

Geologian tutkimuskeskus
Opas 47

Geological Survey of Finland
Guide 47

FOREGS GEOCHEMICAL MAPPING FIELD MANUAL

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FOREGS (Forum of European Geological Surveys) Geochemical Baseline Programme has been initiated to provide high quality environmental geochemical baseline data for Europe. The data will be based on samples of stream water, stream sediment, floodplain sediment, soil, and humus collected all over Europe. High quality and consistency of the obtained data are ensured by using standardised sampling methods and by treating and analysing all samples in the same laboratories.

This programme represents the European contribution to the International Union of Geological Sciences (IUGS)/ International Association of Geochemistry and Cosmochemistry (IAGC) Working Group on Global Geochemical Baselines. The sampling instructions are based on the recommendations of the IGCP 259 and IGCP 360 programmes.

The present report contains comprehensive instructions for sample site selection and sample collection. In addition, a summary of methods intended for sample preparation and analysis is included.

Key words: (GeoRef Thesaurus, AGI): geochemical surveys, baseline studies, stream water, stream sediments, floodplains, overbank sediments, soils, humus, sampling, sample preparation, FOREGS, manuals, Europe

Authors' addresses can be found in the list of members (p. 6).

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Euroopan geologisten tutkimuslaitosten yhteistyöelimen FOREGSin (Forum of European Geological Surveys) geokemiallinen kartoitusohjelma tuottaa korkeatasoista ympäristögeokemiallista tietoa alkuaineiden pitoisuuksien perustasosta koko Euroopasta. Näyttemateriaaleina käytetään purovettä, purosedimenttiä, tulvasedimenttiä, maaperää ja humusta. Kerättävän tiedon laatu ja yhtenäisyys varmistetaan käyttämällä standardoituja näytteenottomenetelmiä sekä esikäsittämällä ja analyoimalla kukin näytetyyppi samassa laboratoriossa.

Tämä kartoitusohjelma on Euroopan FOREGS-maiden osuus kansainväliseen geokemialliseen kartoitusohjelmaan (International Union of Geological Sciences / International Association of Geochemistry and Cosmochemistry Working Group on Global Geochemical Baselines). Näytteenotto-ohjeet perustuvat kansainvälisen geologisen korrelaatiohjelman hankkeiden IGCP 259 (International geochemical mapping) ja IGCP 360 (Global geochemical baselines) suosituksiin.

Opas antaa yksityiskohtaiset ohjeet näytepaikkojen valintaan ja näytteiden keräämiseen. Lopussa on yhteenveto suunnitelluista esikäsittelymenetelmistä.

Avainsanat (Fingeo-sanasto, GTK): geokemialliset tutkimukset, pitoisuuksien perustaso, purovesi, purosedimentit, tulvatasanteet, tulvasedimentit, maaperä, humus, näytteenotto, näytteen-käsittely, FOREGS, käsikirjat, Eurooppa.

Kirjoittajien osoitteet ovat osassa list of members (s. 6).

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Preface

Research conducted since 1988 as part of International Geochemical Mapping project (IGCP 259) indicated that although there are several national and regional datasets describing the geochemistry of the Earth's surface they are incomplete and inconsistent between different national surveys. It has become apparent that the need for a consistent and internationally comparable geochemical dataset is urgent, for example not only for scientists but also for policy makers, especially in the area of contaminated land. It is vital to have as detailed an understanding as possible of background variation in the natural system before defining limits for contamination.

The International Geological Correlation Programme (IGCP) project 259 recommended the collection of a global reference set of materials, which should be collected following predefined, systematic methods, and which should also be consistently analysed in designated laboratories. In this way, a global geochemical database based on a global reference network (GRN) could be created, to provide a reference against which existing national and regional geochemical datasets could be compared. These recommendations are presented in *A Global Geochemical Database for Environmental and Resource Management* by Darnley and others, 1995.

The recommendations were endorsed by the UN Committee on Natural Resources which issued Resolution 3/5 recognizing the urgent need for a global land monitoring programme. The resolution endorsed the need to establish a global geochemical database and recommended that the Global Geochemical Baselines Project forms the core of the programme.

The findings of the IGCP 259 were also reinforced by the findings of the FOREGS Geochemistry Task Force, which was established in 1994 by the Directors of the main European Geological Surveys in or-

der to make an inventory of geochemical databases in European countries. The Group identified 120 separate geochemical databases, but found that the materials collected, the sampling methods and analytical techniques used varied widely so that the GRN was needed urgently to extend across Europe, (Plant et al, 1996).

The FOREGS Directors agreed that there was a need for a European geochemical baseline and decided that the way forward was to integrate this project with the ongoing Global Geochemical Baselines project, (IGCP 360), an international project which aimed to realize the recommendations of IGCP 259. The FOREGS Geochemistry Task Force therefore became the European subcommittee within IGCP 360.

Currently 25 European countries have participated in the work of the FOREGS Geochemistry Task Force. At recent meetings in Aveiro, Portugal and Limbach, Slovak Republic the main sampling and analytical strategies were discussed and accepted, resulting in the definition of a set of sampling protocols. Further refinements at the final IGCP 360 meeting in Vail, Colorado have been incorporated into this document, producing a Field Manual for the collection of a set of reference materials with the aim of producing a standardized geochemical dataset.

The collaboration of professional geochemists which has resulted in this field manual has been widespread both in terms of geographical location and experience. Most of the contributors have been acknowledged as co-authors, but there are many others who have contributed to the preparation of this document. This process has been extremely successful and an active network of European geochemists has developed as a direct result, with a biennially rotating leadership.

Espoo, January 31, 1998

Reijo Salminen

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1. INTRODUCTION

1.1 FOREGS Geochemical Baseline Programme (FGBP)

This programme has been initiated to provide high quality environmental geochemical baseline data for Europe. The data will be based on samples of stream water, stream sediment, floodplain sediment, soil, and humus collected from all over Europe. High quality and consistency of the obtained data are ensured by using standardised sampling methods and by treating and analysing all samples in the same laboratories.

The FGBP is authorised by the directors of the Geological Surveys within FOREGS (Forum of European Geological Surveys). The FGBP also represents the European contribution to the International Union of Geological Sciences (IUGS)/ International Association of Geochemistry and Cosmochemistry (IAGC) Working Group on Global Geochemical Baselines.

The present report contains comprehensive instructions for sample site selection and sample collection. In addition, a summary of methods intended for sample preparation and analysis is included. Detailed descriptions of analytical methods used in FGBP will appear in a separate report.

1.2 The need for environmental geochemical baseline data in Europe.

Data on geochemical baselines are urgently needed in Europe, because environmental authorities are defining limits for levels of contaminants in soils used for different purposes in most European countries. As geochemists know, the natural concentrations of elements are different in the various constituents of overburden, and vary markedly between geologically disparate areas. State authorities, however, are not aware of these significant natural variations, which should be taken into account in defining action limits. There are already examples of action limits that are lower than natural concentrations.

Geochemical mapping produces data, which should be used as one of the major criteria for defining action limits. Existing geochemical data in Europe are not in a form that can be readily used for this purpose (Plant *et al.* 1996, 1997). It is not, therefore, possible, on the basis of existing geochemical data, to define the European Geochemical Baseline even for a single element.

The need for geochemical baseline data in Europe has been stated very clearly by Plant *et al.* (1996) in an inventory of existing geochemical data in

FOREGS countries. The following is an extract from that report.

“Throughout Europe public concern about the environment is growing. In response, national governments and the European Union (EU) are attempting to develop policies, legislation and infrastructure, such as the European Environment Agency (EEA). Attempts are also being made to establish ‘Safe Levels’ of Potentially Harmful Elements and Species (PHES), but these are often based on limited and/or inadequate information. The available data on environmental geochemical baselines and radioactivity are not systematic in coverage or quality and, therefore, are not of the standard required to quantify the distribution of PHES at the European scale as a basis for policy-making and monitoring future change to the environment.

At the present time, knowledge of the geochemistry of the surface environment of Europe is based on different surveys of variable standards carried out by different organisations in the public and private sectors. Whilst there are exceptions, Geological Surveys have, in the past, provided data on rock and stream sediments; Soil Surveys on soils; Hydrological Surveys on ground and surface water and biologists/agriculturists on plant and animal tissue samples.

In general, there is a failure to recognise that the natural geochemical background is highly variable and the natural levels of PHES (such as As, Cd, Pb, NO₃⁻, the radioactive elements and organic pollutants) can be as high or higher than those caused by man-made sources of pollution. Even where synthetic pollutants are concerned, it is the natural geology and geochemistry which frequently exert the fundamental controls on the distribution of the PHES and consequently determine their potential to create hazards.”

Systematic baseline environmental geochemical data are necessary to inform policy makers and provide a sound basis for legislation. According to Plant and others (1996), for this purpose such data are required to be

1. Standardized across national boundaries.
2. Available in digital form for use on GIS so that they can be viewed interactively with other datasets, such as those for landuse and for animal and human morbidity and mortality data.
3. Comprehensive, to include the majority of PHES and ideally as many harmful chemical species as possible, including synthetic compounds.
4. Based on a full suite of sample types including soil, stream sediment, surface water, groundwater and offshore marine and estuarine sediment in the coastal zone.

The report by Plant *et al.* (1996) also gives reasons why Geological Surveys or equivalent governmental institutions are particularly well suited to provide the data needed to establish systematic environmental geochemical baseline databases for Europe.

In Europe, the results of the Global Terrestrial Network (GTN, also called the Global Reference Network) sampling, as recommended by IGCP 259 and 360 projects (Darnley *et al.* 1995), will be used as reference material to normalise baseline datasets of Europe. The final product will then be geochemical baseline datasets of Europe.

The sampling will be carried out in FOREGS countries by the end of September 1998. The FOREGS geochemical sampling forms the European contribution to the IGCP 360 and the International Union of Geological Sciences (IUGS)/ International Association of Geochemistry and Cosmochemistry (IAGC) Working Group on Global Geochemical Baselines. The work is coordinated by the FOREGS Geochemistry Task Force. FOREGS directors have stipulated that the whole project, including analyses and reporting, must be completed by September 2001.

1.3 Sampling media

The choice of sampling media has been made in accordance with the recommendations of the IUGS working group on Global Geochemical Baselines (Darnley *et al.* 1995). These media, described below, are considered to be the most representative of the surface environment, and they are the most commonly used media in previous and present environmental geochemical investigations. Sampling sites are chosen at random, so that the data for each of the chosen media will document the actual situation within Europe.

- Stream water (filtered and unfiltered)
- Stream sediment (mineral sediment, <0.150 mm)
- Residual soil, upper horizon (topsoil) 0 - 25 cm with out the top organic layer (<2 mm)
- Residual soil, lower (C) horizon (subsoil); a 25 cm layer within a depth range of 50 cm - 200 cm (<2 mm)
- Humus where present
- Overbank sediment, upper horizon 0-25 cm (<0.150 mm, optional)
- Overbank sediment, bottom layer (<0.150 mm, optional)
- Floodplain sediment, upper horizon 0 - 25 cm (<2 mm)
- Floodplain sediment, bottom layer (<2 mm, optional)

Stream sediment (<0.15 mm) and floodplain sediment (<2 mm) samples are collected because they reflect the average geogenic composition of a catchment basin. Most Geological Surveys have undertaken national stream sediment studies. The new data

are necessary to link these surveys to a baseline level.

Stream water reflects the interplay between geosphere/hydrosphere and pollution. At the same time it is the main source of drinking water. Many surveys have completed local studies, so the GTN data can be used to link results across Europe.

Soil samples (topsoil 0-25 cm) and subsoil (a 25 cm layer within a depth range of 50 cm - 200 cm) are taken to reflect variations in geogenic composition of the uppermost layers of the Earth's crust. For this purpose it is important to avoid soil sampling at locations that have visible or known contamination. Priority for site selection should be given to

1. forested and unused lands
2. greenland and pastures, and
3. agricultural land.

Comparison with topsoil and subsoil samples will give information about enrichment or depletion processes between the layers. One such process is anthropogenic contamination of the top layer. The <2 mm fraction will be taken according to environmental standards. The <0.18 mm and finer fractions have been widely used in mineral exploration programmes and the GTN data will be used to create a link between environmental and mineral exploration databases.

Humus samples can be used to determine the atmospheric (anthropogenic) input of elements to the ecosystem. To reach this aim samples should be collected in forested areas as near as possible to the other sampling sites. To reflect the atmospheric input, the uppermost few centimetres of the organic layer should be collected immediately under the green vegetation and under the litter (max 3 cm).

The sampling locations are totally randomized and are not designed to show the lowest natural background concentrations in the European environment, but to demonstrate the current geochemistry of the surface environment.

1.4 Organisation and time frame

The sampling will be carried out by the institutions of the FOREGS countries listed in pages 4 to 5, and be completed by the end of September 1998. The whole project, including analyses and reporting, must be completed by September 2001, according to the decision of the FOREGS Directors.

SAMPLING COORDINATOR: The Geological Survey of Finland (GSF) will be the sampling coordinator. GSF will provide detailed sampling instructions (also on web pages), send all maps of random sampling sites within GTN cells, check all the sampling plans and give further information whenever needed.

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2. SELECTION OF SAMPLE SITES

2.1 GTN grid cells

For the purpose of Global Geochemical Baseline the entire land surface of the Earth is divided into 160 km x 160 km cells (Darnley *et al.* 1995). The cells have their origin on the equator at the 0° (Greenwich) meridian. European cells (Fig. 1) have identifiers like N36W01, which is the 36th cell North of the equator and the 1st cell West of the 0° (Greenwich) meridian. The coordinates of the cells will be provided by the Geochemistry Task Force coordinator. They are also available from the web-pages of the GSF (<http://www.gsf.fi>).

Some cells are located in more than one country. In these cases the sampling of that particular cell is coordinated by the country (organisation) in which the centre of the cell is located. The coordinating countries are indicated in the GTN cell list.

Participating surveys should not carry out any sampling on the territory of any other country without agreement with the organisation responsible for sampling in that territory. If one or more of the randomly selected catchments are located in the neighboring country, please contact your neighbor Survey and agree on sampling of those sites.

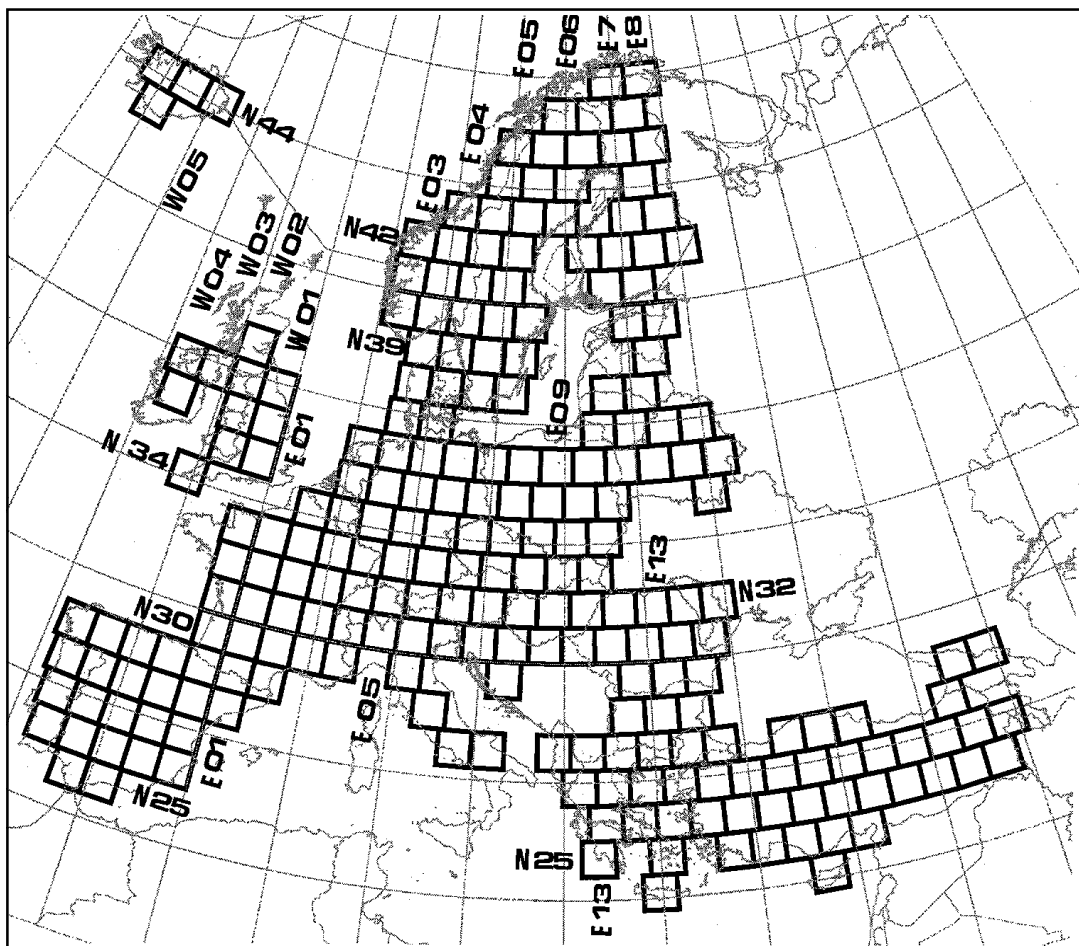


Fig. 1. Global Terrestrial Network (GTN) cells in the FOREGS countries.

2.2 Selecting sample sites

1. From the GTN cell list, find the cells which are coordinated by your country (organisation). The identifier of the GTN cell is for example N43E09. The grid cells are shown on Fig. 1. It is noted that there are more cells in Fig. 1 than in the GTN cell list (file). Some of the cells which are shown in Fig. 1 consist mostly of water area, and only those grid cells mentioned in the GTN cell list (file) should be sampled. However, you are allowed to take samples from some of the cells which are shown on the map but not listed. If you want to sample other GTN cells, please send the list of additional cell identifiers to Timo Tarvainen (Timo.Tarvainen@gsf.fi, address above) to obtain maps of randomized sampling sites.

2. From your national GTN cells randomly select one cell for duplicate sampling. Countries which have 9 or more GTN cells should select 2 cells for duplicate sampling. Duplicate samples of each material must be taken from one geologically representative small catchment (1, 2, 3, 4 or 5) of the selected GTN cell.

3. The GSF will provide small maps of each of your GTN cells with five randomly selected numbered points (Fig. 2). Point number 1 is located in the NE quadrant of the grid cell, number 2 in the NW quadrant, number 3 in the SW quadrant and number 4 in the SE quadrant. Point number 5 is randomly located in the grid.

Selection of small drainage basins in each GTN cell: The randomly selected points should be used to select the five nearest small drainage basins of <100

km² in area. Prepare sample sites for stream water (filtered and unfiltered), stream sediment, residual soil (top and subsoil), humus and optional surficial overbank sediment (0-25 cm) (see Figs. 3, 4 and 5). Soil samples should represent the dominant residual soil type of the small catchment basin. In the case that the randomly selected point happens to be in the sea or in an unreachable area, the nearest useable catchment basin should be used.

Selection of large drainage basins in each GTN cell: The floodplain sediment sample (upper 25 cm) is collected from the lowermost point of the larger drainage basin (area 1000 - 6000 km²) to which the small catchment is connected (see Figs. 3 and 5). If no suitable size drainage basin is available, the floodplain sediment can be taken from a smaller drainage basin (> 500 km²). If the size of the largest related drainage basin is less than 500 km², the floodplain sample should not be taken. Justified exceptions can also be made to this recommendation.

Baltic Soil Survey: Those countries which have taken part in the Baltic Soil Survey (BSS) can use the BSS soil samples in the FOREGS mapping. If you want to do so, please use the randomly selected points provided by the GSF to select the nearest BSS sampling site. The stream sediment, stream water and optional overbank sediment samples should be taken from the same small catchment and the floodplain sediment from the adjacent large drainage basin. You can decide whether you want to use the BSS samples or take new residual soil samples. If you use a BSS sample, mark it on the field observation sheet.

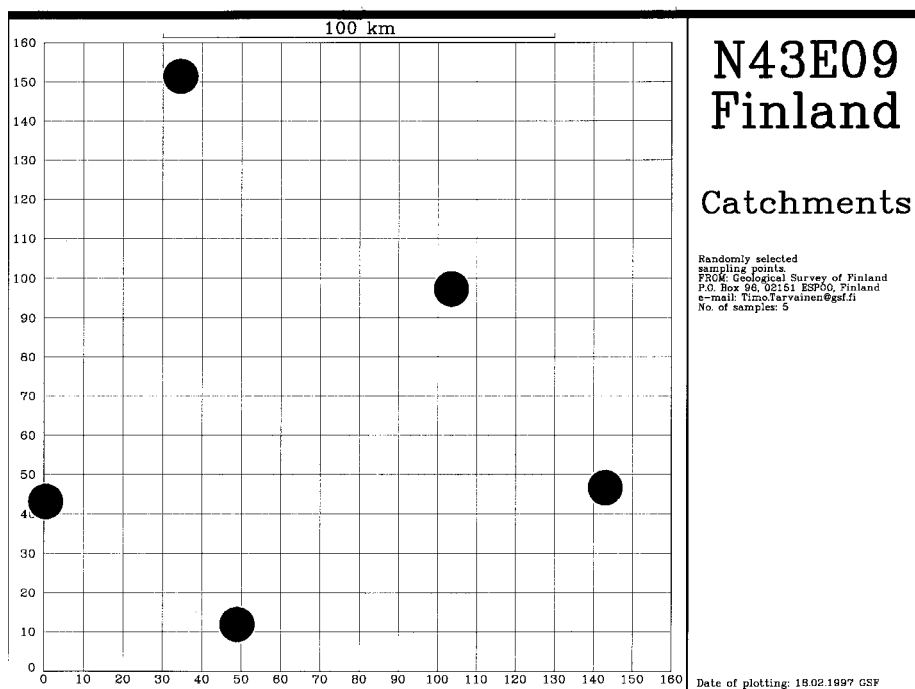


Fig. 2. Example of randomly selected sampling points from one 160 km x 160 km grid cell.

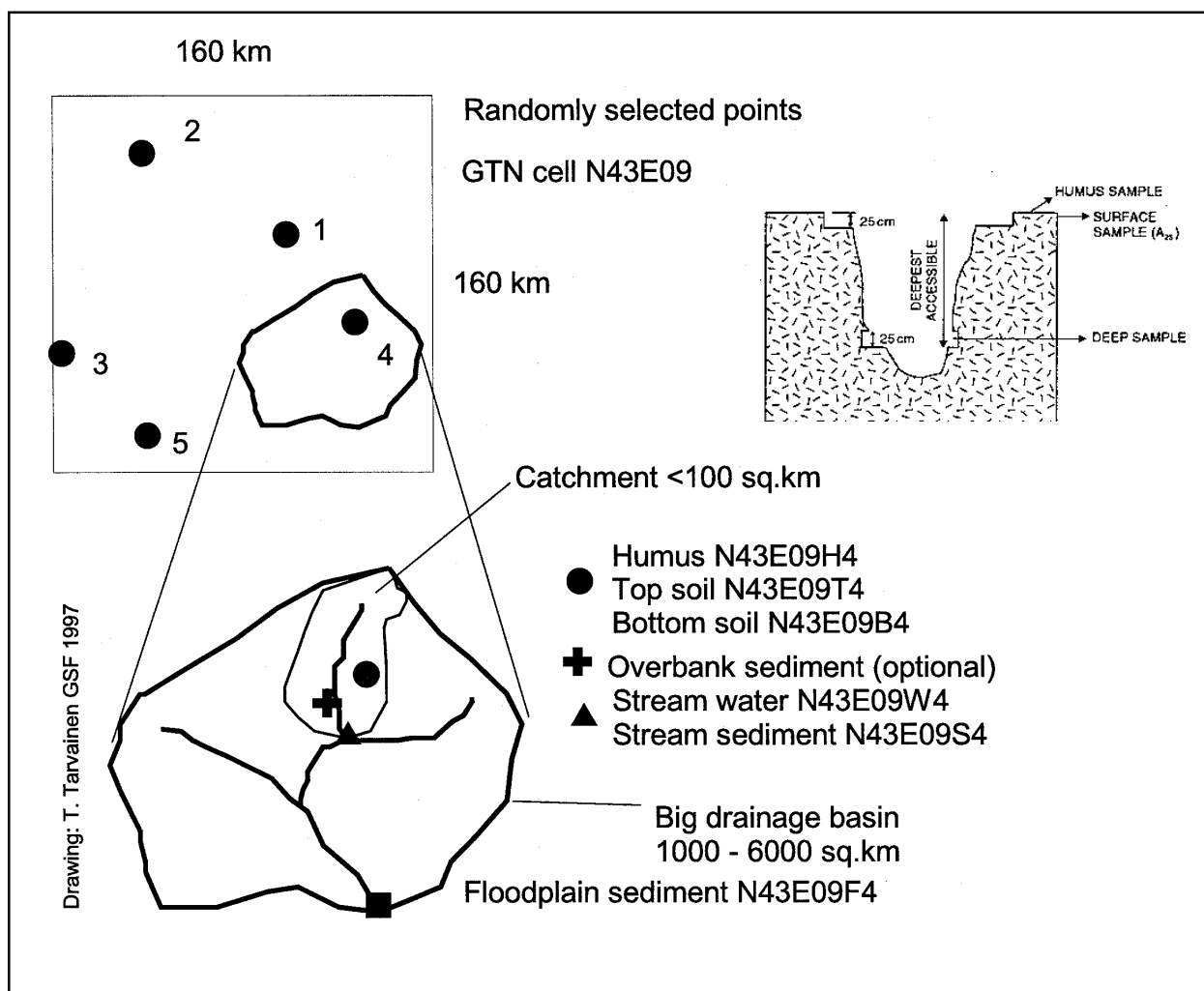


Fig. 3. Selection of sampling sites (Modified after Darnley et al. 1995). Schematic outline of sampling pattern and sampling pit for geochemical reference network. The sample pit applies to all residual soil locations. Deep sample (C): a 25 cm thick section within a depth range of 50 cm - 200 cm.

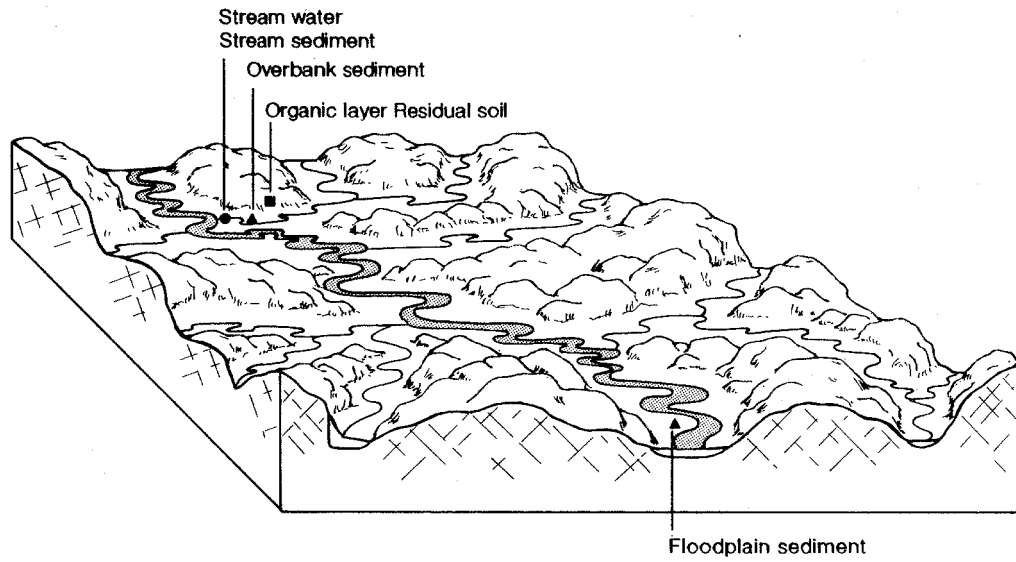


Fig. 4. Block diagram showing possible sampling sites of GTN sampling media (modified after Strahler 1969).

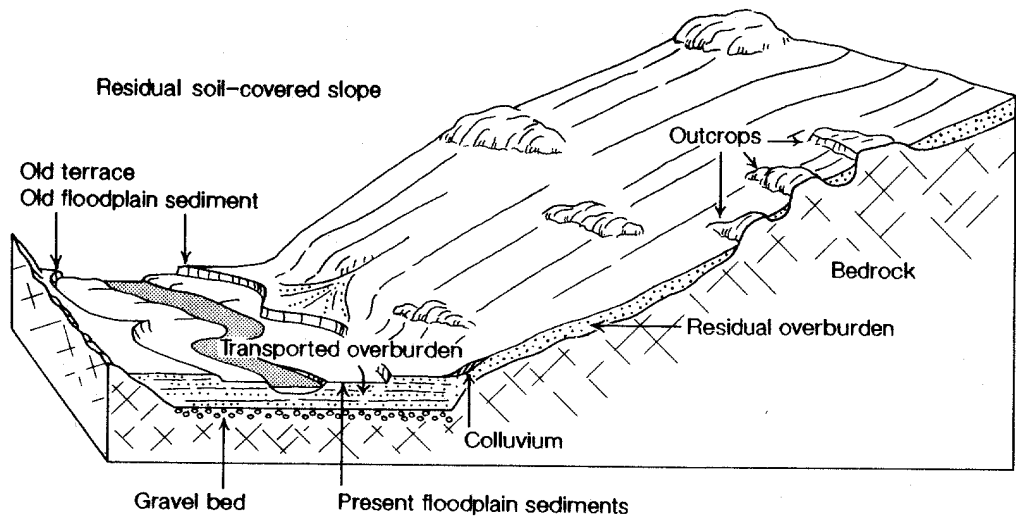


Fig. 5. Block diagram showing residual overburden (soil), colluvium, old and present day floodplain sediments (modified after Strahler 1969).

4. From the small drainage basin (< 100 km²) collect the following sample types:

	Symbol
•Stream water	W
•Stream sediment (mineral sediment)	S
•Residual soil, upper horizon (topsoil); 0 - 25 cm without organic layer	T
•Residual soil, lower (C) horizon (subsoil); a 25 cm layer within a depth range of 50 cm - 200 cm	C
•Soil samples should represent the dominant soil type of the small catchment.	
•Humus where present	H
Optional (will not be analysed in the FOREGS project)	
•Overbank sediment, top 0-25 cm without organic layer	K
•Overbank sediment, lowermost 25 cm	N

5. From the large drainage basin (1000-6000 km²) collect one sample type:

	Symbol
•Floodplain sediment, upper horizon 0 - 25 cm	F
Optional (will not be analysed in the FOREGS project)	
• Floodplain sediment, lower 25 cm thick section	L

2.3 Identifiers

The following example clarifies the sample identification system. The identifier of the stream sediment sample may be

N43E09S4, where
 N43E09 = GTN cell
 S = Sample medium symbol
 4 = Catchment (drainage) basin number

The duplicate sample would be N43E09S4D, where D is the duplicate sample identifier. Identifiers for filtered blank (or zero) water samples are:

GTN cell / W / Catchment basin number / 0 (zero), for example N43E09W40.

3. SAMPLING

Due to the significantly increased performance of analytical methods especially in terms of sensitivity, precision and accuracy, the most dominant source of error is sampling. For this reason it should be

emphasised that sampling procedures should be followed as closely as possible to avoid contamination during sampling

***** All hand jewellery must be removed *****

***** All tools and containers must be free of contaminants *****

3.1 Stream water

Running stream water is collected from the small, second order, drainage basin (< 100 km²) at the same site as the active stream sediment. In dry terrain, such as Southern Europe, streams may not have running water if the sampling is carried out during late spring, summer and early autumn, but ponds with stagnant water may be found. Since the geochemistry of stagnant water is normally different from that of running water no samples should be collected from such ponds.

3.1.1 Water samples to be collected:

1 x 500 ml bottle unfiltered water for major IC ion analysis
 1 x 100 ml bottle filtered water for ICP-MS and ICP-AES analysis
 1 x 60 ml bottle for DOC analysis
 1 x 100 ml bottle for mercury analysis

Sampling quantities: see bottle sizes above.

Duplicate water sample: From one sampling site of a duplicate cell (one in each country) 2 bottles of each type will be required.

3.1.2 Equipment:

3.1.2.1 Equipment to be provided by regional laboratories:

-500 ml new polyethylene bottles (for non-filtered water samples for major IC ion analysis)
 -60 ml new polyethylene bottles (for filtered, unacidified samples for DOC analysis, DOC= Dissolved Organic Carbon)
 -Disposable gloves (Medi-Point vinyl gloves, powder free or comparable)
 -Disposable syringes (e.g. Becton & Dickinson

Plastipak)

-Disposable filters 0.45 µm (e.g. Schleicher & Schuell pyrogen free)
 -Sterile trace element free 100 ml Nalgene bottles (for ICP-MS and ICP-AES analysis)
 -Droplet bottles made of teflon FEP (fluorinated ethylene propylene)
 -100 ml hardened plastic bottles for mercury analysis

3.1.2.2 Equipment to be purchased by each participant:

-Potassium dichromate solution for Hg preservation: 0.2 g of K₂Cr₂O₇ (Pro analysis, PA, quality) / 100 ml nitric acid HNO₃ (Suprapure quality)
 -pH-meter (e.g. WtW pH90 or comparable)
 -EC-meter (e.g. WtW LF92 or comparable)
 -Buffer solutions for calibration of pH-meters
 -Distilled and deionized water and a washing bottle
 -Concentrated HNO₃ 65%, density 1.40 kg/l (Merck Suprapur (R) 100441 or equivalent)
 -Permanent drawing ink markers
 -Cool boxes and batteries for them
 -Maps (topographical maps, preferred scale 1:50 000)
 -Rubbish bags
 -2 polyethylene (1 L) decanters for sample water to measure pH and EC
 -Plastic 100 ml measuring cylinder (for alkalinity measurements, methods A and B)
 -250 ml plastic conical flask (for alkalinity measurements, methods A and B)

If alkalinity is measured using method A (see below)

-Hach Model 16900-01 digital titrator or equivalent with solution delivery straws
 -Hach digital titration cartridges sulphuric acid 1.6 N
 -Hach digital titration cartridges sulphuric acid 0.16 N
 -Bromocresol green acid-base indicator solution: Accurately weigh 0.1 g of bromocresol green into a brown container flask. Add 14.3 ml of 0.01 M NaOH + 235.7 ml distilled and deionized water and swirl to dissolve (Lide 1991).

If alkalinity is measured using method B (see below)

- 1.6 N H₂SO₄: Dilute 44.4 ml of concentrated H₂SO₄ (mass-% = 96, density = 1840 g/l) to 1000 ml with deionized water.
- 0.16 N H₂SO₄: Dilute 10 ml of 1.6 N H₂SO₄ to 100 ml with deionized water.
- Bromocresol green indicator solution: Accurately weigh 0.1 g of bromocresol green into a brown container flask. Add 14.3 ml of 0.01 M NaOH + 235.7 ml distilled and deionized water and swirl to dissolve (Lide 1991).
- Volumetric flasks, capacity of 100 and 1000 ml
- Burette or equivalent equipment, 10 ml capacity, graduated in divisions of 0.02 ml
- Disposable Pasteur-pipettes

Field observation sheets are included in this manual (see Appendix 1).

3.1.3 Water sampling procedure

It would be best if sampling is carried out by persons who have experience in water sampling. If sampling staff are inexperienced, they should be trained and closely supervised for at least a few days.

Avoid sampling during rainy periods and flood events. The water sample must be taken before the stream sediment sample, for obvious reasons (during the collection of the stream sediment, fine-grained material is agitated and transported in suspension). The stream sediment sample is composited from 5-10 subsamples in the field. The water sample should be taken from the first, lowermost stream sediment sampling point.

Water samples to be taken in the manner described below:

1. Write the sample number on 100 ml bottles, 500 ml bottle and on 100 ml Hg sample bottle and the sample number and "DOC" on 60 ml DOC sample bottle.
2. Complete sample card and mark your position and sample number on the map.
3. Rinse twice and fill the polyethylene decanters with stream water, place the electrodes in water and measure the pH and conductivity with calibrated meter (mark with permanent drawing ink marker two decanters for pH and conductivity).
4. Filter one 100 ml sample (Fig. 6)

- Put vinyl gloves on your hands
- Rinse a disposable syringe with sample water and fill it up with water
- Put filter on syringe
- Discard the first 10 ml of filtered water from each new filter unit used
- Take a 100 ml marked sample bottle for acidified sample and rinse the bottle twice with filtered sample wa-

ter

- Fill the bottle up to its neck with filtered water (change filter if needed) and close it tightly. Note that the filtered sample water should go straight into the bottle without contact with your hands
- Take 60 ml marked sample bottle for DOC sample and rinse it twice with filtered sample water
- Fill the DOC sample bottle up to its neck with filtered water (change filter if needed) and close the bottle tightly.
- Note that the samplers are not allowed to smoke, or have the vehicle running, when the water sample is taken.

5. Rinse the marked 500 ml sample bottle with sample water twice and fill it up so that the bottle is completely submerged in the water and no air bubbles are left in the bottle. Fill the bottle as full as you can and close it tightly below water level.

6. Rinse the marked Hg sample bottle with sample water twice and fill it to its neck and close it tightly.

7. Note the pH - (with 1 decimal figure) and EC-value (mS/m) on the field observation sheet. Rinse the electrodes and decanters with distilled and deionized water and keep the electrodes in their measuring decanters until all daily sampling sites are visited. Then place the meters in their cases. The meters are calibrated every day before the first measurements are taken.



Fig. 6. Filtration of a stream water sample (Photo: Jari Väättäinen, GSF).

8. Total alkalinity measurements.

Alkalinity means the ability of water to neutralize acid. It is defined as the quantity of ions, such as CO_3^{2-} , HCO_3^- , OH^- , HSiO_3^- , H_2BO_3^- , HPO_4^{2-} and H_2PO_4^- , in water that will neutralize hydrogen ions. However, in most waters, HCO_3^- is the dominant ion between pH 4.5 and 8.3. The alkalinity is often expressed as milligrams per litre of CaCO_3 . This means that the determined total amount of all ions neutralizing acid is converted to an equivalent concentration of CaCO_3 by calculation. Many digital titrators and standard acid cartridges are, however, already calibrated to read in mg/l CaCO_3 .

The determination of alkalinity is made by titrating the known amount of water with acid (e.g. H_2SO_4 or HCl) to pH 4.5. At this point, all the ions which neutralise acid are used and the colour of indicator (e.g. phenolphthalein or bromocresol green) will change.

There are two methods to determine the alkalinity. Follow either method (A) or method (B) in the field.

Method (A): Using Hach digital titrator and standard acid cartridges.

-Select a sulphuric acid cartridge 1.6N or 0.16N according to the expected alkalinity of samples

-Fit the cartridge to the hand-held digital titrator and push the titrator piston down until it meets the top of the cartridge. Remove the cap from the cartridge and fit a feeder straw into the end of the cartridge. Wind the large wheel in the titrator until all the air is removed from the cartridge and the straw, and a drop of acid leaves the end of the straw. Wipe the end of the straw to remove excess acid. Reset the titrator scale to zero by winding the small wheel to the left of the scale forwards.

-Rinse the measuring cylinder and conical flask with distilled and deionised water prior to rinsing them with a small amount of the first sample. Using the measuring cylinder, measure 100 ml of the first sample into the conical flask.

-Make sure the lid is securely tightened on the sample bottle between each stage of the measurements to reduce degassing of the samples.

-Add a few (two) drops of bromocresol green indicator using a small pipette.

-Add the acid using the large wheel on the titrator until the solution changes from blue to green-yellow and note the reading on the titrator scale when this occurs.

-When the 0.16N cartridge is used the readings should be multiplied by 0.1

-The reading is the total alkalinity expressed as mg/l CaCO_3 . Write up the alkalinity on the sample card.

-The titrator should be reset to zero and the conical flask and measuring cylinder rinsed in a small amount of the next sample prior to the next measurement.

-Titrations should be carried out as quickly as possible to reduce degassing effects.

Method (B): Using normal burette and non stand-

ard acid.

-Transfer 100 ml of the sample water to a 250 ml conical flask with a volumetric glass. Add two drops of bromocresol green indicator with a disposable Pasteur-pipette.

-Select a sulphuric acid bottle 1.6N or 0.16N according to the expected alkalinity of samples.

-Mix and titrate immediately. Add the H_2SO_4 dropwise from a burette while continuously stirring the sample. The end-point is reached when the colour has changed to yellow. Write down the volume of acid used.

-If sample water is very coloured and the end-point is difficult to observe, use a pH-meter to determine the end-point. Titrate solution as above to pH 4.5. After titration, wash the pH-electrode with distilled and deionised water and calibrate the pH-meter.

-If 0.16 N H_2SO_4 was used for titration, the alkalinity of the sample, expressed as milligrams of CaCO_3 per litre is given by equation (1):

$$\text{Alkalinity (mg/l CaCO}_3) = 80 * V_{0.16N} \quad (1)$$

where $V_{0.16N}$ = the volume of 0.16 N H_2SO_4 (ml) used in titration.

-If 1.6 N H_2SO_4 was used for titration, the alkalinity of the sample, expressed as milligrams of CaCO_3 per litre is given by equation (2):

$$\text{Alkalinity (mg/l CaCO}_3) = 800 * V_{1.6N} \quad (2)$$

where $V_{1.6N}$ = the volume of 1.6 N H_2SO_4 (ml) used in titration.

9. The filled sample bottles are placed in the cool box.

10. After every 20th sample (and at least once in every country) the blank water sample is filtered: Filter distilled and deionized water in 60 ml bottle in the same manner as the normal water sample. This sample is treated (acidified and handled) like the normal water samples. The identifier for a filtered 0-sample will be: grid cell / W / sample no. / 0, for example N43E09W40.

3.1.4 Treatment of water samples

1. In laboratory or in comparable conditions, soon after sampling (at least on the same day) add to the 100 ml filtered water sample bottle 1.0 ml of conc. HNO_3 acid with a droplet bottle. Use disposable clean vinyl gloves, because the acid is very corrosive. Do not let the droplet bottle touch the sample water in the bottle. Close the bottle tightly and shake it in order to get the acid well mixed with the water. **Do not add acid to the 60 ml DOC sample bottle!**

2. Add nitric acid and potassium dichromate to water samples in Hg sample bottle: 5 ml HNO_3 - $\text{K}_2\text{Cr}_2\text{O}_7$ to 100 ml water bottle.

3. Place the bottles in a cool unit, e.g. refrigerator.
4. Send water samples to the laboratory soon after sampling.

3.2 Stream sediment

Active stream sediment represents the fine- to medium-grained bed load material (silty-clayey-sandy), which is transported by running water. The active stream sediment is collected from the small, second order, drainage basin (< 100 km²) at a suitable site above its confluence point with the main, third order, channel of the large drainage basin.

Studies into the distribution of trace elements in relation to the size fraction of stream sediments generally show that several elements including Mo, Cu, Zn, Mn and Fe are concentrated in the finest fractions of the sediment. The majority of stream sediment surveys have, therefore, been based on the collection of <0.200 mm material. The IGCP 259 and FOREGS standard sieve mesh is <0.150 mm as this is fine enough to only include the very fine sand, silt, clay and colloidal fractions, but is coarse enough to yield sufficient fine material in the majority of situations.

Studies in the UK have shown the recovery of stream sediments by dry sieving methods is not quantitative owing to the agglomeration of fine material to form larger particles which are then screened out in varying amounts. A system of wet sieving stream sediments wherever possible is therefore recommended for IGCP 259/360 and FOREGS.

It is important to avoid metal contamination at every stage of sampling as follows;

No hand jewellery or medical dressings should be worn during sampling. If medical dressings are worn, heavy duty rubber gloves must be worn at all times to avoid contamination of the samples.

Metal free polyethylene or unpainted wooden spades/scoops should be used.

Metal free nylon sieve-mesh housed in inert wooden or metal free plastic frames should be used.

Metal free funnels and sample collection containers should be used.

If it is not possible to use non-metal equipment (e.g. spades and sieve frames), unpainted steel equipment should be used. Aluminium and brass equipment should be avoided.

Sampling sites should be selected sufficiently upstream of confluences with higher order streams to avoid sampling sediment that may result from a mixing of material from the two channels during flood flow.

Dry sieving is an alternative method if wet sieving (Section 3.2.3.1) cannot be used, as is the case of seasonal streams in Mediterranean countries.

3.2.1 Stream sediment samples to be collected

Each stream sediment sample comprises material taken from 5-10 points over a stream stretch of 250 - 500 m. Prior to stream water and stream sediment collection, it is important to identify the 250 - 500 m stream stretch where obvious signs of contamination can be avoided and suitable sediment can be collected from 5-10 different locations. Sites should be located at least 100 m upstream of roads and settlements. Stream sediment sampling should start from the water sampling point and the other sub-samples should be collected up stream. A composite sample should not be made from samples taken from beds of different nature (ISO 5667-12:1995). From one small catchment basin of a duplicate cell (one in each country) minimum 0.5 kg (dry weight) + 0.5 kg (duplicate sample) of <0.150 mm material is required. From all other small catchment basins a minimum of 0.5 kg (dry weight) <0.150 mm material is required. Larger sample quantities can be taken and stored separately in each country.

3.2.2 Equipment

3.2.2.1 Equipment to be provided by regional laboratories:

- Kraft paper bags
- Polyethylene bags

3.2.2.2 Equipment to be purchased by each participant:

- Heavy duty elbow length rubber gloves
- Metal free polyethylene funnel
- Sieve set with 2 preferably wooden or plastic frames containing nylon 2.0 mm mesh and nylon 0.150 mm mesh screens
- Metal free gold pan or plastic bucket
- Metal free plastic crates
- Metal free plastic buckets or containers with lids
- Trenching tool - metal free, polyethylene (PE) or polypropylene (PP)
- Permanent drawing ink marker (preferably black or blue)
- Permanent ink pen
- Maps (topographical maps, preferred scale 1:50 000)
- Chisel-end geological hammer for dry areas (e.g. Mediterranean countries)
- Bristle brush (dry sediment samples)

Field observation sheets are included in this manual (see Appendix 1).

3.2.3 Sampling procedure

Mark the sample identifier on the Kraft paper bag using permanent ink marker. Mark the exact site location of the first and last subsamples on the field map by means of a small lines perpendicular to the stream flow. Complete the details of the field observation sheet.

Wet sieving is recommended whenever it is possible. Instructions for sampling with wet sieving method are presented in section 3.2.3.1. If it is not possible to wet sieve the stream sediment sample in the field, the collected stream sediment material should be dry sieved. Instructions for sampling and dry sieving are given in section 3.2.3.2.

3.2.3.1 Sampling and wet sieving

Once the site for sampling has been selected, mark the exact location of the first and last sampling points on the field map by means of a small line perpendicular to the stream flow using the ink pen. Mark the sample identifier number on map next to the sampling location. Complete the details on the field observation sheet. Write the sample identifier on the collection bucket and lid using the permanent drawing ink marker.

Rubber gloves are recommended for protection throughout sampling.

Following collection of any water samples, prepare the equipment for stream sediment sampling:

-Wash all stream sediment sampling equipment (buckets, sieves, gold pans, funnel, gloves and spade) with stream water.

-Set-up the gold pan or collection bucket in a stable position (since material will be collected from 5-10 points over a distance of 250-500 m, it is recommended that the sieving site is located at the half-way point).

-Place the sieve with the 0.150 mm aperture nylon cloth in a stable position resting on the gold pan or bucket.

-Place the sieve with the 2 mm aperture nylon cloth over the 0.150 mm sieve.

-It is important that the sieve frames fit closely over the collection pan or bucket to avoid loss of material over the edge of the bucket.

-In rugged terrain, where collapse of bank material into the channel is common, sediment from as near the centre of the stream as possible should be collected to avoid sampling bank-slip material.

-In areas of low relief, active stream sediment in the centre of channels may be enriched in quartz and depleted in clays and other fine particles. In these instances material deposited along stream margins during flood events may be finer grained and more suitable for geochemical sampling.

-Load equal amounts of coarse active sediment from 5 - 10 points on the stream into plastic buckets taking care to drain off excess water (Fig. 7).

-Enough coarse grained material should be collected to yield a minimum of 0.5 kg <0.150 mm material (dry weight).

The amount of coarse material required will vary substantially depending on the underlying geology and terrain. Geochemists should use their knowledge and judgement to assess how much coarse material will be required.

-Mix the buckets of the coarse sediment thoroughly with the plastic stirring rod and carry them to the sieving location

-Load sediment into the top sieve with the spade. If more than one bucket of coarse sediment has been collected, equal amounts of sediment should be loaded into the sieve from each bucket in turn.

-Rub the material through the top sieve wearing rubber gloves for protection.

-Take care to remove large stones from the sediment by hand.

-Once the bottom sieve contains a reasonable quantity of <2 mm sediment, remove the top sieve and discard the >2 mm material.

-The <2 mm sediment in the bottom sieve is washed and rubbed through the sieve with the aid of water and shaken down.

-It is very important at this stage that coarse material which would bias the sample does not enter the collection bucket. This may be avoided by carefully washing the outside of the bottom sieve prior to shaking.

-In order to enhance the trace element signature, it is important that all the <0.150 mm material is collected, therefore, a minimum amount of water should be used to wash the sediment through the bottom sieve and all washing water should be retained in the collection bucket until the sample is allowed to settle.

-The sample should be repeatedly washed and shaken down until all the fine material has passed through the sieve.

-The whole sieving process should be repeated until the bucket contains sufficient fine grained wet sediment to yield 0.5 kg dry weight material.

-If sediment is collected in a gold pan, it should be transferred to a collection bucket with lid for transporting to the field base.

-Once enough wet sediment has been collected, secure the lid on the bucket. The sediment should be carefully transported to the field base and allowed to stand for at least 45 minutes or until all the suspended material has settled and clear water sits on top of the sediment.

-Once the suspended material has settled, excess water on the surface of the sediment should be carefully decanted. Care should be taken to remove only water and not sediment at this stage.

-The remaining sediment should be thoroughly homogenised and mixed using the plastic stirring rod before being transferred into sample bags.

-Using the permanent ink marker pen, write the sample identifier on enough 10 x 22 cm Kraft paper bags to hold all the sample volume. This size of Kraft bag allows ease of drying the samples.

-The number of bags for each sample site should be

recorded on the field sheet and on a sample check-list sheet.

-Once the sample has been homogenized, carefully transfer the sample into the Kraft bags using a clean plastic funnel.

-The Kraft bags should be hung out to air dry at the field base for as long as possible.

-When moving the samples, place each Kraft bag in a 15 x 40 cm polythene bag and secure the top of the bag with a knot to prevent loss or cross contamination of samples during transport.

-The samples should be secured upright in a plastic crate or box and transported carefully to the next location or to the Survey base for further drying.

-At the Survey base or laboratory, the samples should be completely dried at < 40°C. Freeze drying is a recommended as this helps to disaggregate the samples. Dried samples should be sent to LAB I.

All sampling equipment must be thoroughly cleaned between each site to avoid cross contamination.

3.2.3.2 Sampling and dry sieving

The procedure for the selection of sample sites, recording their location on the field map, completion of field observation sheets, wearing of rubber gloves, etc. are the same as for the wet sieving sampling method (refer to Section 3.2.3.1).

Since, water is not available to wet sieve the stream sediment to the required <0.150 mm fraction, collect a bulk composite sample from 5-10 points over a distance of 250-500 meters. The total dry weight of the composite sample (free of stones and other coarse grained material) should be about 5 kg to ensure that the required amount of 500 grams of analytical <0.150 mm material will be obtained after sieving at the domestic lab.

Collect material of finer grain size (or if possible only the top thin layer of silt on the sand bar) from the centre of the stream. Care should be taken to sample stream sediment with as little as possible organic matter, and to avoid the reduced material (mostly dark colour and bad smelling), which occurs at greater depths. Also, in the case of narrow channels take care not to collect material which has fallen from the banks.

The removal of stones and other coarse grained material could be achieved by sieving through a 5 mm nylon sieve, and collecting the material in a plastic bowl. Collect equal amounts of material from the 5-10 subsites. The use of the 2 mm nylon sieve is not recommended for dry sieving, because it is too small for clay agglomerates and slightly moist samples.

Transfer the fine grained sample to the Kraft paper bag and seal. Place the sealed Kraft envelope within a polyethylene bag, and tie a loose knot in the



Fig. 7. Wet sieving of a stream sediment sample (Photo: Fiona Fordyce, BGS)

polyethylene bag to prevent loss or cross contamination during transportation.

A special case is the sampling of small seasonal streams in Mediterranean countries, which must be sampled with extreme care. Some of the seasonal streams have had no water flow for many years, and the stream bed may be covered by fallen bank material in which grass or other plants may have grown. Since, active stream sediment must be sampled, the fallen bank material, covering the “old” active stream sediment, must be removed by digging before taking the sample at each subsite. The pits should be dug near to the centre of the channel.

Air dry the sample, disaggregate if necessary in porcelain bowl and sieve it through a 2 mm aperture nylon sieve in the domestic lab before shipping to the LAB I.

3.2.4 Photographing

At each stream sediment/water sample site take two photographs, the first to show general upstream topography from the lowermost subsite and the second the nature of the stream bed at the best subsite.

3.3 Humus and residual soil samples

3.3.1 Humus and soil samples to be collected

From one sampling site of a duplicate cell (one in each country) collect:

- 2 x 1-2 L humus (where present; normal and duplicate samples)
- topsoil: 2 kg + 2 kg (duplicate sample)
- subsoil: 2 kg + 2 kg (duplicate sample)

From all other sampling sites collect:

- 1-2 L humus (where present)
- 2 kg of topsoil
- 2 kg of subsoil

Larger sample quantities can be taken and stored separately in each country.

3.3.2 Equipment

3.3.2.1 Equipment to be provided by regional laboratories

- Plastic bags (PE) for humus
- Kraft bags for soil samples
- Disposable gloves (1 per sample)

3.3.2.2 Equipment to be purchased by each participant:

- Sampler (cylindrical sampler or spade)
- Plastic or steel scoop
- Plastic spoon (see Fig. 8b)
- Knife
- Maps (topographical maps, preferred scale 1:50 000)
- Wooden folded 2 m long metre
- Permanent drawing ink marker (preferably black or blue)
- Plastic boxes for sample bags

Sampling tools should be made of unpainted wood, polyethylene (plastic) or steel (unpainted spade). Field observation sheets for humus and soil are included in this manual (see Appendix 1).

3.3.3 Humus

In those climatic regions where humus is developed, it is normally found in undisturbed forested areas above the normal mineral soil. Decomposed black coloured organic material (humus) should be sampled.

Mark the sampling site on the field map and complete the field observation sheet after studying the organic layer.

No samples should be taken in local depressions.

The sampling site should be as flatly as possible, unless the sample is collected from a mountainous area (forest, grassland). If the organic layer is present, collect enough humus to fill the plastic bag provided, avoiding contamination (use gloves to collect and handle samples). Each humus sample should be a composite sample from at least five (5) locations within a 50 m x 50 m area.

All points should be selected at a sufficient distance from the nearest trees to avoid "throughfall" precipitation from the trees (a minimum distance of 5 m from nearest tree and 3 m from the nearest bush is recommended, but each sampling team is free to use its discretion in the selection of subsites in forested areas).

A steel or plastic sampling tool is used for sampling (Figs 8 a-d). The living surface vegetation, fresh litter and big roots and rock fragments should be carefully removed wearing plastic gloves. Do not collect raw humus because it is very difficult to define an exact border between litter and raw humus. Only the uppermost 3 cm of humus should be sampled. The mineral soil layer should be carefully removed using a plastic spoon. After collection of each sample clean thoroughly the sampling equipment.

Air-dry samples thoroughly at room temperature, which should not exceed 40°C (loss of Hg!); place the sample on the bag and turn over as necessary. Use gloves! Any residual humidity will promote anaerobic reactions, including sulphate reduction, and must be avoided. When samples are properly dried, transfer to a new bag, if necessary (field bag may still be wet), seal and send to LAB IV.

3.3.4 Residual soil samples

Residual and sedentary soils: Residual soil is collected from the small, second order, drainage basin (< 100 km²) at a suitable site above its alluvial plain and base of slope, where alluvium and colluvium are respectively deposited. The residual soil may be developed either directly on bedrock or on till, as is the case in glaciated terrains. Residual soil distributed by agricultural activities should be avoided where possible since the top soil is usually affected by human activities. It is important to ensure that the material collected is residual and not transported material such as coluvium or alluvium.

Select a suitable area where 3 - 5 pits can be dug. Mark the sampling site on the field map and complete the field observation sheet after digging the pits and studying the soil horizons.

Two different depth related samples will be taken at each site: a topsoil sample from 0-25 cm (excluding material from the organic layer where present) and a subsoil sample from a 25 cm thick section within a depth range of 50 - 200 cm (the C soil horizon). The soil sample should represent the dominant



Fig. 8a. Humus sampling in Finland using cylindrical sampler. (Photographs: Timo Tarvainen).

Humus and soil



Fig. 8c. Remove all mineral soil using a plastic spoon.



Fig. 8c. Pull humus (on left) and roots (on right) apart.



Fig. 8d. Final humus sample.

residual soil type of the small (< 100 km²) catchment basin, it must be a residual or sedentary soil, but definitely not an alluvial soil. Each sample should be a field composite sample from 3 - 5 subsamples in the field. Minimum distance between any two subsamples should be 5 m. Avoid sampling adjacent to roads (minimum distance 10 m) or ditches (minimum distance 5 m), but you are free to use your discretion depending on the traffic density and prevailing local conditions.

Living surface vegetation, fresh litter, big roots and rock fragments (stones) are removed.

In case the whole soil profile does not reach a depth of 75 cm, the lower sample should be taken from a depth, that can be undoubtedly identified as the BC- or C-horizon (do not forget to note this down under remarks on the field observation sheet!). If this is not possible another sample site should be selected.

The subsoil sample is taken first, and then the topsoil sample. This procedure avoids cleaning the surface of the subsoil from fallen top soil, if the latter is taken first.

After collection of each sample clean thoroughly the sampling equipment.

3.3.5 Photographing

At each humus sample site take two photographs; the first to show the general topography of the tree layer and undergrowth, and the second a close-up to show the character of the organic layer. A flash may be necessary for the second photograph. If the humus samples and residual soil samples are collected from the same site, the close-up photo can show both the character of organic layer and mineral soil horizons (see below). In this case, separate photographs for the soil sample site will not be needed.

At each soil sample site two photographs should be taken; the first to show the general view about the sampling site (Fig. 9), and the second a close-up of one of the soil sample pits (Fig. 10). Before taking the second photograph mark with a knife the soil horizons, if they can be distinguished, and place an alternate coloured-section wooden metre as a scale on the face of the pit. If necessary use a fill-in flash when taking the second photograph, because it is

important to show the layers and textural characteristics of the soil horizons.

3.4 Floodplain sediments

A floodplain sediment, representing the alluvium of the whole drainage basin will be collected from the alluvial plain at the lowermost point (near to the mouth) of the large catchment basin (1000 - 6000 km²).

Both floodplain and overbank sediments are fine-grained (silty-clay, clayey-silt) alluvial soils of large and small floodplains respectively, according to the size distinction made by Darnley *et al.* (1995). Floodplain and overbank sediments are deposited during flood events in low energy environments (Ottesen *et al.*, 1989); they should, therefore, be devoid of pebbles, which indicate medium energy environments. The surficial floodplain and overbank sediments are normally affected by recent anthropogenic activities, and may be contaminated. Deeper samples, which are optional sample media, normally show the natural background variation.

3.4.1 Floodplain sediment samples to be taken

From the first sampling site of a duplicate cell (one in each country) collect:

- 2 kg of top floodplain sediment + 2 kg of top floodplain sediment (duplicate sample)

From all other sampling sites collect:

- 2 kg of top floodplain sediment

Enough material must be taken to yield minimum 0.5 kg of <2 mm grain size sediment. Larger sample quantities can be taken and stored separately in each country.

OPTIONAL:

From one sampling site of a duplicate cell 2 kg of bottom floodplain sediment + 2 kg of bottom floodplain sediment (duplicate sample) and from all other sampling sites 2 kg of bottom floodplain sediment.



Fig. 9. General view about the soil sampling site (Photo: Teppo Moisio, GSF).



Fig. 10. Close-up of one of the soil sample pits (Photo: Teppo Moisio, GSF)

3.4.2 Equipment

3.4.2.1 Equipment to be provided by regional laboratories

- Kraft bags for floodplain sediment
- Disposable gloves (1 per sample)

3.4.2.2 Equipment to be purchased by each participant

- Unpainted spade
- Pickaxe
- Knife
- Plastic or steel scoop
- Chisel-end geological hammer
- Wooden folded 2 m long metre
- Permanent drawing ink marker
- Maps (topographical maps, preferred scale 1:50000)
- Plastic boxes for sample bags
- Kraft paper bags for overbank sediments (optional)

Field observation sheets for floodplain sediments and the optional overbank sediments are included in this manual (see Appendix 1).

Sampling tools should be made of unpainted wood, polyethylene (plastic) or steel (unpainted spade). Containers should be made of paper or strong polyethylene.

3.4.3 Sampling procedure

Study the floodplain sediment sequence to begin with, and select a suitable section with many layers of fine-grained material, e.g. silty-clay, clayey silt, deposited in a low energy environment. Pebbles in fine-grained material, and gravel beds indicate medium to high energy environments, respectively. Avoid sites adjacent to roads or ditches (minimum distance 10 m). Mark the sampling site on the field map and complete the general field observations on the card, leaving the grain size to be completed after the sampling.

Two different depth related samples may be taken at each site:

MANDATORY: a top floodplain sediment sample from 0-25 cm (excluding humus where present and surface litter), and

OPTIONAL: a bottom floodplain sediment sample from the very bottom layer (lowermost 25 cm) of the exposed section, just above the water level of the river; note the depth of the sample interval on the field observations sheet. FOREGS laboratories will not provide analyses of the optional sediment samples.

In both cases single floodplain sediment layers must be sampled. If the thickness of the top floodplain sediment layer is less than 25 centimetres, do note its thickness on the field observation



Fig. 11. Floodplain sampling in southwestern Finland (Photo: Reijo Salminen, GSF).

sheet, and the number of layers sampled. The optional deep sample should ideally be collected immediately above the gravel bed (Fig. 5). Since, this is not always possible, the deepest possible layer should be sampled, for the objective is to reach a layer, which is not affected by human activities.

The floodplain sediment and optional overbank sediment samples are collected from a single site (Figs. 11 and 12).

At each exposed floodplain sediment sample site clear the surface humus and litter to begin with, and then cut a vertical section through the exposed floodplain sediment sequence with a steel spade, thus exposing a clean vertical surface for sampling. If you collect both top and bottom samples, sample the bottom layer first at each sample site, and then the top layer. This procedure avoids cleaning the surface of the bottom layer from fallen top sample material, if the latter is taken first.

If it is not possible to sample an exposed floodplain sediment sequence with the aid of a spade, follow the same procedure to select a suitable sample site on the floodplain (e.g., inside meander) and dig a pit down to the required depth.

Living surface vegetation, fresh litter and big roots are removed by wearing plastic gloves.

After the collection of each sample clean thoroughly the sampling equipment.

3.4.4 Photographing

At each floodplain (and overbank) sediment site two photographs should be taken, the first to show the general view about the sampling site with reference to the stream/river channel and the second a close-up of the sample pit. Before taking the second photograph mark with a knife the alluvial sediment layers, if they can be distinguished, and place an alternate coloured-section wooden metre on the face of the pit. If necessary use a fill-in flash when taking the second photograph, for it is important to show the layers and textural characteristics of the alluvial beds.

3.5 Overbank sediments

An overbank sediment, representing the alluvium of the small drainage basin will be collected from its alluvial plain near to the confluence point of the small, second order, stream (< 100 km²) with the main, third order, river.

3.5.1 Overbank sediments to be taken

All overbank sediment samples are optional. FOREGS laboratories will not provide analyses of

the overbank sediment samples.

OPTIONAL: From the first sampling site of a duplicate cell (one in each country) collect:

- 2 kg of top overbank sediment + 2 kg of top overbank sediment (duplicate sample)
- 2 kg of bottom overbank sediment + 2kg of bottom overbank sediment (duplicate sample)

OPTIONAL: From all other sampling sites collect:

- 2 kg of top overbank sediment
- 2 kg of bottom overbank sediment

Enough material must be taken to yield a minimum of 0.5 kg <0.150 mm grain size sediment.



Fig. 12. Floodplain sediment sequence with soil development in Greece. Meter: coloured sections 20 cm. Fine-grained clay and silt down to a depth of 75 cm (low energy floodplain sediment - good for sampling; soil has developed down to a depth of 25-29 cm). Coarse-grained sandy and bobbly unit between 75-100 cm (high energy environment). Sandy-clay unit between 100-134 cm (low-energy environment). Gravel bed below a depth of 134 cm. Photo: A. Demetriades, IGME.

3.5.2 Equipment to be purchased by each participant

- Kraft bags for overbank sediment
- Disposable gloves (1 per sample)
- Unpainted spade
- Pickaxe
- Stainless steel knife
- Plastic or steel scoop
- Chisel-end geological hammer
- Wooden folded 2 m long metre
- Permanent drawing ink marker
- Brush for cleaning equipment
- Maps (topographical maps, preferred scale 1:50000)
- Plastic boxes for sample bags
- Kraft paper bags for overbank sediments (optional)

Sampling tools should be made of unpainted wood, polyethylene (plastic) or steel (unpainted spade). Containers should be made of paper (specially manufactured “Kraft” sample bags) or strong polyethylene.

3.5.3 Sampling

Follow the instructions given in section 3.4.3 for floodplain sediments.

4. FIELD OBSERVATIONS

There are 5 field observation sheets: one for regolith soil; one for humus samples; one for stream water and stream sediment samples; one for floodplain sediment and one for overbank sediment samples.

In the “remarks” section significant supplementary information, if any, about the sampling site should be noted. Any deviations from the sampling routine should also be recorded, e.g. the wearing of jewellery during sampling, smoking, not wearing gloves etc.

Colour photographs are an inherent part of field observations, and should be taken with care and labelled correctly (refer to section 4.1).

It is recommended that gamma ray spectrometer measurements should be taken at each sampling site. See Darnley *et al.* (1995) for instructions, and section 4.2 of this document.

4.1 Photographs

Colour photographs must be taken at all sampling sites (stream water and sediment, residual soil, humus, floodplain sediment and optional overbank sediment), and recorded on the field observation sheets. Each photograph, after processing, should be carefully labelled at the back with a fine permanent ink and sent to the co-ordinator (Geological Survey of Finland) upon completion of the sampling.

Since the photographs will form a unique reference set for all sampling sites, it is important to ensure a good result. This means using a good camera. A reflex camera with a zoom lens (e.g. 35-85 mm) and macro facility is recommended, and an electronic flash.

Negative films of 100 ISO (ASA) rating are good for normal lighting conditions. But since some photographs will be taken in poor light conditions, e.g. in forest, it is recommended to use a colour negative of 400 ISO (ASA) rating.

Extra colour negative films (400 ISO), batteries for the camera and flash must be carried in the field.

Colour photographs should be printed on glossy paper of 10 x 15 cm minimum dimensions.

Photographs of the water/stream sediment sample site could be taken before or after the sampling. Organic layer (humus), residual soil pits, floodplain and overbank sediment pits must be photographed before taking the sample; fill-in flash should normally

be used when photographing the sample pits in order to ensure that all the features of the sample site are recorded.

Two copies of each photograph will be needed: one to be stored with negatives in your own institute, and another to be sent to the coordinator.

4.2 Gamma ray spectrometry

Gamma ray spectrometer measurements should be taken at every sample site:

- (a) lowermost water/stream sediment site
- (b) organic layer (centre of 50 x 50 m area)
- (c) residual soil (near to the first pit)
- (d) floodplain sediment
- (e) overbank sediment (optional)

Darnley *et al.* (1995) recommended taking *in situ* measurements at each subsite of residual soil. This is, however, impractical and is, therefore, recommended to take one measurement near to the first soil sample pit.

It is recommended to use a portable five-channel threshold scintillometer (e.g. Miniscint UG 130 made by Urec Ltd. or similar model). These instruments have the options of taking five measurements at different energy levels, i.e. two total counts, a K+U+Th, a U+Th and a Th.

Measurement must be taken at knee level, which is the normal height used for ground radiometric surveys.

The type of instrument (model, serial no., etc.) should be recorded on the field observation sheet. Ideally, all instruments used in this survey should be calibrated. This may be done before or after the FOREGS field sampling. A meeting will be arranged in a country where there are calibration pads.

4.3 Sampling site coordinates

Sample sites are carefully recorded on 1:50 000 topographical maps, and subsequently digitised in the office, and their coordinates noted on the field observation sheets. Since, the sample sites may be used for future monitoring, the use of a precise GPS instrument (+/- 5 m) is recommended. If a GPS instrument is used, the model, serial no., precision, etc. should be noted.

5. DIFFERENCES BETWEEN FOREGS GEOCHEMICAL SAMPLING AND IGCP 259 RECOMMENDATIONS

Upper horizon of overbank sediments, which is one of the mandatory sample types of the Global Reference Network, will not be sampled during this FOREGS sampling project. However, each country can choose to collect upper horizon samples of overbank sediments as well as deep layers of floodplain sediments and overbank sediments, which are optional materials in the Global Reference Network. No regional analytical services can be provided for these materials.

The field sample weight of soil and floodplain sediment samples is 2 kg (to be sieved). Because 5 samples are taken from each GTN cell, the total

amount of soil sample material is compatible with larger volumes suggested by Darnley *et al.* (1995). The analysed size fraction for soil and floodplain sediment samples will be <2 mm, not the fine soil, and for overbank sediment samples <0.150 mm.

The sample identifiers used in FOREGS mapping are much shorter than those suggested by Darnley *et al.* (1995) or by the Data Management Committee of the IGCP 360 project.

Gamma ray spectrometer measurements at residual soil sample sites are taken at only one subsite (first soil sample pit) instead at each subsite as recommended by Darnley *et al.* (1995).

6. SAMPLE PREPARATION AND ANALYSIS

Sample preparation and analysis will be undertaken by a small number of European laboratories as outlined below. At the time of writing the final assignments for these laboratories have not been made

LAB I Laboratory for sample preparation (soil and sediment samples). Geological Survey of the Slovak Republic.

LAB II Laboratory for analysis of unfiltered water samples, stream sediment and floodplain sediment samples. Ion chromatography (IC) is used for Cl⁻, Br⁻, NO₃⁻, NO₂⁻, PO₄³⁻, SO₄²⁻ and ion specific electrode (ISE) for F⁻ and Total Organic Carbon (TOC) in water. X-Ray fluorescence spectrometry (XRF) analyses for over 30 elements is used for stream sediment and floodplain sediment samples. To be nominated (suggestion: British Geological Survey).

LAB III Laboratory for analysis of filtered and acidified water samples and for XRF analyses of soil samples. Federal Institute for Geosciences and Natural Resources, BGR, Germany.

LAB IV Laboratory for humus analyses and grain size determinations. To be nominated (suggestion: the Netherlands Institute of Applied Geoscience TNO - National Geological Survey).

LAB V Laboratory for Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) analyses for soil/sediment samples. To be nominated (suggestion: Geological Survey of Finland).

LAB VI Laboratory for all Hg analyses (stream water, stream sediment, floodplain sediment and soil). To be nominated (suggestion: Service Géologique National, BRGM, France).

6.1 Handling of collected sample material

All samples will be analysed for the FOREGS regional geochemical mapping project. Send all soil and floodplain sediment samples with copies of your field cards and photographs to LAB I. Keep the negatives and one set of photographs in your institute. LAB I will send back to collaborating countries a smaller amount of prepared sampling material from each sample type.

All stream sediment samples and unfiltered/unacidified water samples should be sent to LAB II. The filtered, acidified water samples should be

sent to LAB III.

Water samples for Hg analyses should be sent to LAB VI.

Humus samples should be sent to LAB IV.

6.2 Sample preparation equipment and methods

The sample preparation undertaken by LAB I will be carried out using the equipment listed below and following the procedures illustrated by the flow sheets Figs. 13 to 15.

6.2.1 Equipment

- Dry box
- Porcelain bowl and pestle
- Pulverisette with agate and balls
- Sieving machine
- 0.15 mm sieve with nylon sieve cloth
- 2.0 mm sieve with nylon sieve cloth
- Rotary divider
- Nylon brush
- Silica sand (to clean agate bowl and balls)
- Acetone
- Deionized water
- 100 ml polyethylene bottles (PE) with colourless caps for analytical samples
- 250 ml PE bottles for duplicate samples
- 1000 ml PE bottles for arbitrated samples
- Self-stick of laboratory labels

6.2.2 Types of prepared samples

- Stream sediment [S], size fraction <0.15 mm
- Floodplain sediment [F], upper horizon 0 - 25 cm, size fraction <2 mm
- Soil upper horizon 0-25 cm [T], without top organic layer, size fraction <2mm
- Soil, lower horizon [C], 25 cm layer within a depth range of 50 cm - 200 cm, size fraction <2 mm

6.2.3 Sample preparation procedures

The sample preparation procedure for dry sieved stream sediment samples is presented in Fig. 13. The preparation procedures for floodplain sediment and soil samples are presented in Figures 14 and 15, respectively.

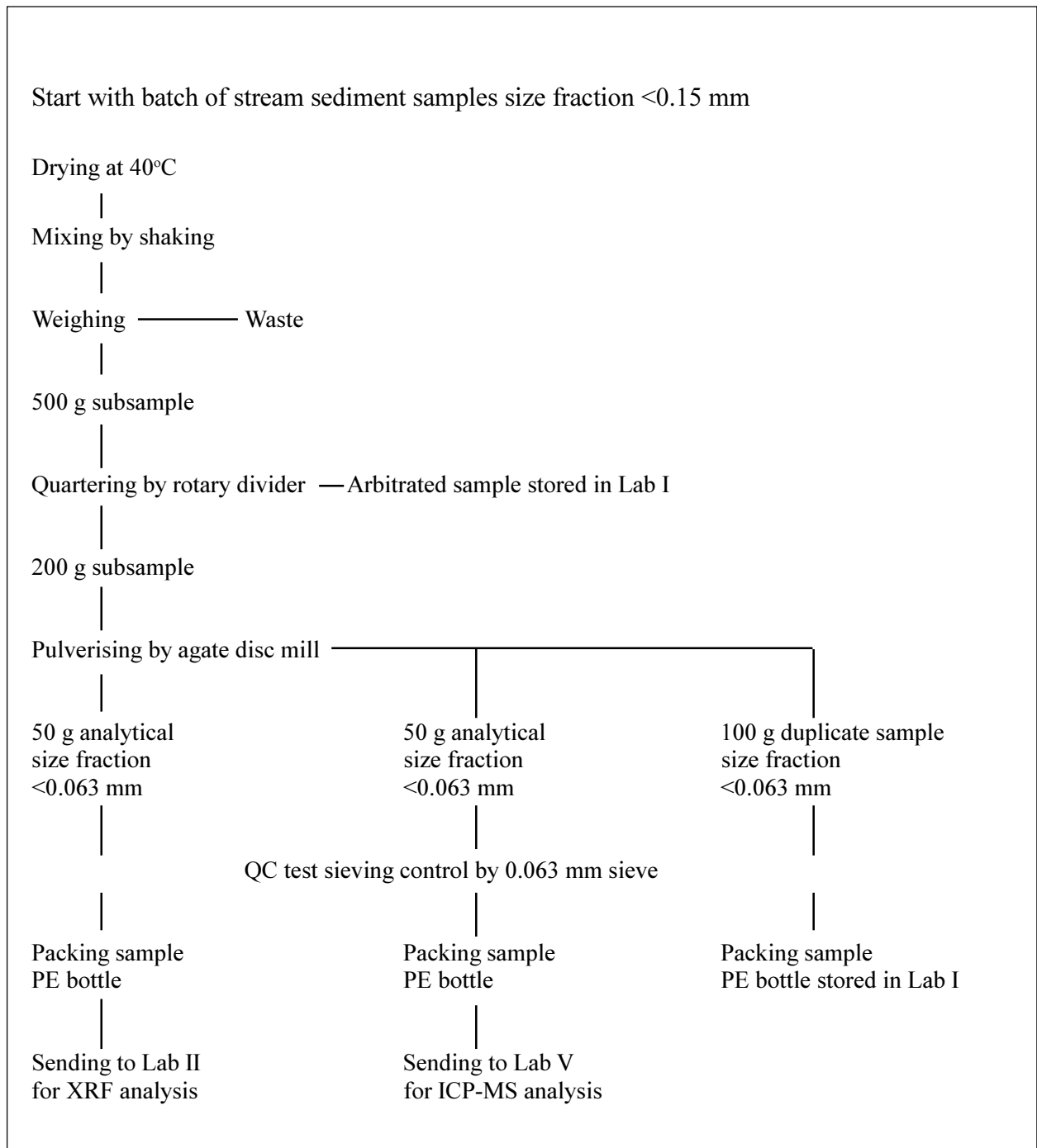


Fig. 13. Screening standard operating procedure for stream sediment samples.

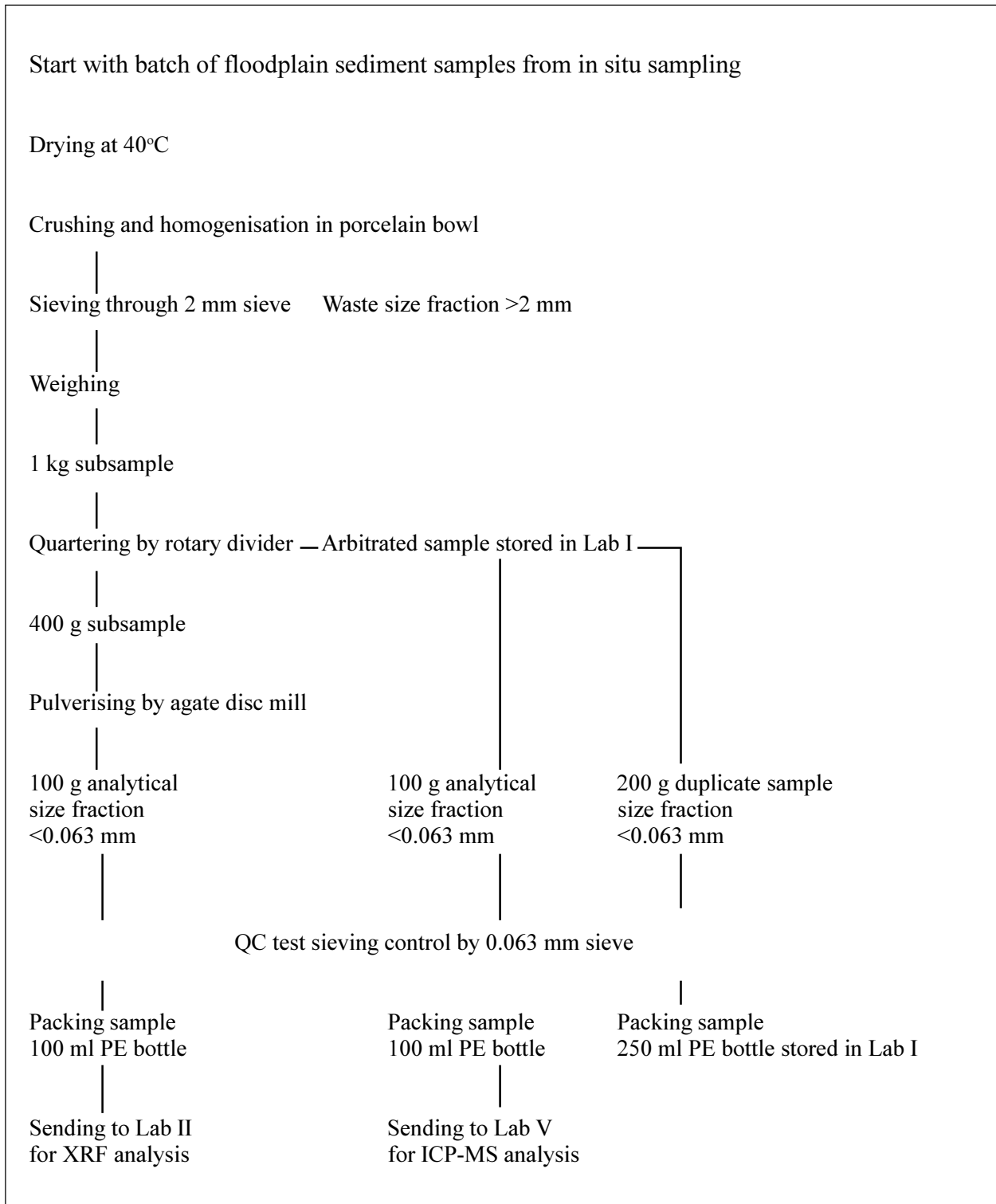


Fig. 14. Screening standard operating procedure for floodplain sediment samples.

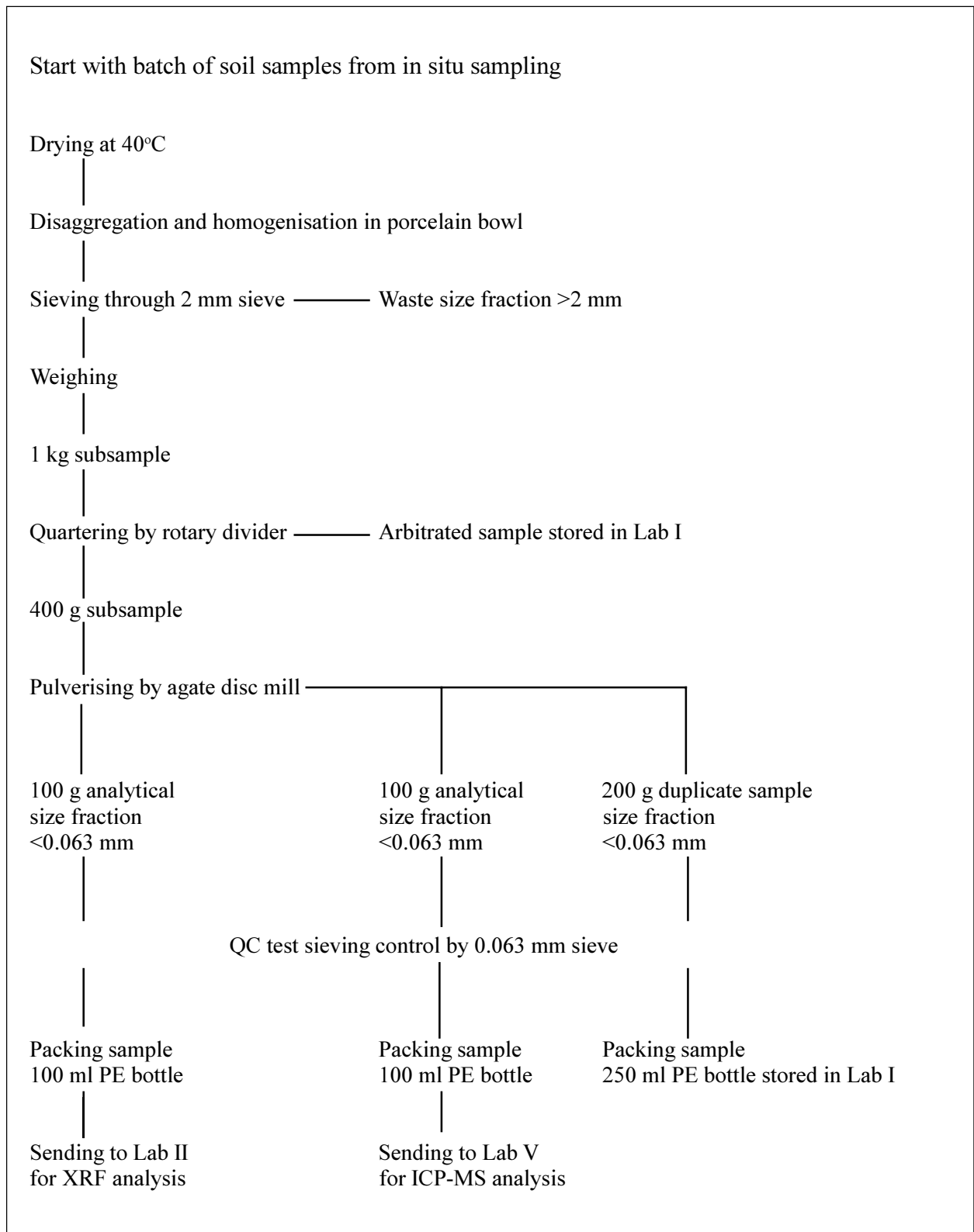


Fig. 15. Screening standard operating procedure for soil samples.

7. REFERENCES

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APPENDIX 1

Field observation sheets:

Stream water / stream sediment
Humus
Residual soil
Floodplain sediment
Overbank sediment

FOREGS GEOCHEMICAL BASELINE PROGRAMME

STREAM WATER+STREAM SEDIMENT

WATER SAMPLE ID _____ Date _____ Sampler _____

STREAM SEDIMENT ID _____ Country _____

Organisation _____

GTN cell coordinator if different from above _____

SAMPLE SITE LOCATION REGION _____ MAP SHEET _____

COORDINATES (Decimal degrees mandatory)

National grid Easting _____ Northing _____

Decimal degrees Longitude _____ Latitude _____ Datum _____

Altitude (m) _____

SITE DESCRIPTION

Approximate size of catchment basin _____ km²

Landscape / topography _____

Land use

 Agriculture, specify crop _____ Pasture, grassland, fallow field Forest: Deciduous Coniferous Mixed Wetland Non-cultivated, moorland etc. Other, specify _____Bedrock lithology _____ Outcrops Yes, specify _____ No outcrops

Type of overburden _____

Channel characteristics Natural Reinforced Man-made (ditch)

Last rainfall before _____ days _____ hours

Water level in stream: Low Normal HighStream flow: Low Moderate HighStream bed: Predominant Boulders and gravel Gravel and sand Sand and silt Mud Vegetation

Possible sources of contamination, specify _____

NUMBER OF SUBSITES (stream sediment) _____

NUMBER OF SAMPLE BAGS (stream sediment) _____

PHOTOS Film and photo ID

Landscape _____

Site _____

GAMMA-RADIATION Total _____ Th _____ U _____ K _____

Instrument _____

WATER CHEMISTRY Normal sample Duplicate sample Instrument

pH _____

EC mS/m 25°C _____

DRYING (Sediment) Freeze drying <40°C

FOREGS GEOCHEMICAL BASELINE PROGRAMME

SOIL

TOP SOIL ID _____ Date _____ Sampler _____

SUBSOIL ID _____ Country _____

Organisation _____

GTN cell coordinator if different from above _____

SAMPLE SITE LOCATION REGION _____ MAP SHEET _____

COORDINATES (Decimal degrees mandatory)

National grid Easting _____ Northing _____

Decimal degrees Longitude _____ Latitude _____ Datum _____

Altitude (m) _____

SITE DESCRIPTION

Landscape / topography _____

Land use

Agriculture, specify crop _____

Pasture, grassland, fallow field

Forest: Deciduous Coniferous Mixed

Wetland

Non-cultivated, moorland etc.

Other, specify _____

Bedrock lithology _____ Outcrops Yes, specify _____

No outcrops

Type of overburden _____

NUMBER OF SUBSITES _____

SOIL TYPE (FAO classification or local name) _____

Ploughing depth (cm) _____

Subsoil, specify soil horizon _____

Sampling interval (cm): Topsoil _____ Subsoil _____

Depth of groundwater table (cm) _____

ABUNDANCE OF SOIL CLASTS %	TOP SUB		TEXTURE	TOP SUB	
	<input type="checkbox"/>	<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>
0	<input type="checkbox"/>	<input type="checkbox"/>	sandy	<input type="checkbox"/>	<input type="checkbox"/>
0 - 2	<input type="checkbox"/>	<input type="checkbox"/>	sandy-loam	<input type="checkbox"/>	<input type="checkbox"/>
2 - 5	<input type="checkbox"/>	<input type="checkbox"/>	loamy	<input type="checkbox"/>	<input type="checkbox"/>
5 - 15	<input type="checkbox"/>	<input type="checkbox"/>	clayey-loam	<input type="checkbox"/>	<input type="checkbox"/>
15 - 40	<input type="checkbox"/>	<input type="checkbox"/>	clayey	<input type="checkbox"/>	<input type="checkbox"/>
40 - 80	<input type="checkbox"/>	<input type="checkbox"/>	clay	<input type="checkbox"/>	<input type="checkbox"/>
> 80	<input type="checkbox"/>	<input type="checkbox"/>			

SAMPLE HUMIDITY	TOP SUB		ORGANIC CONTENT	TOP SUB	
	<input type="checkbox"/>	<input type="checkbox"/>		<input type="checkbox"/>	<input type="checkbox"/>
wet	<input type="checkbox"/>	<input type="checkbox"/>	high	<input type="checkbox"/>	<input type="checkbox"/>
dry	<input type="checkbox"/>	<input type="checkbox"/>	low	<input type="checkbox"/>	<input type="checkbox"/>

Possible sources of contamination, specify _____

PHOTOS Film and photo ID

Landscape _____

Site _____

GAMMA-RADIATION Total _____ Th _____ U _____ K _____

Instrument _____

BALTIC SOIL SURVEY countries only: BSS sample ID _____ is used for this sample.

The BSS sample has already been sent to the NGU yes no, will be sent by _____

REMARKS

FOREGS GEOCHEMICAL BASELINE PROGRAMME**HUMUS**

HUMUS ID _____ Date _____ Sampler _____

Country _____

Organisation _____

GTN cell coordinator if different from above _____

SAMPLE SITE LOCATION REGION _____ MAP SHEET _____**COORDINATES (Decimal degrees mandatory)**

National grid Easting _____ Northing _____

Decimal degrees Longitude _____ Latitude _____ Datum _____

Altitude (m) _____

SITE DESCRIPTION

Type of forest, specify tree/scrub species in order of abundance:

1. _____ 2. _____

3. _____ 4. _____

Tree/scrub density: Typical distance between trees/bushes

 1 - 3 m 3 - 5 m 5 - 10 m > 10 mAge of forest: young mature

Underbrush character: Bushes _____

(Specify species if possible) Low vegetation _____

Underbrush/low vegetation coverage: < 20% 20-80% >80%

Substratum: _____

Total thickness of organic layer (cm) _____

Thickness of humus layer (cm) _____

SAMPLE COMPOSITION Raw humus Raw humus + humus Humus**NUMBER OF SUBSITES** _____**PHOTOS NOTE:** If the humus and residual soil samples are taken from same area and the organic layer is shown in the soil pit photograph, separate photographs for humus are not required. Photographs for humus sample were taken (see below) No separate photographs for humus

Film and photo ID

Landscape _____

Site _____

REMARKS

FOREGS GEOCHEMICAL BASELINE PROGRAMME**FLOODPLAIN SEDIMENT**

TOP ID _____ Date _____ Sampler _____

BOTTOM ID _____ Country _____

(Bottom floodplain sample is optional) Organisation _____

GTN cell coordinator if different from above _____

SAMPLE SITE LOCATION REGION _____ MAP SHEET _____**COORDINATES (Decimal degrees mandatory)**

National grid Easting _____ Northing _____

Decimal degrees Longitude _____ Latitude _____ Datum _____

Altitude (m) _____

DESCRIPTION OF CATCHMENT BASINApproximate size of catchment basin _____ km²

Landscape / topography _____

Land use

- Agriculture
- Pasture, grassland, fallow field
- Forest:
- Wetland
- Non-cultivated, moorland etc.
- Other, specify _____

Predominant bedrock lithology within catchment basin _____

SITE DESCRIPTION

River width _____ m, depth _____ m

Grain size range at sample site sand - silt silt - clay

Abundance of clasts > 2 mm in %: _____

Depth of observed groundwater table (cm) _____

Sampling interval from surface 0 - 25 cm other, specify: _____ cm

Possible sources of contamination, specify _____

PHOTOS Film and photo ID

Landscape _____

Site _____

GAMMA-RADIATION Total _____ Th _____ U _____ K _____

Instrument _____

REMARKS

FOREGS GEOCHEMICAL BASELINE PROGRAMME**OVERBANK SEDIMENT**

Optional sampling material

TOP ID _____ Date _____ Sampler _____

BOTTOM ID _____ Country _____

(Both samples are optional) Organisation _____

GTN cell coordinator if different from above _____

SAMPLE SITE LOCATION REGION _____ MAP SHEET _____**COORDINATES**

National grid Easting _____ Northing _____

Decimal degrees Longitude _____ Latitude _____ Datum _____

Altitude (m) _____

DESCRIPTION OF CATCHMENT BASINApproximate size of catchment basin _____ km²

Landscape / topography _____

Land use

 Agriculture, specify crop _____ Pasture, grassland, fallow field Forest: Deciduous Coniferous Mixed Wetland Non-cultivated, moorland etc. Other, specify _____

Predominant bedrock lithology within catchment basin _____

SITE DESCRIPTION

River width _____ m, depth _____ m

Grain size range at sample site sand - silt silt - clay

Abundance of clasts > 2 mm in %: _____

Depth of observed groundwater table (cm) _____

Sampling interval from surface 0 - 25 cm other, specify: _____ cm

Possible sources of contamination, specify _____

PHOTOS

Film and photo ID

Landscape _____

Site _____

GAMMA-RADIATION Total _____ Th _____ U _____ K _____

Instrument _____

REMARKS