a grid pattern. The vegetation is cut at a height of 1 – 2 cm above ground using a garden scissor. Depending on the purpose, larger cutting heights could be used.

If needed, other material than vegetation is removed from the sampled vegetation prior to weighing, homogenising, drying and ashing. In an acute fallout situation, fresh vegetation would probably be used without treatment. The samples are measured in 560 ml Marinelli geometry and cylindrical beakers (30 ml, Ø 42 mm and 110 ml, Ø 74 mm), using low-background, high-resolution HP Ge detectors. The results are given as Bq/kg fresh weight or Bq/kg dry weight and Bq/m^2. For short periods the samples are stored in a refrigerator and for longer time periods the samples are dried or frozen. After gamma spectrometric measurements the samples may be dried or ashed for storage or for radiochemical treatment.

Geislavarnir, Iceland

The methods used for sampling in Iceland have been chosen with two main criteria in mind: they should as far as possible be based on procedures known and accepted internationally (and/or in the Nordic countries) and they must also be appropriate for Icelandic conditions, where the terrain can often be very inhomogeneous.

The methods have mostly been based on:
IAEA Technical Report Series No 295 *"Measurement of Radionuclides in Food and the Environment - A Guidebook"* (1989)
EML HASL-300 Manual, now the 28th Edition (Rev. 0, February 1997, available on the WWW at: http://www.eml.doe.gov/publications/procman/)
Recommendations given within the NKS RAD-3 project (1990-1993) for vegetation and soil sampling.
IAEA-TECDOC-1092 *Generic procedures for monitoring in a nuclear or radiological emergency* (1999)

When each monitoring / research project is planned, these references are used as guidelines for site selection and for identifying things to look out for when taking samples.

The procedure for sampling is basically based on the one recommended in the NKS/RAD-3 project (1990-1993). Common precautions must be shown (e.g. to sample in an open and undisturbed area, not closer to dusty roads than 100 m and not close to ditches). Usually each sampling site is divided into a few sampling regions. If the site is homogeneous it is divided into sampling regions of similar size, else the division is based upon the types of vegetation cover and soil. Within each sampling region sampling areas are either chosen randomly or they are chosen to represent specific conditions (if the site is not homogeneous). A square 50×50 cm^2 wooden frame is used to define each sampling plot. Normally vegetation from 4 such randomly selected plots within a few meters distance from each other is combined into one sample (total area 1 m^2). More plots are combined into one sample in areas with very poor vegetation.

The vegetation is cut slightly above the mat using garden scissors, typically 2 cm above the surface. The mat must not be included in the vegetation sample and thus the cutting height may have to be increased in some places.

The sample is put in a paper bag and then dried in a slow-airflow drying cabinet at 50 °C. The sample is then ground, homogenised, weighed and put into 1000 ml Marinelli beakers or 200 ml cylindrical beakers (Ø 72 mm) and analysed with high resolution HPGe spectrometry. In a case of emergency the drying may be omitted. After processing and counting the samples are stored in cylindrical plastic beakers at room temperature. The activity is reported in Bq/kg dry weight (and/or fresh weight) and Bq/m^2 decay corrected to time of sampling, or some other reference time if needed (e.g. time of fallout or accident).

AUN, Norway

Vegetation within 1 m^2, or within the diameter of a soil corer (when soil samples are collected), is cut by scissors at about 2 cm above ground. When NH_4-extractions are performed to estimate the bioavailable fraction, the samples are dried at room temperature. For measurements of total concentrations, the samples are dried at 105 °C (dry weight) or ashed at 450 °C. The samples are homogenised in a mill. For gamma-emitters NaI on HPGe detectors are used. For $^{90}Sr/^{90}Y$ analysis, Quantulus liquid scintillation counter is used. Results are given as Bq/kg dw, Bq/kg or Bq/m^2.

NRPA, Norway

No formal agreement or administrative guidelines for sampling of vegetation in case of a nuclear emergency exist at present. There is, however, a wish that a number of sampling sites (approximately 500) could be pre-defined all over Norway, where the Civil Defence could take samples and do field measurements.

Grass is sampled with scissors (cutting height 2 cm above the soil) from a 1-m^2 area and the samples consist of all grass from this area.

The samples are cut by scissors and dried to constant weight at 105 °C. If a rapid result is desired or if the samples contain radioiodine, the samples can be analysed before drying. After drying the samples are homogenised in a food processor and sieved through a 2 mm mesh size. A sub sample is taken for analysis and weighed. The analysis is done with either NaI or HPGe detectors and, depending on sample size and detector type, different types of containers (28, 105, 215 or 250 ml beakers) are used. The result is given in Bq/kg, wet or dry weight, or Bq/m^2.

SSI, Sweden (recommended method for use in an emergency situation)

In Sweden, a number of laboratories (~ 10) are contracted by SSI to make measurements after a nuclear accident resulting in radioactive contamination of the environment. The following method is recommended by SSI to be used by the contracted laboratories, which are supposed to send their results to SSI.

At sampling farms (in Swedish: provgårdar), about 500 farms, grass sampling should be performed by the contracted laboratories during the first four days after the alarm, or until other organisations take over the responsibility for the sampling. The purpose of this sampling is to provide a basis to eventually abolish grazing restrictions in certain areas. At a chosen experiment farm (in Swedish: försöksgård), each contracted laboratory should also perform measurements to gain some knowledge of the transfer factors from vegetation to milk in the particular fallout situation. This should be done by measuring the activity in grass and milk in controlled experiments.

At the sampling farms the vegetation is cut at a height of 5 cm above ground from a circular area of 1 m^2. This area is outlined by a 56.4 cm rod, connected to a central pole, which is driven down into the ground. The cutter is equipped with a device to keep the distance constant at 5 cm above ground. Three sampling plots, 1 m^2 each, are selected diagonally over the field and pooled. The total sample is then weighed. At the experiment farms six sampling plots, 0.5 m^2 each is distributed in a W-shaped pattern over the field.

The analysis, by high-resolution gamma spectrometry, is made on a fresh sample if the sample contains radioiodine or if a quick result is needed. In other circumstances the sample can be chopped and/or dried. The samples are kept, frozen or dried. A special form is used for reporting, which for the experiment farms includes e.g. type of sample, farm, fertilizing procedures, weight and activity (Bq/kg, Bq/m^2). A special form is also used for reporting the collection and analysis of the milk samples. For the sampling farms the reporting form is similar.

4.2. Soil sampling

Risø, Denmark

In the general procedure for soil sampling, a soil corer with inner diameter 6.5 cm is used. At each sampling site 13 samples down to 50 cm depth are taken over an area of 4×4 m^2. The samples are sliced in layers, varying from 3 to 10 cm, and the layers are pooled for the 13 samples. For freshly deposited material, soil samples (including vegetation) are taken down to 5 cm depth on a known area (e.g. 10×10 cm^2). The samples are dried, sometimes sieved for stones, mixed and weighed prior to analysis.

The samples are weighed and dried, weighed and analysed with Ge-detector gamma spectrometry. The results are given in Bq/kg fresh weight, Bq/kg dry weight and Bq/m^2.

STUK, Finland

Soil samples are collected on flat and open areas with undisturbed soil, avoiding shadowing by trees, bushes or other objects. The samples are not taken next to roads, ditches or buildings and preferably bare ground is chosen for sampling. The soil is sampled with steel augers (Ø 70 mm or 106 mm) and for surface layers of soil a frame (e.g. 20×20 cm^2) can be used. Within the frame the soil is sampled by a trowel or small spade. The area represented by the sampling varies, but can be e.g. 30×30 m^2 and within this area 4 to 12 separate samples are taken, usually with a depth of 5 to 15 cm, in an emergency situation even thinner (to 2 cm). The soil is subdivided with a knife into the following sections: surface vegetation, 0 – 2 cm, 2 – 5 cm, 5 – 10 cm and 10 – 15 cm depth. In an emergency situation a thin sample may also be analysed in one piece. The soil samples are weighed, dried and sifted (2 mm) prior to gamma-spectrometric measurements. If a rapid result is needed the soil samples can also be analysed without drying. Acid digestion, acid extraction or buffered acidic solution extraction can be used before radiochemical separations.

The samples are measured in 560 ml Marinelli geometry and cylindrical beakers (30 ml, Ø 42 mm and 110 ml, Ø 74 mm), using low-background, high-resolution HP Ge detectors. The results are given as (Bq/kg fresh weight,) Bq/kg dry weight (without stones) and Bq/m^2. For short periods the samples are stored in a refrigerator and for longer time periods the samples are dried or frozen.

Geislavarnir, Iceland

For the last decade the NKS/RAD-3 procedure of sampling vegetation and soil has been used. In the summer of 2000 a new procedure and a new corer were taken into use. For a while the old procedure will be used for comparison for some samples

Old method

The same method of choosing plots was used as described in the section on sampling pasture. One of these 4 plots was chosen for taking soil samples. First the vegetation above the grass mat was cut and removed as described in the section on pasture sampling. Within a frame where vegetation had been sampled, 3 soil cores were collected, distributed in a triangular geometry. Each of these cores was divided into 5 cm thick slices (0 – 5 cm, 5 – 10 cm, 10 – 15 cm etc.) and the corresponding slices from all 3 cores were subsequently combined into one sample. The number of slices could vary according to the observed depth penetration of nuclides of interest and the thickness of the soil. Typical lengths of soil cores would be 15 – 30 cm (3 – 6 slices).

Initially a 10 cm long corer was used with an inside diameter of 5.7 cm. This was soon replaced by longer corers with inside diameter of 5.0 cm.

Each combined sample was put into a plastic bag, transferred to a paper bag and dried in a slow-airflow drying cabinet at 50 °C. Subsequent steps were as in the new method.

New method

The terrain sampled has often been very inhomogeneous and combing 3 cores using the old method easily gave results with a high degree of variability. Therefore the following method was introduced, mostly adopted from the EML procedures.

A composite sample of about 20 cores is used. The aim is to get at least 200 ml of dried soil for analysis from each section. If the terrain is reasonably homogeneous, then the cores are taken in a straight line at a regular interval (minimum 30 cm, EML method).

If the terrain is inhomogeneous, then a procedure based on the EML 2.4.3.4 method is used to the degree appropriate. Briefly this involves identifying the number of different type of covers (strata) within the area. Then a straight line (e.g. 100 m**)** is selected in a random manner and the proportion of each cover along the transect is estimated. Each stratum that represents more than 5% of the total transect is sampled by the core method at a fixed interval along the transect if possible.

Areas very limited in size and/or irregular and/or with small-scale variations may make it necessary to alter the procedure given above. The sampling team must make sure that the sampling spots chosen reflect the area of interest. Hummocks up to 1 m in diameter are a very distinct character of many grazing areas in Iceland. The transect line may need to be less than 100 m in length and consideration must be given to having high and low areas (incl. hummocks and hollows) represented in the proper proportions in the total sample. In all cases the features of the area must be described and the sampling method chosen must be documented.

The samples from the same depth layers in each stratum are combined and analysed to obtain information pertinent to each stratum. Each slice is 5 cm thick (same thickness as in the old

method). The total inventory in the area is estimated by summing the proportional contribution from each stratum.

A corer with an inside diameter of 17 mm and capable of taking 30 cm long cores is used. Core sections belonging to the same depth interval are placed together in a canvas bag and dried in a slow-airflow drying cabinet at 50 – 100 °C (depending on planned subsequent analysis). The sample is then homogenised and sifted (2 mm) prior to a gamma spectrometric measurement in a 200 ml cylindrical beaker. After measurement the samples are stored in cylindrical beakers at room temperature. The activity is reported in Bq/kg dry weight (and/or fresh weight) and Bq/m^2 decay corrected to time of sampling, or some other reference time if needed (e.g. time of fallout or accident).
In a case of emergency involving fresh fallout, the sampling may be simplified and limited to undivided cores to a depth of 10 cm, using a 5 cm diameter corer, and without drying the samples.

AUN, Norway

Four soil cores (5-15 cm diameter, 5-20 cm depth) are collected within 1 m^2, one from each quadrant. The cores are sectioned in 1 or 2 cm slices. When sequential extractions are performed to estimate the mobility of radionuclides in soils (or sediments), samples are dried at room temperature. When total concentrations are to be determined, samples are dried at 105 °C (dry weight) and ashed at 450 °C (loss on ignition). The samples are homogenised and sieved through a 1 mm mesh sieve. For analysis of gamma-emitters, HPGe is applied. For ^{90}Sr, the samples are dissolved in aqua regia and subjected to liquid-liquid extraction of ^{90}Y. Quantulus liquid scintillation counter is used for measurement. Results are given as Bq/kg dry weight, Bq/kg wet weight or preferentially Bq/m^2.

NRPA, Norway

Soil cores are sampled from an area of 1 m^2, which is divided into four equal squares. From each square, one sample is taken with a soil corer of 5-15 cm diameter (usually 10 cm diameter) to 5 cm depth. The soil cores are sliced in 1 cm slices (in some cases 2 cm).

Stones and big roots are usually removed and are not considered when calculating the specific activity. The soil is dried to constant weigh at 105 °C, homogenised in a mortar and sieved through 2 mm mesh size. The analysis is done with HPGe in different measurement geometries (28, 105 or 250 ml beakers), depending on sample size. The results are given in Bq/kg dry weight or Bq/m^2.

SSI, Sweden (recommended method for use in an emergency situation)

The recommended method for soil sampling is that three bore-cores (diameter 50 – 150 mm) are sampled at each sampling plot, distributed in a triangular geometry (~30 cm between the holes). The samples should be taken on a flat, open area. The sampling site is photographed and the type of vegetation is noted. If the sampling is performed in connection with field-gamma spectrometry, the soil samples should be taken close to the position of the detector. If it is important to measure volatile elements and thus using a wet, unhomogenised, sample three additional bore-cores can be taken at the same plot. The bore-cores are sliced in three layers: 0 – 2, 2 – 5 and 5 – 10 cm.

The gamma-spectrometric analysis of the samples can be made according to different steps, depending on the situation. A complete, unsliced, bore core can be analysed for ^{134,137}Cs and ^{131}I. The sample is then dried to determine the wet/dry weight. In addition the bore core can

be sliced and measured in a wet condition or dried and homogenised before analysis. The fourth method is to pool each layer for the three cores. The method used for sample preparation will depend on the circumstances and the composition of the deposition. SSI will notify the contracted laboratories the method to use. Special report forms have been worked out to facilitate the reporting of results.

4.3. Deposition sampling

Risø, Denmark

Precipitation is sampled by a 10-m^2 stainless steel collector with an ion-exchange column mounted at the outlet. The ion-exchange column is exchanged monthly. Collectors in the sizes 1 to 10 m^2 are used for collection of precipitation at Risø and of 0.147 m^2 in the rest of Denmark for routine monitoring. The water from the other collectors than the 10-m^2 collector is pooled to form yearly samples. Dry deposition is collected together with wet deposition by washing the 10-m^2 collector with a dilute acid solution. Aerosols are sampled with a high-volume sampler. Smaller aerosol-sampling systems for mobile teams are also available.

Prior to measurement the ion-exchange resins are dried at 100 °C. Gamma spectrometric measurements are made with Ge-detectors with the sample placed in cylindrical boxes. The precipitation is analysed for ^{137}Cs and ^{90}Sr and in the case of fresh fallout the precipitation is analysed directly with gamma spectrometry. The results are given in Bq/m^3 and Bq/m^2 and transferred to a database. Results from the Risø site are published in internal reports twice a year.

STUK, Finland

Two types of deposition samplers are currently in use. One type has an area of 0.07 m^2 and it collects wet and dry deposition together. The other type samples wet and dry deposition separately and has a collecting area of 0.5 m^2. The sampler has two funnels (0.5 m^2 each) and a lid, which changes place according to weather conditions. The lid covers the dry deposition funnel in case of precipitation and covers the wet deposition funnel when it is not raining. A light heating inside the funnel is used to facilitate sampling in the wintertime by melting snow and ice gathered in the funnel in both types of samplers. The precipitation is gathered into plastic cans from both types of the samplers for wet deposition. Dry deposition sample is taken by wiping the collecting surfaces of the sampler. The collectors are made of stainless steel and are designed at STUK.

The collecting surfaces are rinsed, first with dilute nitric acid and thereafter with distilled water, which is added to the precipitation samples. After sampling 3 ml HNO_3 (65%), 20 mg Sr-Cs carriers and 1 mg Ba-Ce carriers are usually added to the samples. The amounts of carriers depend on the radionuclides and the method used for analysing the samples. The samples are usually concentrated by evaporation and the final drying is made in 30 ml cylindrical geometry, which is also used in the gamma spectrometric measurements. If there are signs or suspicions of fresh fallout the samples are measured in Marinelli geometry without any pre-treatment. The dry deposition sample is usually analysed in 30 ml cylindrical geometry.

The samples are analysed using low-background, high-resolution gamma spectrometry. The samples are stored by adding HNO_3 to the sample containers, which are kept in room

temperature before treatment and analysis. After gamma-spectrometric measurements the samples are usually used for radiochemical analysis for, e.g. ^{90}Sr. The results are reported as Bq/m^2 per sampling period.

Geislavarnir, Iceland

Precipitation is sampled by the Icelandic Meteorological Office and these samples can be used for measurements of radionuclides. Most of the samplers have a collecting area of 0.02 m^2 (200 cm^2). Currently rainwater is monitored on a regular basis for Cs-137 from three of these stations, using a chemical separation process (AMP) followed by gamma spectrometric HPGe analysis using a 200 ml cylindrical beaker (Ø 72 mm). Results can be reported in Bq/litre and Bq/m^2 decay corrected to time of sampling, or some other reference time if needed (e.g. time of fallout or accident).

In a case of emergency, where higher levels are suspected, samples would be measured without pre-treatment using high-resolution HPGe gamma spectrometry and using either a 1000 ml Marinelli beaker or a 200 ml cylindrical beaker (Ø 72 mm).

No special system is currently in use for collecting dry deposition, but one high volume air sampler is currently in use and another will be installed in 2001. Filters will be changed and analysed on a daily basis in the latter one. In both cases high resolution HPGe spectrometry is used.

If appropriate, dry deposition could be collected from high-walled pots (EML procedure 2.3.2), taking care to rinse the deposition with a weak acid and suitable carriers. Deposition is not collected currently in this way on a routine basis, but fallout of ash from volcanic eruptions has often been collected at meteorological stations.

AUN, Norway

In the field, GM tubes or NaI-detectors are used to identify hot spots (radioactive particles). At hot spots, a 1 cm layer within a small, well-defined area is scraped off by a spoon. The samples are dried at room temperature and subjected to autoradiography and gamma spectrometry (HPGe-detectors). If hot spots are identified from the film, further analysis of particles is performed by scanning electron microscopy and x-ray microanalysis.

NRPA, Norway

Continuous air filter analyses by HPGe at 5 locations. In case of a new deposition, dry deposition is measured by air filters. Wet+dry deposition is measured by analyses of soil samples with an HPGe detector.

Department of nuclear physics. Lund University, Sweden

A precipitation collector (4 m^2) is used for the collection of precipitation at Lund. The collector is built from two slanting sheets of aluminium with a gutter between them. The precipitation is collected in plastic cans and dry deposition is collected by wiping the collectors using a mild detergent.

The water is transported to the laboratory and poured through an ion-exchanger system (both anion and cation resins). The ion-exchange resins are oven-dried and both the resins and filter paper are packed into plastic cans (60 ml or 180 ml).

The samples are analysed using low-background, high-resolution gamma spectrometry and the results are reported as Bq/m^2. The samples are stored in room temperature both before and after measurement. In addition to deposition sampling, air filter measurements and a large system of precipitation collectors in the province of Skåne (southern Sweden) can be used to calculate the deposition, based on the activity concentration in precipitation and on the amount of precipitation.

5. Summary of sampling methods

5.1. Sampling of pasture

A summary of sampling methods for vegetation (pasture) is shown in Table 1. The cutting height of the grass varies between 1 and 5 cm above the ground and the sampled areas are usually about 1 m^2. The positioning of the sampling plots on the investigated area (field) is usually at random and the number of plots on the area varies.

The analysis is made with NaI or high-resolution gamma spectrometry on fresh and/or dried samples, and in some cases also on ashed samples. Both fresh weight and dry weight are determined. The results are given in Bq/kg fresh weight, Bq/kg dry weight and Bq/m^2. The samples are usually stored dried, frozen or ashed.

Sampling of pasture is discussed in IAEA-TECDOC-1092 *Generic procedures for monitoring in a nuclear or radiological emergency* (1999). The report recommends that at least 1 kg of vegetation is sampled from an area of 1 m^2 or more and that the grass is cut 2 cm above the ground. These recommendations agree well with the procedures in use in the Nordic countries (among the laboratories participating in this study).

5.2. Soil sampling

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

The analysis is made with high-resolution gamma spectrometry on fresh or dried samples, depending on the expected radionuclides present. Scanning of a complete soil core to determine the depth distribution is also used. Both fresh weight and dry weight are determined for comparison with field-gamma spectrometry and determination of soil water content. The results are given in Bq/kg fresh weight, Bq/kg dry weight or Bq/m^2.

Sampling strategy and methods for soil sampling are also discussed in IAEA-TECDOC-1092. The document gives some recommendations concerning the choice of sampling site, which seems to agree with the criteria used by the laboratories participating in this study. Also the sampling method described (5 cm deep ring corer) is utilised, with modifications, in the Nordic countries.

5.3. Deposition sampling

A summary of sampling methods for deposition is shown in Table 3. Precipitation collectors of a wide variety of sizes are used to determine the activity concentration in the precipitation. The precipitation collector may also be wiped to determine dry deposited radionuclides. The analysis is made either directly on a water sample or by ion exchange resins. The analysis is made with high-resolution gamma spectrometry and the results are given in Bq/l and/or Bq/m^2. Hot spots in the deposition are also investigated using autoradiography on a surface soil layer.

Table 1 Summary of methods for pasture sampling and analysis

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN, Norway	NRPA, Norway	SSI, Sweden
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	25×25 cm^2, 40×40 cm^2 or other sizes	One or more square metres	1 m^2 or at significant growth 0.25 m^2 or collected from the top soil core (area defined by the core)	1 m^2	3×1 m^2 or 6×0.5 m^2
Area definition tool	Ruler, measuring tape	Frames of different sizes	50 x 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 scissor	Garden scissors	Scissors	Scissors	Cutter equipped with a device to keep the distance constant at 5 cm above ground

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

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN, Norway	NRPA, Norway	SSI, Sweden
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	NaI or HPGe	NaI or HPGe	HPGe or Ge
Measurement geometry	Fresh samples: 1 l Marinelli. Ashed samples cylindrical container	560 ml Marinelli, 30 ml & 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 (fresh, dry), Bq/m^2	Bq/kg (fresh, dry), Bq/m^2	Bq/kg (fresh, dry), Bq/m^2	Bq/m^2 or Bq/kg (fresh, dry)	Bq/m^2 or Bq/kg (fresh, dry)	Bq/kg (fresh, dry), Bq/m^2

Table 2 Summary of methods for soil sampling and analysis

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN, Norway	NRPA, Norway	SSI, Sweden
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	4×4 m^2. For fresh deposition e.g. 10×10 cm^2	Varying size, e.g. 30×30 m^2	*Old method* 50 × 50 cm^2 *New method* Straight line (e.g. 100 m)	From an area of 0.25 m^2 0.5 cm depth is scraped of	1 m^2 (if frozen ground, 0.5 cm depth is scraped off from an area of 0.25 m^2)	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 cm^2)	*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-m^2 square.	4 samples taken from each quadrant in a 1-m^2 square.	3 samples in a triangle with side length 30 cm. Eventually duplicate samples
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

Table 2 Summary of methods for soil sampling and analysis, continued

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN, Norway	NRPA, Norway	SSI, Sweden
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 (LOI) ,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 fresh weight, Bq/kg dry weight and Bq/m^2	(Bq/kg fresh weight,) Bq/kg dry weight (without stones) and Bq/m^2.	Bq/kg 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 dry weight and Bq/m^2	Bq/kg dry weight and Bq/m^2	Bq/kg fresh weight, Bq/kg dry weight and Bq/m^2, decay-corrected to sampling

Table 3 Summary of methods for deposition sampling and analysis

	Risø, Denmark	STUK, Finland	Geislavarnir, Iceland	AUN, Norway	NRPA, Norway	NPL, Sweden
Method for wet deposition sampling	10 m^2 stainless steel collector with an ion-exchange column mounted at the outlet	1) Stainless steel collector (area 0.07 m^2) with light heating inside the funnel in winter. 2) Stainless steel sampler (0.5 m^2) with light heating inside the funnel in winter. Samples gathered into plastic cans.	Most of the samplers have a collecting area of 0,02 m^2 (Icelandic Meteorological Office)	GM-tubes or NaI detector at the ground or 1 m above ground	Wet+dry deposition measured by analysing soil samples with HPGe detector.	Aluminium precipitation collector (4 m^2). 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 m^2) 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 with 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/m^3 and Bq/m^2	Bq/m^2 per sampling period	Bq/litre and Bq/m^2			Bq/m^2

Appendix A. Questionnaire

Sampling methods –

A survey of methods in use in the Nordic Countries

(NKS/BOK-1.1: Laboratory measurements and quality assurance
a sub-project within the NKS/BOK-1 project)

Questionnaire

Introduction

As stated at the NKS/BOK-1.1 Web Site, the aim of the BOK-1.1 sub-project is to assist participating laboratories to improve and demonstrate the quality of their measurements, taking into consideration the needs of emergency preparedness and radioecology. The project is divided into three periods:

- 1st period: December 1998 - August 1999
- 2nd period: August 1999 - autumn 2000
- 3rd period: autumn 2000 – autumn 2001

where each such sub-period will focus on some defined tasks and end with a workshop where the results of the work will be presented along with proposals for new tasks in the next period.

In the first period the following work tasks are being dealt with:

- Sampling methods - A survey of methods in use in the Nordic Countries
- Intercomparisons on radioactivity in environmental samples
- Effects of the introduction of a new standard, ISO/IEC DIS 17025, on the process of accreditation of laboratories

The present work constitutes the first task, where the aim is to compile information on the techniques currently in use for sampling radionuclides in the Nordic countries, with special emphasis on techniques for emergency preparedness and monitoring (not scientific studies of special processes). At present the project concentrates on the sampling of:

- **vegetation**
- **soil**
- **depositions**

It would be of great value for this project if you could contribute with descriptions of your specific sampling methods by responding to this questionnaire. *If you have additional information, not covered by the questions, please feel free to add anything you think could be of value for the survey*. Since the project will be reported by the end of 1999 I would appreciate if you could give me your answers not later than October 31st.

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Questionnaire

Vegetation sampling

1. How is the sampling area chosen?
2. What is the size of the area usually sampled?
3. What kind of tool is used to define the area?
4. How are the sampled areas distributed on a sampling site?
5. At what height is the vegetation cut? (If the cutting height varies for different types of vegetation, please indicate this).
6. What kind of tool is used for cutting?
7. How are the samples prepared for measurement (weighing, drying, chemical pre-treatment, etc.)?
8. What kinds of detectors are used for the activity determination?
9. What measurement geometry is used?
10. How are the samples stored before and after measurement?
11. How are the results reported?

Soil sampling

12. How is the sampling area chosen?
13. What kind of tool is used for the soil sampling?
14. What is the size of the area usually sampled?
15. How are the samples distributed on a sampling site and how many samples are taken?
16. What is the depth sampled in an emergency situation?
17. Do you subdivide the soil? How?
18. How are the samples prepared for measurement (weighing, drying, chemical pre-treatment, etc.)?
19. What kinds of detectors are used for the activity determination?
20. What measurement geometry is used?
21. How are the samples stored before and after measurement?
22. How are the results reported?

Deposition sampling

23. Please describe the method you use for wet deposition sampling (size of precipitation collectors, ion exchange systems, etc.)?
24. Please describe the method you use for dry deposition sampling?
25. How are the samples prepared for measurement (weighing, drying, chemical pre-treatment, etc.)?
26. What kinds of detectors are used for the activity determination?
27. What measurement geometry is used?
28. How are the samples stored before and after measurement?
29. How are the results reported?

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Abstract	This report is a survey of sampling techniques currently in use for radioactivity measurements in the Nordic countries, but restricted to sampling techniques for pasture, soil and deposition in emergency situations. It is found that the participating laboratories apply similar sampling procedures for pasture, including cutting height and size of sampled areas. Soil samples are generally taken by some sort of corer of different diameter. The number of cores taken varies, different sampling patterns are used, and pooling of the samples is done by some of the laboratories. The analysis of pasture and of soil is made with NaI-detectors or by high-resolution gamma spectrometry on fresh or dried samples. Precipitation collectors of a range of sizes are used to determine the activity concentration in precipitation and of dry deposited radionuclides. The analysis is made with high-resolution gamma spectrometry, either directly on a water sample or on ion exchange resins.
Key words	Radionuclides, sampling, emergency preparedness, pasture, soil, deposition

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