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Sampling in forests for radionuclide analysis – General and practical guidance

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Abstract

The NKS project FOREST was established to prepare a guide for sampling in forest ecosystems for radionuclide analysis. The aim of this guide is to improve the reliability of datasets generated in future studies by promoting the use of consistent, recommended practices, thorough documentation of field sampling regimes and robust preparation of samples from the forest ecosystem.

The guide covers general aims of sampling, the description of major compartments of the forest ecosystem and outlines key factors to consider when planning sampling campaigns for radioecological field studies in forests. Recommended and known sampling methods for various sample types are also compiled and presented. The guide focuses on sampling practices that are applicable in various types of boreal forests, robust descriptions of sampling sites, and documentation of the origin and details of individual samples.

The guide is intended for scientists, students, forestry experts and technicians who appreciate the need to use sound sampling procedures in forest radioecological projects. The guide will hopefully encourage readers to participate in field studies and sampling campaigns, using robust techniques, thereby fostering competence in sampling.

Key words

Forest, sampling, radioactivity, boreal

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SAMPLING IN FORESTS FOR RADIONUCLIDE ANALYSIS

- GENERAL AND PRACTICAL GUIDANCE

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NKS Project FOREST
January 2009

FOREWORD

This sampling guide was prepared in collaboration between four Nordic research organisations:

FOI, Sweden
IFE, Norway
Metla, Finland
STUK, Finland.

The FOREST project, financed by the organisations involved and Nordic Nuclear Safety Research (NKS) in 2005 and 2006, was established to improve knowledge regarding sampling in radioecology and forestry investigations and, thus, the quality of samples used and the data obtained in studies on forest radioecology. The project has been co-ordinated by Elisabeth Strålberg, IFE, Norway.

Experts on forest research and terrestrial radioecology have critically reviewed this guide to sampling methodology. The peer reviewers were:

Eiliv Steinnes, Norway
Pasi Rautio, Finland
Harald Grip, Sweden

The authors acknowledge the financial and intellectual support provided by NKS and other organisations involved in the project, and appreciate the substantial inputs of experts who reviewed the guide.

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1. Introduction

There has been a need for a detailed sampling guide since the late 1980s, when collaborative, multidisciplinary forest radioecology analyses were prompted by the Chernobyl disaster. However, inadequate documentation of the sampling procedures and field sites has prevented many otherwise interesting datasets from being used in assessment models or comparisons of sites. The aim of this guide is to improve the reliability of datasets generated in future studies by promoting the use of consistent, recommended practices, thorough documentation of field sampling regimes and robust preparation of samples from the forest ecosystem.

Sampling strategies, which should reflect the research objectives, are also influenced by factors such as the available human and financial resources. This guide covers general aims of sampling, the description of major compartments of the forest ecosystem and outlines key factors to consider when planning sampling campaigns for radioecological field studies in forests. Recommended and known sampling methods for various sample types are also compiled and presented.

The guide focuses on sampling practices that are applicable in various types of boreal forests, robust descriptions of sampling sites, and documentation of the origin and details of individual samples. Various types of regional sampling regimes, radioactivity studies and monitoring procedures are illustrated in Appendices. The report is based on recently published general guidelines (Eurachem, 2006; IAEA, 2004; ICRU, 2006; Nordtest, 2007), and expertise and practical experience of the involved organisations.

The guide is intended for scientists, students, forestry experts and technicians who appreciate the need to use sound sampling procedures in forest radioecological projects. The guide will hopefully encourage readers to participate in field studies and sampling campaigns, using robust techniques, thereby fostering competence in sampling.

2. Sampling in forests - general aspects

Information on the activity concentrations and/or contents of radionuclides in forest compartments or sub-compartments is generally acquired from analysis of samples. Thus, sampling is a key step in the acquisition of information regarding their abundance and distribution in environmental compartments of interest, prior to analyses by laboratory or field station equipment. The results of laboratory analyses are generally expressed following conversion of the units, for instance Bq kg^{-1} fresh weight to Bq kg^{-1} dry weight, or vice versa. Hence, the dry matter contents of undisturbed (carefully packed, transported and stored) samples in specified conditions must be appropriately determined. Samples to be used for determining the biomass, and/or abiotic components, present in a forest compartment can either be taken simultaneously with samples for radionuclide determinations or collected separately. Direct alpha, beta or gamma spectrometric analyses of radionuclides can be used, in conjunction with biomass estimates obtained from appropriate models, to obtain generalised overviews of radionuclide activities in forests. However, for accurate scientific determinations of their abundance and distribution laboratory analyses of samples, using validated techniques, is essential.

2.1 Expected quality of samples

The sampling strategy employed in a particular study will depend on the research objectives, and will differ (for instance) between studies of internal and external exposure to radiation, and between studies of human and biotic exposure. The desired, and expected, representativeness of the sampling needs to be spatially and temporally defined in accordance with the aims of the study, and the site conditions. In environmental radioactivity surveys the sampling may be either systematically based on prior knowledge (e.g. previous radioecological observations) or some kind of random sampling (e.g. using totally random placed, or evenly spaced plots, see Appendix A). Also the accuracy of results to be obtained has to be specified. If the contamination history and management of the site is not known, a preliminary study preceding larger-scale or more focused sampling may radically improve the planning of the sampling campaign and provide valuable indications regarding the most suitable techniques to use.

Sampling has a major effect on the reliability of the results obtained in subsequent measurements. Hence, representative sampling is crucially important, as emphasised, for instance, in the *Soil Sampling for Environmental Contaminants* report published by the International Atomic Energy Agency (IAEA, 2004). The cited report describes (*inter alia*) potential sources of errors in obtained results, including the sampling design, sampling methodology, sample heterogeneity and analytical procedures. In addition, various methods for estimating uncertainties originating from measurements and sampling are presented. Methods for estimating sampling uncertainty can also be found in the Nordtest Handbook (Nordtest, 2007) and the Eurachem guide (Eurachem, 2006). In addition, guidance for quality assurance in sampling is provided in these reports. For instance, a straightforward (albeit sometimes costly) way to reduce random effects on

analytical results and measurement uncertainties, due for example to variations in the composition of the samples, noted by the Nordtest Handbook (Nordtest, 2007, page 8), is to increase the number of samples taken. Guidance for calculating sampling statistics, such as estimates of spatial or temporal variations is provided in the report *Sampling for radionuclides in the environment (ICRU, 2006)*, which together with the IAEA's report on soil sampling (IAEA, 2004) provides a selection of designs for spatial sampling (Appendix A).

Examples of earlier studies that highlight differences in objectives, approaches and the intensity or frequency of sampling in space and time are given in Appendix B.

2.2 Factors to consider in sampling plans

In order to map or survey a large area, relevant statistics must be considered, notably the number of samples that will be needed to map or represent the area of interest with acceptable uncertainty. This will depend on the size of the area and the choice of sampling design. The number of samples and the most appropriate sampling design will in turn be influenced by the available resources, which may limit the timing and frequencies as well as the number of samples that can be collected. Sometimes it may be necessary to pool samples because of a lack of sample material or measurement capacity, but then the variation in the set of samples will inevitably be lost. However, the advantage of combining several systematically collected samples before analysis is that the pooled samples will be more representative of the overall area than any single sample. Effects of sampling and radionuclide heterogeneity are thus decreased, which might be important in certain field experiments. On the other hand, if the focus is on the range of radionuclide concentrations, then samples should be analysed individually rather than pooling them.

The limit of detection (LOD), of the radionuclide determination, and even more so the limit of quantification (LOQ, i.e. the lowest amount of the relevant radionuclide in the sample that can be quantitatively determined with acceptable precision and accuracy) affect the required quantities of samples (ICP Forests, 2008). The radionuclides of interest and available analytical techniques must also be considered when assessing the minimum quantities of samples required. The LOQ will vary depending on the sample type. Not only the method of determination, but also the representativeness of the sampling, sample preparation and the need for archiving samples are important aspects to consider.

Regarding the use of a forest site for sampling, permission often has to be acquired in advance from the landowner. In Nordic countries there is a legally enshrined "everyman's right" that permits anyone to roam with very little restriction, and (*inter alia*) to pick berries and mushrooms. However, such rights are much more limited in other countries, and access to sites for sampling needs to be ensured in advance.

2.3 Documentation and packaging of samples

Samples should be packed, individually, in clean bags. If the samples are to be further prepared quite soon after sampling and can be stored in a dry, well-ventilated place, use of paper, rather than plastic, bags for plant material is recommended. However, if samples are frozen before further pre-treatment, the paper bags may expose the samples to freeze-drying processes, which must be considered if the fresh weight or some other variable of fresh samples is relevant to the outcome of the analysis. A further practical point to bear in mind is that if samples are wet (e.g. moist mosses) paper bags can readily break down.

Each sample should be identified by recording all relevant information about it, the sampling site and the technique used to acquire it on a label, which should be attached to the outside of the bag containing it. The following information is needed to identify each sample:

- date of sampling
- sampling site
- code identifying the sample (i.e. details such as the specific sampling point, when multiple samples are collected from a site)
- for soil and vegetation: the surface area sampled (i.e. frame size)
- for fungi and vegetation: the name of the species collected (if the sample is species-specific)
- for trees: the tree part
- for soil: the layer, its thickness and its depth from the soil surface
- any observation that might influence interpretation of the results, and
- the name of the collector.

Any information that applies to all samples collected at a site can be recorded on a sample sheet or form and enclosed with the series of samples. This will save time in the field. Such information may include:

- the location of the sampling site
- the surface area sampled (i.e. frame size).

Sample sheets should also provide a description of the site, as discussed in Chapter 3.2.

2.4 Sample treatment

At all times when handling samples and sampling devices at the site (for instance when samples are being divided into fractions), when handling unpacked samples at the site, and preparing them for analysis in the laboratory, operatives need to avoid cross-contamination, otherwise uncontrollable errors may arise in all later determinations of their radionuclide contents.

After sampling, the samples have to be packed, transported to the laboratory and stored for further analysis, using techniques that preserve the original contents of radionuclides and other substances to be analysed. A storage temperature should be chosen at which the original form of the samples is maintained until they are prepared for analysis in the

laboratory. Sometimes it may be best to dry the samples during storage, but in other cases there may be a clear need to preserve their original water contents, e.g. if they are to be analysed for water-bound tritium. In such cases samples may need to be frozen.

Additional questions are:

- Is extraneous material present in the samples?
- Will the sample preparation treatments limit the possible sample size?
- Can the samples be stored without any preparation?
- Will sample preparation cause losses of analytes from the sample?

Sampling instructions should also remind personnel of the need to ensure that samples remain undisturbed during transit from the field to the laboratory and (thus) that appropriate packaging materials are available for their transport.

2.5 Considerations for specific radionuclides

When planning a sampling campaign it is important to realise that radioisotopes of different elements differ in terms of the time-dependent processes that affect their distribution in forest compartments. It is also important to be aware of the origins of the investigated radionuclides, since anthropogenic and/or natural radionuclides involved in nutrient transfers and abiotic partitioning in forests may originate from atmospheric deposition, the bedrock or hydrological sources.

For sampling of water for analysis of radionuclides, the requirements concerning the containers are much the same as those for other trace elements. It is important to ensure that the materials used will not irreversibly adsorb the radionuclide(s) of interest or that techniques can be used to prevent their irreversible adherence to container surfaces. This is particularly important for those radionuclides that have no naturally occurring carrier elements (i.e. stable isotopes of the same elements). For instance, isotopes of polonium are all radioactive and a real risk for losses exists due to surface adsorption. Polyethylene containers are useful for many radionuclides, particularly when adding carrier elements and adjusting pH is considered as early as possible before storage of samples.

Pre-treatment methods vary according to the nuclides and types of samples studied. They may also vary between laboratories, which can affect the comparability of the results. Plant materials and soil are often dried at 60 ^{o}C or 105 ^{o}C, although soil is sometimes dried at just 40 ^{o}C. Dry ashing at temperatures lower than 500 ^{o}C and $\leq$ 600 ^{o}C is recommended for determinations of caesium and strontium, respectively. For determinations of isotopes of technetium, plutonium and lead wet ashing is favoured, and microwave digestion is often applied.

For determinations of some radionuclides, e.g. ^{14}C and organically bound tritium (OBT), in samples containing organic materials the samples are dried. Automated facilities for combustion of samples, absorption of CO_2 and preparation of liquid samples for beta measurement are currently used that ultimately yield separate liquid

samples for determinations of ^{14}C and OBT. Both of these isotopes can be measured using a liquid scintillation spectrometer.

Losses of water-bound tritium in samples can occur if water is allowed to evaporate from fresh samples during collection, handling in the field and transport. It is therefore essential to pack the samples in airtight containers as quickly as possible after sampling and store them in suitable containers until they are to be analysed in the laboratory. Such samples are usually deep frozen for storage. Generally, knowledge of the chemical characteristics of all relevant radionuclides and elements to be studied has to be acquired well in advance of the study, during the planning stages.

The amounts of samples required, which will depend on the radionuclide(s) under investigation (and their abundance), for the analysis must also be considered. For gamma spectrometric measurements the amounts of dried, homogenised plant material or soil collected must correspond to the volume of calibrated sample containers used. For alpha- and beta-emitting nuclides the method to be used to analyze them has to be considered, and sufficient sample material must be collected to ensure that uncertainties of the final results are sufficiently low to meet the study's requirements.

3. Forest definitions and guidelines for site description

3.1 Forest definitions

According to the Food and Agriculture Organization of the United Nations (FAO) forest land is defined as land covering more than 0.5 hectares with trees taller than 5 meters and a canopy cover of more than 10 percent, or trees capable of reaching these thresholds *in situ*.

The total forest land area in the Nordic countries amounts to ca. 60 million hectares according to the FAO definition, and is distributed by countries as follows (Finnish Statistical Yearbook of Forestry, 2008):

Denmark	500 000 ha
Finland	22 130 000 ha
Iceland	43 000 ha
Norway	9 387 000 ha
Sweden	27 871 000 ha

Each country also has its own definitions of forest land (see, for instance, the Finnish Statistical Yearbook of Forestry, 2008; Gjertsen *et al*., 2004; SSB, 2006). Forest type classifications have been provided in the reports "European forest types - Categories and types for sustainable forest management reporting and policy" (European Environment Agency, 2006) and "Vegetationstyper i Norden" (Påhlsson, 1994).

3.2 Guidelines for site description

Site descriptions are extremely important, for example when comparing datasets from different areas or when data from different areas are compiled and used in model predictions. Types of information that should be included in site descriptions, where applicable, may include:

- location
  - geographical location: coordinates (specifying the type of coordinates), map
  - elevation
  - topography
  - vegetation zone
- site properties
  - soil type (for more details, see Chapter 4.1)
    - organic or mineral soil, rocky soil etc.
    - thickness and type of the organic layer
    - thickness of overburden
    - stoniness
    - textural class of mineral soil (i.e. particle-size distribution)
  - hydrology
  - vegetation type
  - typical plant species (indicator species) and their percentage cover

- tree stand
    - tree species
        - main species
        - secondary species
        - full scientific names, common names
    - main tree stand characteristics, e.g.
        - age
        - volume, density (trees/area, basal area)
        - mean height, diameter at breast height, length of living crown
- land-use history (if available):
    - timing and descriptions of forest management measures applied
    - former agricultural history
    - original site types of peatlands drained for agriculture or forestry

Most of this information has to be recorded based on measurements and observations of the site characteristics obtained during the sampling.

4. Soil

4.1 Description of the compartment

Soil is a vital part of the environment. It controls the flow of water and chemical compounds between the atmosphere and the earth, and acts as both a source and store for gases (like oxygen and carbon dioxide) in the atmosphere. It influences the distribution of plant species and provides a habitat for a wide range of organisms. The aim of soil sampling may be to define the concentration (Bq/g) or the content per unit ground area (Bq/m^2) of a radionuclide or a compound in the soil.

Soil is unconsolidated debris overlaying hard unweathered bedrock, created when exposed bedrock crumbles and decays. The unconsolidated layer is called the regolith and varies in thickness from virtually nonexistent in some places (i.e. exposed bare rock) to tens of meters in other places. The regolith material has often been transported many kilometres from the site of its initial formation and then deposited over the bedrock, which it now covers. Thus, the degree to which the regolith is related to the bedrock below it may vary widely. There are three classes of soil: organic, sediment and till. The mineral soils are also classified according to the size of the soil particles.

Horizontal layers, called horizons of soil (see Figure 1), develop through abiotic and biotic processes. The development of these horizons in the upper regolith is a unique characteristic that sets it apart from the deeper regolith material. Soil profiles can provide warnings about potential problems in using the land, and provide abundant information about the environment and history of a region. In some soil profiles the horizons have very distinct colours, with sharp boundaries that can be easily seen, while in other soils, the changes in colour between the horizons may be very gradual, and the boundaries more difficult to discern. However, colour is only one of many properties by which one horizon may be distinguished from the horizon above or below it.

A number of soil classification systems are used in different parts of the world, for example the US Soil Taxonomy (Soil Survey Staff, 2003) and the FAO/UNESCO (FAO, 2001) systems. In this text the US Soil Taxonomy will be used. There are six categories of classification in the Soil Taxonomy system: 1) order, 2) suborder, 3) great groups, 4) subgroups, 5) family and 6) series. Each of the world's soils are assigned to one of the 12 orders listed below:

- Entisols-Recent, little if any profile development.
- Histisols-Deep accumulation of organic materials, wet conditions.
- Andisols-Mild weathering on volcanic ejecta.
- Inceptisols-Mild weathering, various conditions, inception of B horizon.
- Gelisols-Very cold, permafrost.
- Aridisols-Desert shrubs, grasses, dry.
- Vertisols-High base status, high activity clays, dry season.
- Alfisols- Moist, mildly acid, clay accumulation.
- Mollisols-Semi-arid to moist grassland, mollic epipedon.
- Ultisols-Wet tropical and subtropical forest, acid silicate and Fe, Al oxides.

- Spodosols-Cool, wet, sandy acid, coniferous forest.
- Oxisols-Wet, tropical forest, extreme weathering, low activity, clays, Fe, Al oxides.

Most of the soils in the Nordic countries belong to seven of these orders (Entisols, Histisols, Andisols, Gelisols, Inceptisols, Vertisols and Spodosols) but soils of other orders may occur in small areas.

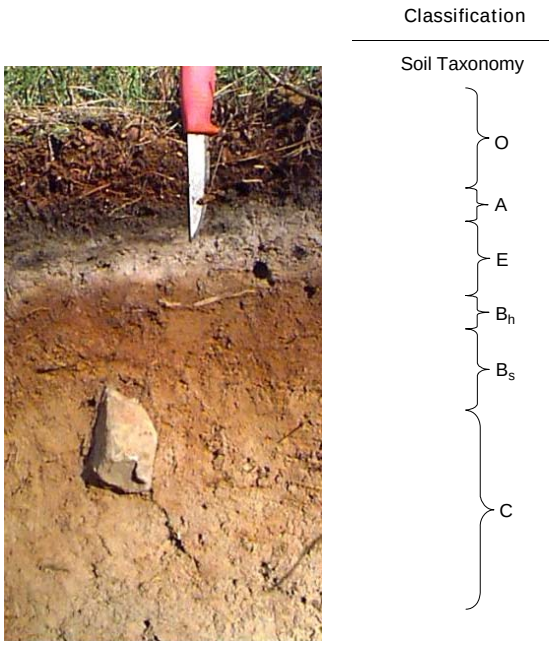

Figure 1. Classification of the soil horizons in a Spodsol according to the Soil Taxonomy system.

Some of the soil layers illustrated in Figure 1 are defined as follows:

- **O** Layers dominated by organic material. Some O layers consist of undecomposed or partially decomposed litter.
- **A** Mineral horizons that have formed at the surface or below an O horizon, in which there is an accumulation of humified organic matter, strongly mixed with the mineral fraction, and characteristics of E or B horizons are weak or absent.
- **E** Mineral horizons in which the main feature is the loss of silicate clay, iron, aluminium or some combination of these substances, leaving a layer largely composed of sand and silt particles.
- **B** Enrichment horizon
  - B_h Illuvial accumulation of organic matter
  - B_s Illuvial accumulation of sesquioxides and organic matter.
- **C** A soil layer that is little affected by pedogenic processes.
- **R** Bedrock

Four major components of soil are air, water, mineral matter and organic matter. The relative proportions of these components greatly influence the behaviour and productivity of soils (Brady and Weil, 1999).

The uptake conditions of radionuclides can be modified by forest management treatments such as various harvesting methods and additions of fertilizers or other kinds of amendments, e.g. lime or ash, to the soil. However, no detailed descriptions of soil treatment techniques are provided in this guide, since they often have no effect on the sampling procedure.

4.2 Sampling methodology

Soil samples can be collected using simply a spade or some kind of auger, depending on the kind of soil to be sampled (mineral soil of various textures or organic soil). However, if soil augers or spades are used to collect soil cores the resulting samples are often disturbed, and thus should not be used for volume estimations of the soil. If undisturbed soil samples need to be collected steel cylinders of 5-10 cm should be used, which should be driven perpendicularly into the soil. The soil surrounding the cylinder should be carefully removed, then the top and bottom soil should be carefully tended. Undisturbed soil samples are needed to estimate the dry volume-weight of the soil in order to estimate the content per unit ground area (Bq/m^2) of a radionuclide. Undisturbed soil samples are also essential for measuring many physical variables of the soil, such as its porosity and hydraulic conductivity.

Various strategies can be used to select collection points of specific soil cores/samples, as further described in Appendix A and the IAEA (2004). Collection of samples with a random strategy (Figure A1) may save some time. However, cores/samples need to be collected from the entire area to be surveyed in order to obtain the most reliable estimates. Collecting soil cores/samples in a grid pattern (see Appendix A) may require more time.

The division of each sample into layers (see Figure 1) will provide information on the depth distribution and movements of elements in the profile. Dividing a soil sample into appropriate layers before analysis strongly affects the potential utility of the results. Even when soil samples are to be used for calibration of an instrument it is important to divide them correctly. Diverse physicochemical properties, including density and water content, differ between soil layers. Hence, one should try to divide them accordingly. If soil samples are not properly divided into layers the element concentration data obtained will give erroneous indications about the depth distribution of elements of interest, and thus lead to inaccurate estimates of their contents per unit ground area.

The depth that needs to be spanned by the samples depends on the elements to be studied, the origin of the elements (bedrock or deposition), time since their deposition (if deposited) and the mechanisms involved in their redistribution in the ecosystem. When collecting soil samples to calibrate field measurements, samples should be collected to sufficient depth to ensure that they contain all of the activity contributing to the measurements.

Proper sampling can provide an accurate representation of concentrations of elements in the field. If individual samples are analysed separately they can also be used to produce

element concentration maps for the studied area. It is more costly, however, to analyse a large number of individual samples than a few composite samples. Alternatively, therefore, individual soil cores/samples can be mixed to create composite samples, which will provide good indications of average concentrations of elements in the field, but will not provide data on variations in potentially important variables. The heterogeneity of variables such as concentrations of elements in the soil will influence the number of soil samples that need to be collected. Additional information that individual soil samples provide facilitate the elucidation of ongoing environmental processes. Furthermore, to avoid errors due to seasonal variations, soil samples should be collected at approximately the same time each year, or in time series at appropriate intervals throughout the year.

The following factors should be considered before going out into the field to collect soil samples:

1. The kind of sampling strategy to use.
2. Whether individual samples or composite samples should be collected.
3. The number of individual cores/samples to collect per composite sample.
4. Sampling depths, depending on what is to be sampled.
5. Division of cores by depth. The ideal thickness of the samples depends on the aims of the study, the soil profile and/or processes to be studied
    a. Surface sample
    b. Subsurface sample
    c. Deeper subsurface sample
    d. Samples from additional depths originating from the parent material

When sampling the soil the vegetation layer should be removed or harvested before collecting the soil samples (collection of understorey plants is described in Chapter 7). This procedure limits the risk of cross-contamination between soil and vegetation samples.

If a defined volume of soil is to be collected everything in that volume needs to be collected, including roots and stones. If for any reason roots or stones are to be excluded from the analysis they should be removed at the laboratory, after determining the density of the sample.

4.3 Sampling equipment

- Spade (always useful, even if a soil corer is used)
- Soil auger/corer, see Figure 2.
- Steel cylinders for undisturbed samples
- If a spade is used for sampling and a defined sample volume need to be sampled it could be very useful to bring a frame corresponding in size to the area to be sampled. If this is not possible use a ruler.
- Knife
- Plastic bags
- Waterproof marker
- Ruler

- Protocol
- Camera, which could be useful for documentation.
- A plastic sheet to cover the ground if there is a risk of the soil surface contaminating samples.

Figure 2. Examples of sampling devices for different soil types: soil corers for till soils (a) and sediment soils (b), and a peat sampler (c).

5. Soil solution and streamwater

5.1 Description of the soil solution

The soil solution consists of water held by capillary and adsorptive forces in the pore spaces of the soil or percolating downwards due to gravitation. In an unsaturated soil water occupies only the smallest pores while, the soil air occupies the larger ones. Depending on the pore size distribution of each individual soil there is a unique functional relationship between soil water content and tension, i.e. the magnitude of the force needed to extract water at a given water content. Most chemical reactions in a soil take place in the water phase at the soil particle surfaces, and the closer to the surfaces the higher the electrolyte content of the soil solution.

Sampling of the soil solution is an essential component of many studies addressing diverse biogeochemical questions. The composition of soil solutions obtained from the field may vary, depending on the method of extraction. Artefacts introduced by sampling methods and the difficulties in extracting representative samples from soils in space and time can often explain divergent results. Several methods can be used for collecting soil solution samples, e.g. centrifugation or lysimetry, using either zero-tension or tension lysimeters. Artefacts that should be considered include mineralization due to the effects of cut roots in zero-tension lysimeters, adsorption/desorption from tension lysimeters and changes in solute concentrations due to bulking of soil samples when centrifugation is used. See, for example Giesler (1996), for more details regarding advantages and disadvantages of the three methods mentioned here.

5.2 Sampling methodology

To extract soil water by high speed centrifugation undisturbed soil cores are taken in the field by pressing a steel cylinder of suitable size into the soil at the selected depth. The filled cylinder is then excavated, its external surfaces are cleaned and it is capped at each end. In the laboratory the soil samples in the cylinders are centrifuged, using appropriate tubes (often double-bottomed, in a swing-out rotor), and the artificial gravity field applied to the moist samples draws the soil water into a collection cup. It should be noted that since this method of sample collection is destructive, it is not possible to repeat the sampling at a different time at the same spot.

A zero tension lysimeter often consists of a porous plate, sealed at the bottom and drained by a tube. The plate is introduced from the side into the soil, where a suitable space has been excavated, which is sometimes backfilled to maximize the plate's hydraulic contact with the surrounding soil. In principle this type of device will sample draining water from large pores with weak tension. A zero tension lysimeter is a practical option when time series of soil water samples are needed and when the investigation concentrates on percolating water.

A tension lysimeter consists of a porous cup made of a suitable inert material with low ion exchange capacity inserted, in good hydraulic contact with the soil, at the chosen

sampling depth. The cup is connected to a collection bottle via a tube and the whole system is evacuated to drain soil water. Such devices can extract water with tensions lower than ca. 80 kPa and are suitable when time series of soil water are required.

5.3 Description of runoff formation and streamwater

Every point in a landscape is part of a catchment delineated by a water divide. Precipitation falling inside a water divide that is not evaporated will eventually reach the outlet of the catchment, while precipitation falling beyond the water divide will reach the outlet of another catchment. Precipitation or snowmelt in the catchment will infiltrate the soil, and if not taken up by plants and transpired, percolate down to the groundwater surface. In the upper parts of slopes (the "groundwater recharge area") water has a flow component oriented towards the groundwater zone, while at the bottom of slopes (the "discharge area") the water has a flow component directed away from the groundwater body. With stable isotopes it has been shown (e.g. by Sklash and Farvolden 1979; Rodhe 1984) that even during peak flow events most streamwater consists of water that was in the catchment before the events. Thus, most of the water in a stream generally consists of former groundwater discharged from discharge areas.

The infiltration capacity of forest soils in the Nordic countries is largely sufficient to absorb heavy showers and snowmelt. Overland flows on top of unsaturated soils do not, therefore, usually occur in Nordic recharge areas. In discharge areas, on the other hand, surface runoff or runoff close to the surface of saturated soils does commonly occur. A major implication of this pattern is that it will take a long time for hydrologically transported substances, e.g. various radionuclides, deposited on recharge areas to reach discharge areas and later streams. In contrast, elements deposited on discharge areas will leak to the streams quite quickly. Further, since discharge areas generally constitute only a small part of their respective catchments, most fall-out will remain for long periods in the catchment.

Streams draining discharge areas converge, forming successively larger streams and rivers, then eventually discharge into lakes or the sea. The flow regime of a river depends on annual variations in the local climate. In northern parts of Scandinavia and Finland the winter is long, consistently cold and snow accumulates throughout it. Thus, the main flow event during the year is associated with snowmelt in spring. In large parts of the Nordic countries July is the wettest month of the year, but at the same time evaporation is highest, leaving less water for runoff. In some regions there are also high flows in the autumn, due to a runoff-enhancing combination of high precipitation and low evaporation.

The flow through a stream varies during the hydrological year and depends on the aquifers involved. During periods of direct runoff (storm periods or snowmelt) both stored surface water and precipitated water/melted snow will be carried via saturated overland flow, and thus increase discharge rates, while during periods when stream flow is lower the main contributor will be discharged groundwater. Stormflow events generating saturated surface runoff are not evenly distributed during the year. Instead, runoff rates in the Nordic countries are generally low during the winter (November to

April) and summer (June to August) and highest during the spring (the end of April to beginning of May) and autumn (September to October). In Southern Fennoscandinavia the maxima occur during late autumn. As a consequence of the seasonality, the discharge of nutrients from land to rivers and lakes is likely to be highest during the spring in northern Fennoscandinavia and during the autumn in the southern parts.

During saturated overland flow or during groundwater movement, water can pick up nutrients and soil contaminants and transport them out to streams. Radionuclides with a superficial distribution in soil will migrate to some extent during such periods, or snowmelt, and be transported via riverines to the outlet. Percolating water will also carry nuclides down through the regolith and finally reach the outlet. Rates of radionuclide removal in this manner are generally very low, and influenced by a number of factors in addition to water saturation (*inter alia*, soil texture, competing cations and the type of radionuclide). Hence, activity concentrations are generally very low in runoff water, and quite low in streams even during fall-out events. However, despite the low concentrations the activity carried via runoff is not negligible in the bioaccumulation processes in lakes, and runoff is an important source of contamination of rivers and lakes, indeed it may be the most important factor, besides the redistribution of lake sediments, for the uptake of radioactive caesium in fish in long-term perspectives.

5.4 Sampling methodology

To estimate yearly runoff from a catchment the streamflow intensity at the outlet of the area must be estimated at sufficiently high temporal resolution. This can be done by monitoring water stage (the elevation of the water surface) in a control section upstream, using an appropriate recorder, and converting it to streamflow by means of a specific rating curve for the control section. Ideally, the control section should be an artificial construction in the water course, for example a V-notch weir with a 90-degree opening. Typical natural control sections are the top of rapids where the state of water movement changes from subcritical to supercritical. Rating curve parameters must be calibrated by simultaneously measuring stage and flow at different flow rates. For small streams the volume flow rate can be determined simply by measuring the time required to fill a bucket of known size. The salt dilution method is suitable for larger streams and integration of the velocity field measured by a current meter across large rivers will give their flow intensity.

The timing and number of samples required for element concentration analysis depend on the number of events and the water flow variability. The amount of water that needs to be collected at every sampling occasion is dependent on the concentrations of the elements of interest and the analytical methods used, and may vary from a few millilitres to tens of litres. One must also (as always) be very careful not to contaminate the samples.

6. Fungi

6.1 Description of the compartment

The Kingdom Fungi includes some of the most important organisms, both ecologically and economically. Fungi are eukaryotic organisms, i.e. their DNA is contained in a nucleus. Many of them may look plant-like, but unlike plants fungi do not synthesise their own carbohydrates.

Almost all fungi participate in nutrient cycling. This is best known for saprotrophs (decomposers), particularly those that degrade wood (cellulose and lignin). Two other roles are equally important, however, in this ecosystem function. Firstly, fungal mycelia are large sinks for organic carbon and nutrients in the soil. In ectomycorrhizally-dominated forests, a significant proportion of the carbon fixed by host plants may be transferred to the mycorrhizal structures and fungal mycelium, thereby supporting this symbiotic association (Smith and Read, 1997). A mycorrhizal symbiosis is a mutualistic symbiosis in with both partners benefit from the association; carbohydrates move from the plant to the fungi, while nutrients and perhaps water derived from the soil move in the opposite direction. Some fungal mycelia and ectomycorrhizal root tips turn over rapidly and thus return nutrients to the soil, whereas other fungal structures such as rhizomorphs retain nutrients in relatively long-lived structures or shunt it to other parts of the mycelium as some sections die. A key feature of fungi, in this context, is that they are major sinks that maintain nutrients on site, thereby preventing nutrient leaching and loss from soils. Secondly, fungi exude polysaccharides similar to root exudates, and these 'organic glues' (Perry *et al.*, 1989) play important roles in creating and stabilizing soil microaggregates and soil micropores, which make major contributions to soil aeration and water movement. Since most terrestrial organisms are strongly aerobic, this fungal contribution to soil microaggregation is critical for the overall maintenance of soil health and productivity. Furthermore, fruit bodies of fungi (mushrooms and truffles) are vital food sources for wildlife.

We know little about fungal ecology in general, and until recently we did not have tools that allowed us to define individuals, let alone populations. Further, the nutritional mode of many fungal species is uncertain, making it difficult to use 'food source' as a habitat variable.

The mass of thread-like mycelium (thallus) that gives rise to fruit bodies is frequently hidden from view. Attempts to characterize the size and distribution of discrete thalli have identified individuals encompassing areas ranging from square centimetres (Gryta *et al.*, 1997) through square metres (Baar, Ozinga and Kuyper, 1994) to hectares (Smith, Bruhn and Anderson, 1992). Dahlberg and Stenlid (1990) have also shown that ecological factors such as forest age can influence the size of individuals and alter the mechanism (sexual or vegetative) whereby new thalli are established. The range and variance of the sizes and distribution of individuals are unique to each fungal species, and thus dictate both the scale at which population boundaries are likely to be encountered, and the minimum size of appropriate study areas.

Perhaps surprisingly, it is not possible to state with any certainty the numbers of species present in a particular locality. Fungi differ from plants in that, with the exception of perennial species like some brackets, it is not possible to predict when or whether their fruiting structures will appear from year to year. They are also very easy to miss, since most species produce fruit bodies that decay and disappear within a few days.

6.2 Sampling methodology

Sampling of fungal fruit bodies
A number of problems are associated with fruit body sampling, which should always be borne in mind. Notably, the abovementioned sporadic production of fruit bodies – as few as 5% of species observed at sites in one year may recur in the following year (O'Dell, Ammirati and Schreiner, 1999) – and the difficulties in detecting various easily-overlooked fruit bodies, such as resupinate forms found on the underside of woody debris. Thus, many repeated visits to a site are required to obtain representative samples and to determine the presence or absence of a species at a given site. Furthermore, even intensive fruit body surveys poorly reflect the composition of belowground ectomycorrhizal communities, making interpretations of acquired data difficult at the community level (Gardes and Bruns, 1996; Dahlberg, Jonsson and Nylund, 1997; Kårén *et al.*, 1997; Jonsson *et al.*, 1999). Therefore, to ensure adequate replication of the species of interest fruit body sampling should generally be carried out over the course of several years, and samples should be collected frequently (during peak fruiting seasons, significantly fewer species may be detected in sampling campaigns with two-week intervals than otherwise identical weekly campaigns). However, repeated visits to a site may be impractical unless an intensive study is being conducted.

Preferably, fruit bodies of the same developmental stages should be collected for analysis. Decomposition processes could have started in old fruit bodies, which should therefore be avoided, and fruit bodies in prolongation stages (very young specimens) should also be avoided if possible, because their elemental composition may not be representative.

Collected fruit bodies should ideally be brought back to the laboratory in paper bags marked with the sampling date, plot, species and developmental stage. If it is not possible to define the species in the field it can be done at the laboratory. Further, all specimens should be re-examined at the laboratory to correct species identification errors made in the field. The fruit-bodies should then be weighed, sliced and dried to constant weight at 60-70 °C (organic material should not be exposed to higher temperatures to avoid losses). Dried fruit bodies can be stored in bags at room temperature for years.

Mycorrhizal root tip sampling
Molecular approaches are routinely used to study the diversity of fungi in ecosystems (Gardes and Bruns, 1993; Bruns *et al*., 1998) with analyses of the internal transcribed

spacers (ITS) of rDNA being especially popular. Molecular techniques allow species that cannot be grown in culture using current methods to be studied, and the observed diversity of fruit bodies using such techniques can be compared with that of root tips. (To sample ectomycorrhizal root tips, they can be separated from soil cores or samples obtained from areas where root tips of plants of interest are present, using a dissecting microscope). In addition, molecular techniques are useful for taxonomically classifying fungal symbionts to the family level in forests where species fruit infrequently or not at all. Similarly to morphotyping, studying the mycorrhizal community using molecular techniques is challenging and time-consuming. Thus, the size of samples that can be investigated may be highly restricted. Consequently, the wide variance typically found in ectomycorrhizal root tip communities may not be adequately reflected, so the acquired results should be interpreted carefully. It is also important to remember that the root tips should be separated and morphotyped as soon as possible to avoid saprotrophic fungi expanding and growing in stored samples. Morphotyping is also easier when fresh root tips are used.

6.3 Sampling equipment

- Knife
- Paper bags
- Waterproof marker
- Ruler
- Protocol
- Soil corer/spade
- Plastic bags

7. Understorey vegetation

7.1 Description of the compartment

In boreal forests the understorey consists of lichens, mosses, fungi[1] and vascular plants. Three layers of vegetation can be distinguished**,** namely the:

1. bottom layer, consisting of lichens, mosses and fungi,
2. field layer, consisting of herbs, grasses, ferns and dwarf shrubs, and
3. shrub layer, consisting of tree saplings and shrubs.

The composition and abundance of species in the understorey vary from site to site depending on variables affecting growth, such as the light regime, nutrient availability, pH and water content of the soil. In forests, the number of species present, biomass (net primary production, g m^{-2} a^{-1}) and the rate of litter decomposition increase with soil fertility. At any given site vegetation gradually changes over time due to natural succession. Forest management practices can also change the relative abundance of the species present.

Understorey vegetation plays important roles in the annual biomass production and nutrient cycling of boreal forest ecosystem (e.g. Mälkönen 1974, 1977). Nutrients bound in the understorey are returned to the soil with litter and are released through decomposition. Transfer of radionuclides from the soil to plants occurs via the same processes as nutrient uptake by roots, as reviewed by Ehlken and Kirchner (2002), and varies both between radionuclides and according to the soil type and nutrient status of the site.). The uptake of certain radionuclides also varies between plant species. Possible contamination of plant surfaces by soil containing radionuclides and atmospheric deposition also warrants consideration. Depending on the element and its chemical form, such radionuclide contamination may pass, to varying degrees, through the cuticle into plants.

Radionuclides are accumulated to varying degrees, depending on the nuclides, in various parts of vascular plants. Thus, the distribution in plants is not homogeneous and should be considered when sampling and separating different parts of plants for analysis. In addition, radionuclide concentrations in plants or plant parts may either decrease or increase over time. Hence, the variation between radionuclides, sites, plant species and plant parts, as well as temporal changes in their distribution should all be taken into account when sampling plants.

Lichens and mosses differ from vascular plants in that they obtain nutrients **–** and accumulate radionuclides, heavy metals and other pollutants – directly from rainwater and airborne particles absorbed on their surfaces. Thus, atmospheric pollutants accumulate in them, which is why they are often used as bioindicators.

[1] Descriptions and sampling methodology for fungi have been presented in Chapter 6.

7.2 Sampling methodology

The international organizations responsible for overseeing the use and disposal of nuclear materials and setting measurement standards – the International Atomic Energy Agency (IAEA), International Commission on Radiation Units and Measurements (ICRU) and International Organization for Standardization (ISO) – have not, as yet, published recommended methods for sampling forest vegetation to be used for radionuclide analyses. The only recommended sampling method related to radioactivity in terrestrial plants, other than human food, is the method for sampling pasture published by the IAEA (1999) for estimating the contamination of animal feed in nuclear or radiological emergencies, which has limited applicability for sampling forest vegetation.

Lichens, mosses and mushrooms have been suggested by the IAEA to be used as indicators in environmental monitoring of radionuclides during normal discharges from licensed facilities and following emergencies (IAEA, 2005, pages 66 and 69). Sampling is suggested to be carried out once a year, for mushrooms at harvest. No other practical instructions are presented in the report; so the methods used should be decided on a case-by-case basis.

Timing of vegetation sampling

The best time for collecting vegetation samples is when plants are fully developed (usually after the middle of the summer), but before they have begun to senesce.

Sampling design

Sufficient sampling points at the sampling site should be selected to provide reliable estimates of radioactivity over the whole area, and the expected distribution of the radioactivity should be considered when deciding how to distribute the sampling points. If the radioactivity distribution in vegetation is expected to be heterogeneous, then systematic (grid) sampling is recommended. Random sampling should only be used in cases where the distribution is homogeneous. Guidance for designing statistically based sampling approaches for field studies is provided in the report “Sampling for radionuclides in the environment” (ICRU, 2006).

Sampling tools for determining radioactivity in vegetation per ground area

Sampling is carried out by using a frame, which defines the land area covered by the ground vegetation to be sampled. The frame may be square or round and may be made of metal, wood or plastic. Frames used for collecting samples of pasture or ground vegetation have varied in size from 0.06 to 1.5 m^2 (Isaksson, 2000; Rahola *et al.*, 1999; SSI, 1999; Suomela *et al.*, 1999). However, forest ground vegetation is generally far more heterogeneous than pasture, and frames with less than 1 m^2 areas are likely to be too small to provide representative samples. This area can also be formed as the sum of smaller areas. The amount of samples required for radionuclide analysis also depends on the detection limit of the analytical techniques to be used. This should be taken into account when choosing the frame size or the size of (combined) vegetation samples for analysis.

Sampling procedure
The frame should be laid on the ground and above-ground parts of all plants within it should be collected by cutting the plants with scissors, at a height defined according to the aims of the study. For example, it should be near the soil surface (above the litter layer and decaying or brown plant parts, which should be regarded as litter) if the objective is to collect all above-ground biomass for determining the total activity content of vegetation. Below-ground parts of plants are components of soil samples, if they are to be collected at the same sampling point. Care should be taken to avoid touching the plants with scissors or hands during the operation, since soil particles could contaminate the sample, and the operation should be repeated at each sampling point of the sampling site.

If the objective is to determine radionuclide concentrations in Bq/kg or contents in Bq/plant of specific plant species, or groups of species, samples collected by the frame method can be subsequently divided by species (e.g. SSI, 1999; Suomela *et al.*, 1999). Samples (whole plants or certain parts of plants) can also be collected over the whole study area to obtain a representative pooled sample to determine activity concentrations in Bq/kg for the site. In the laboratory, dead parts of plants are usually removed (and analysed if required). Sub-samples can be prepared by separating different parts of plants, according to the aims of the study (e.g. leaves, stems and roots, if below-ground biomass was collected; different parts of plants of varying ages can also be separated). This will provide information on radionuclide distributions in the plants.

Division of the sample defined by the frame
Each sample should be treated (according to the aims of the study) either as a bulk sample consisting of the total above-ground biomass of the ground vegetation, or as sub-samples consisting of specific species or groups of species (in which case all plants in the frame must be collected and classified according to species or group of related species). If species-specific samples are required, it is important to sort them directly in the field to prevent contamination, rather than in the laboratory from a bulk sample. The advantages and disadvantages of sub-samples versus bulk samples are:

- Sub-samples will be smaller than bulk samples and will require longer gamma-ray spectrometric measurement times. Sub-samples may also be too small for radiochemical analysis of certain nuclides (e.g. ^{90}Sr and $^{239, 240}$Pu).
- Total radionuclide contents per unit ground area (in Bq m^{-2}) have to be calculated from summed activity contents of sub-samples, whereas results can be obtained directly from bulk samples, with less uncertainty.
- Sub-samples will provide information on the variation between plant species or species groups, or plant parts, whereas a bulk sample represents a varying mixture of species (which may be either advantageous or disadvantageous, depending on the objectives of the study).

Sampling procedure using a frame:

1. Set the frame on the ground at the selected sampling point. Avoid sampling points with large stones or roots, logs, holes, trails etc. If necessary, change the sampling point according to pre-defined guidelines.

2. Collect all plants inside the frame by cutting the stems with scissors/pruning shears, at a cutting height in accordance with the aims of the study. Avoid contamination by soil particles.
3. Identify all plant species if required (preferably in the field)
4. Place all plants in a bag, if a bulk sample is required, or sort them by species and put each sample in a separate bag if species-specific samples are needed.
5. Record all required information regarding the sample for identification on the outside of the sample bag.
6. Record all information regarding the sampling site required to describe the site.

Making composite samples

Samples from one sampling site can be analysed either as single samples or as a composite sample. Compositing or pooling samples will naturally increase the sample size, which may be essential for subsequent laboratory analysis. If samples are pooled, the number of samples pooled, and their mass (fresh weight) need to be recorded.

Other sampling methods

If the objective is to determine the total radionuclide content of the ground (in Bq m^{-2} or Bq kg^{-1}), soil and ground vegetation need to be collected from the same area to obtain estimates of total activity content at each sampling point. Associated soil and plant samples are also needed to estimate levels of radionuclide uptake from the soil. The root zone of plants of interest should define, in theory, the required depth and horizontal area of soil samples. In practice, uptake levels have also been determined from soil and plant samples representing whole study areas (rather than individual plants), thereby providing estimates of average uptake for the respective areas.

In studies of atmospheric deposition using lichens or mosses as indicators, several sampling methods have been used. A common criterion for selecting sampling points is that they should be located below openings in the tree canopy to avoid collecting samples affected by direct throughfall or stemflow water (ICP Vegetation Coordination Centre, 2005). Sampling points should be covered with lichen or moss, and few other plants, litter, cones etc. (which are removed and not usually analysed) should be present. Bioindicators should be sampled regularly (annually or at constant intervals) using exactly the same methodology on each sampling occasion.

Methods used in the International Cooperative Programme on Effects of Air Pollution on Natural Vegetation and Crops (the ICP Vegetation programme) can be applied for sampling mosses (ICP Vegetation Coordination Centre, 2005). For lichens no international standards or guidance for sampling are available. However**,** frames of various sizes are usually used for sampling them in studies related to deposition. Lichens need to be collected when moist to prevent them crumbling. If the lichen is dry it should be watered before sampling. In some studies (e.g. Paatero *et al.,* 1998; Puhakainen *et al.*, 2007) the soil layer below the lichen carpet has also been sampled. The lichen can also be fractionated into different layers to obtain information on the vertical distribution of radionuclides (in studies of intake of reindeers; see Tuominen and Jaakkola, 1973).

Berries are collected over the whole study area. A representative sample can be obtained, for example by collecting along lines set in W or X shaped patterns across the study area (Environment Agency, 2007, page 66) or along several parallel transect lines. Depending on the aims, it may be useful to collect berries from a known surface area. Any extraneous material should be removed from the sample when it is collected. Results should be reported as Bq kg^{-1} fresh weight, and fresh:dry weight ratios should be provided, regardless of the unit of activity concentration used. Samples should be stored in airtight containers until weighed to prevent changes in moisture content.

7.3 Sampling equipment

- Frame
- Scissors and/or pruning shears
- Paper bags (or plastic bags if samples are to be stored frozen until analysis)
- Waterproof marker
- Labels
- Guidebook for species identification
- (Plastic gloves)
- (Metric measure)
- (Airtight and watertight containers for collecting berries)
- (Knife and wash bottle filled with water for collecting lichen)
- Protocol

8. Overstorey vegetation

8.1 Description of the compartment

The overstorey consists of trees and shrubs, which are not included in the understorey vegetation (see Chapter 7). A tree is a perennial woody plant, usually with a single main stem from which branches extend to form a characteristic crown of foliage, while a shrub often produces new shoots from the ground level. Generally, trees also differ from shrubs by reaching greater heights at maturity, branching at greater distances from the ground, and increasing in size by producing new branches and expanding in girth rather than producing new shoots from ground level. However, both trees and shrubs vary considerably, and they do not have clear distinguishing features.

The basic parts of a tree are its roots, stem (trunk), branches and leaves. Roots absorb water and nutrients from the soil, the stems consist mainly of support and transport tissues (xylem and phloem), and the leaves differ strongly between deciduous trees (which have broad leaves that are shed at the end of each growing season) and evergreen trees (conifers), which have needle-like leaves that are shed after a few years. Thus, evergreens retain green foliage in all seasons.

Foliage analysis provides information on the nutritional status of trees. In addition, the chemical analysis of materials on and in the foliage may, under some circumstances, provide information on the loading of pollutants or radionuclides. Due to the rapid mobility of some elements (i.e. internal translocation) it is essential to know from which part of the tree (e.g. the vertical and horizontal position, and needle age class) foliage samples have been collected before interpreting the results. The sampling time will also affect the results of analysis.

If the objective is to study the distribution of radionuclides in trees or forests, it is essential to know the elemental concentrations in all parts of trees, e.g. their branches, stems, stumps and roots. Biomass determinations are also needed if an objective is to examine their radionuclide contents.

Biomass is considered as living or dead organic material both above-ground and below-ground. Above-ground biomass includes all biomass above the soil surface (stump, stem, bark, branches, seeds and foliage), while below-ground biomass includes all biomass of roots. However, fine roots less than 2 mm in diameter are sometimes excluded because they often cannot be separated from soil organic matter or litter.

The diameter of a tree is the most important feature used for estimating its trunk volume or the biomass of different tree compartments. As a general standard, tree diameter is measured at 1.3 m above ground (Figure 3), referred to as diameter at breast height (dbh) in forestry literature (e.g. Brokaw and Thompson, 2000). The height of small trees can be measured using a measuring rod, while larger trees can be measured using a hypsometer (e.g. a Vertex Laser Hypsometer), and the biomass of tree stands can be

estimated by using appropriate allometric biomass functions (e.g. Marklund, 1988; Zianis *et al.*, 2005; Repola *et al.*, 2007).

The ages of trees can be determined by counting annual rings in their stems, which indicate the diameter growth of the tree each year (annual growth). This should be done by coring in order to avoid destroying the study object. The ages of pine and spruce trees can also be determined by counting branch whorls, but this is a less reliable method than counting the annual rings.

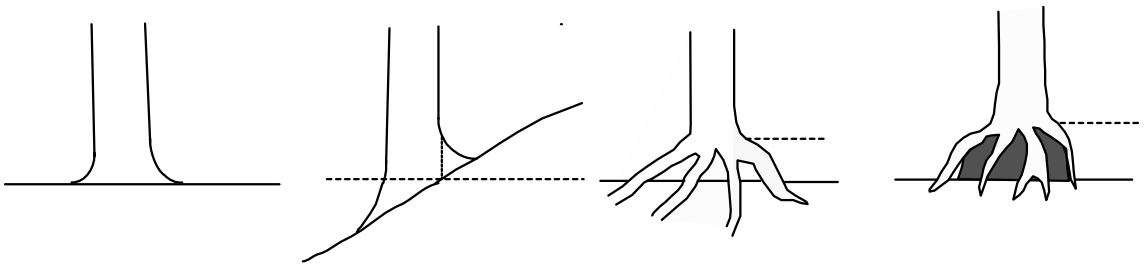

Figure 3. Examples of baselines for determining breast height (1.3 m above ground). The dashed horizontal lines show correct start levels in relation to the birth origins of trees that have grown (from left to right) on a slope, a hummock or a stump that is no longer present at the time of measuring, or a stone). Redrawn from Kangas and Päivinen (2000).

8.2 Sampling methodology

Timing of sampling

Foliage should be sampled regularly, and at the same phenological stage, in monitoring programmes, especially for monitoring changes in environmental radioactivity in forests. Further, during the growing season a large proportion of the foliage mass consists of varying amounts of starch, which complicates data interpretation. Therefore, deciduous species should usually be sampled in late summer (when they have stopped growing but not yet started to senesce), while evergreen species are best sampled in the dormant season (i.e. from October-November to February-March, depending on climatic and geographical conditions). The same considerations should be applied to sampling branches, if they are to be analysed.

Normally foliage samples are collected with branches, then needles and leaves are detached from branches in the laboratory. Samples of other parts of trees can be taken at any time. However, sampling should be avoided during strong growth periods of trees, i.e. during spring and early summer. If the objective is to determine the total radionuclide distribution in a forest, it is advisable to collect all samples at the same time, or at least to collect all samples of overstorey vegetation within as short a time as possible.

Sampling of foliage and branches

The optimal method for sampling foliage and branches depends on the purpose of the study. However, established sampling methodology should be used wherever possible, e.g. the methods used in the Pan-European Forest Condition Monitoring Programme (United Nations Economic Commission for Europe, 2004). As well as helping to ensure that the results will be robust, this facilitates cross-study comparison of the results with time-series of nutrient or pollutant concentrations of tree species growing in similar conditions.

At least 3-5 trees of each main species should be sampled annually in monitoring programmes, but for statistical reasons it is advisable to sample more trees (e.g. ten). Due to heterogeneities in soil properties, sampled trees should be situated close to each other. A composite sample for each tree species should be prepared in the laboratory by mixing equal quantities of foliage from each individual sample, or if the aim of the study is to examine spatial variations in element concentrations in the study area, then each sample tree should be analysed individually.

According to the manual of the United Nations Economic Commission for Europe (2004) the trees selected for sampling should:

- be spread over the total plot area, or around the plot if the stand is homogeneous over a larger area
- belong to the predominant and dominant crown classes (in forests with closed canopy) or have heights within ± 20% of the average height of the forest canopy
- be in the vicinity of locations where other samples were taken (or are to be taken) for analysis
- representative in terms of the mean defoliation level of the plot

Sample trees should be randomly selected from trees fulfilling these criteria.

For monitoring purposes the same trees should be sampled each year, and the trees must therefore be numbered. No trees should be felled for foliar sampling. The sampled leaves or needles should have developed in full light. Usually, the current-year needles of evergreen species are the most convenient for assessing the trees' nutrient status but, for a number of elements, comparing element concentrations in older needles with those in current-year needles may provide more useful results.

The foliage should be sampled from the upper third of the crown, but not from the uppermost (1-4) whorls in conifers (Figure 4). For broad-leaved trees the current-year leaves should be sampled, while at least current-year (c) and previous-year (current+1, c+1) foliage of evergreen species should be sampled (Figure 5). For all species, only mature leaves should be sampled, preferably from south- and west-facing parts of the sample trees. If only one orientation is to be sampled, or foliage from all orientations are represented in samples, it should always be properly documented.

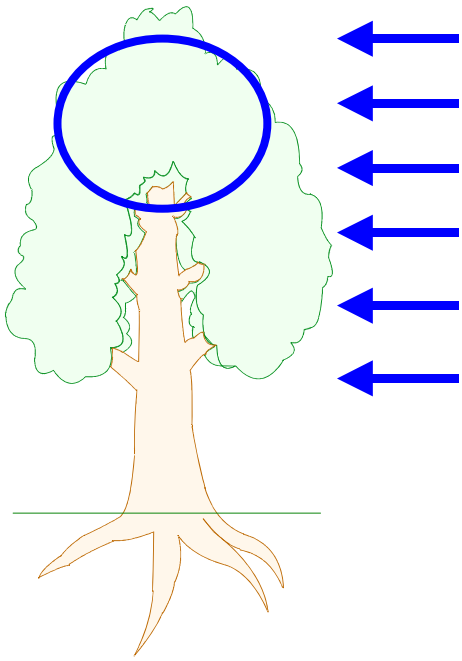

Figure 4. Foliage should generally be sampled from the upper third of the crown (circle). Sampling from other vertical positions (arrowed) may also be justified, depending on the study plan, but if they are the positions should be carefully documented.

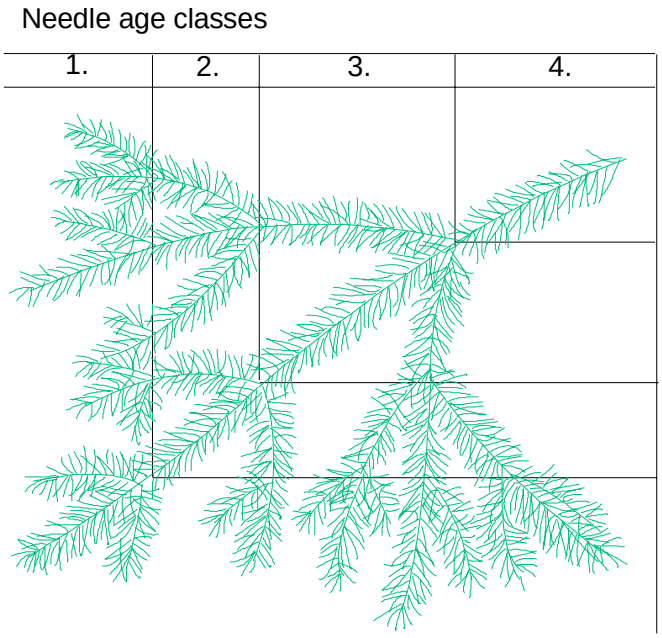

Figure 5. Sketch illustrating age classes of Norway spruce needles (from Salemaa et al.*, 1996).*

The abovementioned recommendations have to be modified when sampling understorey trees or shrubs which do not belong the understorey vegetation or either the pre-dominant or dominant crown classes. Finally, foliage samples can also be taken – and have been taken – in very different ways from those described here. There are no official constraints on using other, non-conventional methods, but any such methods used should be carefully documented when reporting the results. Further, unusual sampling practises may inconvenience comparisons of results with those obtained in other studies.

When studying radionuclide distributions in forests, data on older branches in a range of vertical positions, and data on biomass distributions, are also needed. Sampling for acquiring such data should follow the guidelines for empirical biomass determination, if general allometric biomass functions are not used. Otherwise, branches should be sampled that represent the whole living crown (see Figure 4).

Sampling of other tree fragments

For stem samples 5-7 trees should be sampled in each plot, and the numbers should be increased if the plot area is larger than 1,500-2,000 m^2, depending on the sampling uncertainties deemed to be acceptable. Sampled trees should be randomly selected, but they should also be representative of the stand at the studied site, which may be assured by making random selections within specified size classes, based on the size distribution parameters of the trees obtained from stand measurements. Stem discs can be sawn from trees at fixed heights or relative heights – e.g. at stump or breast height (1.3 m), 6 m, or relative heights of 25%, 50% and 75% of the total stem height (Figure 6), depending on the objective of the study.

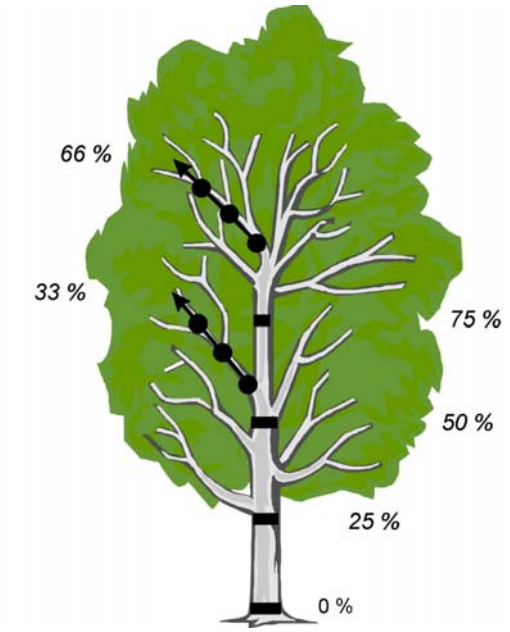

Figure 6. Illustration showing relative sampling heights of a birch crown and stem (Aro and Rantavaara, 2004).

Sampling of timber
In cases where industrial-scale surveillance of radionuclide concentrations in timber is required, following radionuclide contamination of forests, practices described by Rantavaara (1996) can be used. After felling timber for construction material, the origin of loads of logs is generally known and can be documented when they reach the sawmills where they are to be processed. When the contaminated forest region of interest has been defined, loads to be systematically sampled can be selected.

The butt logs are most suitable for sampling because they can be easily identified in the loads of logs, and vertical differences in contamination of stemwood can be assessed after separate analysis of the vertical distribution of the radionuclide concentration in the stems. Samples should be discs a few centimetres (perhaps 2-4 cm) thick, sawn from the upper end of butt logs. The number of logs to be sampled per load should be defined by considering the pre-defined acceptable uncertainty of sampling.

The samples should be analysed for radionuclides after dividing them into bark plus phloem, and wood. The dry matter content in the analysed samples has to be determined. Generally, in radionuclide analyses samples have to be homogenised, but in a few laboratories specialised measuring systems are available that accept solid samples of any geometry (4π measuring geometry) and thus do not require samples to be homogenised. This is advantageous since grinding dried wood samples is time consuming, and dry ashing may cause losses of radioactive caesium.

Sampling biofuel
Biofuel is here considered to consist of logging residues (e.g. branches with or without foliage, top parts of stems and stems which have no use in the forest industry) and stumps that are stored in a forest before being transported to power plants. Sampling methods for logging residues (chips) are described in the standard technical specification CEN/TS 14778-1 (European Committee for Standardization, 2005). Stacks of logging residues can be sampled before making chips from them using a frame and chain saw. Alternatively, either systematically or randomly located area sub-plots can be used. Stem, bark, branches of different sizes and foliage can be separated from logging residues before analysis if required.

8.3 Sampling equipment

There is no need to give detailed guidelines regarding sampling devices. Any sampling technique can be used, provided it is suitable for the planned sampling and minimises the possibility of sample contamination. However, needle/leaf and branch samples of living trees are usually collected using branch cutters that can be extended up to 18 m (Figure 7), and chain saws are usually used to sample stems (generally by cutting discs), stumps and branches (of trees that have been cut down). Phloem and bark are separated in the laboratory if stem wood is not analysed as bark over stem. Generally, radionuclide concentrations in wood and bark are recommended to be analysed separately, especially for deposition-related measurements, since the activity concentrations of bark may be orders of magnitude higher than those of stem wood. In such cases it is also highly important to avoid contamination between sub-

compartments. Roots can be sampled with a suitable soil auger or dug up, with a shovel for example. If the objective is to determine radionuclide contents in roots per unit area, volumetric or area-based samples should be collected using a soil auger of known diameter. As with bark, the activity concentrations of the bottom layer after a deposition event may be orders of magnitude higher than those of roots or soil. In such cases vertical sampling must be done layer by layer with clean equipment for each layer.

Figure 7. An example of an extendable branch cutter used for sampling foliage (Photos: J. Ilomäki / Metla).

8.4 Additional comments

Analyses of both live material (to determine foliage chemical contents) and dead material (shed foliage, to determine litter chemical contents, see Chapter 9) are important for assessing nutrient and radionuclide fluxes and the nutritional status of forest trees.

Contamination by the soil surface of all samples of overstorey vegetation should be avoided, e.g. by spreading a plastic cover over the soil surface. Special care is needed when handling tree parts that are known to have the highest concentrations of radionuclides, i.e. the youngest shoots of trees and bark, to avoid cross contamination of samples.

The required quantities of sampled material strongly depend on the pre-treatment of the samples and eventual analyses, and should be estimated before starting sampling. Larger samples should be taken if samples are to be archived for further analyses. The mass of 100 leaves or 1,000 needles should be determined using sub-samples dried at 105 ˚C.

9. Deposition and litterfall

9.1 Description of deposition and litterfall

Deposition and litterfall are important input factors when studying element or radionuclide cycling in forests. Elements may be deposited from the atmosphere via wet or dry deposition. Wet deposition refers to the washing out of particles and ions from the air by precipitation and is not influenced by surface conditions. The dry deposition rate, on the other hand, depends to a great extent on the surface roughness, which in turn depends on canopy height. Deposition collected from below a forest canopy is called **throughfall**, while deposition draining down a trunk is called **stemflow**. The composition and amount of throughfall and stemflow differ from that of deposition collected above the canopy or in open areas because tree canopies intercept some of the dry deposition and modify the chemical and radionuclide composition of the incoming wet deposition.

Several methods can be used to measure or estimate deposition in forests. Throughfall measurements are relatively simple and cheap in most circumstances. In addition to throughfall, bulk deposition should be measured. Bulk deposition refers to all deposition via precipitation (i.e. wet deposition plus, to varying degrees, dry deposition) and can be readily collected in continuously exposed funnels placed in an open field.

Litterfall is a key link in the biogeochemical cycle, between the tree components of the biosphere and both water (hydrosphere) and soil (pedosphere) components. Knowledge of both the biomass of the litter and its chemical contents (including heavy metals and radionuclides) are needed to quantify the annual return of elements and organic matter via litterfall to the soil. Litter decomposition is a major pathway of nutrient and radionuclide fluxes, and a major contributor to forest soils' organic matter inputs.

9.2 Sampling methodology for throughfall

Type and number of deposition collectors and their siting

Several types of collectors (e.g. funnels, such as the one shown in Figure 8) can be used for monitoring deposition. The collector and its containers, tubes, glue etc. should be made from materials that will not affect the sample solution. Polyethylene equipment, which is acid-washed in the laboratory before use, is recommended for studies of macro-ions and heavy metals (United Nations Economic Commission for Europe, 2004), and similarly for many radionuclides. See Chapter 2.5 for information on containers and the risk of losses of radionuclides during storage of water samples.

In most cases funnels of 20 cm diameter should collect sufficiently large samples. The collection bottle should also be sufficiently large to contain the largest amounts of precipitation expected during the sampling period at the sampling location (i.e. 2-5 litres). In areas where the precipitation is generally low and the sampling frequency high, use of funnels of larger diameter is recommended (United Nations Economic

Commission for Europe, 2004). The throughfall collectors should be placed at a height of approximately 1 m above the ground level to avoid contamination by soil. The sampling bottles should be kept cool and in the dark, e.g. in a pithole.

Figure 8. An example of a throughfall collector with a 20 cm diameter (Photo: A. Ryynänen/Metla).

Samples obtained from a throughfall collector correspond only to the small area covered by the collector. Thus, to take into account the generally large local variations in throughfall deposition in forest stands, sufficient numbers of collectors must be used. As a guideline, 10 collectors or more are needed to cover the variability in deposition over a 30 x 30 m sampling plot (United Nations Economic Commission for Europe, 2004).

It is recommended to carry out a pilot study of the variation within the plot, using a large number of collectors, before the final sampling strategy is selected to obtain guidance regarding the size and number of collectors required. To minimize the analyses only one variable, e.g. conductivity or potassium contents, needs to be analysed in such a pilot study. A criterion for deciding the appropriate number of collectors is that it should be possible to measure all major ions across the plot to within ±20% (United Nations Economic Commission for Europe, 2004).

The collectors should be placed in such a way that they provide a representative measure of the total deposition in the forest stand, and sited around selected trees (tree-wise) or either randomly or systematically in the plot (plot-wise) (see e.g. Starr *et al.*, 2007). The exact locations, places and height above the ground, of the collectors within the plot should be recorded along with other data concerning the study site (United Nations Economic Commission for Europe, 2004).

For many tree species stemflow is limited, but in others it can constitute a substantial part of the precipitation reaching the ground. Since trunks and branches are heavily

exposed to dry deposition, the element concentration of stemflow may be high, and even if it only accounts for a small proportion of precipitation it may carry a substantial proportion of the total element deposition.

Stemflow collectors usually consist of cut plastic tubing wound, spirally around a trunk, leading to a collection bottle. The tubing is fixed to the trunk by nails and leakage is prevented by placing glue between it and the bark. As a guideline at least 10 stemflow collectors are needed per plot.

Sampling
Sampling should be carried out monthly, weekly or at some other suitable time interval for the prevailing climate and method used. However, evaporation and growth of algae in the sample containers should be avoided, so sampling periods should not be excessively long. The risk of sample losses due to contamination should also be considered. The frequency at which containers are emptied should be the same for all deposition measurements (throughfall, stemflow and bulk deposition). More information is available in (*inter alia*) the ICP Forests manual (United Nations Economic Commission for Europe, 2004).

9.3 Sampling methodology for litterfall

Figure 9. An example of a litterfall collector with an upper diameter of 80 cm (Photo: A. Ryynänen/Metla)

Type and number of litterfall collectors and their siting
Many types of collectors can be used for monitoring litterfall (one of which is shown in Figure 9). It is recommended that the collectors should be fixed sufficiently high above the ground to ensure water drainage, and their opening areas must be horizontal. Litterfall is sampled in litter bags attached to a frame. The sampling area of each collector must be sufficiently large to determine both the amount and quality of the

litter, preferably 0.25 m^2 at least, and for tree species with large leaves the collecting area of samplers must be increased (e.g. up to 0.5 m^2).

The fixed litterfall collectors may be placed randomly or systematically (e.g. at regular intervals) and in sufficient numbers to represent the whole plot, rather than just the dominant tree species. Since litterfall is not a tree parameter, litterfall collectors should be distributed all over the study area. It is recommended to sample litterfall from at least 10 collectors per plot and even up to 20-30 collectors, depending on the size of the plot and tree species involved in the assessment (e.g. leaves from deciduous species are more susceptible to turbulent air movement than conifer needles). More information is available e.g. in the ICP Forest manual (United Nations Economic Commission for Europe, 2004).

Sampling

It is recommended to empty litterfall bags at least monthly, to avoid pre-collection drainage and decomposition of the collected litter due to long residence times in the collectors. The samples may be pooled to obtain periodic samples once the monthly variations in amount and quality of litter have been investigated.

The bags must be carefully labelled before sampling with relevant information on the study site, species, sample type, collector number, and date of collection. If the bag is attached to the bottom of the collector the sampling bag can be conveniently replaced at each plot visit. The samples should be transferred immediately to the laboratory where they should be dried and stored before dividing them into appropriate fractions, according to the aims of the study.

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APPENDIX A: Examples of aerial sampling

Detailed information on sampling strategies that can be used can be found in IAEA (2004), however the main types of strategies are as follows.

Judgemental sampling is subjective, and based on historical knowledge of the sampling site in question, visual inspection of the site and professional judgement.

Random sampling refers to strategies in which each sampling point within the sampling site is arbitrarily selected. No point has a higher probability of being selected than any other point, and all points are selected independently of the others. This strategy is well suited for sampling areas that are suspected to be homogeneously contaminated. Figure A1 illustrates this sampling approach.

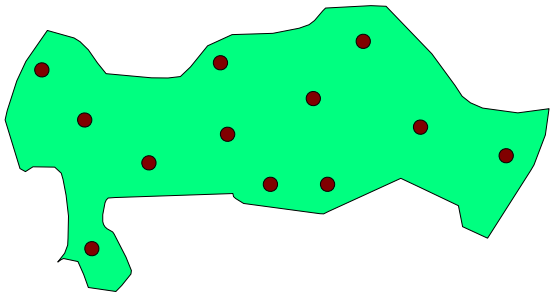

Figure A1. Random sampling

Stratified random sampling refers to cases in which the area is divided into smaller areas called strata, each of which is more homogeneous than the area as a whole. The division should be based on available historical and screening data from the site. Typical strata may be depth or contamination level. The sample locations are randomly selected within each stratum. This kind of sampling design is useful when estimating radionuclide concentrations within specific depth intervals or areas of concern.

Systematic grid sampling refers to cases in which the area is divided into smaller areas (usually squares or triangles) and samples are collected from the nodes. The grid is placed in the terrain by selecting a random point and extending the grid from that point. The size of each square or triangle is determined by the area of the sampling site and the number of samples needed. When the aim of the sampling is to assess the contamination levels and concentration gradients in an area, systematic grid sampling is the most appropriate strategy. Figure A2 illustrates a systematic sampling grid. When elevated concentrations are detected at one node, further samples should be collected from adjacent squares or triangles to define the area of contamination.

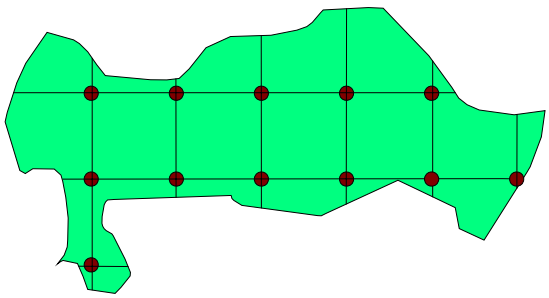

Figure A2. Systematic grid sampling

In **random grid sampling** (or systematic random sampling) the same kind of grid as described above is used, but the samples are collected at a random location within each square or triangle. This is a useful approach when the aim is to estimate the average concentration within each cell in the grid. Figure A3 illustrates a random sampling grid.

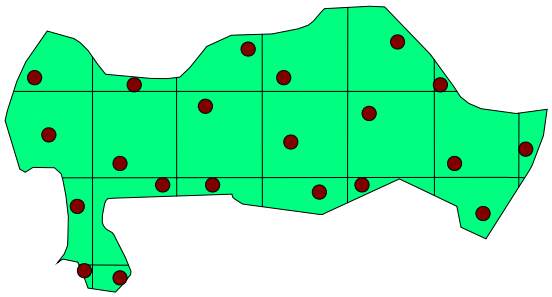

Figure A3. Random grid sampling

When the purpose of sampling is to locate hot spots, either of these two grid sampling approaches can be used, and the grid spacing should be determined by the expected size of the hot spots. The spacing must be sufficiently narrow to detect small hot spots, but can be larger when the hot spots are expected to cover larger areas.

Transect sampling involves the establishment of one or several transect lines across the whole sampling site. The lines may be parallel, but not necessarily. Samples are collected at regular intervals along the lines. If several lines are established, this design will approach a systematic grid sampling design. Transect sampling approaches are used to obtain indications of concentrations of contaminants and to define concentration gradients. Figure A4 illustrates transect sampling.

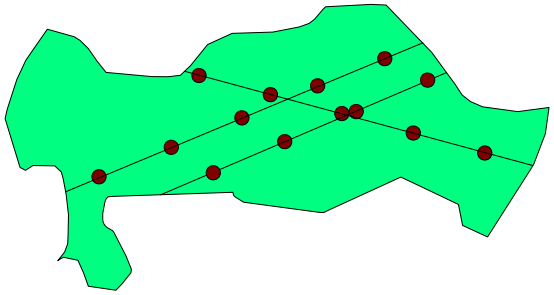

Figure A4. Transect sampling

APPENDIX B: Examples of sampling related to radioactivity studies in forests

B 1. Inventories and distributions of radionuclides

Sampling campaigns are often undertaken, and required, to study short and/or long term radionuclide transfer processes and mechanisms following a contaminating event.

A regular sampling campaign may be a part of a more spatially and/or temporally extensive surveillance programme. To obtain a complete radionuclide inventory of a forest is a laborious task, demanding the collection of several hundred samples (e.g. SSI, 1999). However, the required accuracy of the results (and the numbers of samples needed) may vary both between studies and between radionuclides. Therefore, critical evaluation of the data required in a study will save time and resources during the course of the investigation. Some of the processes that influence the redistribution of radioactive nuclides in the forest ecosystem are outlined below.

Radioactive nuclides are deposited in a forest ecosystem either by **dry** or **wet deposition** and are mainly transferred from the vegetation to the forest floor by **leaching** (throughfall) and **litterfall**. In the short term after primary deposition of radionuclides the particles intercepted by the canopy may gradually settle to the forest floor or other vegetation surfaces. Some nuclides may even be **taken up directly** through the leaves. In trees and other plants internal nutrient cycling transfers radionuclides between different plant parts (**translocation**). When plants are **grazed** by animals, radionuclides are transferred to the animals. Litter containing radionuclides must be **decomposed** by microorganisms before the radionuclides are available for uptake again. Animals living in the soil that dig or burrow play important roles in mixing the soil (**bioturbation**) and translocating organic material containing radioactive nuclides from one horizon up or down to another. Positively charged nuclides are attracted to negatively charged surfaces of minerals and humus where they are held in exchangeable form, available for uptake, but partially protected from leaching. Some nuclides of appropriate size may be trapped within cavities in the crystal structures of certain clays. **Weathering**, physical and chemical breakdown, of clay particles will make the nuclides available for uptake and leaching again at sites where clays occur in the root zone. Radionuclides can be lost to groundwater and nearby streams, where they are transported to lakes or out to the sea (see Figure B1).

The processes and mechanisms related to the redistribution of radionuclides in forests in the early phase (in the first weeks, months and first year after contamination) are not well known in quantitative terms. However, sampling for scientific purposes, rather than simply for monitoring, should provide valuable new information for filling gaps in our knowledge regarding radionuclide dynamics (i.e. time-dependent changes in post-incident concentrations). The data acquired from such analyses should help to improve predictive models developed for assessing radionuclide dynamics and radiation exposure.

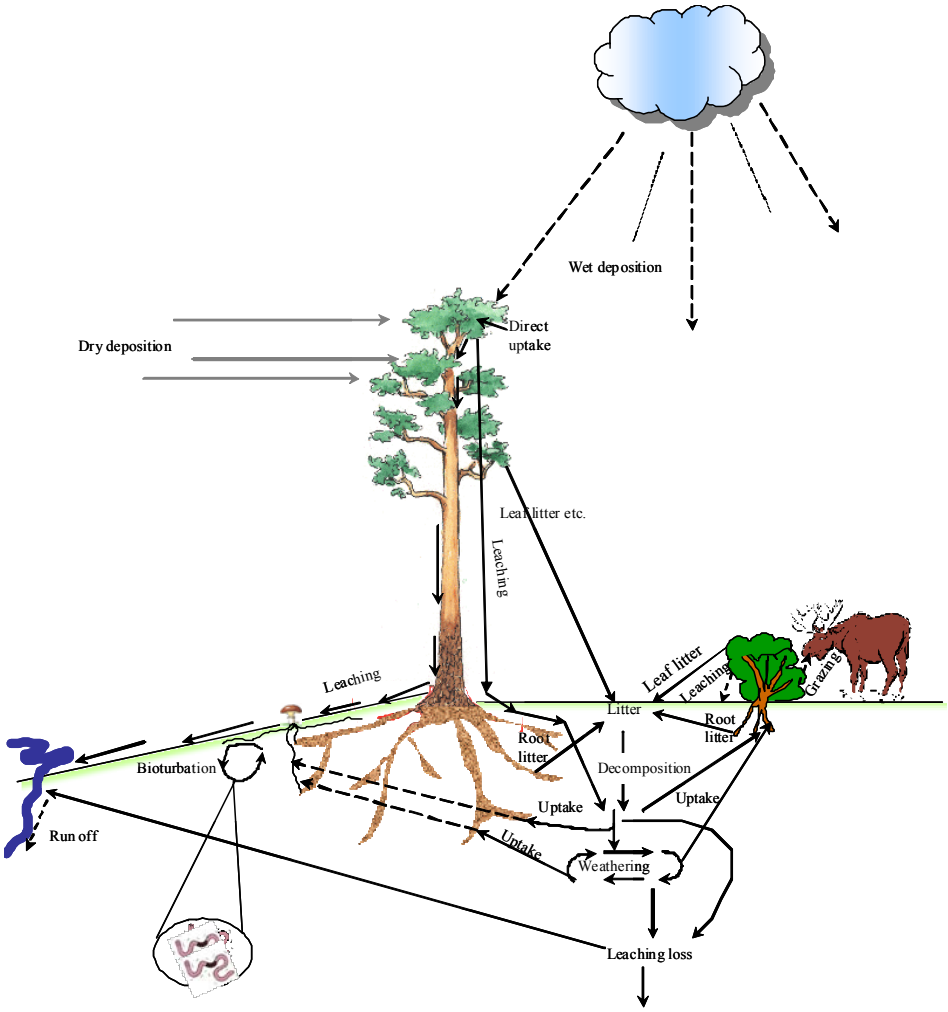

Figure B1. Radioactive nuclide cycling, showing some of the pathways involved in cycling radionuclides through the atmosphere-soil-plant-animal system.

B 2. Human exposure

Immediately after an accident, time is critical and it will not therefore be advisable to use time-consuming procedures for dose assessments. However, assessments must provide reliable results to avoid unnecessary waste of forest products or abandonment of sites that have not been contaminated, or use of products or sites that are more heavily contaminated than estimated, thus leading to exposure of the public occupying the site and/or consuming forest products.

Humans may be exposed in several ways through their use of forests, e.g. through outdoor activities, ingestion of forest produce, and/or the use of firewood, timber or associated products. People that may be potentially exposed will therefore include large proportions of the population, from forest and production workers to consumers of game meat, berries and mushrooms, and members of the public using the forests for recreation

(Shaw *et al.*, 2001). The exposure pathways will vary amongst different groups of the population, hence the data that needs to be collected for dose calculations will also vary.

B 3. Exposure of biota

There are currently no international standards or criteria that specifically address issues related to the protection of biota from ionising radiation (Zinger *et al.*, 2007). However, several international programmes have recently been carried out with the aim to develop a system for estimating radiation doses to biota (Larsson *et al.,* 2004; Brown *et al.*, 2003; Beresford *et al.,* 2007). They can now be estimated, for instance, using a software package developed in the ERICA project (available at www.erica-project.org). The selected reference organisms for forest environments used in Erica can be found in Beresford *et al.* (2007).

A basis for developing approaches to enable regulatory assessments is currently being formulated in an EU project called PROTECT. Deliverable 3 for this project includes a review of approaches for protecting the environment from chemicals and ionising radiation (Hingston *et al.,* 2007).

The international organisations International Atomic Energy Agency (IAEA), International Commission on Radiological Protection (ICRP) and United Nations Scientific Committee on the Effects of Atomic Radiation (UNSCEAR) have also addressed issues regarding the protection of biota (e.g. IAEA, 2003; ICRP, 2003; UNSCEAR, 1996).

B 4. Monitoring radionuclide contamination

The general objective of monitoring radiation is to ensure the protection of the public and the environment from exposure to radiation (IAEA, 2005). Monitoring is carried out, for instance, to control discharges of individual radionuclides from nuclear facilities and assessments of baseline levels of environmental contamination during the normal operation of nuclear power plants and other facilities involved in the nuclear fuel cycle. Areas contaminated by past activities are monitored to protect the public and for planning and surveillance of restoration projects. Monitoring is also required in connection with disposal of radioactive waste. The aims of radiation monitoring have been described in a report published by the IAEA (2005).

Sampling campaigns in forests may be parts of a regular monitoring programme of atmospheric releases from the nuclear industry. In Finland, for example, such monitoring includes analysis of radionuclides in samples of soil, wild berries, mushrooms and selected bioindicators (hair moss, reindeer lichen and pine needles), using sampling methods described by Ilus *et al*. (2008).

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Abstract

The NKS project FOREST was established to prepare a guide for sampling in forest ecosystems for radionuclide analysis. The aim of this guide is to improve the reliability of datasets generated in future studies by promoting the use of consistent, recommended practices, thorough documentation of field sampling regimes and robust preparation of samples from the forest ecosystem.

The guide covers general aims of sampling, the description of major compartments of the forest ecosystem and outlines key factors to consider when planning sampling campaigns for radioecological field studies in forests. Recommended and known sampling methods for various sample types are also compiled and presented. The guide focuses on sampling practices that are applicable in various types of boreal forests, robust descriptions of sampling sites, and documentation of the origin and details of individual samples.

The guide is intended for scientists, students, forestry experts and technicians who appreciate the need to use sound sampling procedures in forest radioecological projects. The guide will hopefully encourage readers to participate in field studies and sampling campaigns, using robust techniques, thereby fostering competence in sampling.

Key words Forest, sampling, radioactivity, boreal

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