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Design of monitoring of contaminated sites 2 – Sediments, soils, biota, technical matrices

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29/04/2021

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Target

Examples of different methods used for determination and monitoring of pollutants in sediment, biota, soils, and technical matrices

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Sediment sampling

Errors in sampling lead to large errors during resulting activities

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Soils and sediments

- High environmental burdens – important for global balances
- Slow response times
- Background soils and sediments reflect spatial differences in cumulative atmospheric deposition/net air-surface exchange
- Very heterogeneous - important questions of depth, ecosystem type etc.
- Can show spatial trends, but poor for time trends

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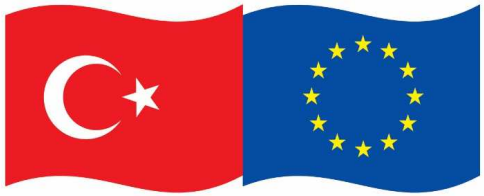
Sediments - introduction

- Heterogeneous polyphase system - silt, sand, gravel
- Secondary source of pollution
- Potential waste
- Selection of appropriate procedures and techniques
- Evaluation of sediment toxicity
- Proper sampling - a representative sample
- Improper sampling - bias to complete devaluation of the final result of the analysis
- Lack of standards and regulations (the use of sediment on agricultural land)
- U.S. EPA methodology



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Sediment sampling

Historical data

- collection of samples from depth horizons of bottom in the river where the moving of bottom is minimal

Data concerning to actual state:

- sampling by using of suitable sampler from upper horizons of bottom to 15 cm
- sampling by using of sedimentation bottles (traps) over bottom, on the bottom on in the level of bottom

The used method depends on the granularity of sediments.

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Sediment sampling

The most important:

- Sediment fraction with granularity less than 1 mm the main part of sorption of alogennous components
- Coloid and rough dispersions of endogennous and autigennous components.

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Sediment sampling

- very non-homogenous material
- great differences in granularity
- problematic sampling of representative sample

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Sediment sampling

- **Fine granular sediment** (fine gravel, gravel-sand, sand, sand-clay, clay, swamp) – sampling by using of check piston or overseal samplers from plastic, metals (glass).

- **Medium granular sediment** – sampling by using of grabs (dredgers) various construction and materials

- **Rough granular sediment** – sampling by using of grabs and freezing probes

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Selection of sampling equipments

- Purpose of sampling (fresh sediment, the upper layer of bottom sediment profile horizontal, vertical profile - history of contamination)
- Type of analysis
- Physico-chemical parameters and heterogeneity (grain size, stratification, pH, redox potential, cohesiveness)
- Availability of sampling sites (depth, width flow velocity)
- Amount of sample

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Types of samples

- **Physical state:**
  - gas
  - liquid
  - solid
- **Homogeneous or heterogeneous material**
- **Sampling plan:**
  - Representative
  - Selective
  - Random
  - Composite samples

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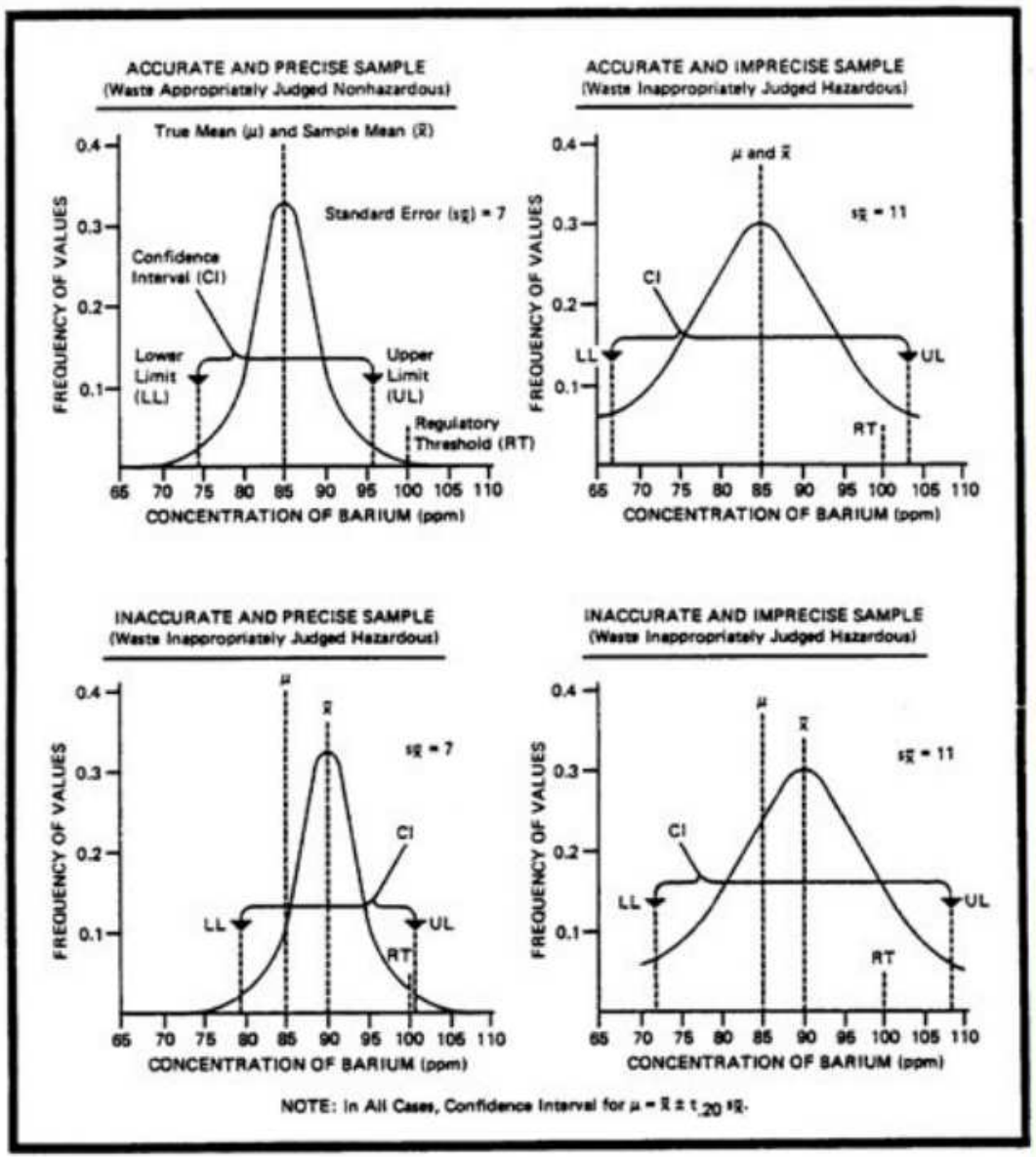
Representative sample

How do we get an accurate sample?

It must be one that accurately represents our material

Accurate sample



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Representative sample

This is a sample that is typical of the parent material for the characteristic under inspection

- knowledge of the method used for the analysis is also important.
- state of the parent material
  - Homogeneous
  - Heterogeneous
  - Static (contained)
  - Dynamic conditions

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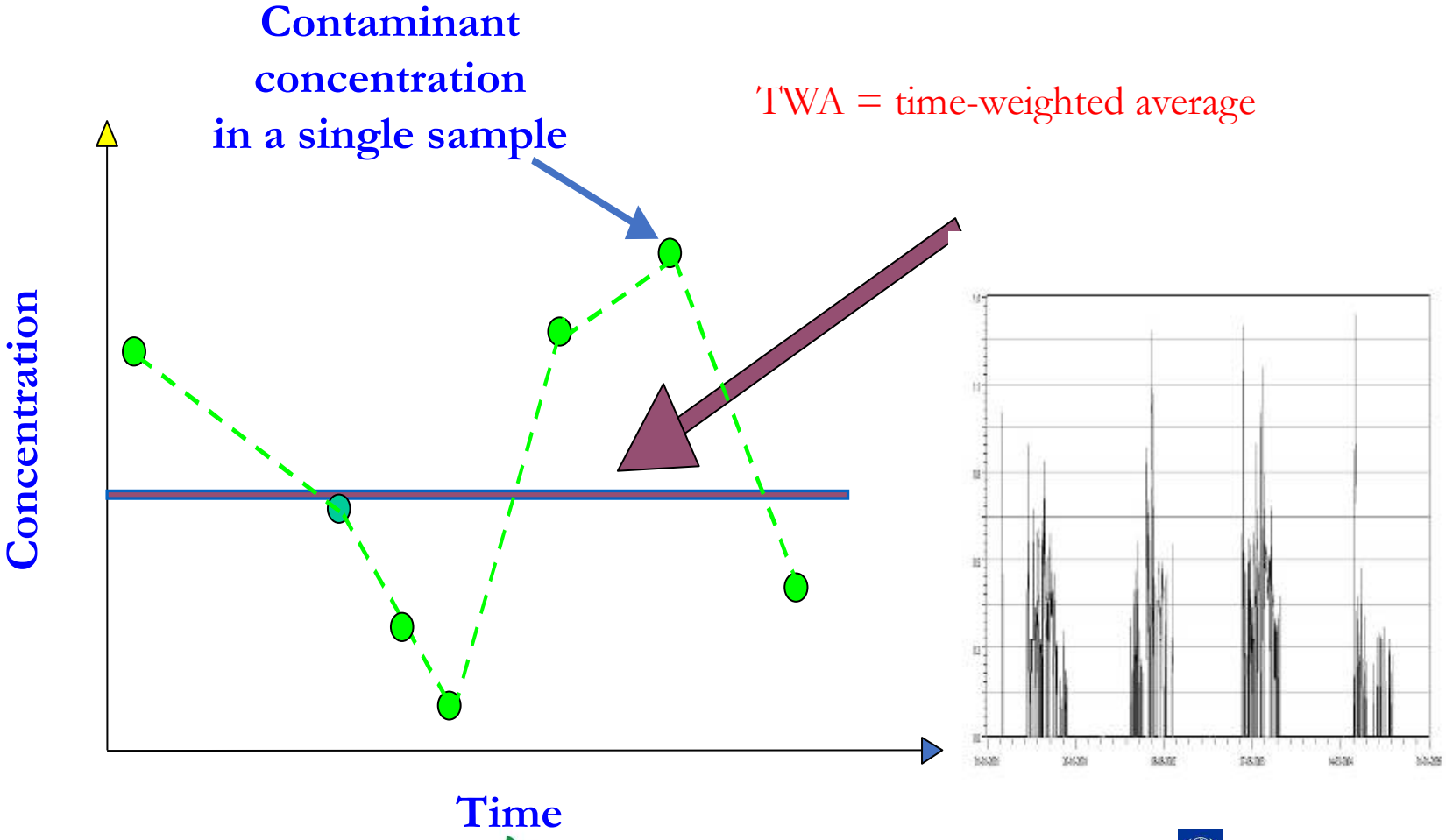
Dynamic system - contaminant in a river



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Selective sample

This is a sample which is deliberately chosen by using a sampling plan that:

- screens-out materials with certain characteristics and/or
- selects only material with other relevant characteristics.

This may also be called directed or *focused* sampling

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Composite sample

- **Composite sampling** is a way of reducing the cost of analysing large numbers of samples. A composite sample consists of two or more portions of material (collected at the same time) selected so as to represent the material being investigated.
- **The ratio of components** taken to make up the composite can be in terms of
  - Bulk
  - Time
  - Flow
- **The components of the composite sample** are taken in proportion to the amount of the material that they represent

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Sampling plan

IUPAC: ***"A predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as samples."***

- **Sampling schedule - when there is a regular requirement for analysis, the sampling plan is referred to as a sampling scheme or**
- **Sampling programme - a combination of procedures where several related sampling schemes are combined.**
- **Standards – e.g. sampling procedures for monitoring water quality (e.g. ISO 5667-1)**

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Sampling strategy

- Sampling plan = all aspects of the sampling process
  - Number of samples
  - Site
  - Sample volume
  - Instructions for the preparation of laboratory sample
  - Sampling frequency
- Sampling schedule
- Sampling program
- Legal requirements

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Sampling plan

- **Responsive to regulatory objectives**
  - **Representative samples – represent average properties of material sampled to be compared with regulatory thresholds (e.g. EQSs, drinking water standards etc.)**

- **Responsive to scientific objectives**
- **Sampling plan must demonstrate the variability of contaminants in time and space**
- **This may require a statistical approach to determining the number and distribution of samples to be collected**

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Sampling parameters

- Collection of a representative sample from a heterogeneous material
- Homogeneity test
- Sampling procedure must be validated for collected sample amount
- Analytical problem - control samples to check the procedure
- Problem that has to be solved

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Real environmental sampling

Sampling locations and number based on site conditions

- Surface and groundwater flow paths
- Known distribution of pollution sources
- Evidence of contamination
- Site access
- Historical data
- Cost

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Sampling plan components

Background information

- Site location
- Site description
- History
- General geology, hydrogeology, hydrology
- Known nature and extent of contamination

Sampling procedures

- Health and safety considerations
- Media specific sampling procedures
- Media specific sample handling procedures
- Record keeping and QA/QC

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Methods involving preparation prior to transport to field

- Passive samplers are subject to contamination with pollutants during preparation
- Exhibit variable background levels that need to be estimated by using construction blanks
- Similar problems are met with biomonitoring where test organisms, even from a relatively clean environment, have some background contamination
- Estimated using random sample from pool of available test animals
- For passive sampling it is also necessary to use field blanks that are transported to the deployment site, and exposed during the deployment and retrieval processes in a manner similar to the test samplers.
- BSI (2006) PAS 61 gives guidance for passive samplers

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Natural and technical conditions of sampling

Locality – screening determination of area in the map, km river profile, name of village in the surroundings, reservoir, tributary etc.

Object - well, bore, spring, source, basin

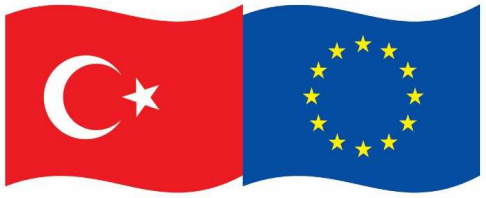
Sampling site – exact determination – areal or spatial

Individual sampling - 1 sample evaluated individually

Line sampling – few samples in temporal and spatial consequences in ratio to flow

Single sample – sampling of suitable volume of water in selected point and time

Mixed sample – mixing of the same volumes of single samples (it is a possible also from continuous sampling)



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Climatological conditions of sampling (meteorological)

Intensity of deposition – local effects on water composition

Low temperature – disabling of sampling

High temperature – distort the determination of gases and volatile compounds

Wind + dust, sand – contamination of samples, damage of measuring equipments

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Sampling plan components

Health and safety considerations

- Toxicity characteristics of contaminants
- Hazardous site conditions
- Equipment hazards
- Natural hazards
- PPE

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Types of sampling

Probability Sampling

- **Simple random sampling** involves taking increments from the bulk material in such a way that any portion of the bulk has an equal probability of being sampled
- **Stratified sampling** requires the consignment to be subdivided into groups (strata) according to predefined criteria
- **Systematic sampling** involves taking increments from the bulk material at predetermined intervals, as defined by the sampling plan.

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Types of sampling

Non-Probability Sampling

- **Judgement sampling** involves using knowledge about the material to be sampled, and the reason for sampling, to select specific samples.
- **Quota sampling** requires the consignment to be subdivided into groups (as for stratified sampling described previously). Once the material has been grouped, judgement sampling is used to select samples from each group.
- **Convenience sampling** involves selecting samples on the basis of availability and/or accessibility.

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Types of sampling

Bulk Sampling

- Involves the taking of a sample from material which does not consist of discrete, identifiable or constant units. The bulk material may be gaseous, liquid or solid.

Acceptance Sampling

- Acceptance sampling involves the application of a predetermined sampling plan to decide whether a batch of goods meets the defined criteria for acceptance either by attributes or by variables

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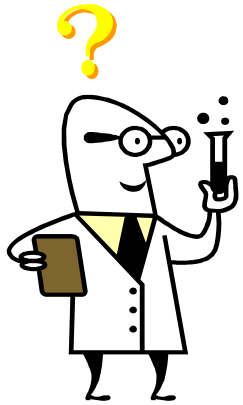
Sample numbers and sample size

The sampling plan should specify

- the number and size of primary samples which need to be obtained from the lot/batch.
- how the laboratory sample is to be obtained
- how the validity of any analysis will be affected



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Number of primary samples

- Sector specific
- Empirical rules often used
- Number of samples to be taken from a lot:

$$n = 3 \times \sqrt[3]{N}$$

- Lot/batch size
- Inspection level (single, double, multiple sampling
- Inspection type (normal, tightened or reduced)
- Acceptance quality limit (AQL)

Tabulka 3.5a. *Počty primárních vzorků, odebíraných z konzervovaných výrobků*

Počet plechovek, balení nebo obalů v dávce	Nejmenší počet primárních vzorků
1 – 25	1
26 – 100	5
101 – 250	10
> 250	15

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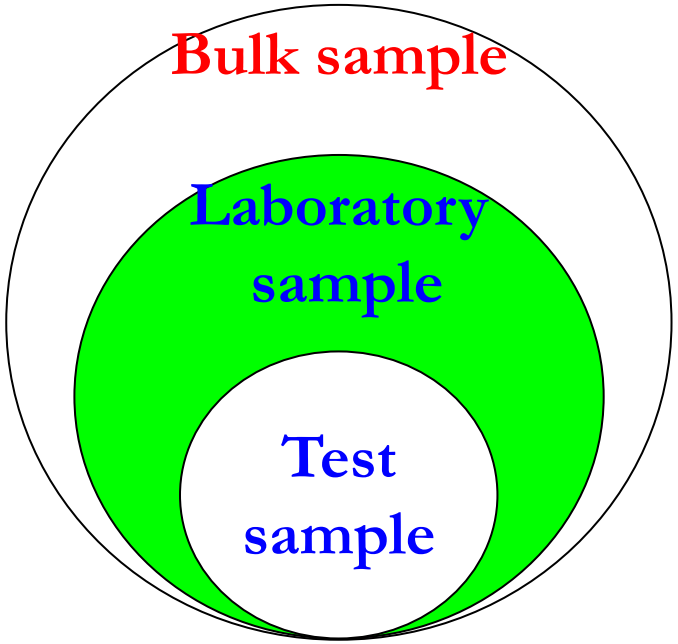
Subsampling

- A subsample is a portion of a sample, prepared in such a way that there is some confidence that it has the same concentration of analyte as that in the original

- There should not be any significant inhomogeneity between subsamples

- Error becomes more important as the concentration of the analyte of interest diminishes



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Sampling constant K_s

- minimum size of a subsample
- estimate the minimum size of the test portion
- test portion size must be confirmed as part of method validation
- mass of the test portion necessary to ensure a relative subsampling error of 1%

$$C_v = \frac{100s}{\bar{x}}$$

$$CV = \sqrt{\frac{K_s}{m}} \text{ or } K_s = (CV)^2 m$$

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Top layer sediment sampling

- For chemical status assessment
- Only few mm of top sediment layer
- Sampling of fresh sediment
- Use showel or a spoon
- Sieving through 2 mm mesh sieve onsite

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Stagnant sediment sampling

- **Core samplers**
  - Sediment corer Beeker type
  - Multisampler
  - Piston sampler
  - Free fall corer
  - Peat sampler (takes a half round core)
  - Vrijwit auger
  - Core samplers
- **Grab samplers for top sediment layers:**
  - Van Veen grabs
  - Ekman grab
- **Grab samplers for stagnant water:**
  - Kemmerer water sampler
  - Van Dorn water sampler

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Flowing sediment sampling

- Watertrap (all water levels except river bed)
- Delft bottle sampler (water near bottom)
- Arnhem bed load sampler (stones, gravel, coarse sand)

Various

- Secchi disc (visibility)
- Turbidity meter (turbidity)
- Sediment level stave ("feels" sediment level)
- Sediment detection pole (optical)

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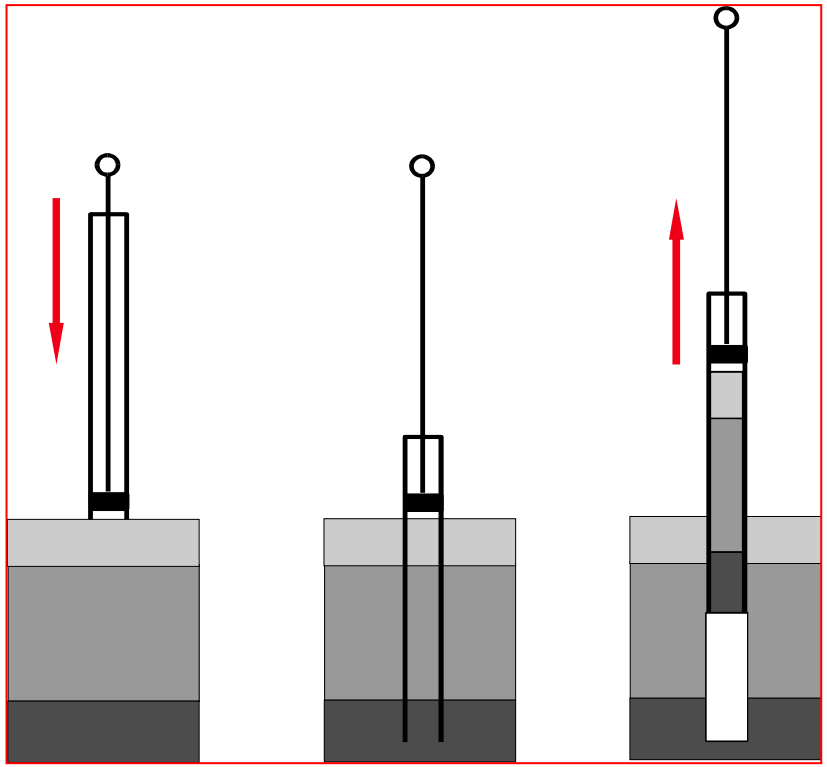
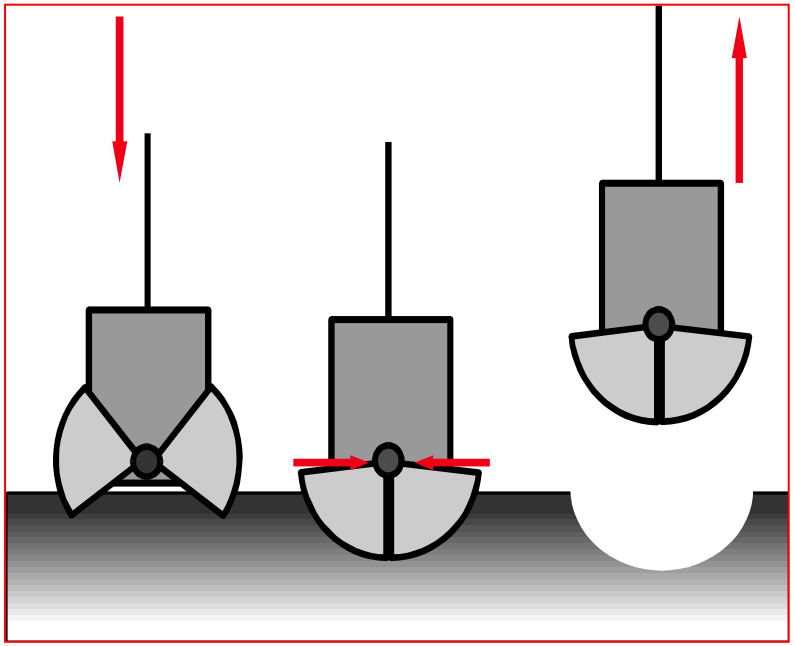
Sediment sampling



Piston probe



Grab probe

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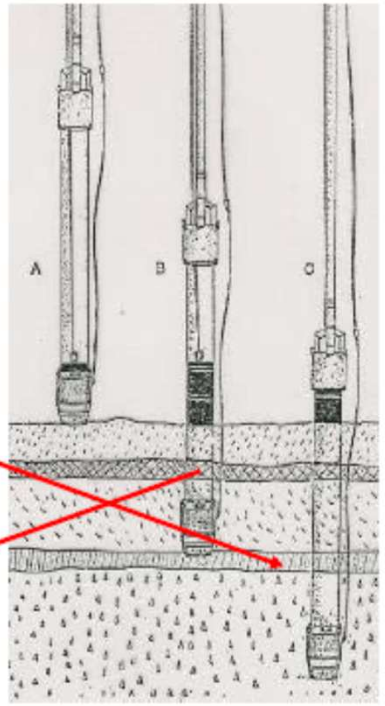
Stagnant sediment sampling

- Working principle of core sampler with a piston.
  - Aim: Take an more or less undisturbed column of the sediments.
  - Without piston the sample will be compressed during sample taking
  - Piston is located at a stationary position. Sample tube is pushed down.
  - Sampler will show full length of sample.



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Stagnant sediment sampling

- 04.23.SA + SB Sediment corer Beeker type
  - Benefits
    - No loss of sample in **any** case
    - Cutting head can be closed with air pressure
    - **Piston** assures identical core sample length
    - Can be **hammered** to sample all sediments
    - For sample lengths up till 150 cm
    - **Transparent**: Easiest profile description
    - Contaminant free stainless steel body
    - Set B allows for 10cm sub sample transfer
  - Remarks
    - Cannot be used in unsaturated sediments (piston !)

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Sediment sampler Beeker type

- **Main applications**
  - Core sampling (<1.50 m) at depths < 10 m of soft and harder sediments
  - Core sample must be expelled in the field
- **Main sectors of application**
  - Waterboards, dredging and engineering coms, harbour, lake and dam authorities, environmentalists, schools
  - Also scientists

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Sediment sampler Beeker type - operation








Sediment sampler Beeker type - operation

- Taking sediment samples (photo's: Beeker sampler old type)

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Stagnant sediment sampling

- 12.42 Multisampler
  - Benefits
    - For sediment sampling with a small budget
    - Professional tool with transparent tube
    - Open cutting head for undisturbed samples
    - Ball valve to sample sewers, bassins a.s.o.
    - Piston assures perfect core sample length
  - Remarks
    - No hammering possible
    - Loss of sample in watery or coarse sediments
    - Cannot be used in unsaturated sediments

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Stagnant sediment sampling

- 01.09 Piston samplers
  - Benefits
    - Thin walled: Lowest penetration resistance
    - Samples up to two meters of saturated sand
    - Various lengths available
    - Straight forward use
  - Remarks
    - Coarse sands or watery sludge will flow out
    - No control over correct sampling of top layer (invisible)
    - Risk of compaction or de-mixing by user
    - Cannot be used in unsaturated sediments (piston!)
    - Check correct operation of piston regularly (invisible)

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Vrijwit auger

- 04.22 Vrijwit auger
  - Benefits
    - Perfect for shallow water bottoms with **roots**
    - Sharp stainless steel body for all soils and analyses
    - Takes a beautiful rectangular sample
    - Wedge shape and core catcher prevent sample loss
  - Remarks
    - Double rods are unhandy in deeper waters

- Main applications → PKD
  - Root and top soil research in estuaries, shallow lakes

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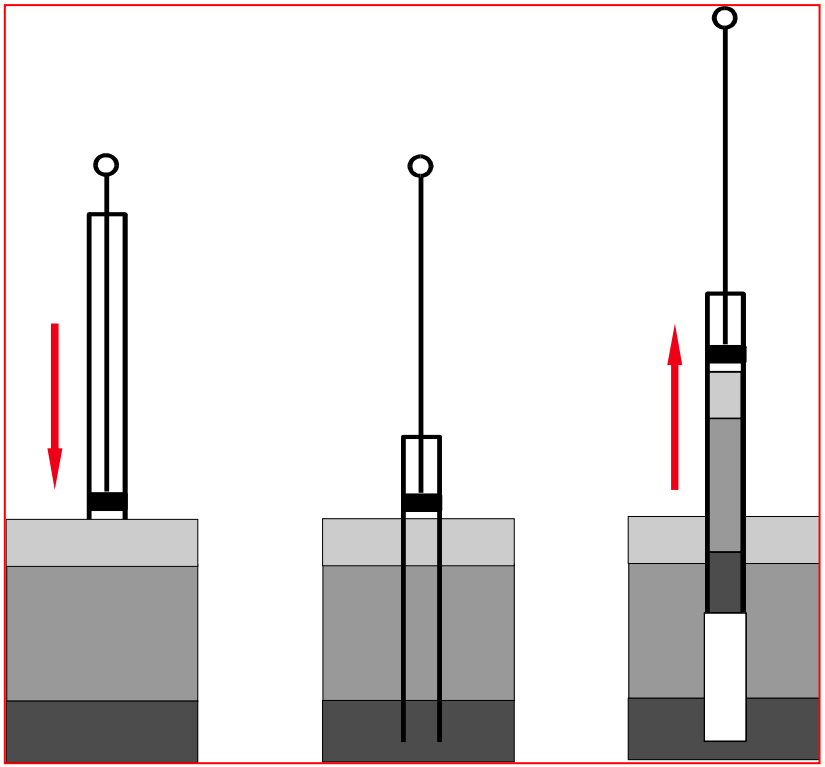
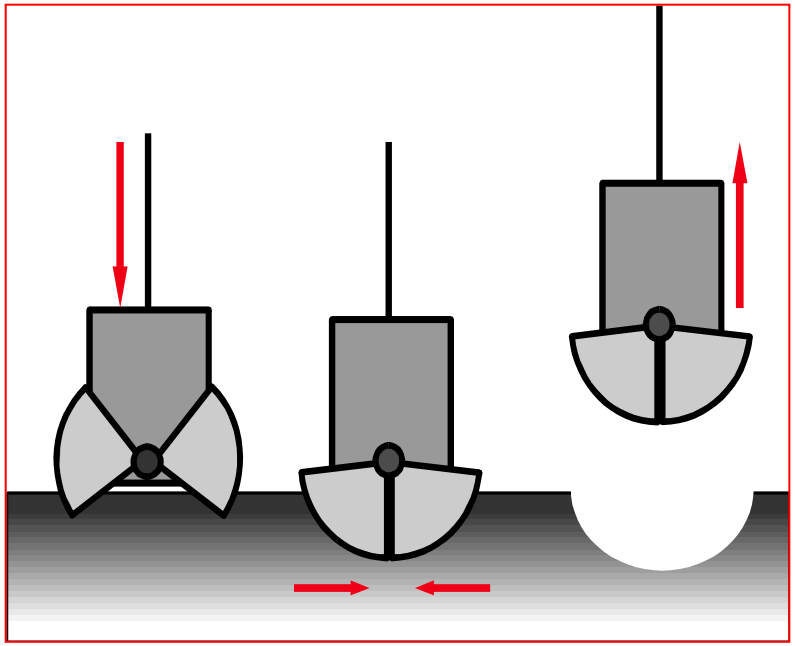
Sediment sampling



Piston probe



Grab probe

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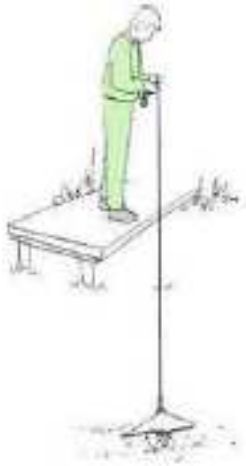
Van Veen grabs

- **Benefits**
  - Fastest indication of sediment type
  - Inert stainless steel construction
  - Anyone can do the job



- **Remarks**
  - Will sink through (and miss) “thick water” layer.
  - Larger models operated with a winch

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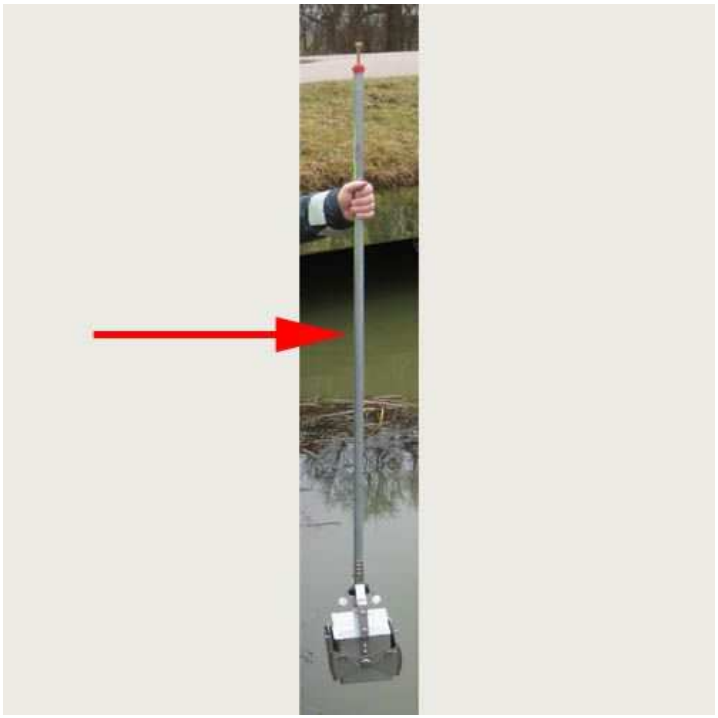
Van Veen grabs

- 04.30 Van Veen grabs
  - Main applications → PKD
    - Rapid indication of top layer (if not thick water)
    - Deeper waters
  - Main Markets → PKD
    - Engineering coms, water boards, dredging companies, harbour, lake and dam authorities, environmentalists

Ekman grab

- Ekman grab, stainless steel, dim. 152x152x152 mm, contents 3.5 litres, complete with messenger, 30 m synthetic line and carrying case.
- Handle for Ekman grab, length 150 cm.
- Main applications
  - Accurate square sample of top layer
  - Shallow waters with rod
  - Deeper waters with line
- Main users
  - Engineering coms, water boards, dredging companies, harbour, lake and dam authorities, environmentalists



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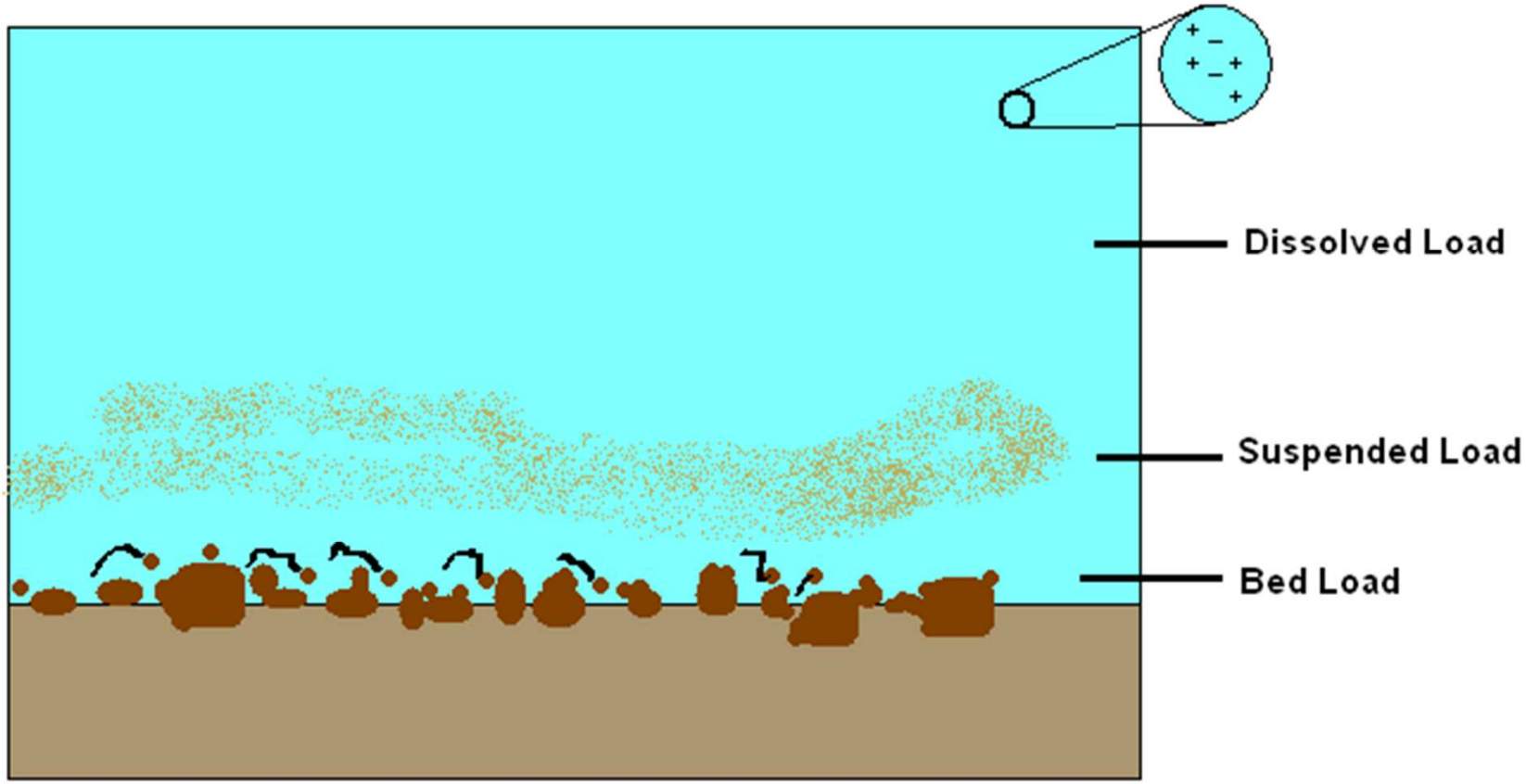
tarafından ortaklaşa finanse edilmektedir.

Sediment sampling (flowing)



Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Suspended particulate matter trap

Principle: partial diversion of the water flow to a vessel, where the flow velocity is decreased and particles sediment

System supports coagulation of particles, which enables separation efficiency up to 70%.

Sampling 14 days

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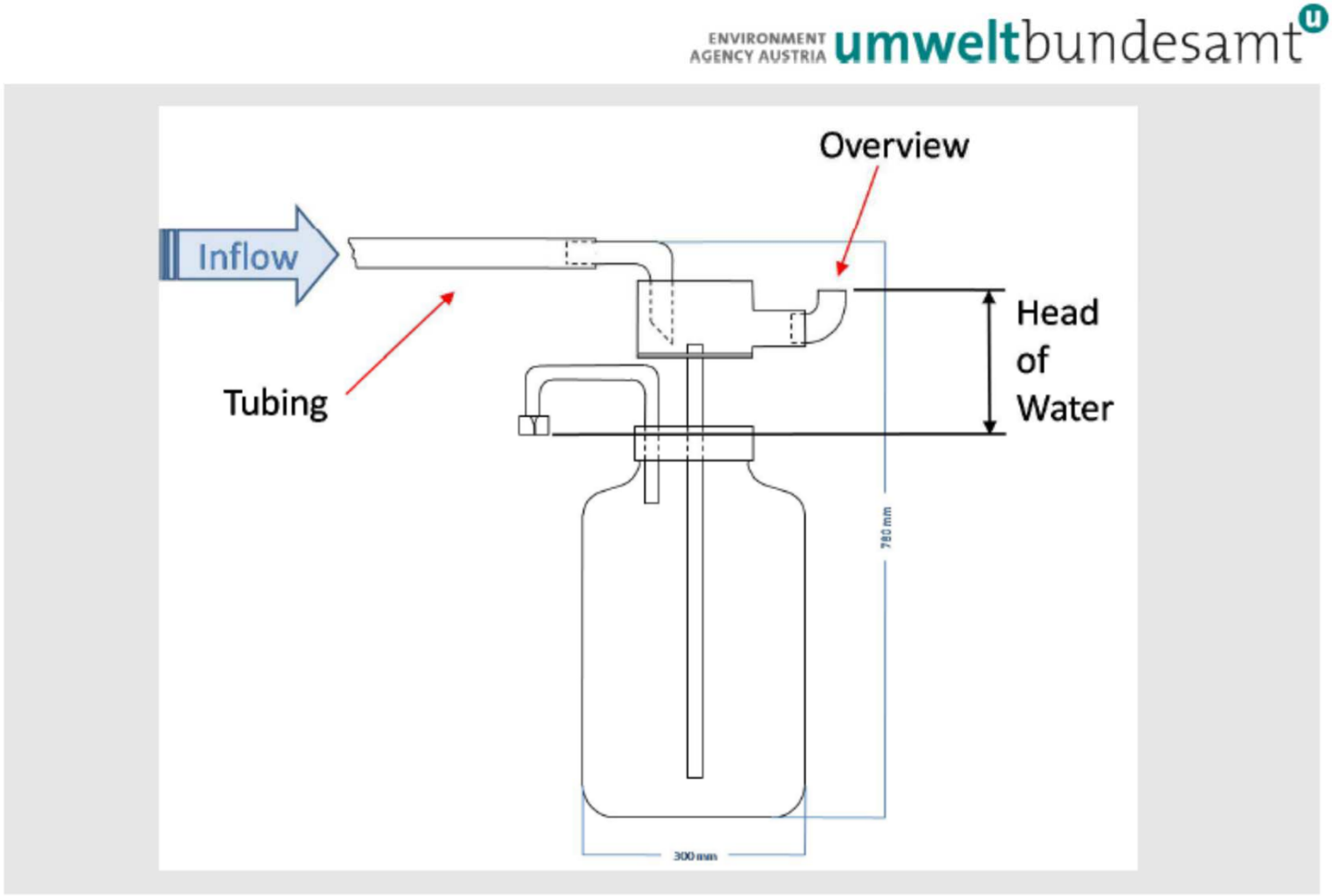
Principle of the suspended sediment trap



Suspended sediment trap

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Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti
tarafından ortaklaşa finanse edilmektedir.

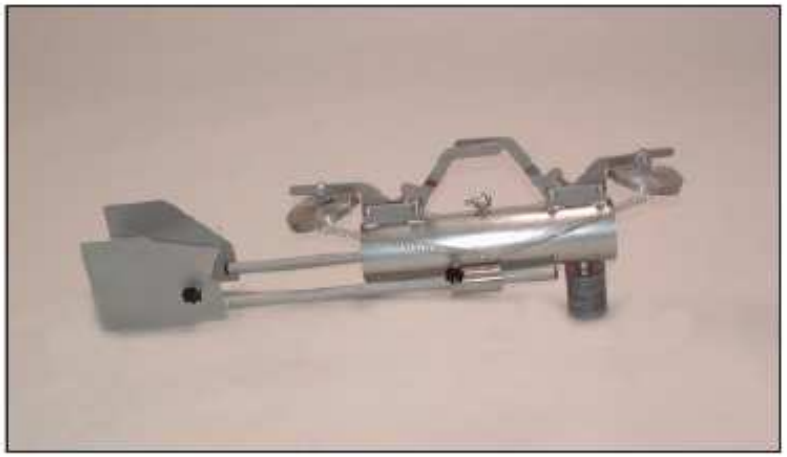
Time proportional suspended matter traps

- Combination of advantages of water and sediment sampling
- Sedimentation time known
- Calculation of fluxes possible
- Lots of substances adsorb to suspended matter particles
- Pumping necessary if there is no slope

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Sediment sampling (flowing)

- Watertrap
- Benefits
  - Easiest determination of sediment load
  - Simple operation: Lower, drop messenger and retrieve sample
  - Cable operated; works at any depth
  - Stainless steel construction
- Main applications
  - River research on sediment transport
- Main users
  - Engineering coms, water boards, harbour, lake and dam authorities, erosion authorities, biologist
- Remarks
  - Not for strong currents



Suspended load sampler Delft Bottle type

Benefits

- Frame lowered on bottom
- Accuratesampling height from the bottom
- Bronze body, sea water proof

Main users

- Engineering companies, water boards, harbour, lake and dam authorities, erosion authorities, scientists

Remarks

- Only from larger boat with davit and winch

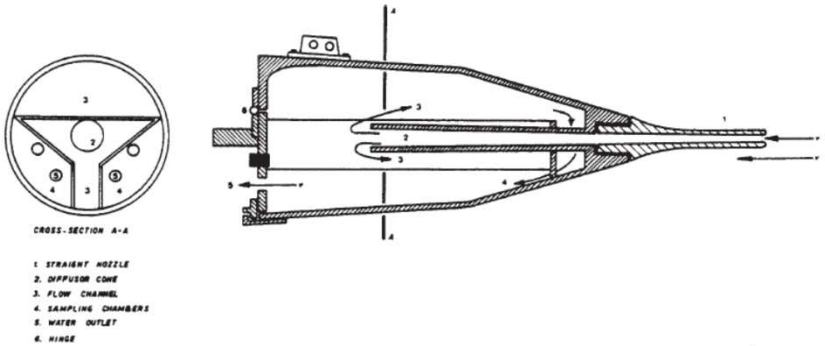


Figure 1 Longitudinal section of the sampler

Suspended load sampler Delft Bottle type

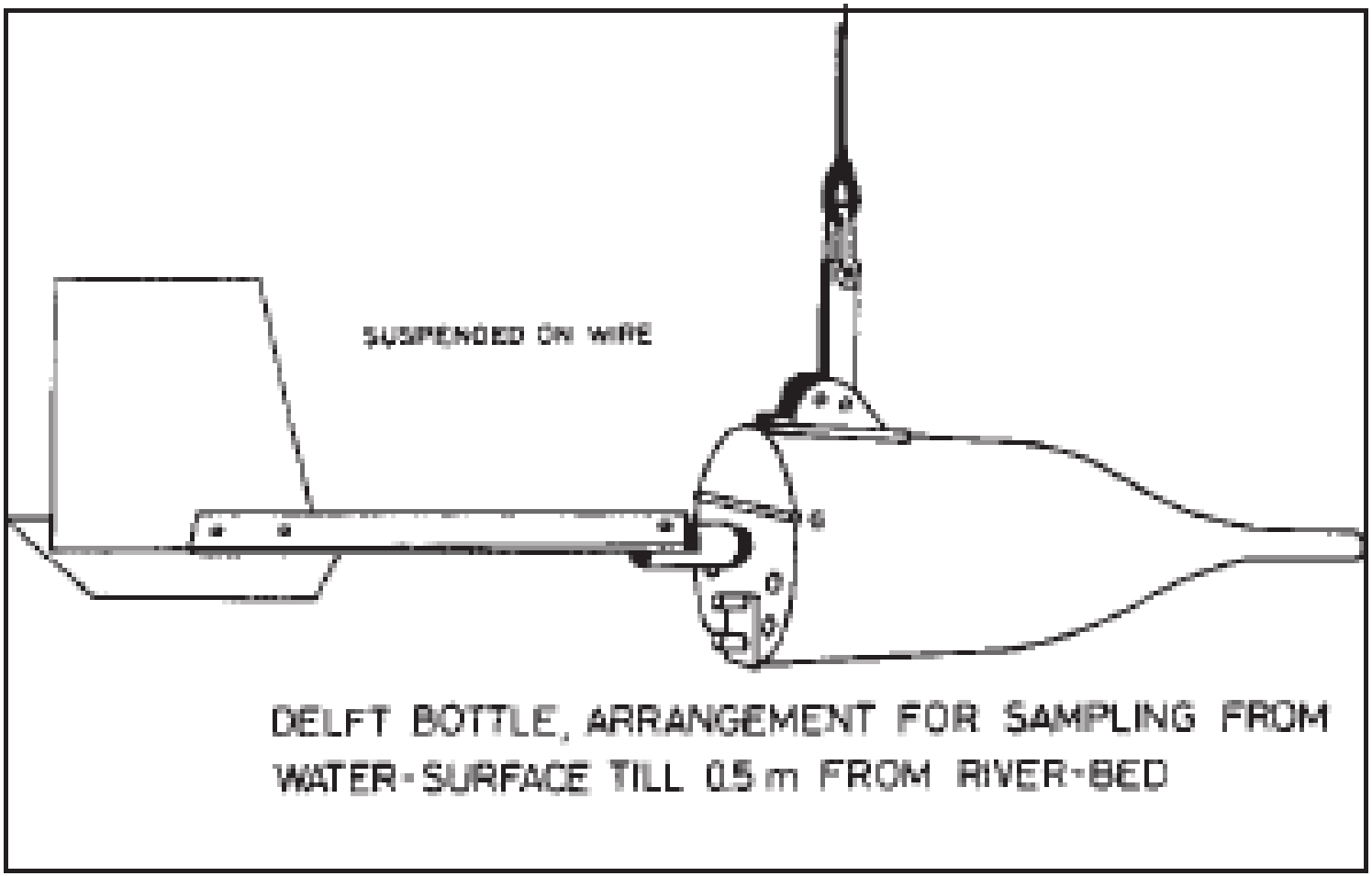
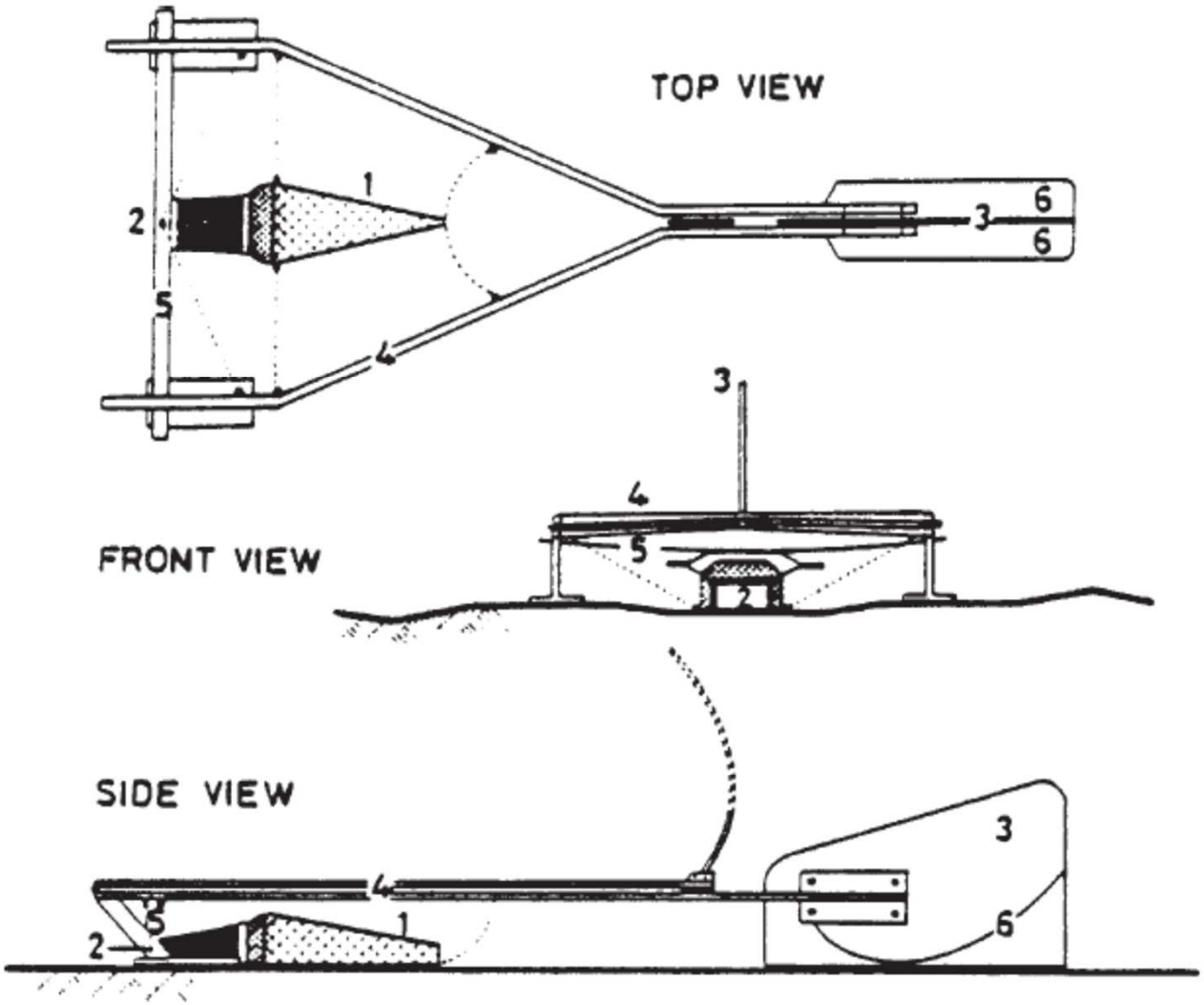


Figure 2 *Sampler suspended on a cable*

Bed-load transport meter Arnhem type

- Collects stones, gravel and coarse sand from river bed while water flows through collector..
- Streamlined
  - The streamlined sampler is mounted in a frame and consists of a mouth followed by a basket of fine wire meshing (width of the mesh 300 mu).
  - the sharp decrease of the velocity in the wide sampling chambers causes the sediment material to settle there
  - A tail fin mounted on the frame keeps the sampler in up-stream direction
  - The results of the measurements are influenced by the shape of the bed (ridges, dunes, flat beds, etc.)
- For a successful measurement knowledge of the relief of the bed is essential. The sampler can be discharged on board using the sample trough.

Bed-load transport meter Arnhem type

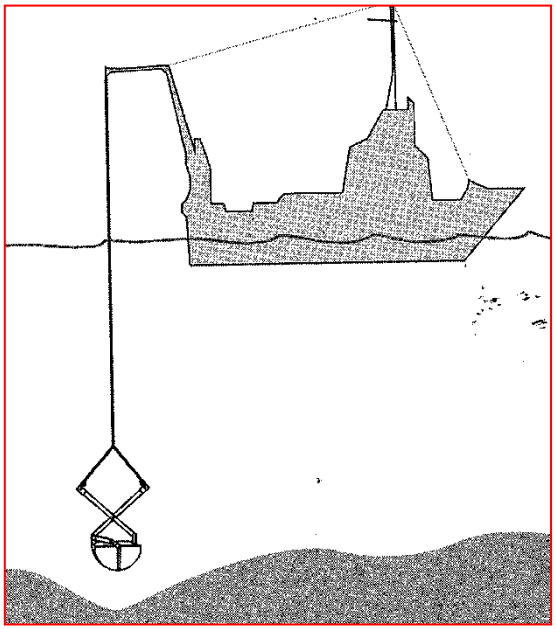


Sampling equipments

Subscribe upper sediment layer

Grab samplers (Ekman, Ponar, Van Veen)

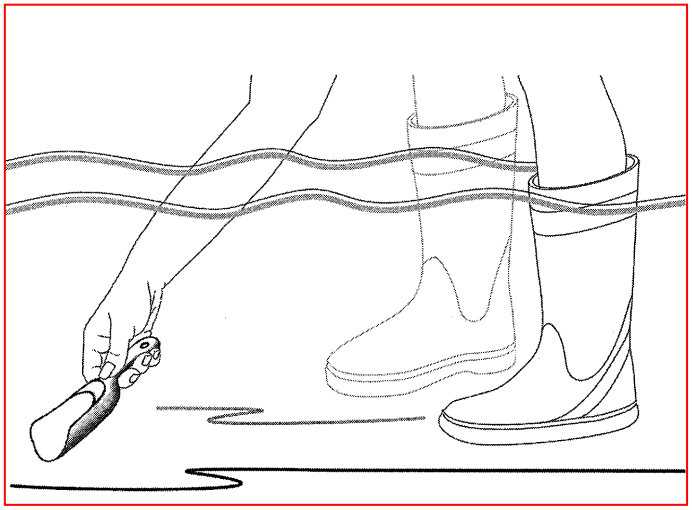
- Easy handling
- Disrupted sediment - when passing through the water column loss
- Fine particles



Sampling equipments

Subscribe upper sediment layer

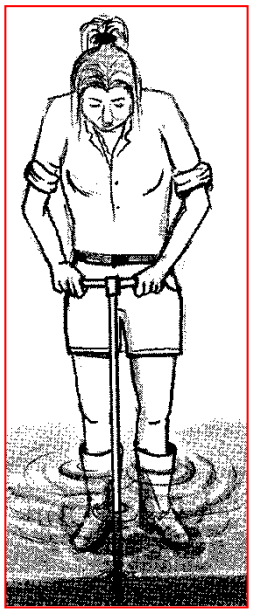
- stainless steel or plastic scoop
- smaller thickness of the layers of sediment (small streams)
- smaller depth
- less material

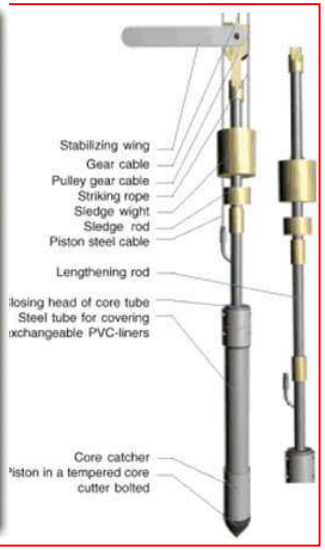


Sampling equipments

Subscribe vertical profile

- core (piston) samplers (freely falling LSS, Beeker)
- samplers for large depth (Livingstone, Niederreiter)
- history of contamination
- geological characterization
- determine the depth and zonal stratification
- ecotoxicological bioassays, toxicity testing of individual layers
- minimization of contact of the sample with oxygen





Sampling equipments

Consumption of fresh sediment

- Sediment trap
- Sampling (particle sedimentation) extends a certain time - captured material represents the actual gain in a given time
- Toxicology potential sedimented material
- Susceptibility to resuspension and pollution flow again

Sampling equipments

Sampling of suspended solids:

- Watertrap water probe (measuring transmission solids)
- Type Delft bottle (measurement of sediment transfer)
- Arhem type (measurement of suspended sediments of sand and gravel)

Processing and preservation of sediment samples

After collection:

- The removal of impurities (stones, branches, leaves)
- Bottle (glass, PTFE, HDPE)
- Transport (samples in a cool dark place)
- Transport fridge
- Storage
- Immediate analysis
- Freezing -20°C

Sediment sampling – sediment depth collected by different samplers under optimal conditions

Sediment Depth Collected by Different Samplers Under Optimal Conditions (About 2 m of Fine-Grained Sediment)

Sediment depth sampled	Sampling equipment
0—10 cm	Lightweight, small-volume grabs (for example, Birge-Ekman, Ponar and mini-Ponar, mini-Shipek)
0—30 cm	Heavy, large-volume grabs (for example, Van Veen, Smith-McIntyre, Petersen)
0—50 cm	Single gravity corers (for example, Kajak-Brinkhurst and Phleger corers)
	Box corers
	Multiple corers
0—2 m	Single gravity corer (for example, Benthos and Alpine corers)
Deeper than 2 m	Piston corers

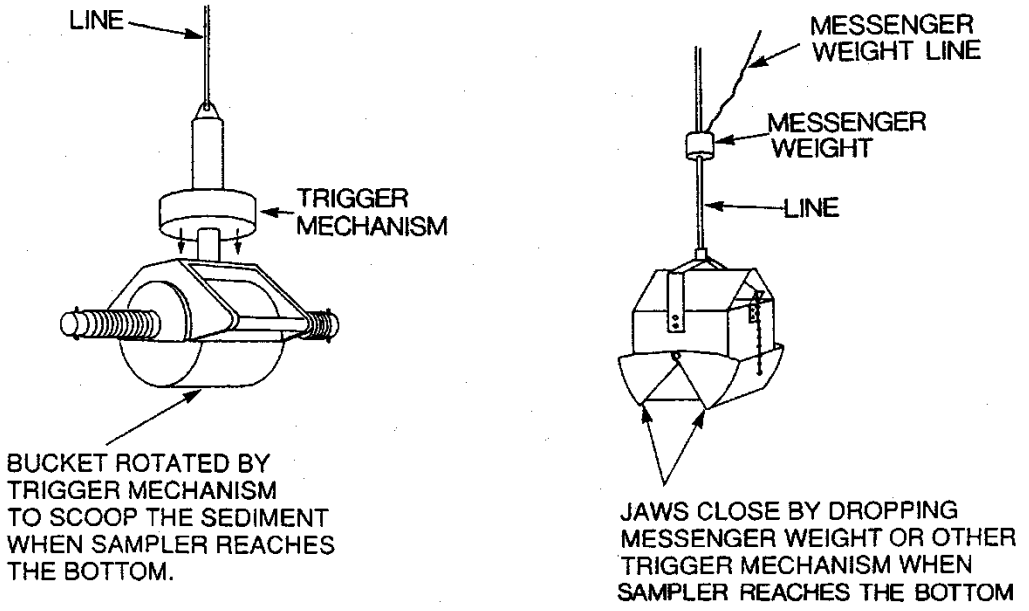


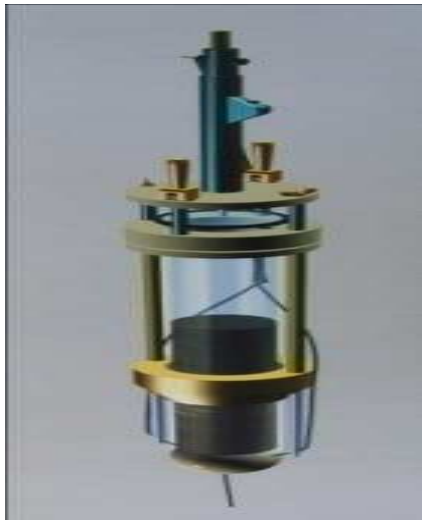
FIGURE 1. Grab samplers with their essential parts.

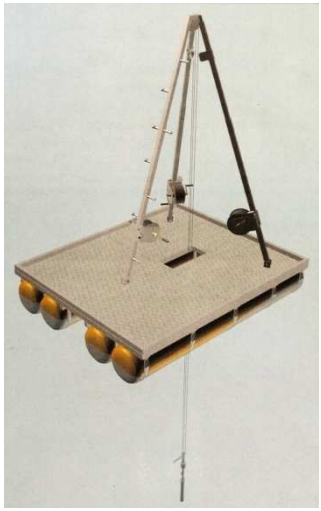
Sediment sampling

Probe for sampling of fine compact sediment

Sediment sampling

Deep probe

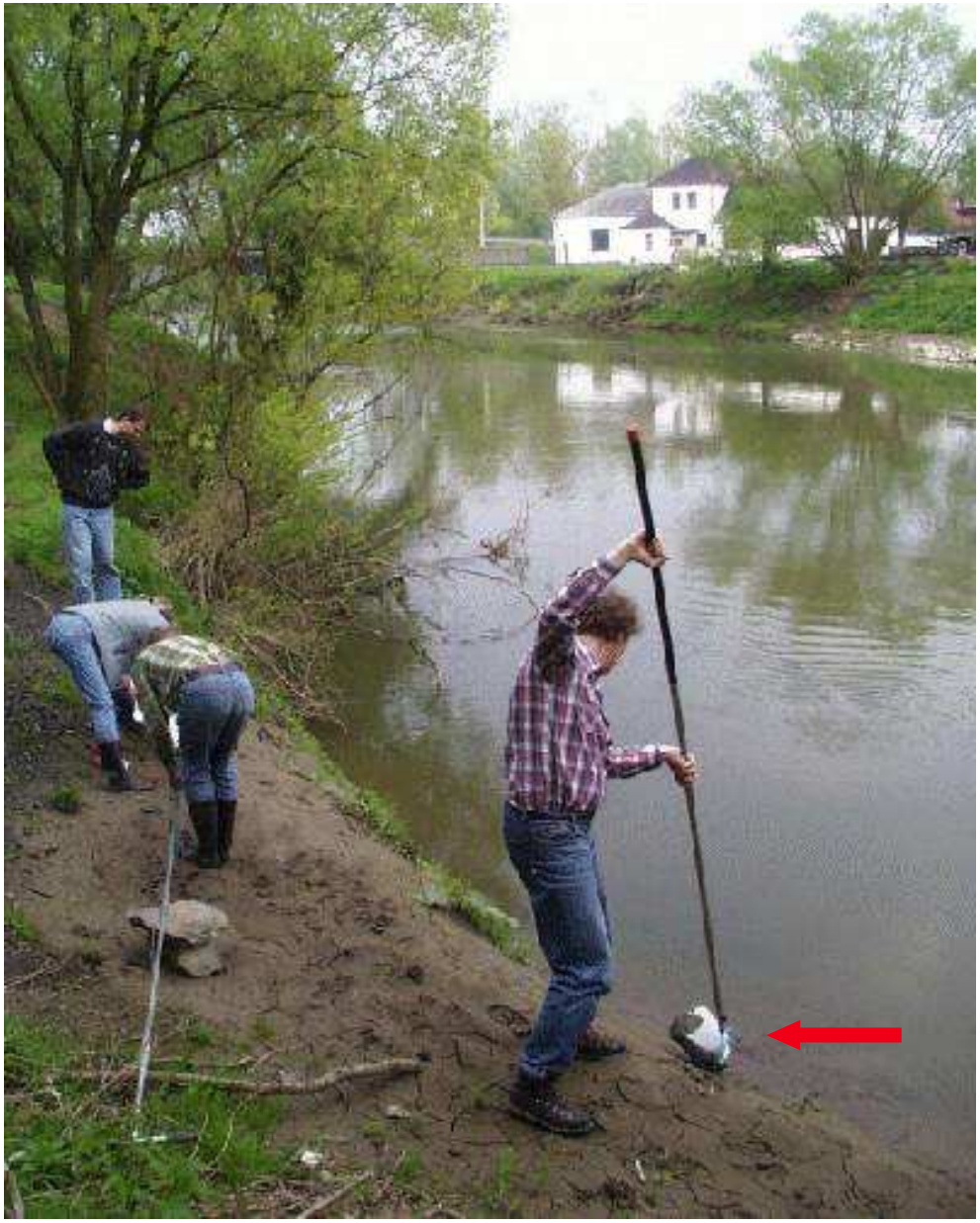




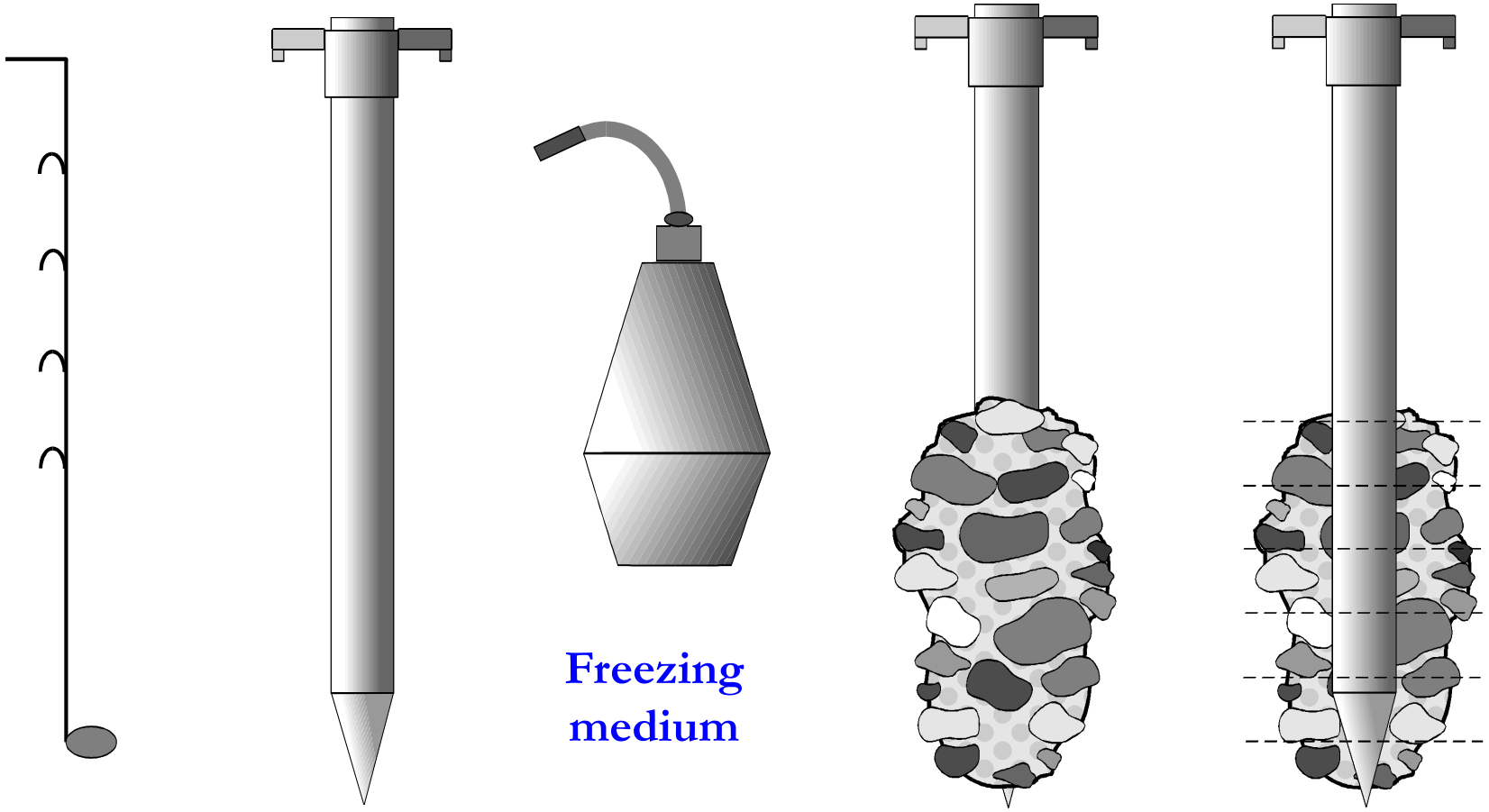
Sediment sampling

Grab probe

Sediment sampling by „scraper“ sampler



Freezing sampling



Disperser of media flow

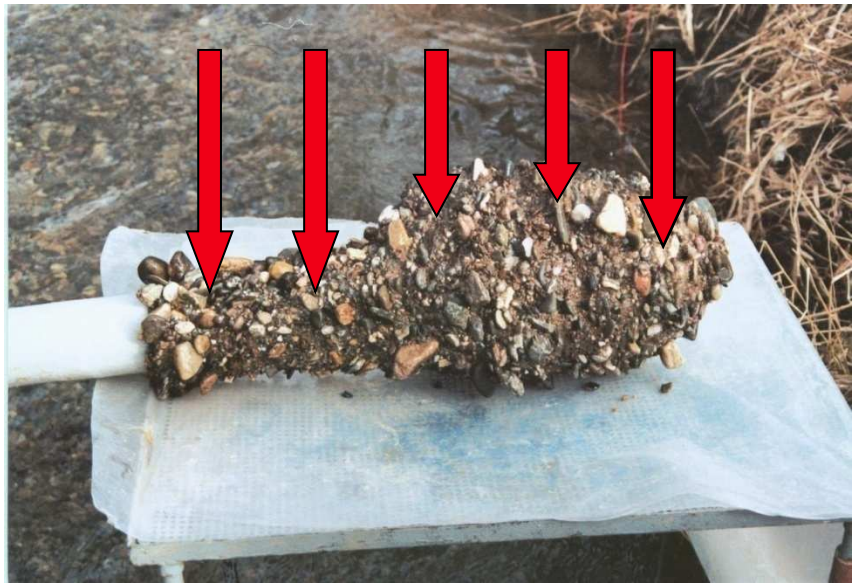
Tube probe

Freezing medium

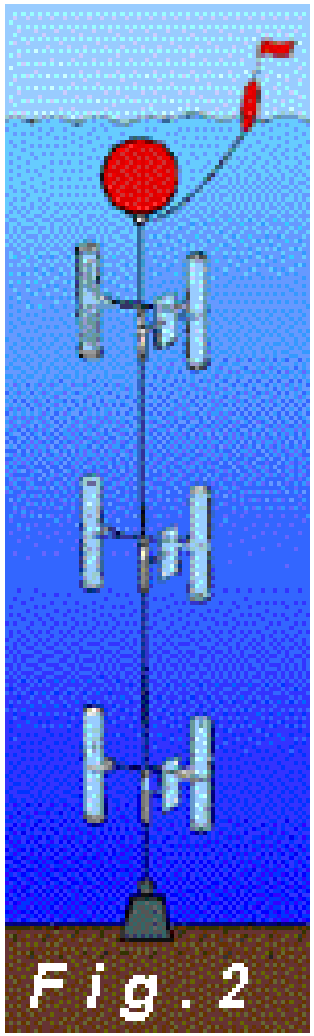
Frostbitten sediment

Freezing sampling – installation of probe and whip out of frostbitten sediment

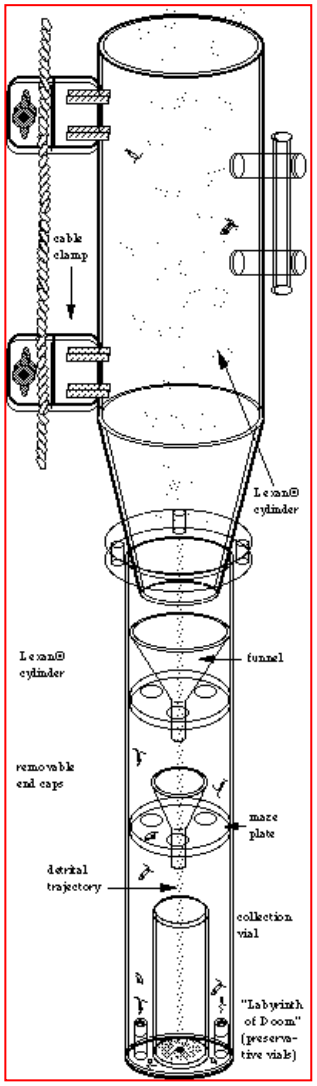
Freezing sampling – separation of individual horizons

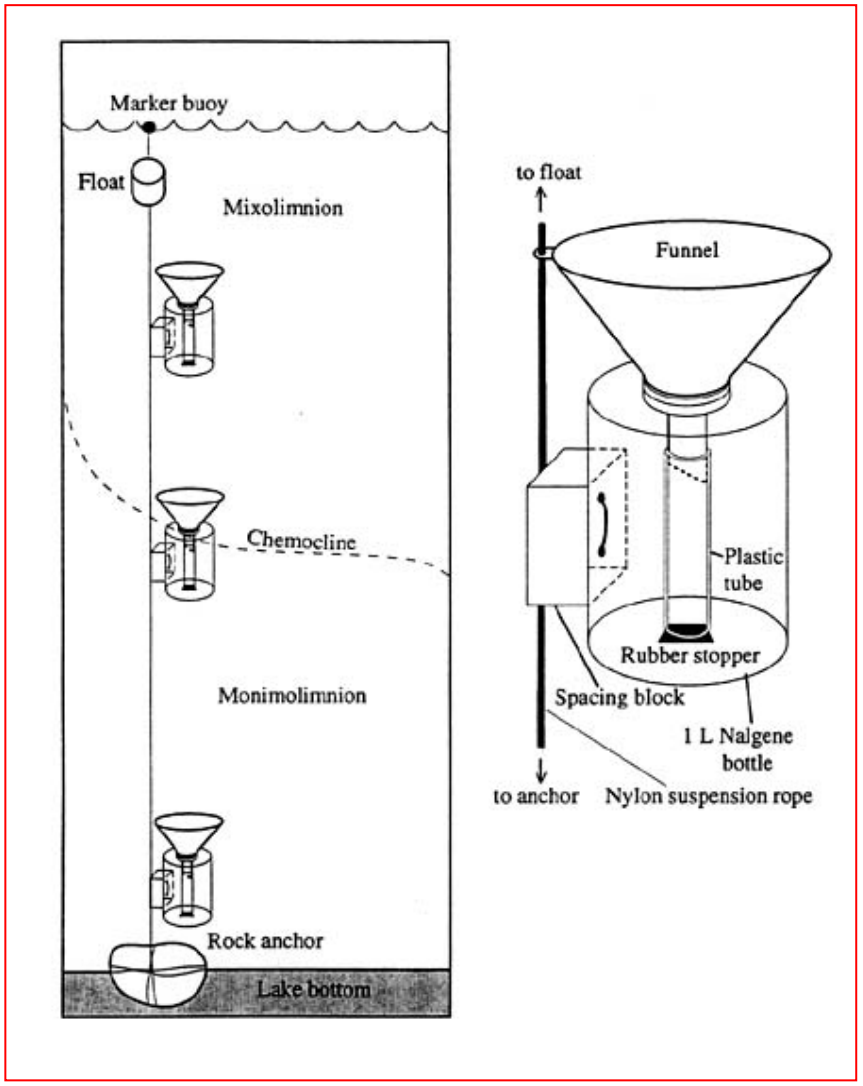


Sediment traps



Sediment traps





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Sampling by diver

Summer

Winter

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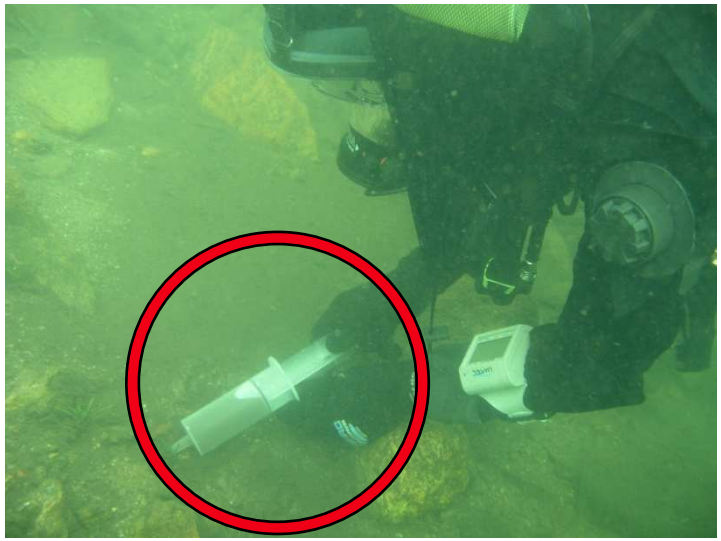
Sampling by diver



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Sediment sampling

Collection of samples from reservoirs

- Based on the horizontal broken of bottom
- Samples are not collected randomly, but during the transect cross the reservoirs (lake, pond, dam).
- Individual samples have to characterized thickness of sediment of predicted depth of sampling in given place.
- Sampling protocol have to include direction of transect including the localization of sampling sites.
- The recommend mass of one mixed sample is approximately 1 kg dry weight (2-3 kg of wet sample)

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Sediment sampling

Colletion of samples from streams and rivers

- One mixed sample is collected every 1 000 m over the length of stream 2 000 m.
- Every mixed sample consists from minimal 3 individual samples located one from the other at minimum 100 m.
- Samples are collected in cross profile of stream.
- Sampling sites are describe in sampling protocol.
- The recommend mass of one mixed sample is approximately 1 kg dry weight (3-5 kg of wet sample).

ISO norms

- ISO 5667-1:2006 Water quality -- Sampling -- Part 1: Guidance on the design of sampling programmes and sampling techniques
- ISO 5667-3:2012 Water quality -- Sampling -- Part 3: Preservation and handling of water samples
- ISO 5667-4:1987 Water quality -- Sampling -- Part 4: Guidance on sampling from lakes, natural and man-made
- ISO 5667-5:2006 Water quality -- Sampling -- Part 5: Guidance on sampling of drinking water from treatment works and piped distribution system
- ISO 5667-6:2005 Water quality -- Sampling -- Part 6: Guidance on sampling of rivers and streams
- ISO 5667-7:1993 Water quality -- Sampling -- Part 7: Guidance on sampling of water and steam in boiler plants
- ISO 5667-8:1993 Water quality -- Sampling -- Part 8: Guidance on the sampling of wet deposition
- ISO 5667-9:1992 Water quality -- Sampling -- Part 9: Guidance on sampling from marine waters
- ISO 5667-11:2009 Water quality -- Sampling -- Part 11: Guidance on sampling of groundwaters
- ISO 5667-12:1995 Water quality -- Sampling -- Part 12: Guidance on sampling of bottom sediments
- ISO 5667-13:2011 Water quality -- Sampling -- Part 13: Guidance on sampling of sludges
- ISO 5667-14:1998 Water quality -- Sampling -- Part 14: Guidance on quality assurance of environmental water sampling and handling
- ISO 5667-15:2009 Water quality -- Sampling -- Part 15: Guidance on the preservation and handling of sludge and sediment samples
- ISO 5667-16:1998 Water quality -- Sampling -- Part 16: Guidance on biotesting of samples
- ISO 5667-17:2008 Water quality -- Sampling -- Part 17: Guidance on sampling of bulk suspended solids
- ISO 5667-19:2004 Water quality -- Sampling -- Part 19: Guidance on sampling of marine sediments
- ISO 5667-20:2008 Water quality -- Sampling -- Part 20: Guidance on the use of sampling data for decision making -- Compliance with thresholds and classification systems
- ISO 5667-21:2010 Water quality -- Sampling -- Part 21: Guidance on sampling of drinking water distributed by tankers or means other than distribution pipes
- ISO 5667-22:2010 Water quality -- Sampling -- Part 22: Guidance on the design and installation of groundwater monitoring points
- ISO 5667-23:2011 Water quality -- Sampling -- Part 23: Guidance on passive sampling in surface waters

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Subsampling

- Obtaining a representative subsample is the most uncertain step in most analyses
- Risk of contamination - nearly all sample treatment techniques require a close physical contact between the sample and laboratory equipment (and the analyst) – potential contamination of samples
- Potential analyte loss
- Sample preparation techniques should be communicated with the customer and should be agreed on before the sampling starts

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Subsampling procedure

Solid Material

- milling/grinding by mechanical means to produce a mixture containing particles
- of the appropriate size;
- mixing/homogenization by using a ball mill;
- subdivision of the ground and mixed sample using coning and quartering or
- riffling techniques

Relationship between particle size and test portion

Particle mass (mg)	Test portion (g)
10	10
1	1
0.1	0.1

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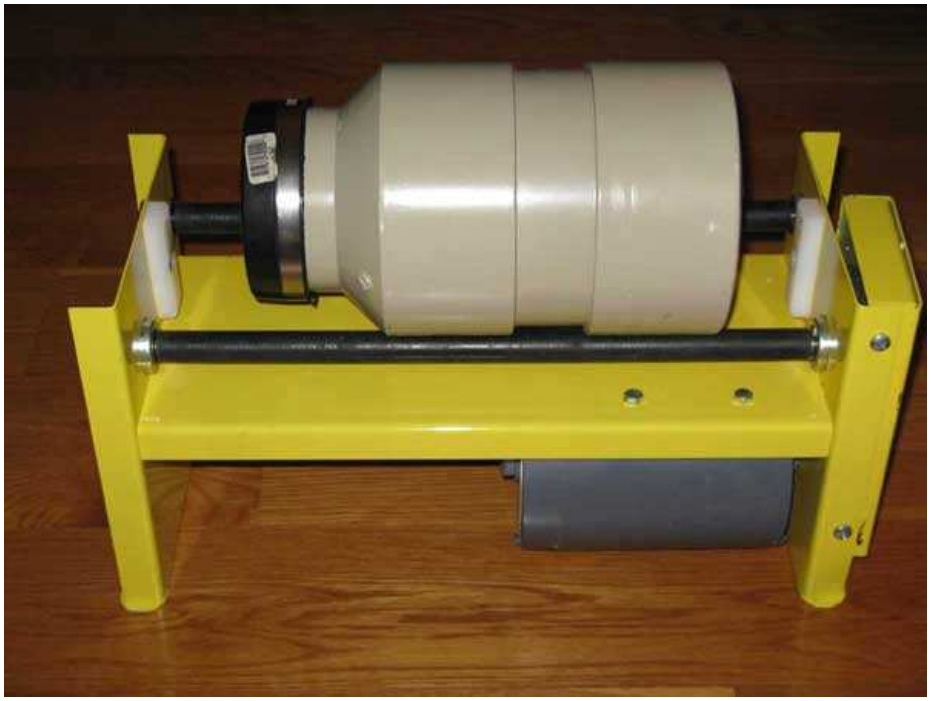
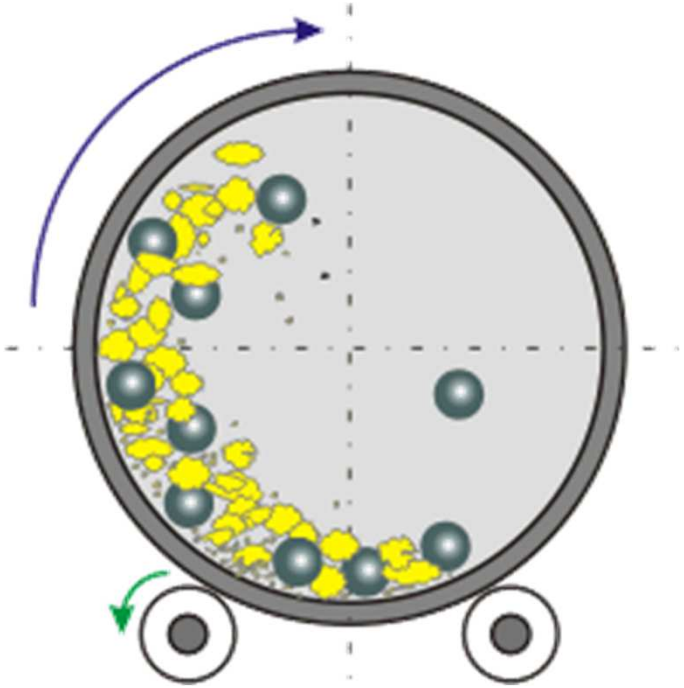
tarafından ortaklaşa finanse edilmektedir.

Subsampling solid materials – ball mill





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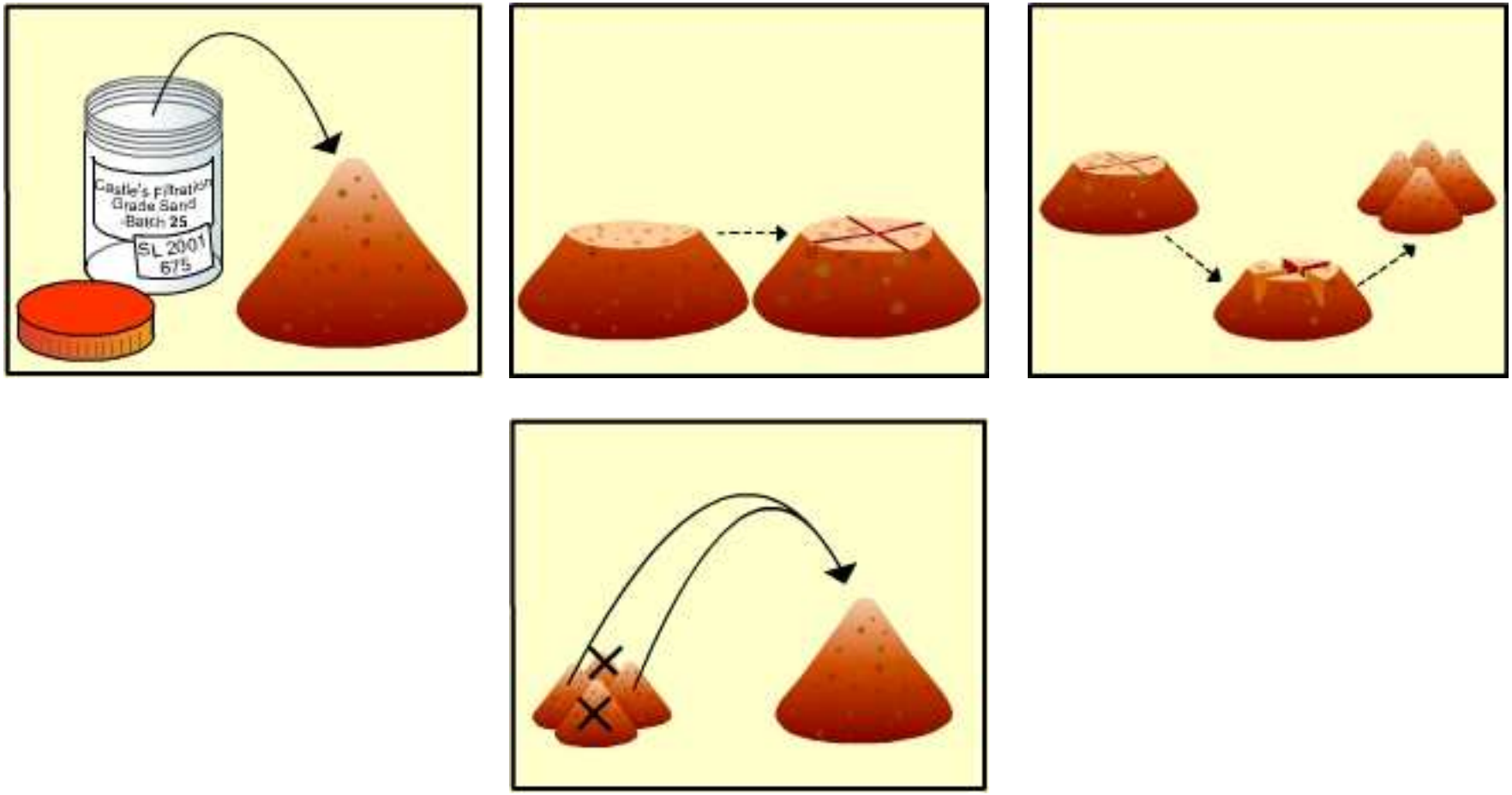
Subsampling solid materials – coning and quartering



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Subsampling solid materials – riffling

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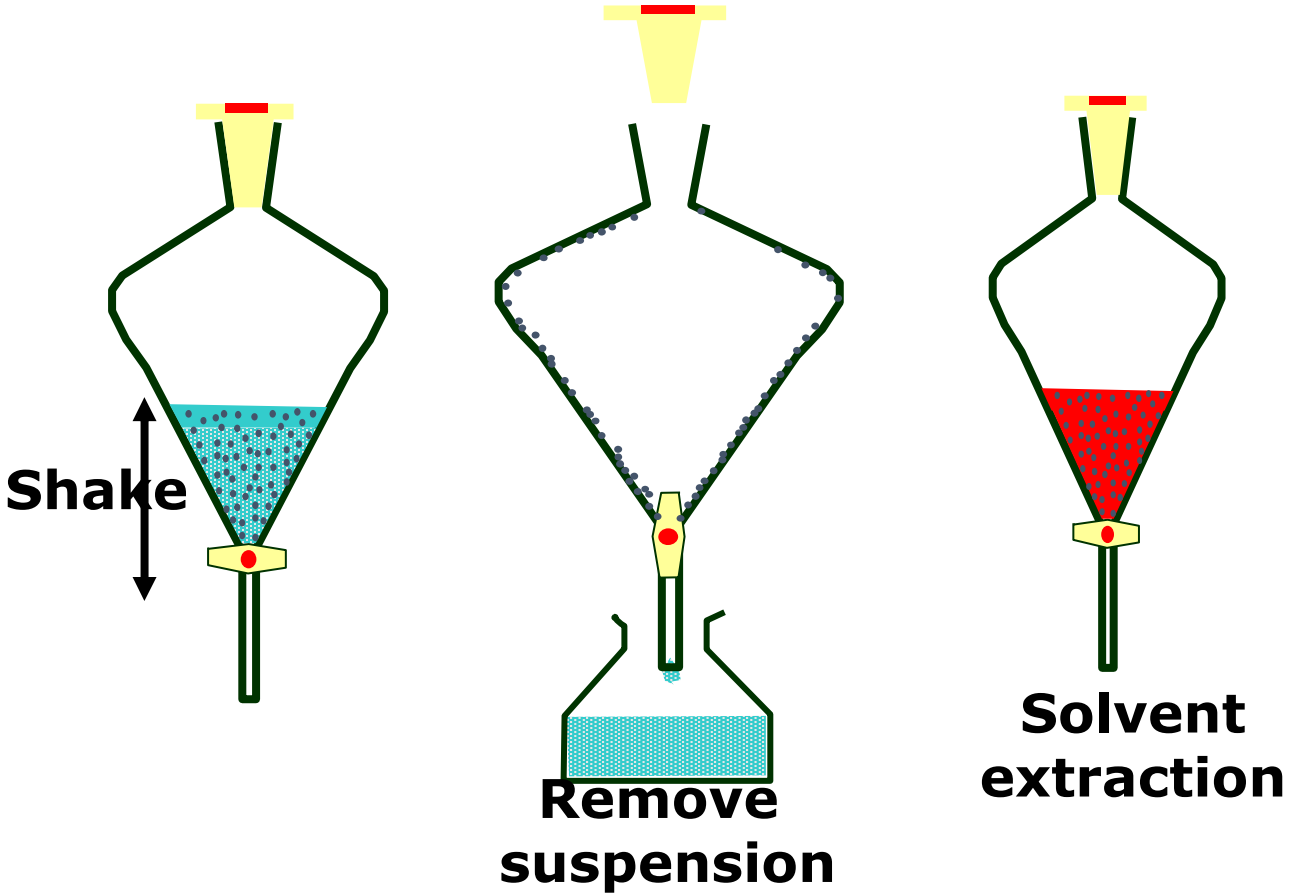
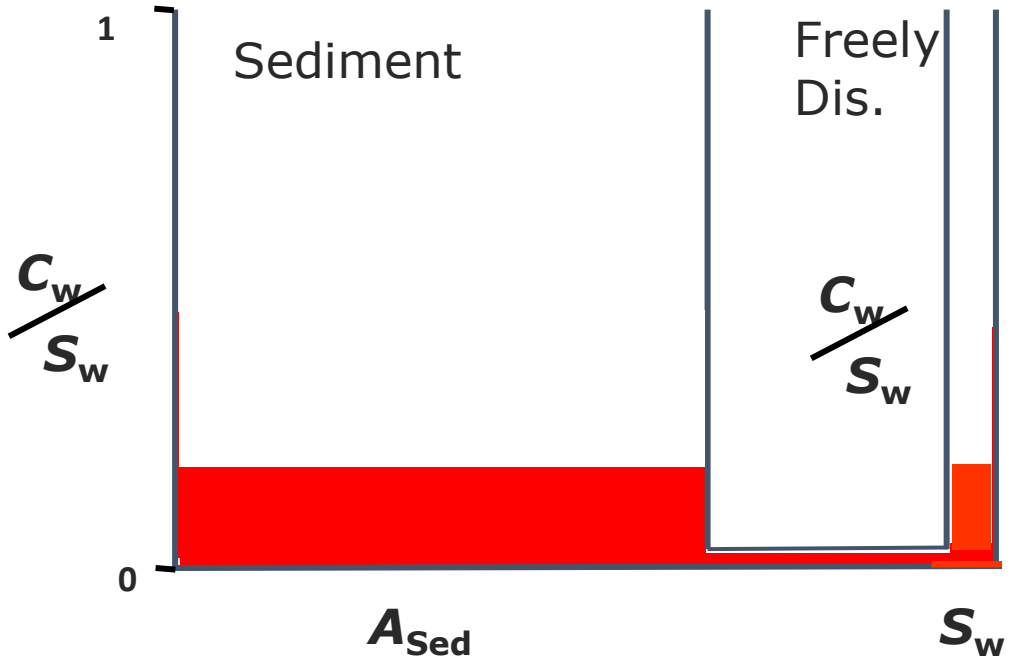
Extraction with Tenax



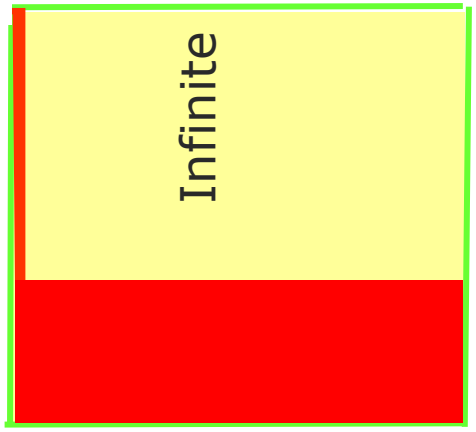
Drain principle – infinite sorption



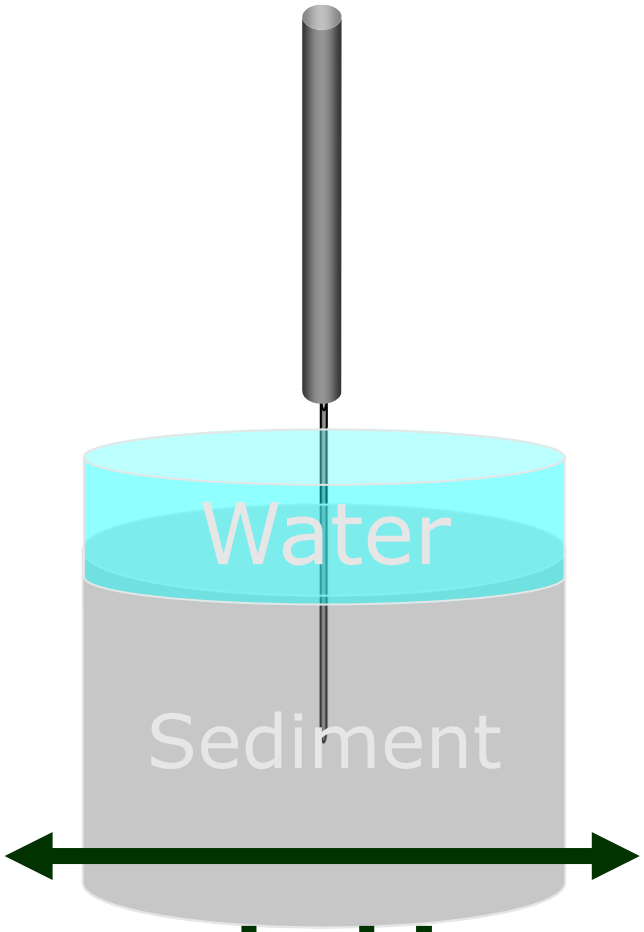
Tenax

XAD

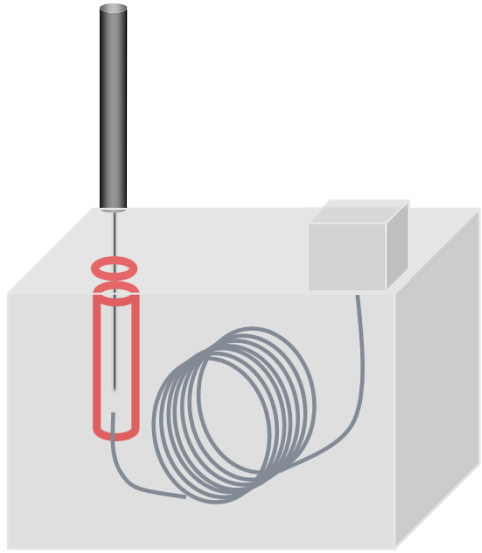
Etc.



SPME in sediment water system



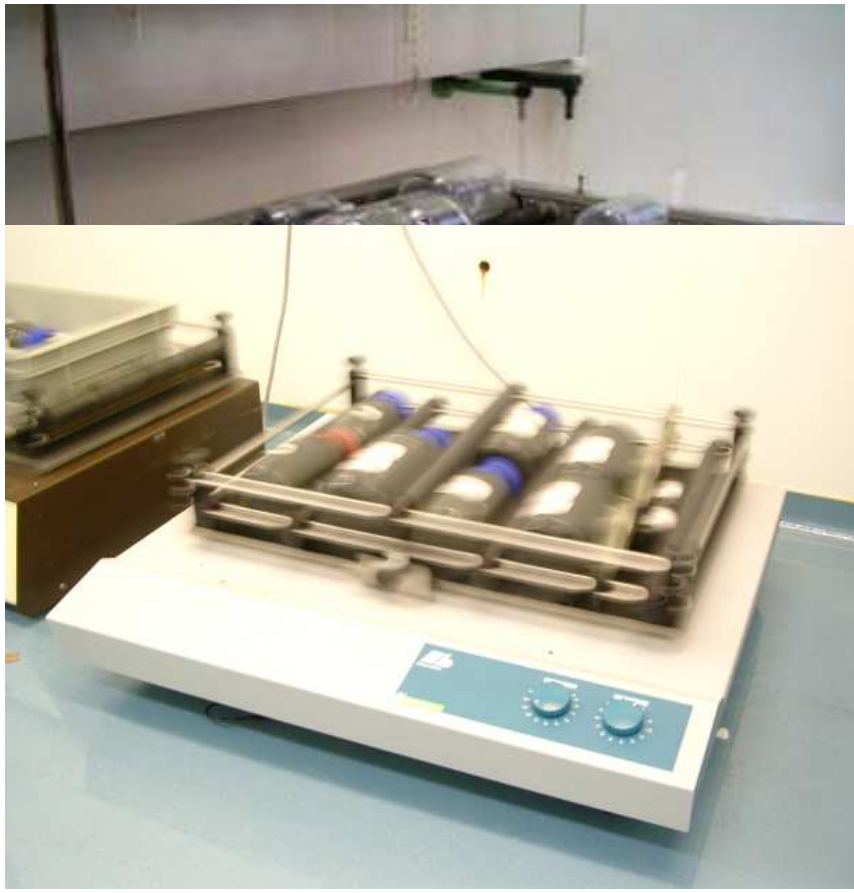
shaking

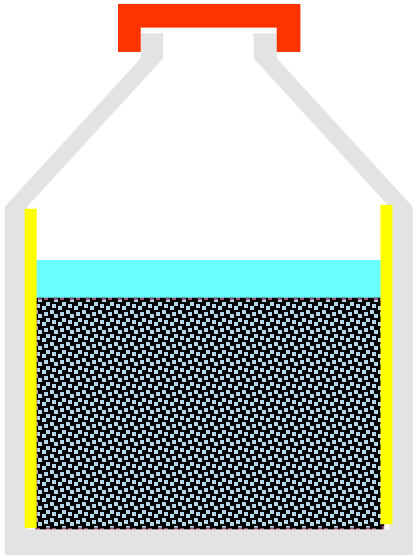


GC and detector

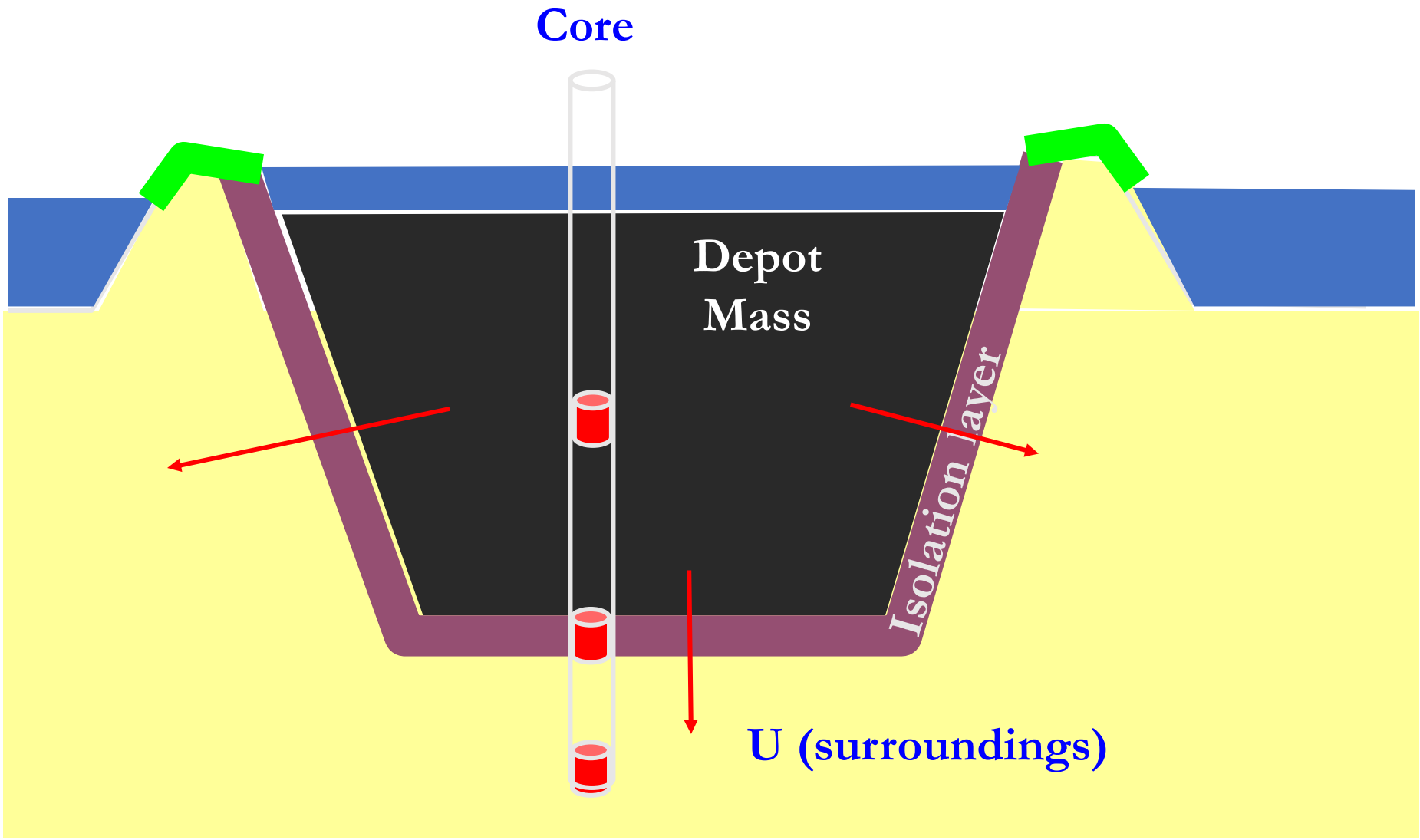
Passive Sampling with coated bottles

Silcone rubber film and sediment in glass bottle





Study migration, leakage from Sludge depot



Contents

Sediment sampling – example 1

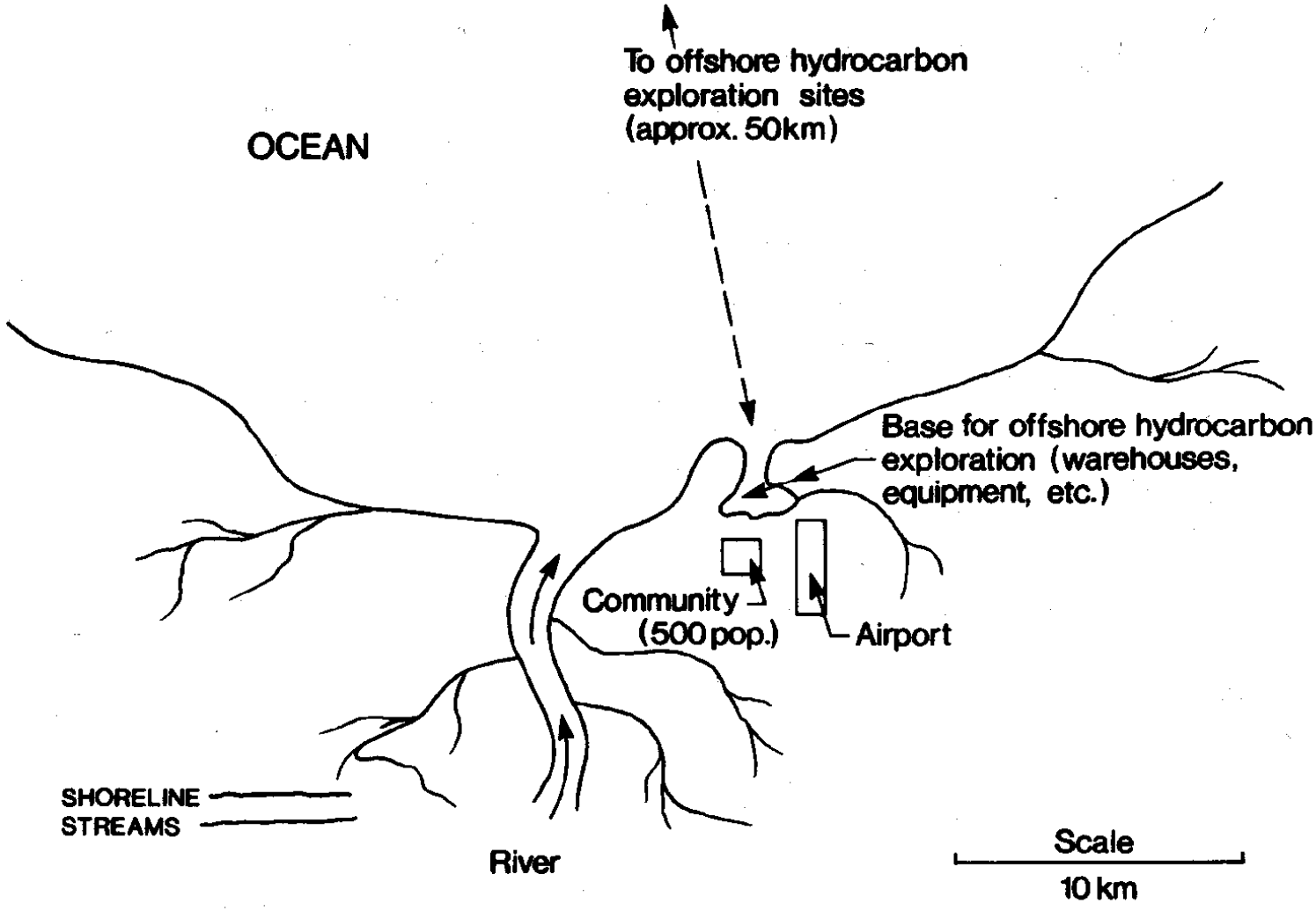


FIGURE 1. Example 1.

Sediment sampling – example 2

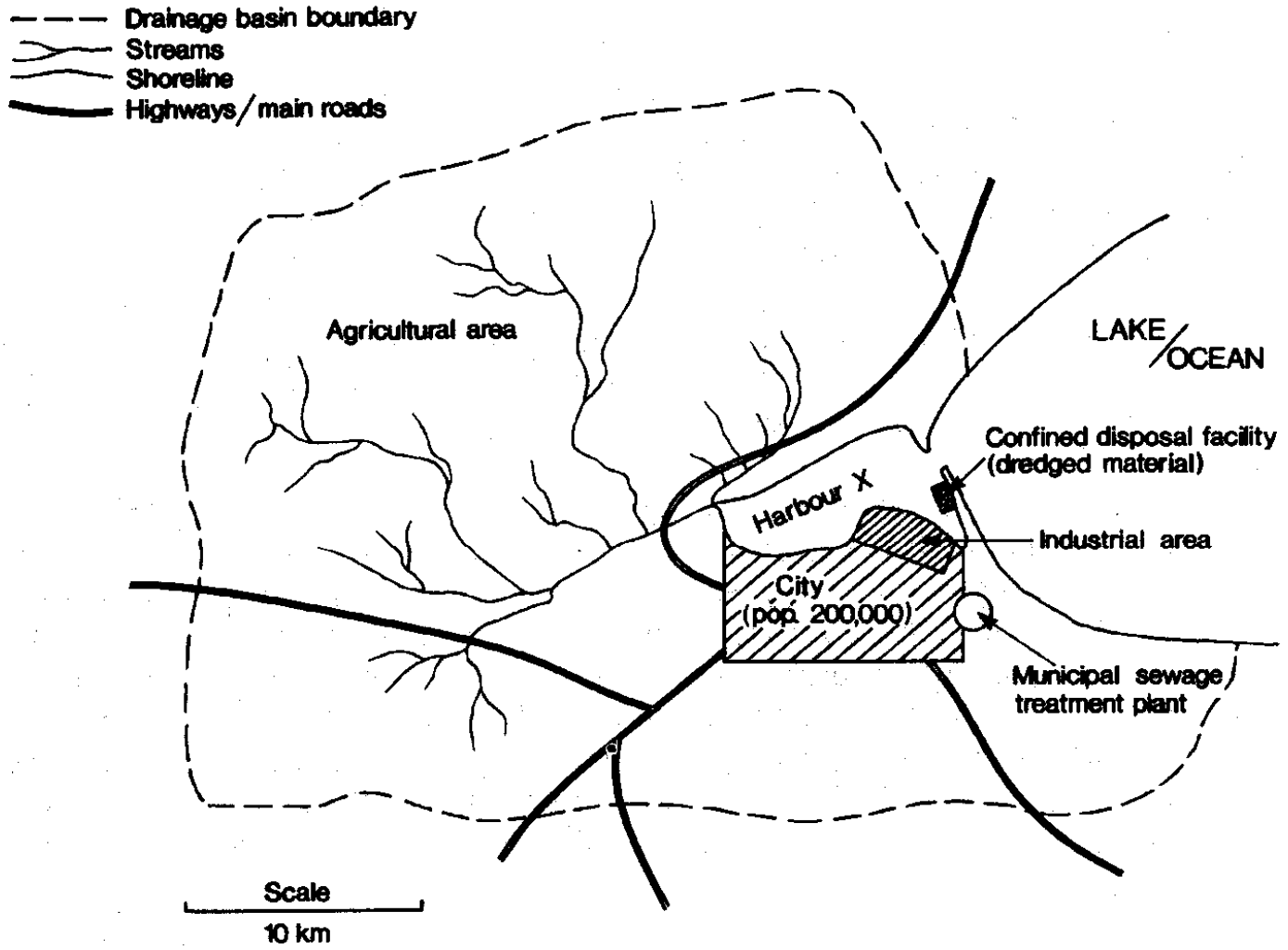


FIGURE 2. Example 2.

Sediment sampling – example 3

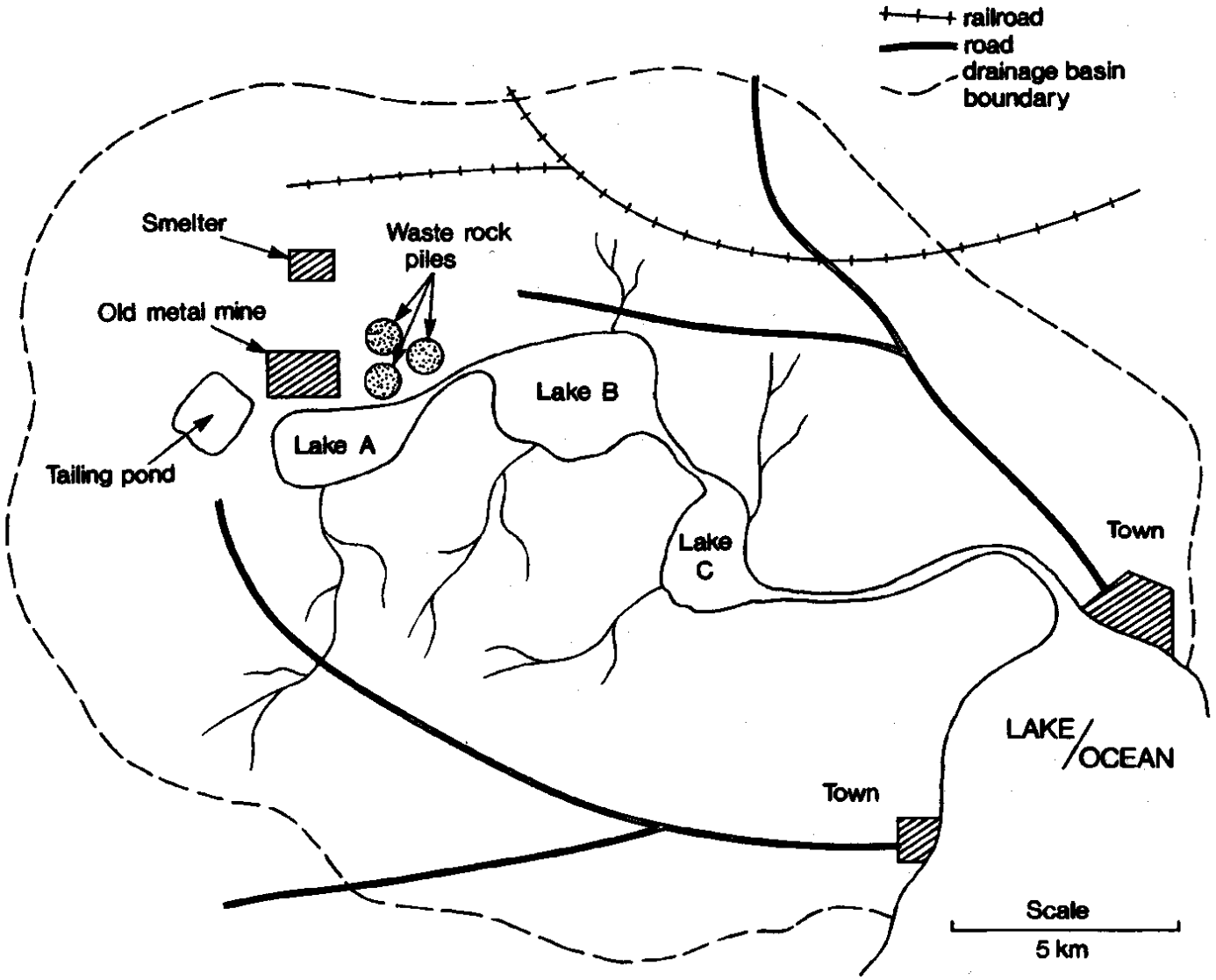


FIGURE 3. Example 3.

Sediments – sampling grid at a point source

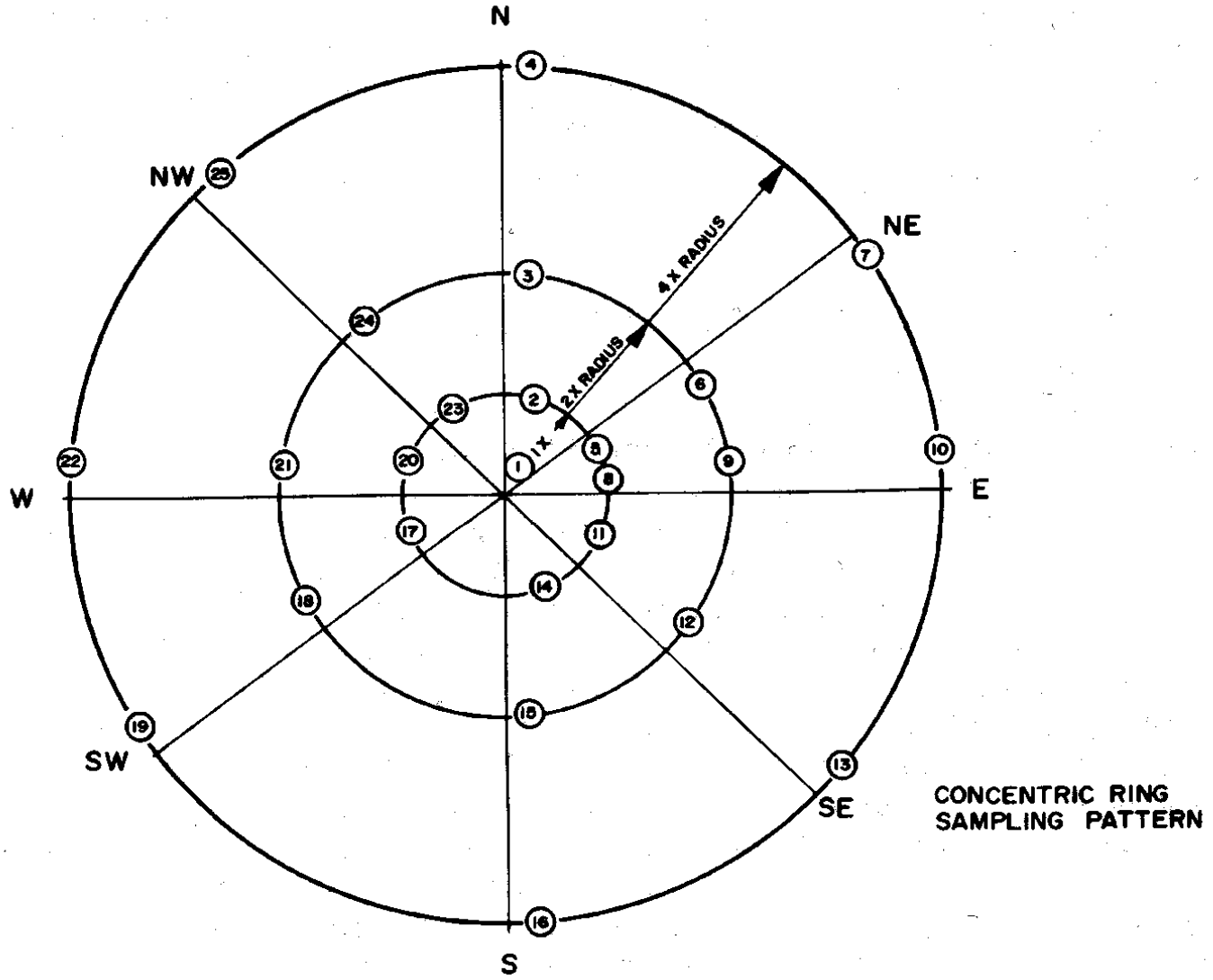


FIGURE 1. Sampling grid at a point source.

Sediments - random selection no prior knowledge

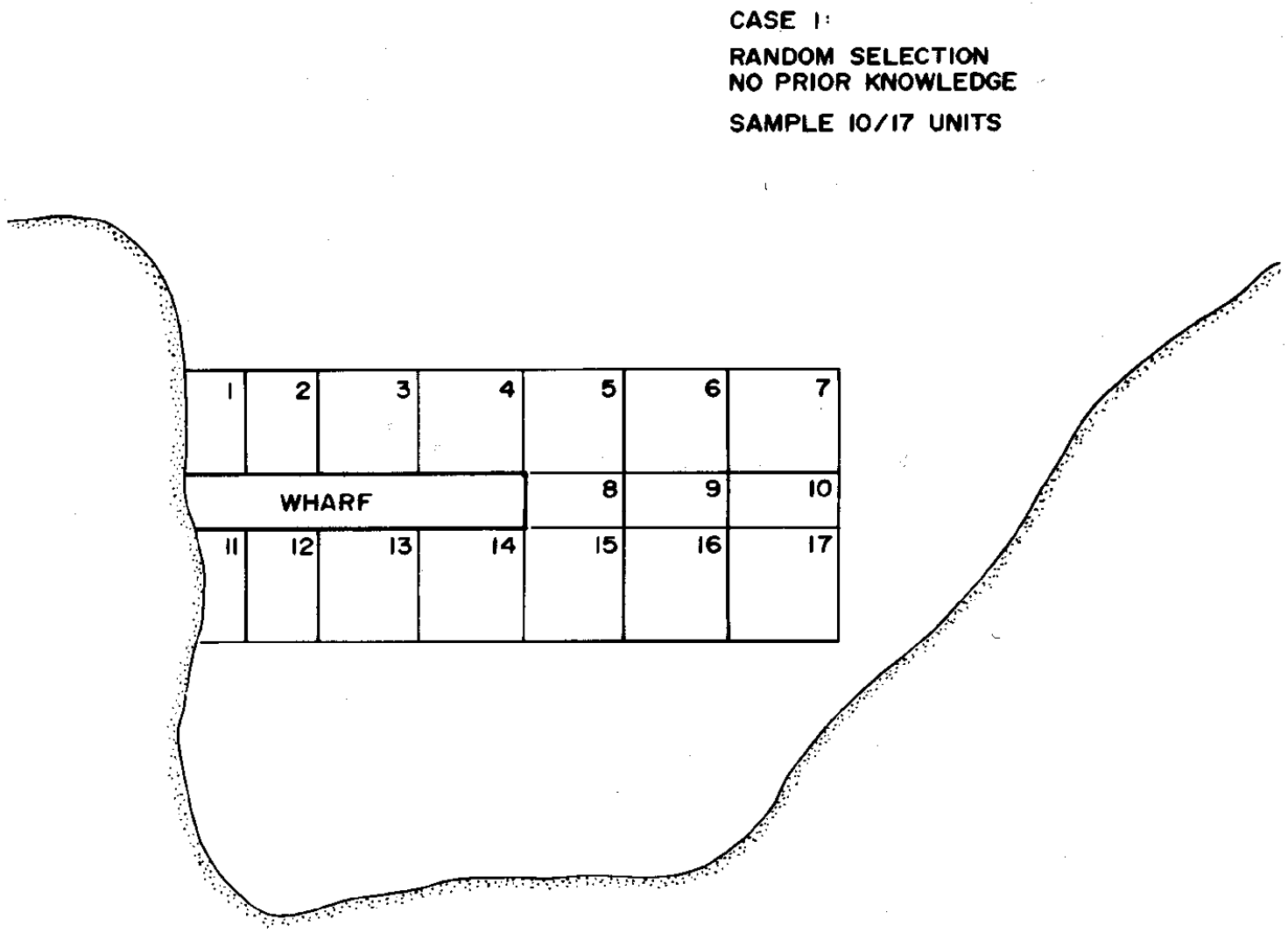


FIGURE 2. Random selection of sampling stations with no data available on the project.

Sediments – selection of sampling stations with available information on point sources

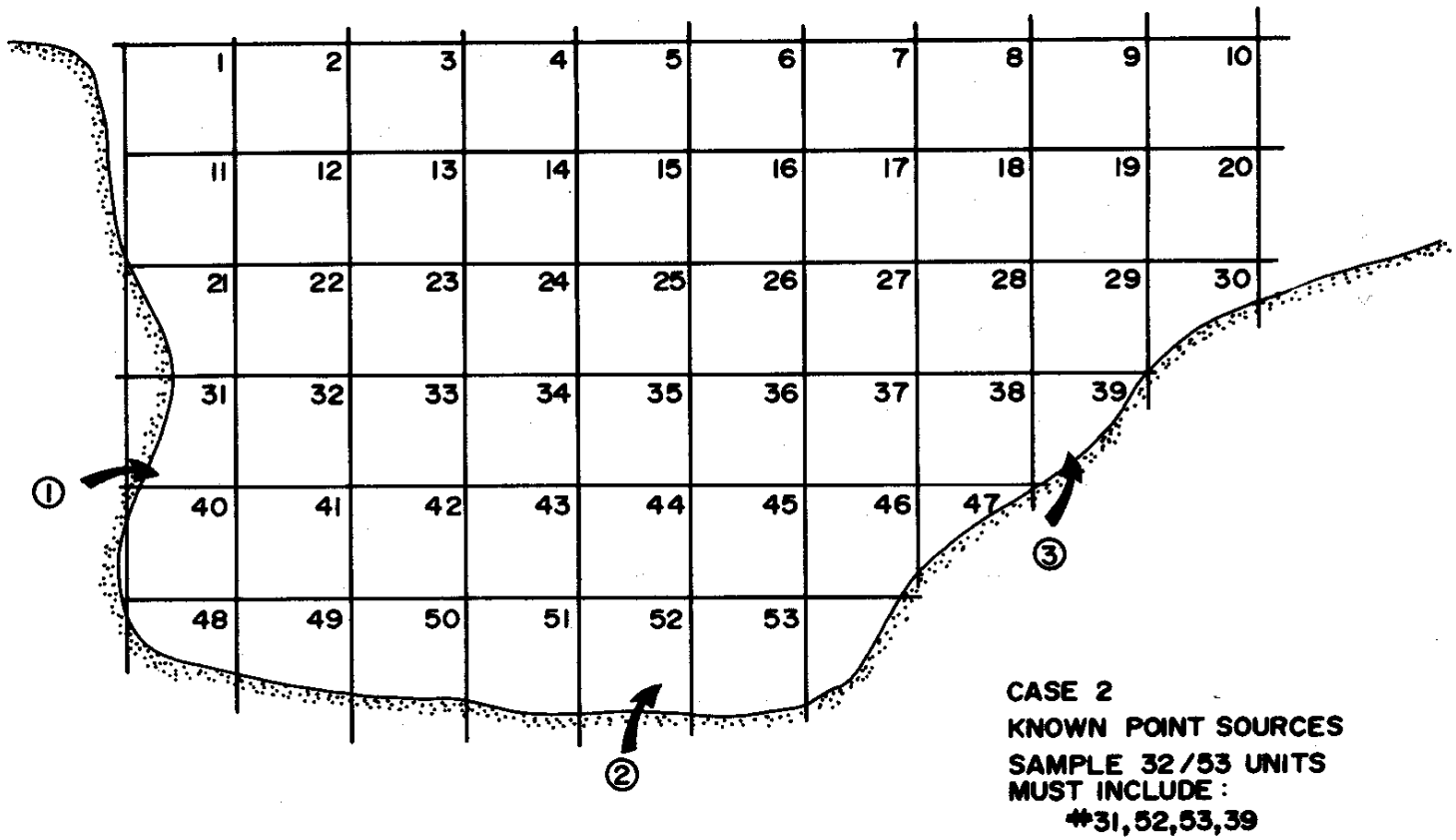


FIGURE 3. Selection of sampling stations with available information on point sources.

Sediments – selection of sampling stations with available data on sediment particle size

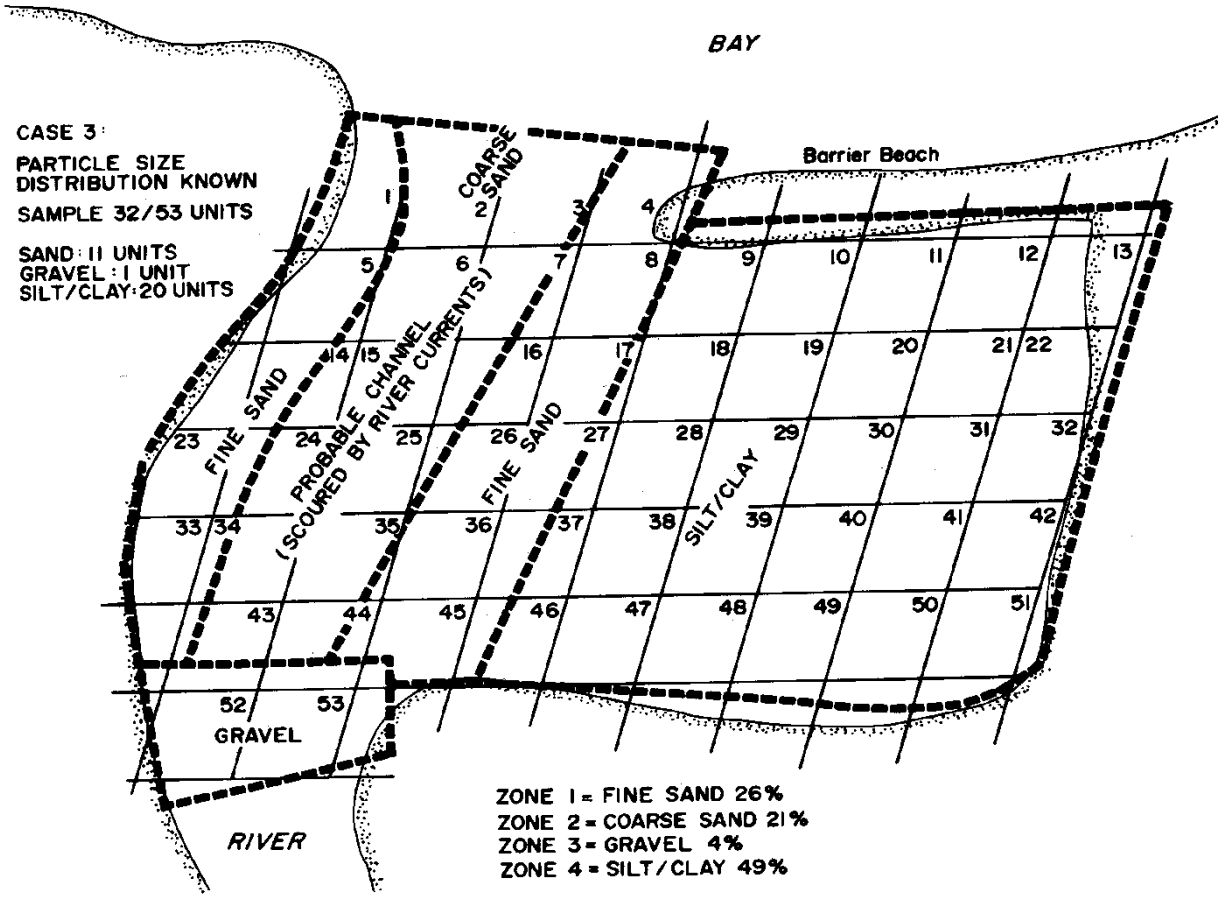
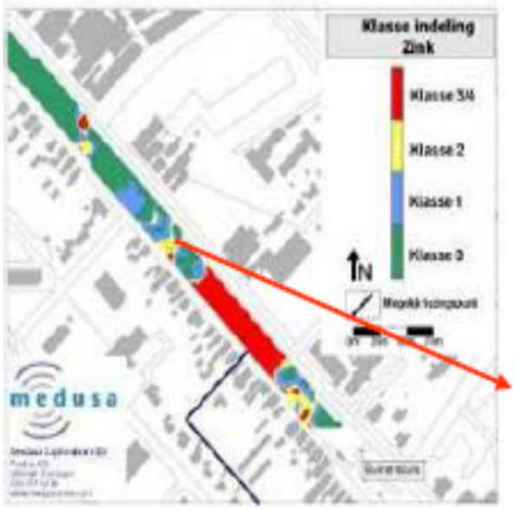


FIGURE 4. Selection of sampling stations with available data on sediment particle size.

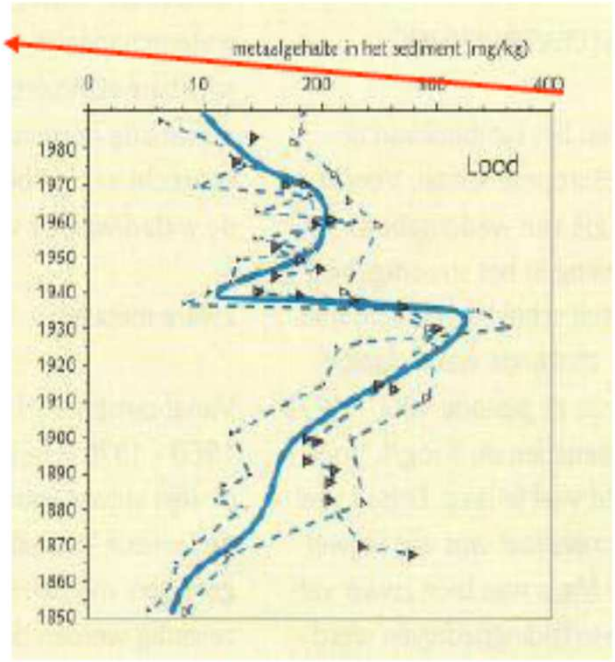
Sediment sampling

- Typical research purposes (stagnant sediment)
  - Determine pollution levels of layers prior to dredging



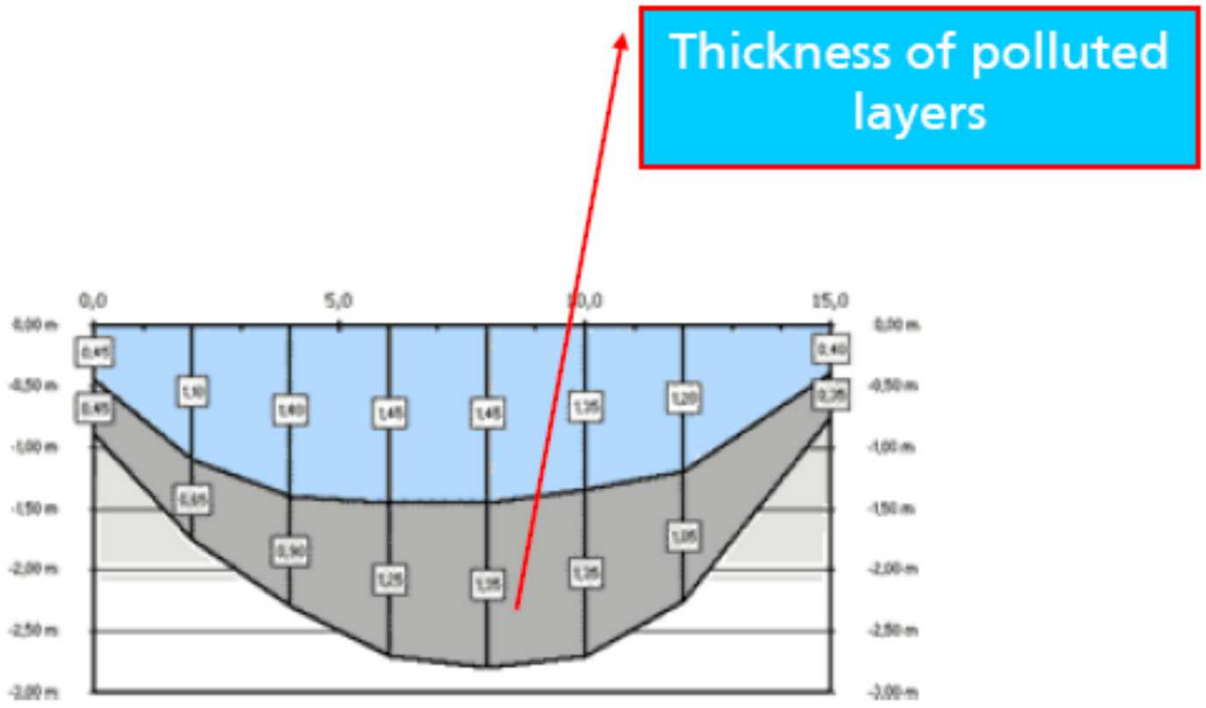
Zinc pollution levels in a canal

Lead pollution over the years in the Rhine sediment



Sediment sampling

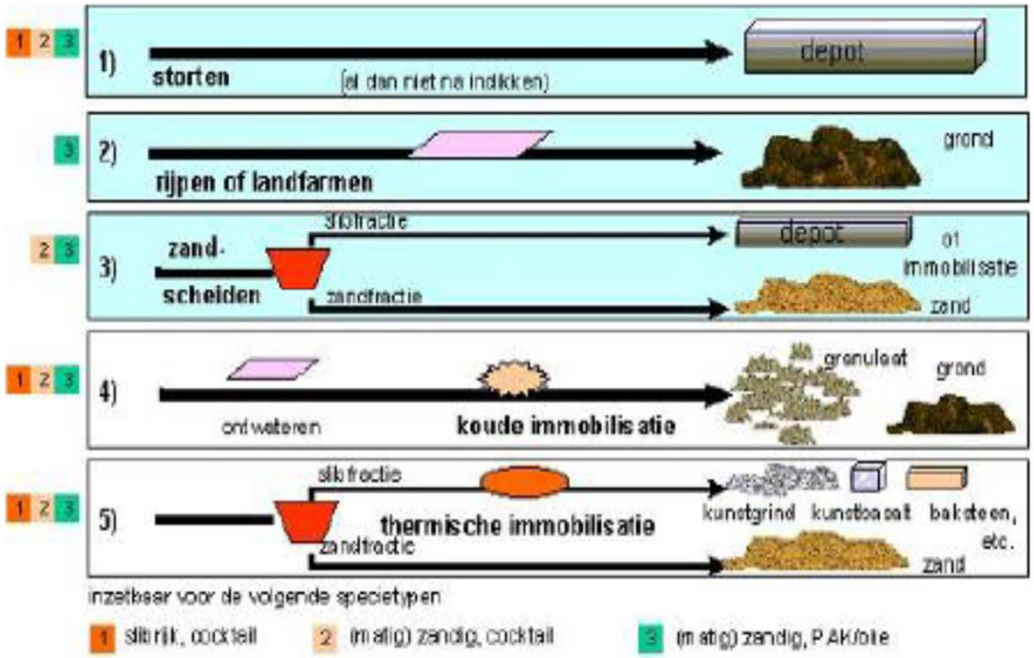
- Typical research purposes (stagnant sediment)
  - Determine thickness of sediment layers prior to dredging



Sediment sampling

- Typical research purposes (stagnant sediment)
  - Determine treatment policy like landfarming, hydrocyclonage, cold or hot immobilisation, re-use or storage

Treatment techniques of dredgings



Sediment sampling

- Typical research purposes (stagnant sediment)
  - Determine transport and storage policy

150.000.000 m^3 dredgings storage for the port of Rotterdam

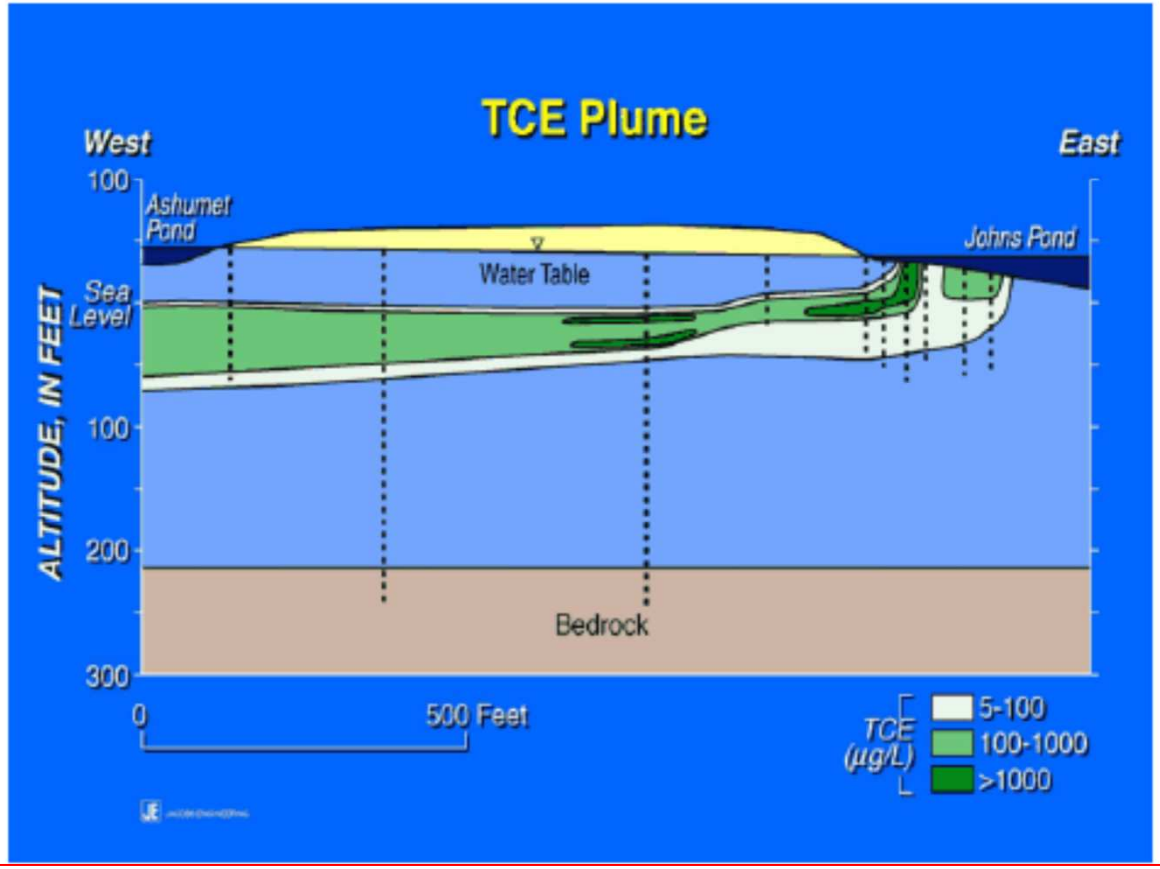
Small scale project or large scale

Sediment sampling

- Typical research purposes
  - Determine possibilities of recycling dredgings

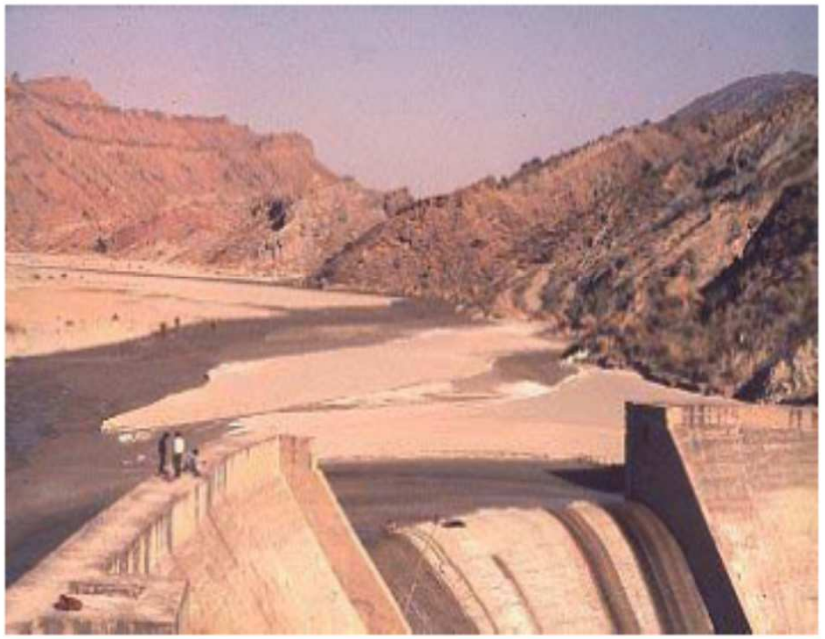
Sediment sampling

- Typical research purposes (stagnant sediment)
  - Behaviour and migration of pollution near canals and rivers



Sediment sampling

- Typical research purposes (flowing sediment)
  - Determine sediment load (up stream erosion level)
  - Determine down stream effects (silting up of dam reservoirs)

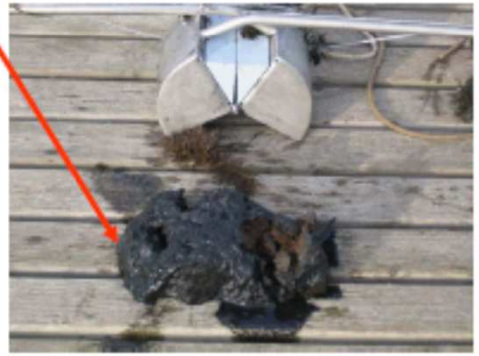


Sediment sampling

- Typical research purposes (flowing sediment)
  - Determine see through visibility (swimming water)
  - Determine composition (clay-silt-sand-stones) over the depth and over the seasons

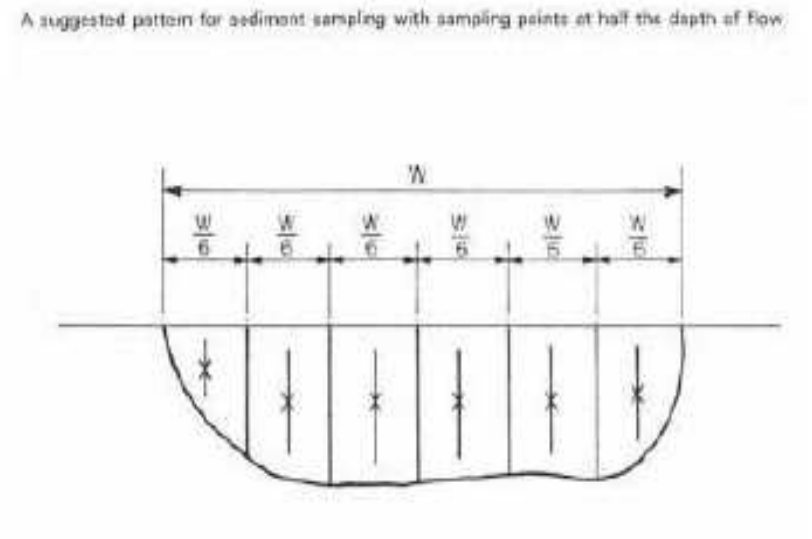
Sediment sampling

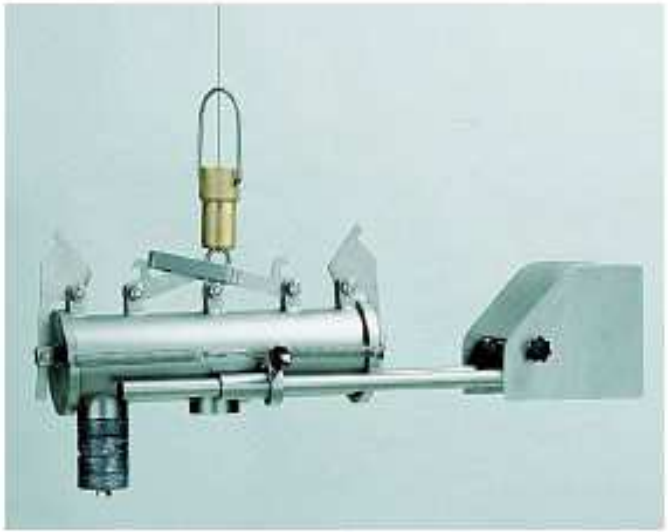
- Working principle of **stagnant** sediment samplers
  - Grab samples
    - Short samples with an indicative quality
  - Core samples
    - Cores must show all important (top) layers



Sediment sampling

- Working principle of **flowing** sediment samplers
  - Grab samples
    - Sample is taken at specific depths across the river
      - Many samples must be taken to get an average





Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Compunds and matrix selection for sediment and biota monitoring

- The choice of the matrix to be monitored depends firstly on the physico-chemical properties of the substances
- The priority list of the WFD contains several (classes of) substances which have a
  - low solubility in water,
  - high octanol/water partition coefficient (log K_{OW})
  - high potential for bioaccumulation and bioconcentration

Contents

- Sediment monitoring – active sampling
- Sediment monitoring – passive sampling
- Design of sediment sampling
- Biota monitoring – approaches, sampling
- Soil – sampling, monitoring
- Technical matrices – sampling, monitoring
- Plastics

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Selection of compounds to be monitored in biota

Organic compounds that bioaccumulate

- when the biomagnification factor (BMF) is >1 or
- when the bioconcentration factor (BCF) is >100
- log K_{ow} > 3 can be considered as an indicator for bioaccumulation potential
- If rapid degradation (ready biodegradability or hydrolysis half-life <12h at pH 5-9, 20°C) – no monitoring
- molecular size can be an indicator of limited bioaccumulation potential of a substance
- metabolisation and depuration efficiency of the different species should also be taken into consideration

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti
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Selection of compounds to be monitored in biota

Metals that bioaccumulate

- biomagnification of metals
  - rarely observed and,
  - if it does occur, it usually involves the organo-metallic forms of metals (e.g. methylmercury)
- lack of biomagnification should not be interpreted as a lack of exposure or an absence of concern for trophic transfer. Even in the absence of biomagnification, aquatic organisms can bioaccumulate relatively large amounts of metals and this can become a significant source of dietary metal to their predators.
- For metals, a BCF should not be used; this is because the model of hydrophobic partitioning, giving a more or less constant ratio C_{biota}/C_{water} with varying external concentration, (does not apply to metals)

Sampling strategy for chemical monitoring in biota

- The natural variability within biota samples should be reduced by an appropriate sampling design
- Contaminant concentrations can be affected by differences in
  - Age
  - Size
  - Sex
  - sexual maturity status can affect the measured concentrations of contaminants.

- Biota sampling should only take place when fish and bivalves are
  - in a stable physiological state
  - outside the normal period of spawning

- Fishes should be collected from areas characterised by relatively low natural variability
- No use of species that are endangered or that require special protection in compliance with "Habitat Directive" requirements or any other national or international action plan for nature conservation.
- Active biomonitoring, such as caging and transplantation procedures must avoid the introduction of allochthonous species to waterbodies.
- Non-native species should not be used in active biomonitoring.
- Sampling strategies for biota monitoring should seek continuity with pre-existing monitoring programmes when relevant.
- harmonisation with the biota sampling performed for the purpose of the classification of the ecological status can be useful

Recommendations for the selection of biota species

- a relationship exists between contaminant concentrations in the species and average concentrations in the surrounding environment;
- the sampled organism is a potential food for predatory organisms or humans;
- the species accumulates the contaminants;
- the species is sedentary (migrating species should be avoided) and thus represents the sampling location, and does not originate e.g. from aquaculture plants
- the species is widespread and abundant in the study region, to allow comparisons between different areas
- the species lives long enough so that more than one year-class can be sampled, if desired;
- the species is large enough to yield sufficient tissue for analysis
- the species is easy to collect and hardy enough to survive unfavourable conditions
- the species is easy to identify

Selection of sites: general considerations

- The geographical representativeness of a sample in lakes varies with, for example, species and size
- The fish should be collected from a sampling site representative of the area. The site should not differ from the general picture of the area of concern such as for example an isolated bay. Differences between a lotic and a lentic environment, high-flow and low-flow rivers, and feeding behaviour of the species should be highlighted.
- In rivers the sampling sites have to be representative of the respective ecosystem, and/or of the respective sampling region. This means that they must not be close to local sources of emissions. The minimum distance from such pollution sources depends on the type of emissions and on numerous hydrologic and hydrogeographic factors, e.g. water depth, water width, surface and volume of the water body, degree of mixing, pH-value, oxygen content, water hardness, conductivity, trophic level, flow rate, wind direction, wind strength, character of the riparian zone, exposure, etc. The minimum distance from the nearest source of emission must therefore be ascertained separately for each sampling site.
- For active monitoring with zebra mussels, a secure, undisturbed and sheltered position should be chosen.. Natural sources of irritation, e.g. too strong a current or a risk of siltation, need to be avoided, as do possible irritations by river boat traffic. Otherwise, the exposure spots need to be readily accessible, even in bad weather. In the selection and demarcation of sampling sites for the sampling of free-living populations (passive monitoring), the population must be of a sufficient size, density and stability in order to ensure good long-term sampling. Furthermore, long-term use of the sampling sites and access to the exposure spots must be secured by contract as a basic principle. The detailed arrangements will depend on the level of protection and the ownership structure.

Sampling period

- Concentrations of chemical pollutants in tissues of bioindicator organisms can be influenced environmental and biological factors, independent of the variations in anthropogenic inputs. In particular, seasonal fluctuations must be carefully considered for the correct interpretation of the results, and to discriminate natural variability from changes due to human impact.
- environmental factors which modulate bioavailability and the tissue burden of chemicals are
  - fluctuations of temperature,
  - organic matter,
  - presence of nutrients
  - water fluxes and circulation
  - up-welling phenomena
  - freshwater or river inputs
  - land runoff
- Seasonal changes of tissue concentrations have also been reported during phytoplanktonic blooms, which can modulate the bioavailability of several chemicals
- Other biological variables, including intrinsic species-specific features such as
  - phase of reproductive cycle,
  - weight fluctuations
  - changes in relative tissue composition
  - massive development of gonadic tissues during gametogenesis
  - loss of weight during spawning,
- Depending on the strategy and objectives of the monitoring plan, it can be recommended to select the sampling periods in advance or to consider the most important variables which might influence the results obtained.
- When designing large-scale and/or long-term (years to decades) biomonitoring projects to assess temporal trends of contamination, the influence of seasonal variability can be reduced by defining in advance the sampling period(s) which will be kept constant for all subsequent years. Carrying out sampling of biota during a period in the year when contaminant concentrations are not being significantly affected by changes in physiological mechanisms is essential for consistency of sampling. Such periods of minimal change are generally related to periods outside the spawning cycle and when food supply is relatively constant.

Sampling frequency

- Sampling frequency should consider
  - biological half life of contaminants
  - aim of monitoring
  - presence of anthropogenic inputs/pressure,
  - availability and quality of previous results or trends
- In general terms, a surveillance programme could be based on a low-frequency (six-months/annual) sampling strategy, especially if the monitored area is not challenged by marked anthropogenic pressures.
- higher frequency (monthly to seasonal) should be recommended in areas characterised by the presence of specific impacts and/or specific forms of pollutants (e.g. petrochemical sites, industries, river estuaries, harbours, etc.).
- specific monitoring project, i.e. to evaluate the impact of a temporary activity (such as dredging) should include sampling periods before, during different phases of and after the end of operations.
- an "investigative" monitoring programme in an area where the source of pollution is unknown should begin with a high frequency (i.e. 1–2 months) which might be lowered depending on the results obtained and, again, the possible presence of anthropogenic impacts.
- It is recommended for the purpose of trend monitoring to start at least with a cycle of one examination every 3 years. After several cycles it may be appropriate to downscale the frequency to one every 6 years

Trend Analysis

- The main characteristics of the data collected for the purpose of temporal trend analyses are the following:
  - Collection of biota annually at the same time within each year.
  - The time should be principally outside the spawning period.
  - The same size range of the target species is sampled each year.
- Sampling guidelines are necessary to provide some control over both
  - between-years biological variation (e.g. mean length, condition, stock composition) and
  - within-year biological variation (e.g. individual fish length).
- The organisms chosen must be typical for the water body type and as far as possible resident species that occur frequently in the water body under investigation.
- It may be desirable to study two different fish species per monitoring point so that different feeding habits can be taken into account and to ensure that, if a fish species disappears, reference can at least be made to the trend in the other species

Sampling methods (passive)

- Fish may be captured by trawling, netting, creels and other appropriate methods, depending on the species and location.
- Electrofishing can be also used for small, shallow rivers (commonly chalk streams), drained canals or full navigational waterways with a maximum depth of 2.5 m.
- When fish can be sampled from either research vessels or commercial vessels, the former is the preferred option, since research vessels are likely have better facilities for processing and storing scientific samples. In both cases, the following precautions must be taken when selecting samples from the trawl catch to ensure that contamination is kept to a minimum:
  - trained personnel must be present when a trawl comes on board to ensure that the sample can be isolated from possible sources of contamination during the release of fish from the net;
  - the trawling time should not exceed one hour and the trawling speed should be as slow as possible to reduce damage and stress to the fish;
  - fish which are visibly damaged or in bad condition must not be selected;
  - clean containers should be available on deck to hold the samples temporarily before they are taken to the ship's laboratory. Containers used for holding fish collected from the ship's normal trawling operations must not be used;
  - personnel must wear clean gloves when the samples are taken from the net. The samples should be transferred to the ship's laboratory as quickly as possible and rinsed with clean sea water to remove any material adhering to the surface;
  - equivalent precautions should be taken on modern fisheries research vessels, when the catch is released from the net directly into facilities below deck; only material suitable for the subsequent analyses should be retained

Technical Report - 2010 - 041

COMMON IMPLEMENTATION STRATEGY FOR THE WATER FRAMEWORK DIRECTIVE (2000/60/EC)

Guidance document No. 25 ON CHEMICAL MONITORING OF SEDIMENT AND BIOTA UNDER THE WATER FRAMEWORK DIRECTIVE

EUROPEAN COMMISSION

environment

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti
tarafından ortaklaşa finanse edilmektedir.

Biological early warning

Chemical monitoring:

- analysis of pollutant in water, sediment or biota
- well defined compounds (but only those analysed)
- concentration known
- comparison with standards

Biological monitoring:

- detection of biological effects
  (in cell, organ, organism or population)
- integrated signal
- broad spectrum

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Why is fish or mussel used for monitoring of pollutants?

- detection limit (water concentration too low)
- accumulation in food chain
- accumulation via water and food
- interpretation of biological effects
- secondary poisoning
- temporal integration
- load in a distinct site (mussel)
- spatial integration (fish)
- concentrations in food (fish)

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Sampling strategy for chemical monitoring in biota

The biota sampling strategy for a given water body should include:

- the choice of the substances to be monitored
- the selection of the species representative for that specific water body
- the selection of the sampling sites
- the monitoring frequency
- the monitoring techniques

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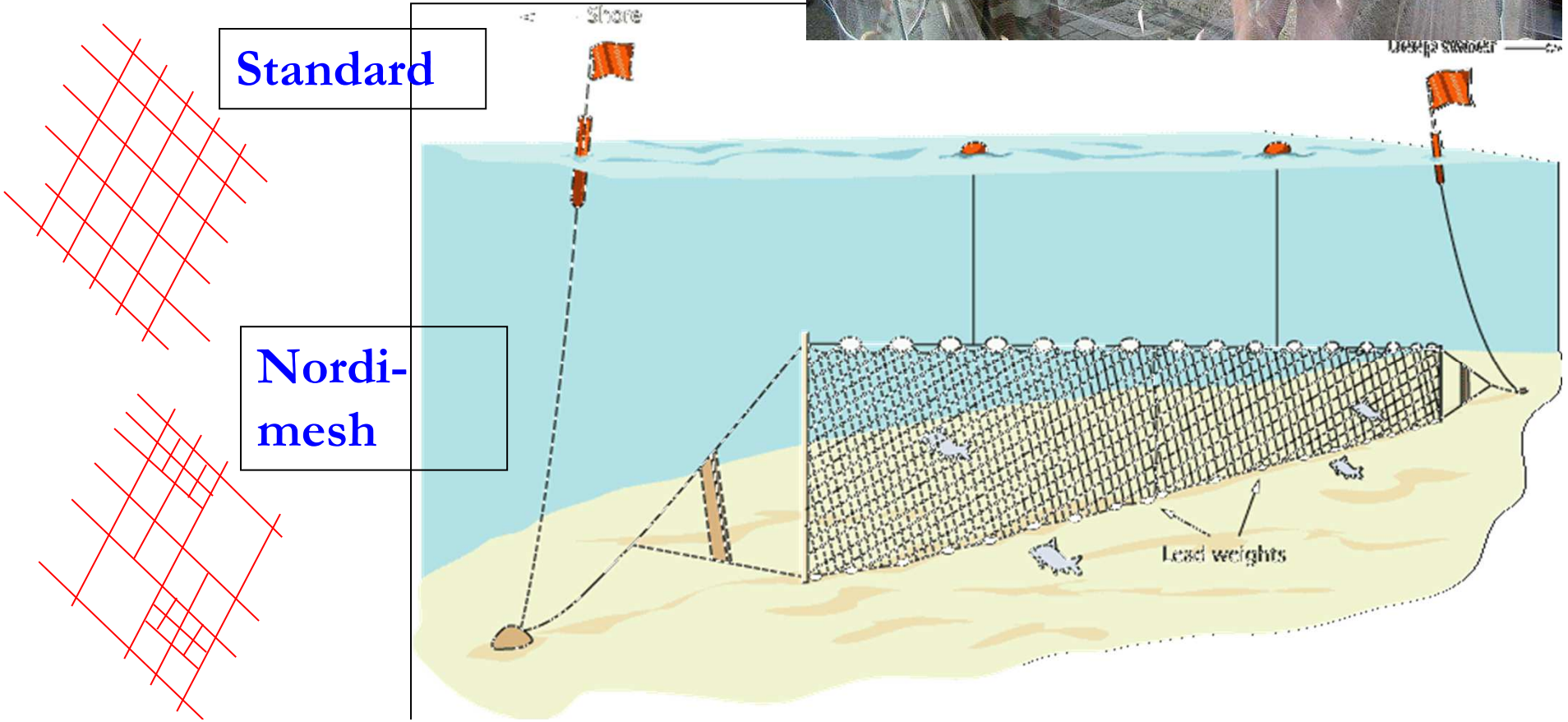
Passive Gear

Entanglement – gill nets

Considerations: habitat, depth, mesh size, mortality

Standard

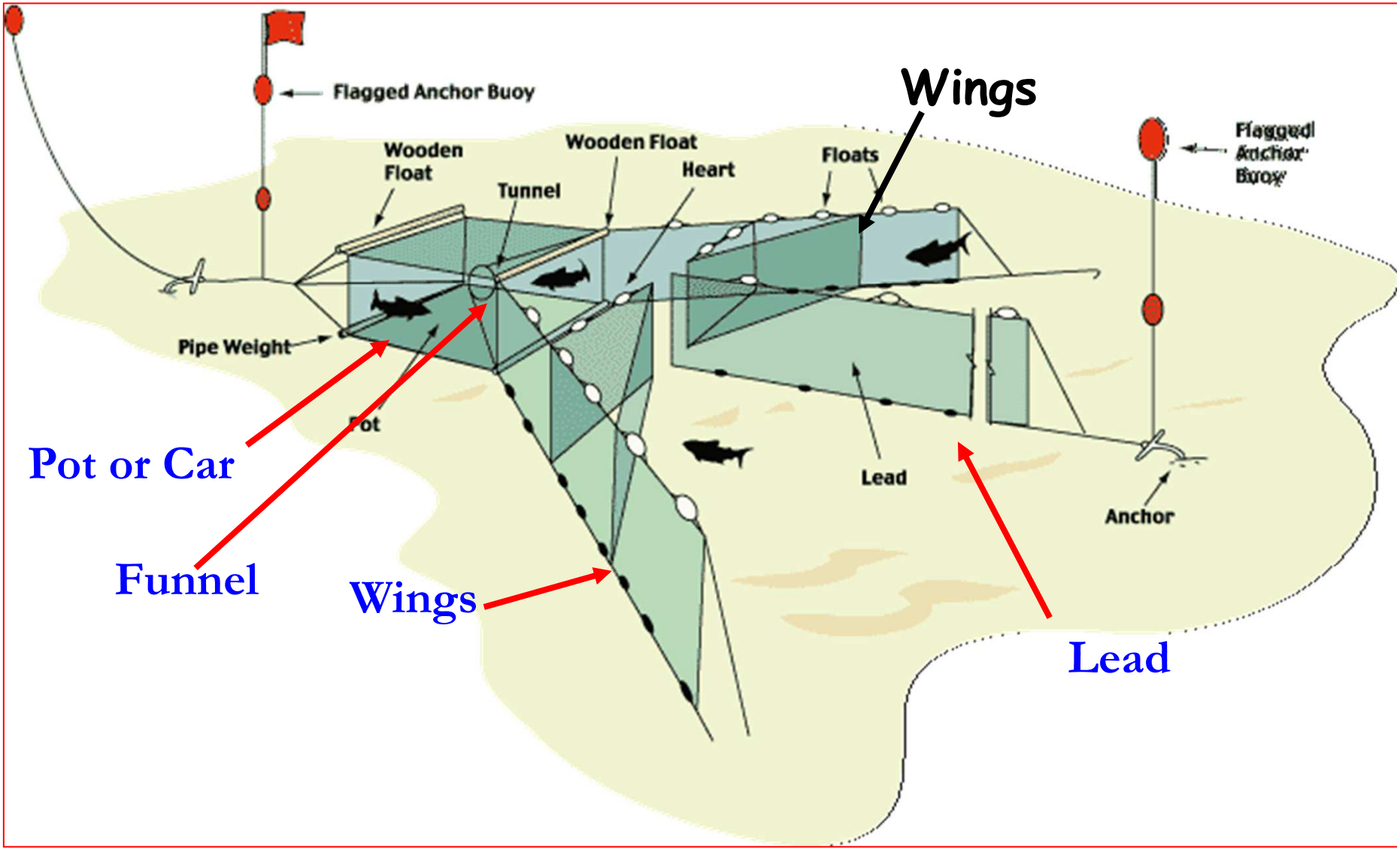
Nordi-mesh



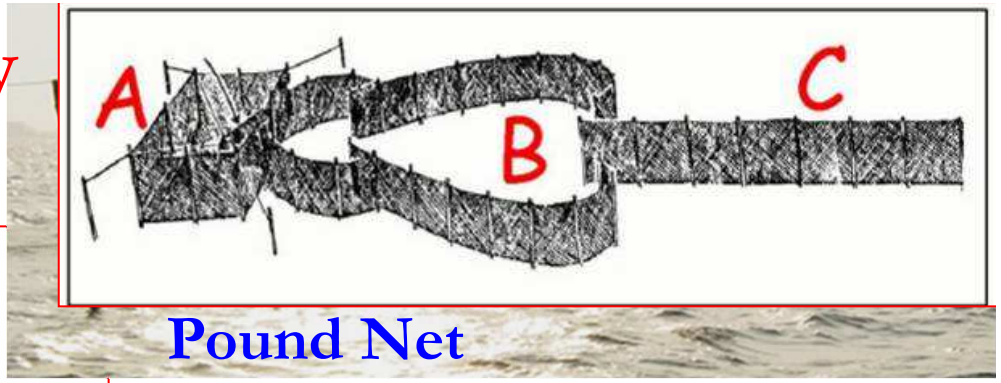
Passive Gear

Considerations:
location, mesh size, funnel diameter

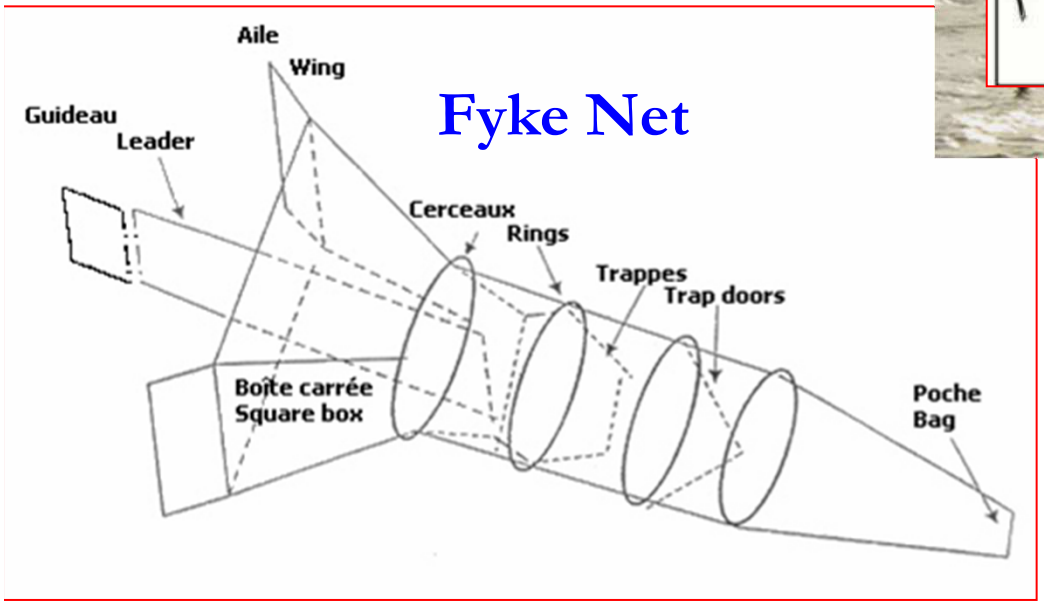
Entrapment- trap nets



Trap nets, lots of variety

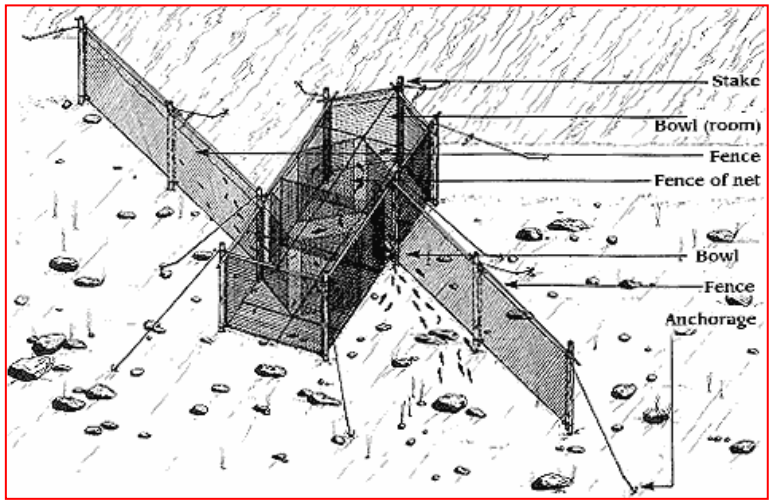


Pound Net

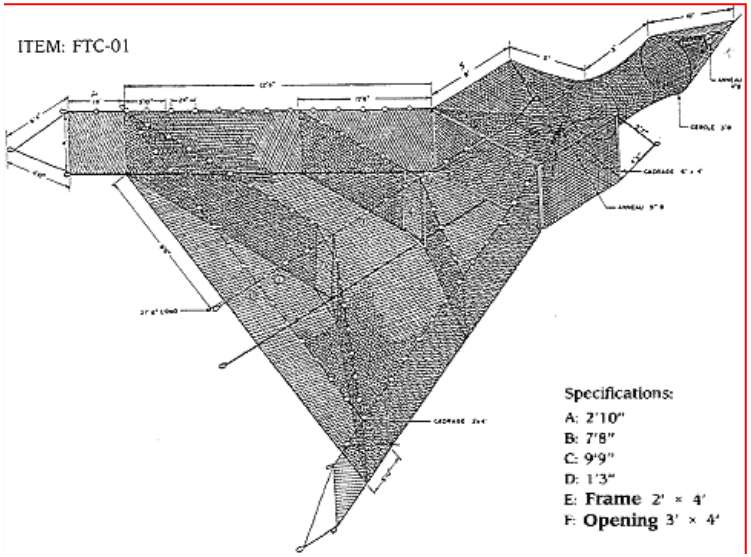


Fyke Net

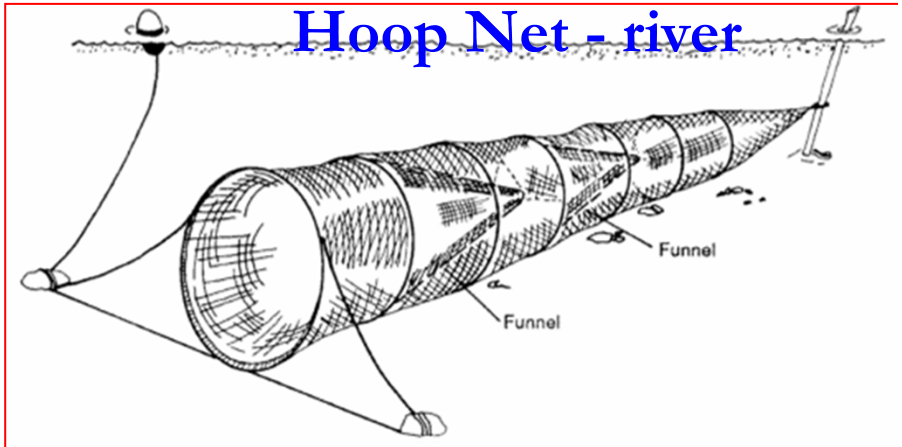
River Trap Net



PA style trap Net



Hoop Net - river



Passive Gear – Fish Weirs

Block entire stream, catch everything going up or down

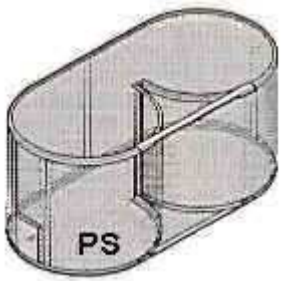
Considerations: location, high level of effort, non-target effects

Passive Gear

Minnow trap – can mean anything

Considerations: location, mesh size, funnel diameter, trap fullness = more or less caught, predation

Gee style



B style

Glass style

Square

Plastic style

Passive Gear

Angling – sort of a weird mix between active/passive

Considerations: time and place, what lure, certain types of fish more vulnerable, behavioral effects

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Active gear - electrofishing

- Electrical current immobilizes fish
- Excessive current kills fish or damages fish
- Alternating current (AC) stuns fish in place
- Direct current (DC) draws fish into field
- Many factors affect effectiveness

Active gear - electrofishing

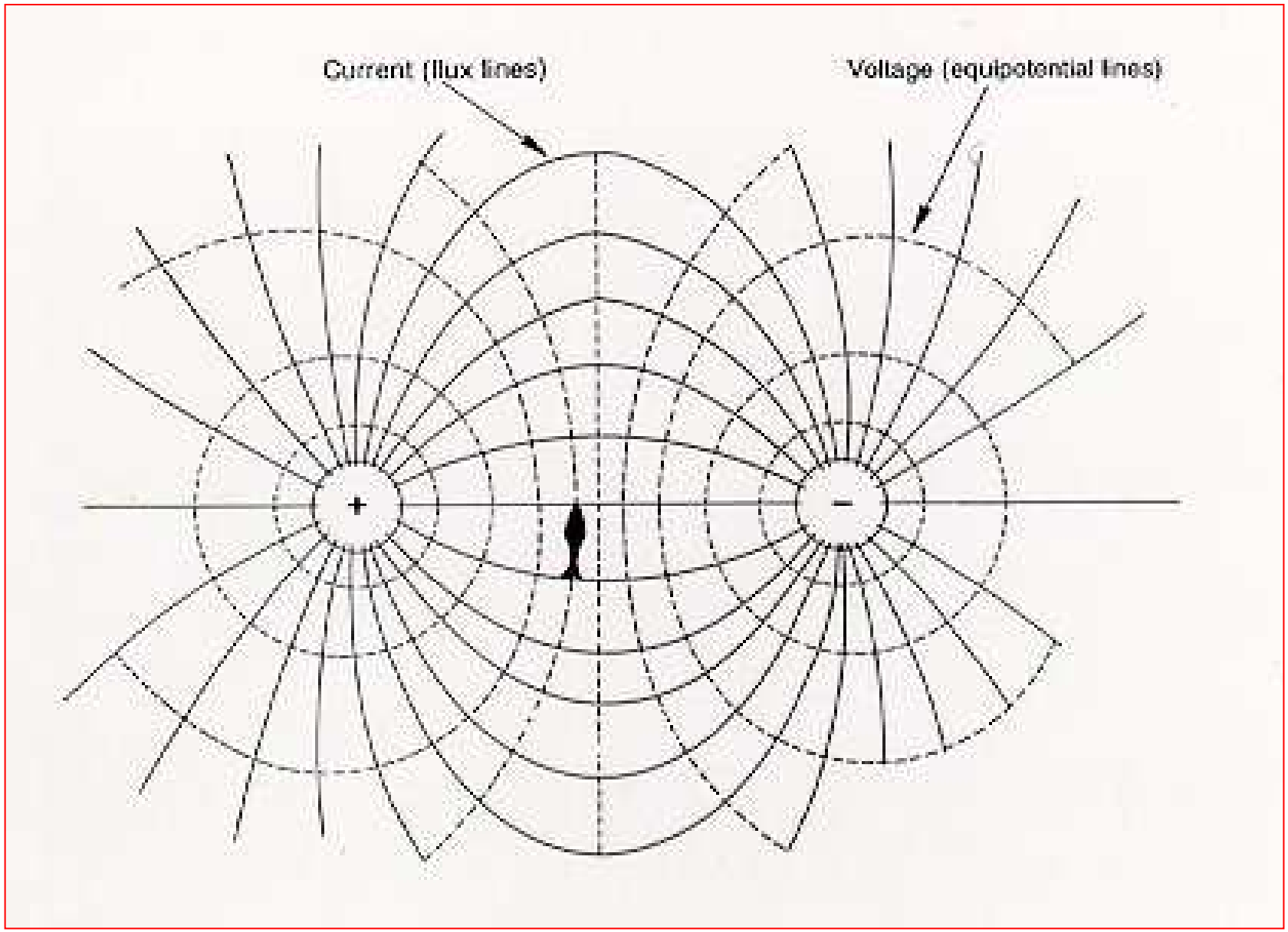
Stream electrofishing - backpack, tow boat, bank based, electric seine

Considerations: safety – fish and researchers, not effective in all habitats

Active gear – boat electrofishing

Considerations: depth, safety again

Electrofishing



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Biological early warning

- Organisms under (semi) controlled conditions
- Exposure to (semi) water flow regime
- Recording of suitable biological functions:
  - response parameter quantifiable
  - rapid reliable detection
  - physiological or behavioural
- Evaluation of measurements
- Detection of abnormal situation

T.C. ÇEVRE VE ŞEHİRCİLİK BAKANLIĞI
Çevre ve İklim Eylemi Sektör Operasyonel Programı
Kalıcı Organik Kirleticiler
UNDP

Biological early warning

- Minimum false alarms
- Organisms inexpensive and easy to handle
- Automated, stand alone
- 24 h/day
- Minimum maintenance
  - Biological
  - Technical

zebra mussel
Dreissena polymorpha

quagga mussel
Dreissena rostriformis bugensis

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Biological early warning

- Different organisms used:
  - Bacteria
  - Algae
  - Daphnia
  - Invertebrates
  - Bivalves (mussels, oysters, clams)
  - Fish
- All respond to a wide range of pollutants
- Dose-effect relation will be different

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti
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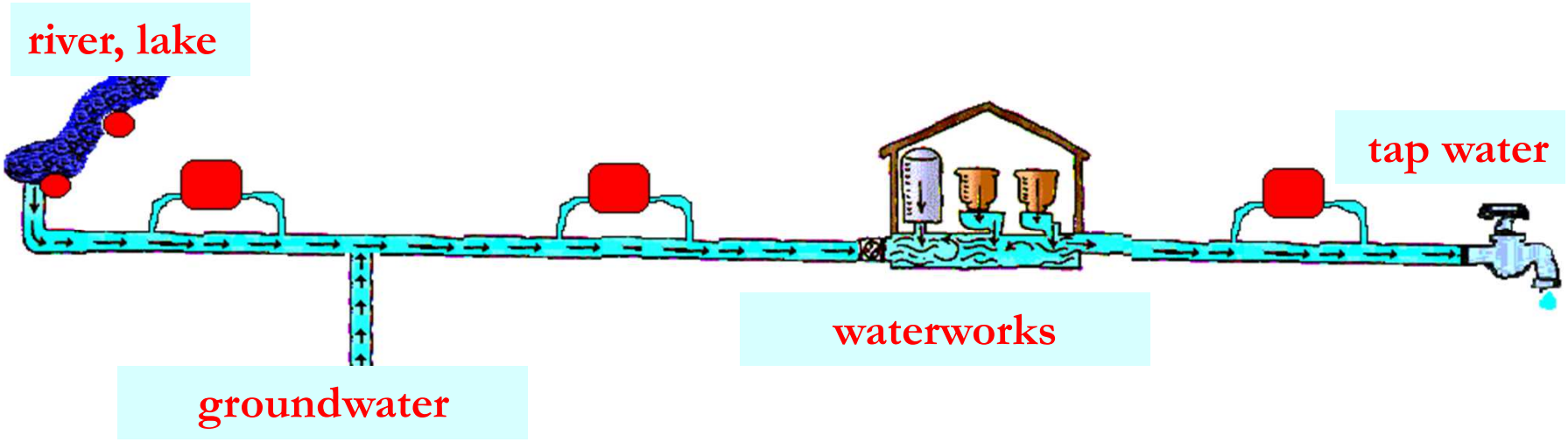
Examples of applications

- Surface water monitoring
- Testing of drinking water inlet
- Domestic/industrial effluents
- Monitoring drinking (tap) water (!)
- Protection of aquaculture (fish / mussel farms)
- Toxicological studies (EC50, NOEC)
- Chemical speciation studies
- Control of chlorination of cooling water

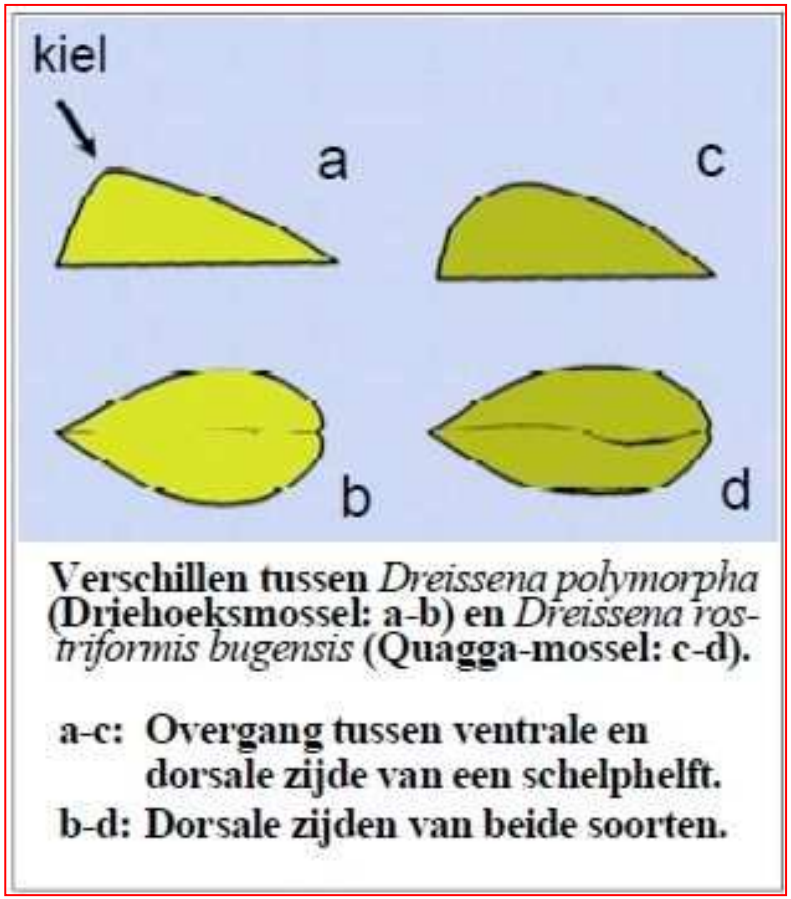
Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Applications



Valve movement response of mussels

- Usually mussels are open
- Mussels close under stress: 'escape' behaviour
- Changes in environmental conditions:
  - Decrease % open
  - Closure
  - Increase frequency



Verschillen tussen *Dreissena polymorpha* (Driehoeksmossel: a-b) en *Dreissena rostriformis bugensis* (Quagga-mossel: c-d).

a-c: Overgang tussen ventrale en dorsale zijde van een schelphelft.
b-d: Dorsale zijden van beide soorten.

zebra mussel *vs.* quagga mussel

After: Bij de Vaate & Jansen, 2008

Bivalve species

Corbicula fluminea

Unio sp.

Anodonta signea

Mytilus edulis

Dreissena polymorpha

Perna viridis

Crassostrea gigas

Contents

Basic soil components

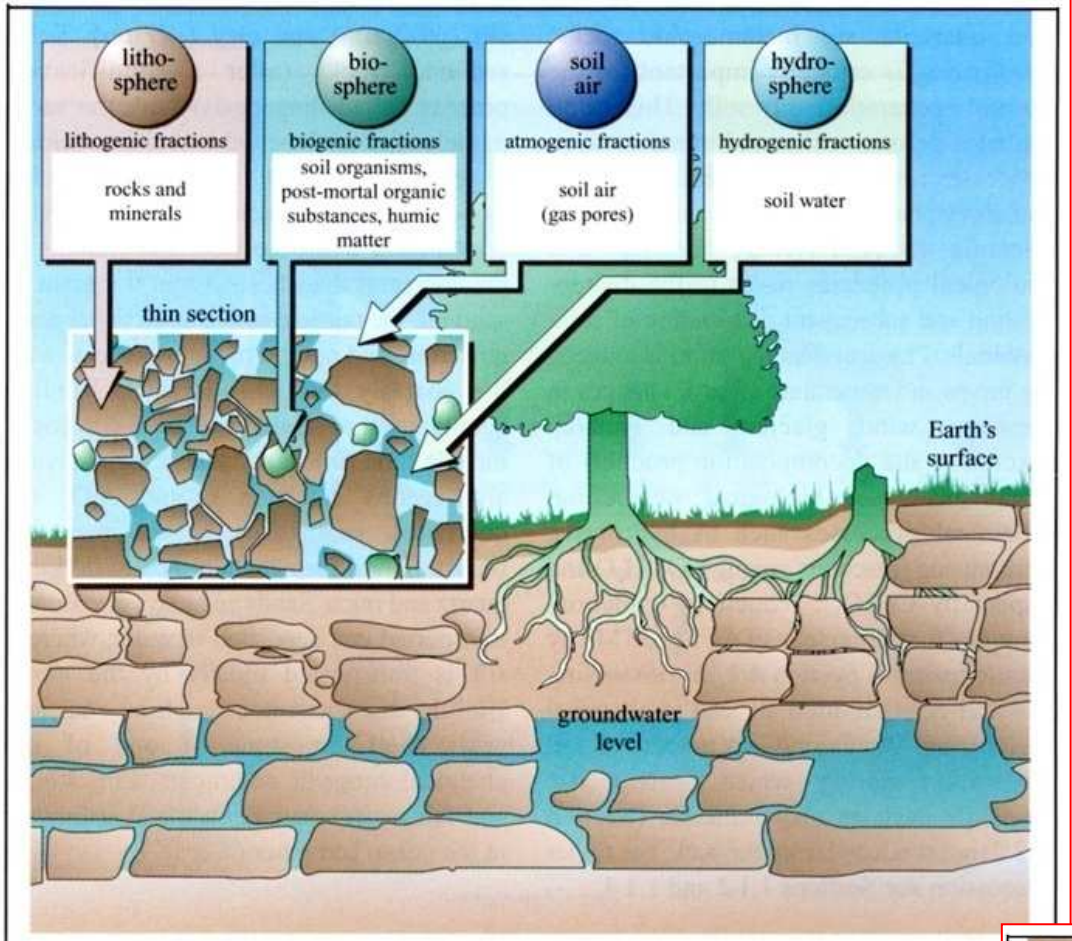


Figure 4.1.2 Components of the litho-, bio-, hydro- and atmosphere

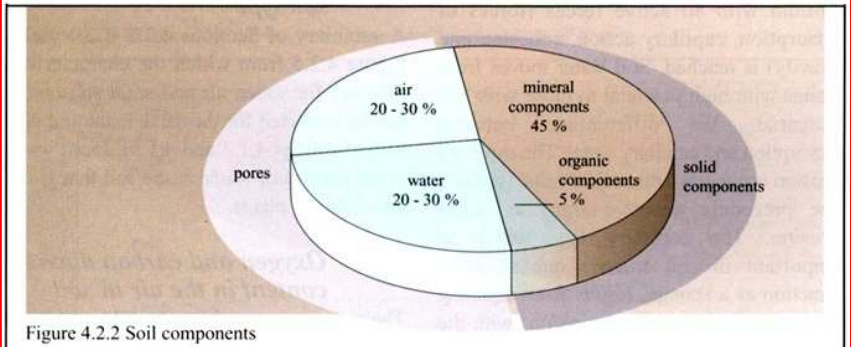
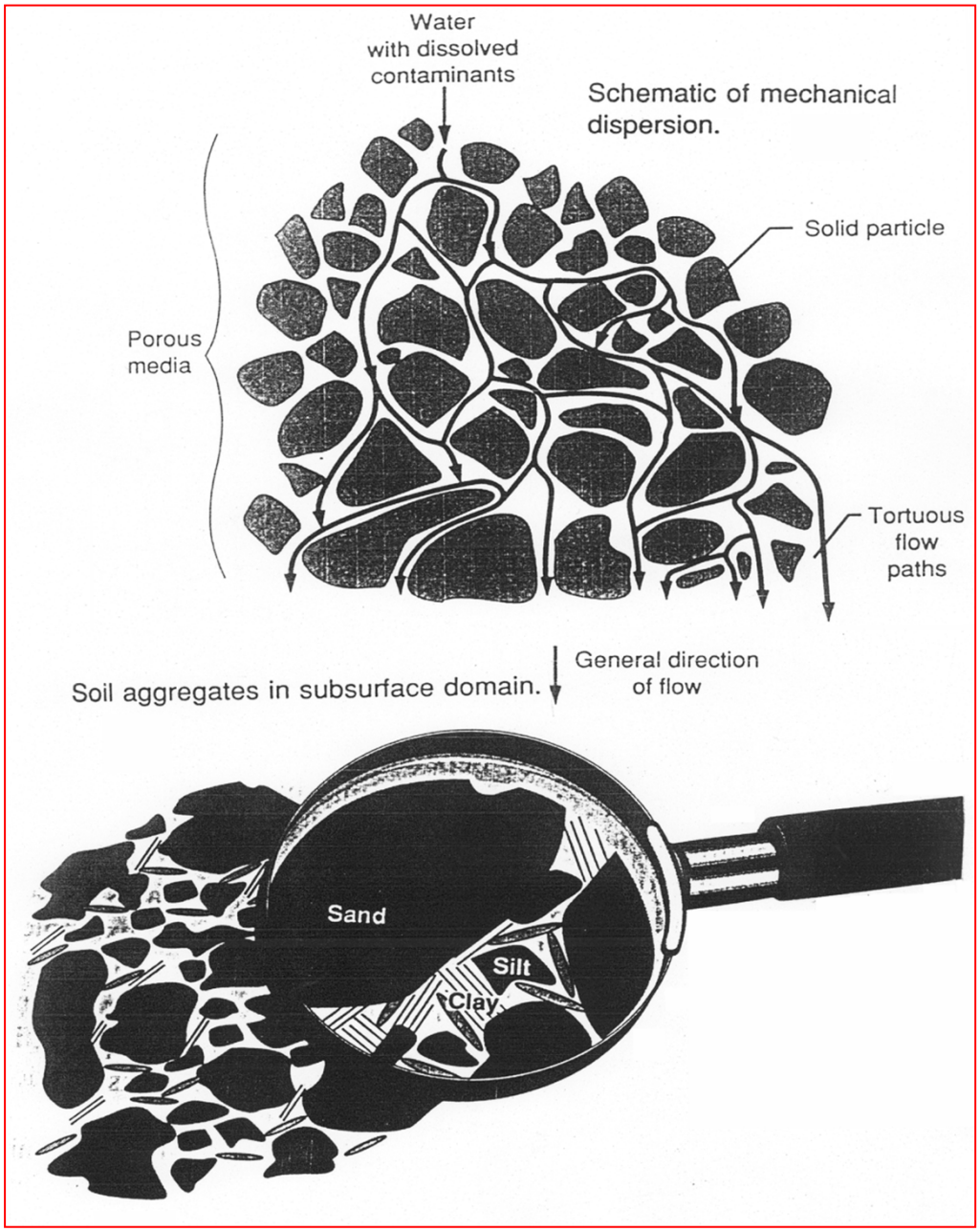
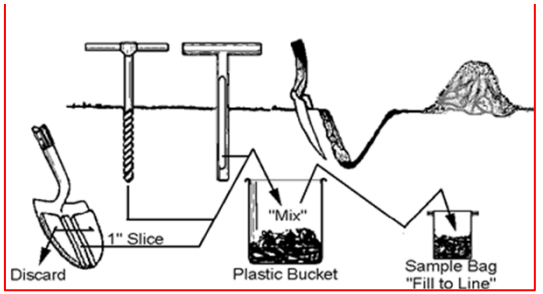


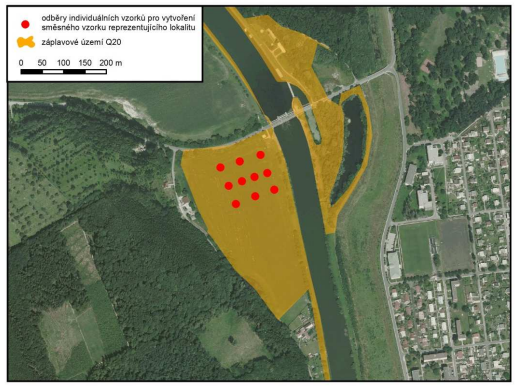
Figure 4.2.2 Soil components

Structure of soil



Soil sampling



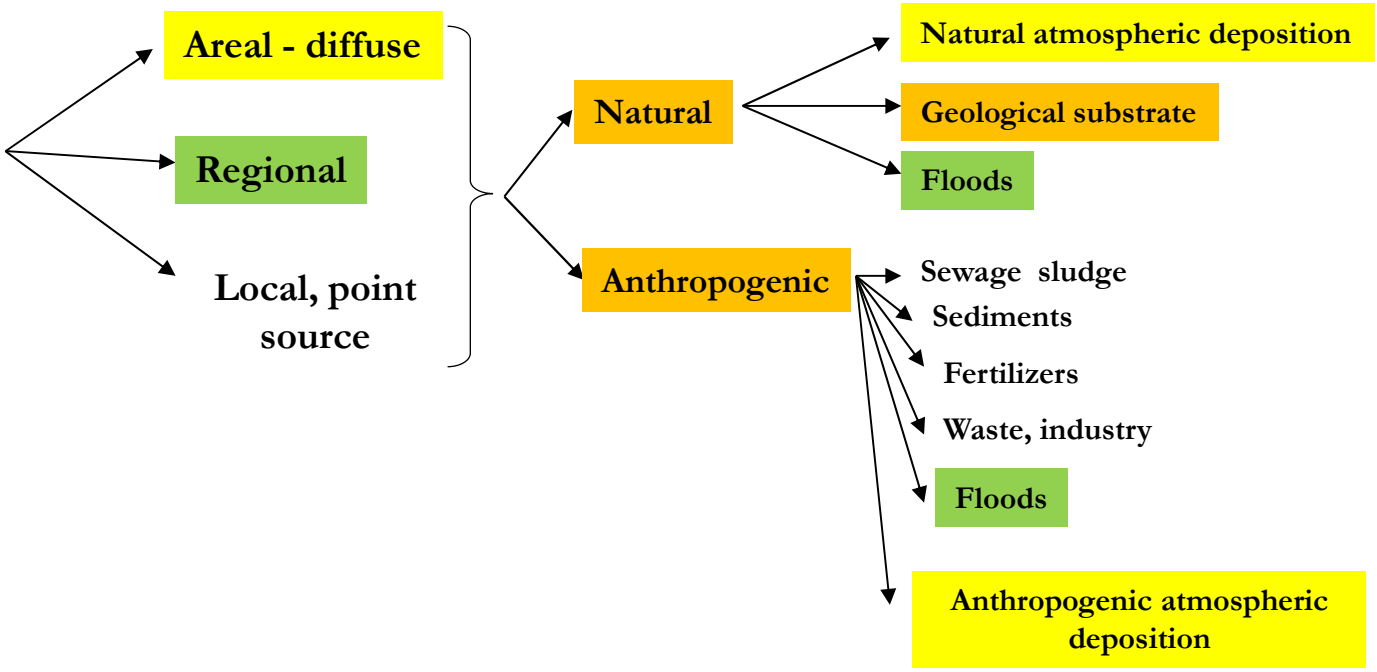


Considerations for soil sampling

- Soils are very complex! Horizontal and vertical spatial distribution
- The oil sample must be representative
  - Composite sample (homogenization)
    - Aliquots needed, same volume
  - Sampling design and tool used mainly depends on purpose of sampling and type of contamination

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Types of soil contamination



Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Types of soil monitoring

- Agricultural, arable soils various types (field, gardens..)
- Grasslands
- Forest soils
- Protected areas
- Industrial areas

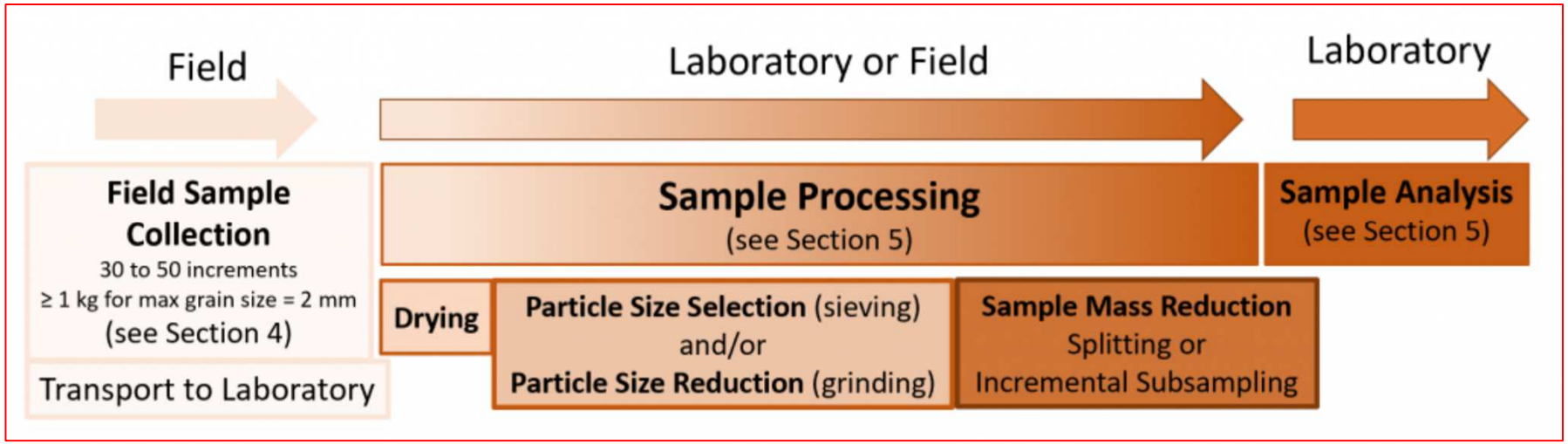
Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Goals of soil monitoring

- Understanding of level of destruction and contamination
- Determination of trends
- Specification of potential negative changes, quantification
- Make a safe feedback – development of conditions for stopping of negative trends (or improvement of actual state)
- Getting of background information for legislature
- Establishment of database for fundamental and applied research

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Soil sampling



Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Methods of soil sampling

Area of sampling:

- Arable soils and grassland – ca 7 - 10 ha (according to soil conditions and production area
- Hop-garden – area for the collection of 1 mixed sample – 3 ha
- Vineyard - 2 ha
- Database – one sample per 1km^2; area for the sampling of 1 mixed samples – at minimum 30 nicks uniformly cover whole investigated area

Depth of sampling:

- Arable land - 30 cm
- Grasslands - 15 cm with removal of upper turf layer
- Hop-garden - 40 cm with removal of upper 10 cm layer
- Vineyards – sampling of two layers 0 - 30 and 30 - 60 cm
- Intensive orchards - 30 cm.

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Soil sampling

Based on the type of analysis

Sampling probe tube, soil borer (mg kg^{-1})

Depth of sampling – based on the goal of monitoring

0 - 5; 5 – 10 cm ....pesticides applied by spraying, ambient air

0 - 30; 30 – 60 cm ....(based on the depth of root systems)

heavy metals, pesticide residua, xenobiotics, nutrients: N, P, K

Average sample - 30 stabs (nicks)

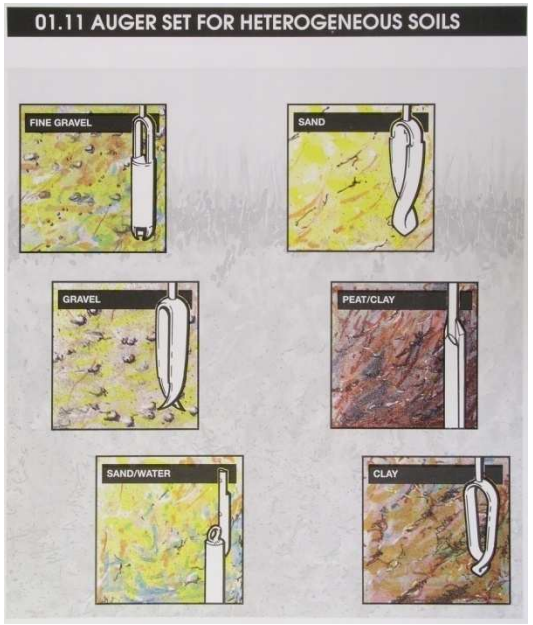
- lowland - 9,5 ha one sample
- mountains - 6,5 ha

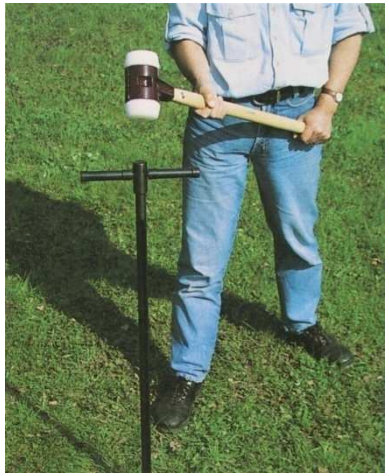
Distribution of stabs regularly whole area (cross the field)

Soil sampling

Sampling sets with various types of probes and soil borer – according to the type of soil

Soil sampling



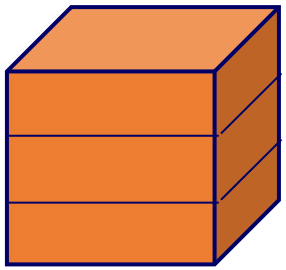


Soil sampling

Areal contamination (accidents)

Sampling of ashlars - 30 x 30 x 30 cm - horizons

0 – 5 cm
5 – 15 cm
15 – 30 cm



In exactly selected localities *(with respect of deposition shadow, wind directions, local anthropogennic activities)*

Frequency of sampling – according to the goals of monitoring

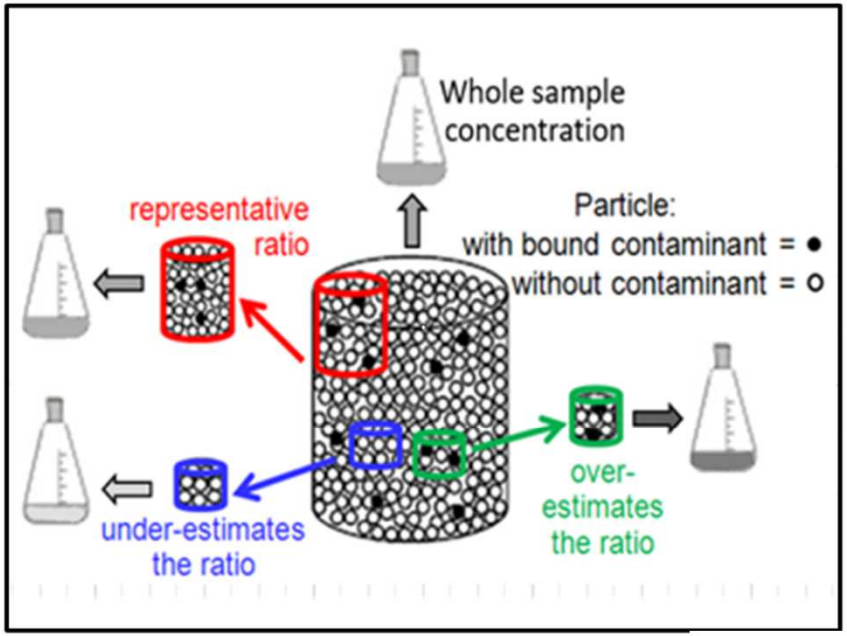
Samples - PE (paper) bags with description, dispatch note

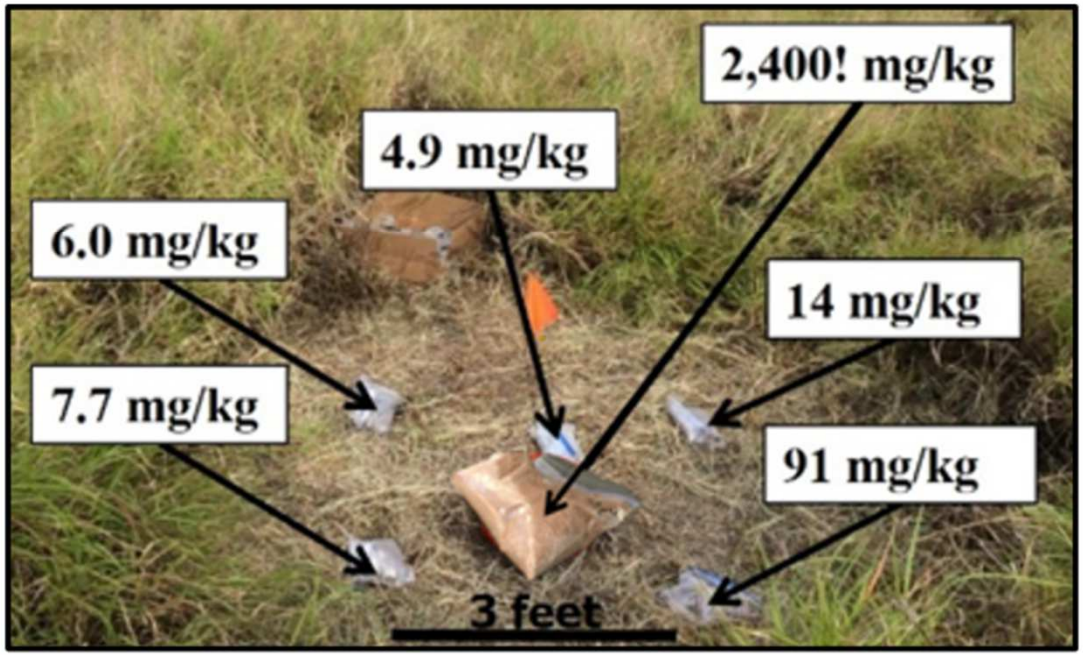
In the case when the degradation or transformation can be expected – cooling bags

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Soil sampling

Soil heterogenicity



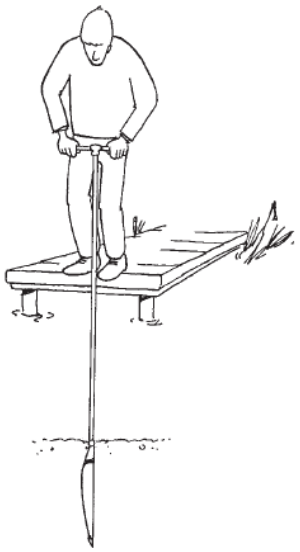


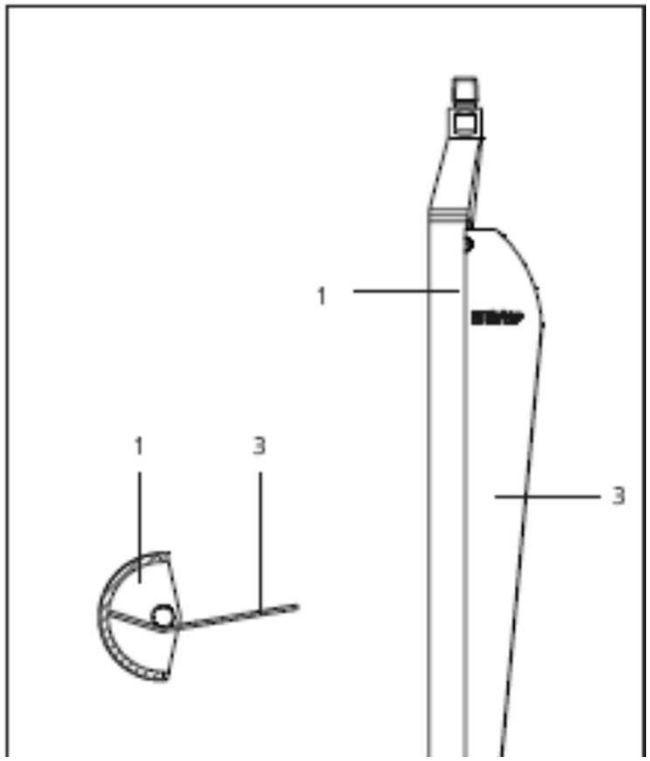
Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

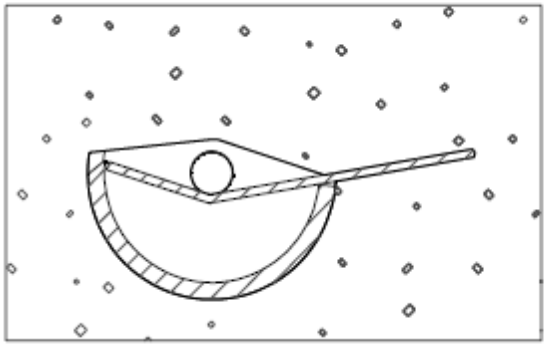
Peat samplers

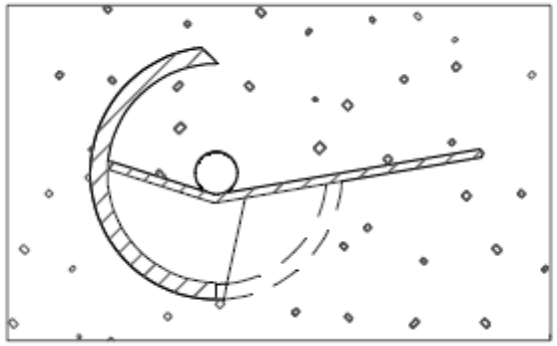
- 04.09 Peat sampler
  - Benefits
    - Samples saturated AND unsaturated material
    - Stainless steel sample body for all analyses
    - Simple gouge-with-a-flap principle
    - Effective to sample young peat and sediment
    - Takes point samples at any depth (if very soft)
  - Remarks
    - Thick point limits penetration in stiff material
    - Water plant roots will be pushed aside
    - Water and watery top soil will flow out

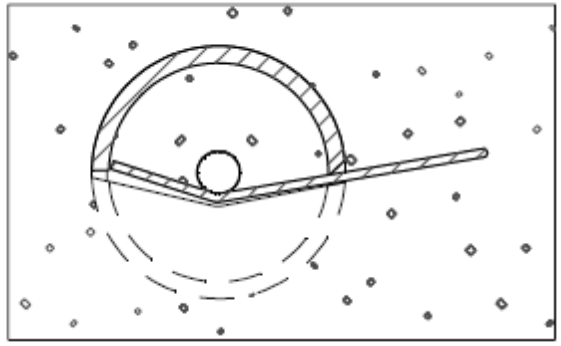
Peat sampler operation









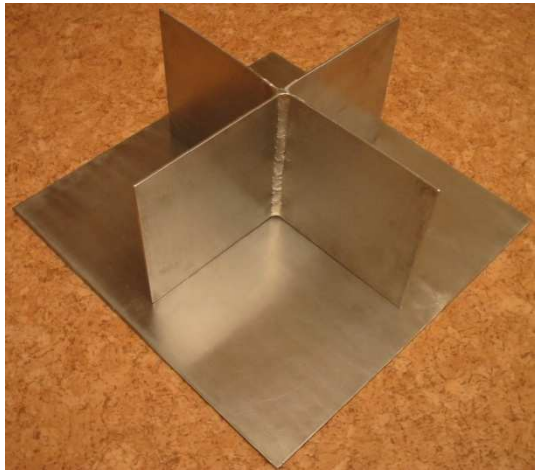


Homogenisation, sieving, dividing of samples

Crushing, grinding, homogenization

Sieving

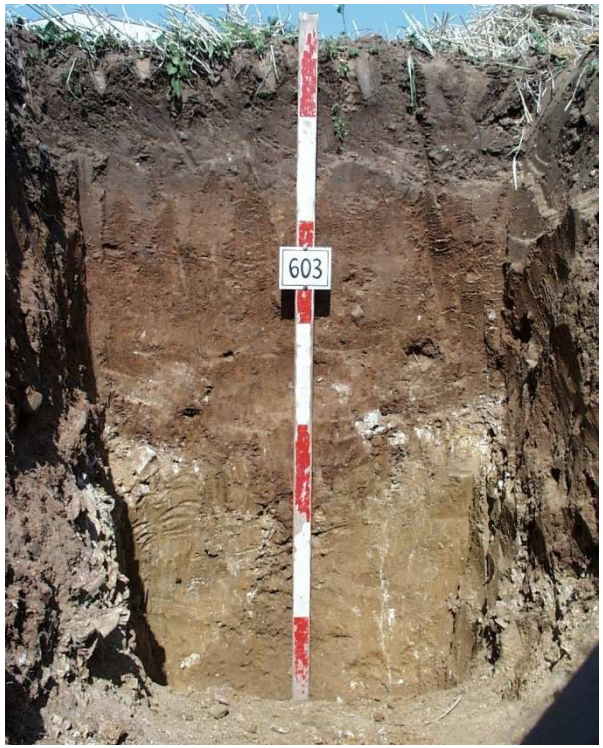
Dividing of sample



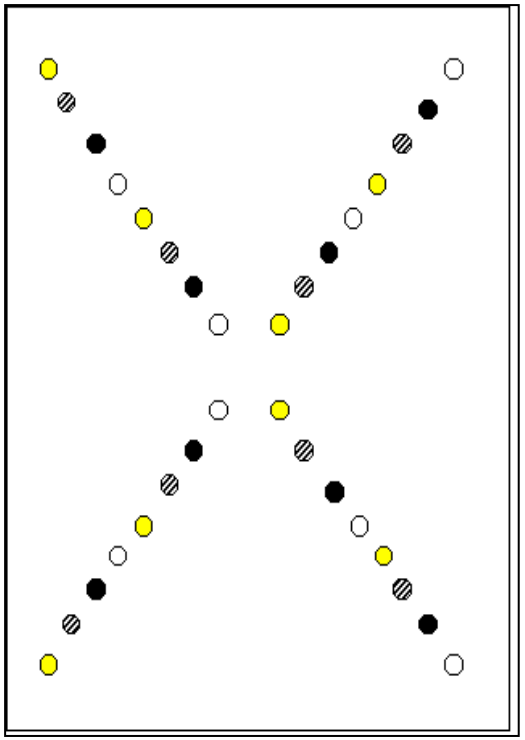
Quarter cruise

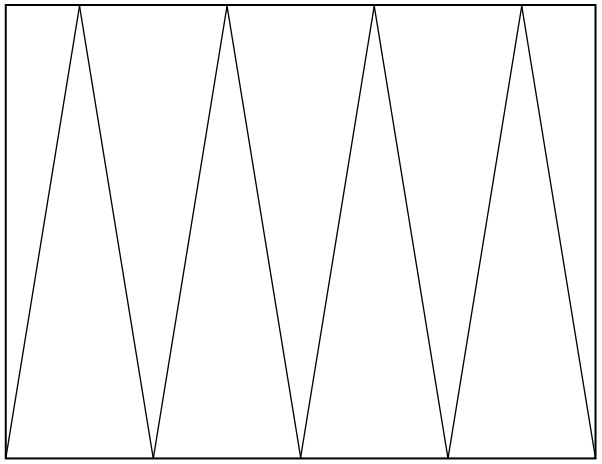
Sampling scheme on the monitoring plot

soil pit



25 m





40 m

no. 1 no. 2 no. 3 no. 4

individual sampling to get four composite samples

Basal soil monitoring program - setting the soil characteristics

Unrepeated parameters monitored at establishing the monitoring plot

- recording of the identification information of a monitoring plot
- soil pit description
- analyses of physical parameters (complete analyses of disturbed and undisturbed soil samples)

Parameters monitored in six years period

- active and exchangeable soil reaction
- contents of available nutrients - P, K, Mg, Ca analysed by several methods
- contents of microelements (B, Mo, Mn, Zn, Cu, Fe)
- sorption capacity (S, T, V)
- organic matter content (Cox)
- risk element contents in nitric acid (As, Be, Cd, Co, Cr, Cu, Ni, Pb, V, Zn) and aqua regia (Al, As, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Ni, P, Pb, V, Zn) extraction and total Hg content

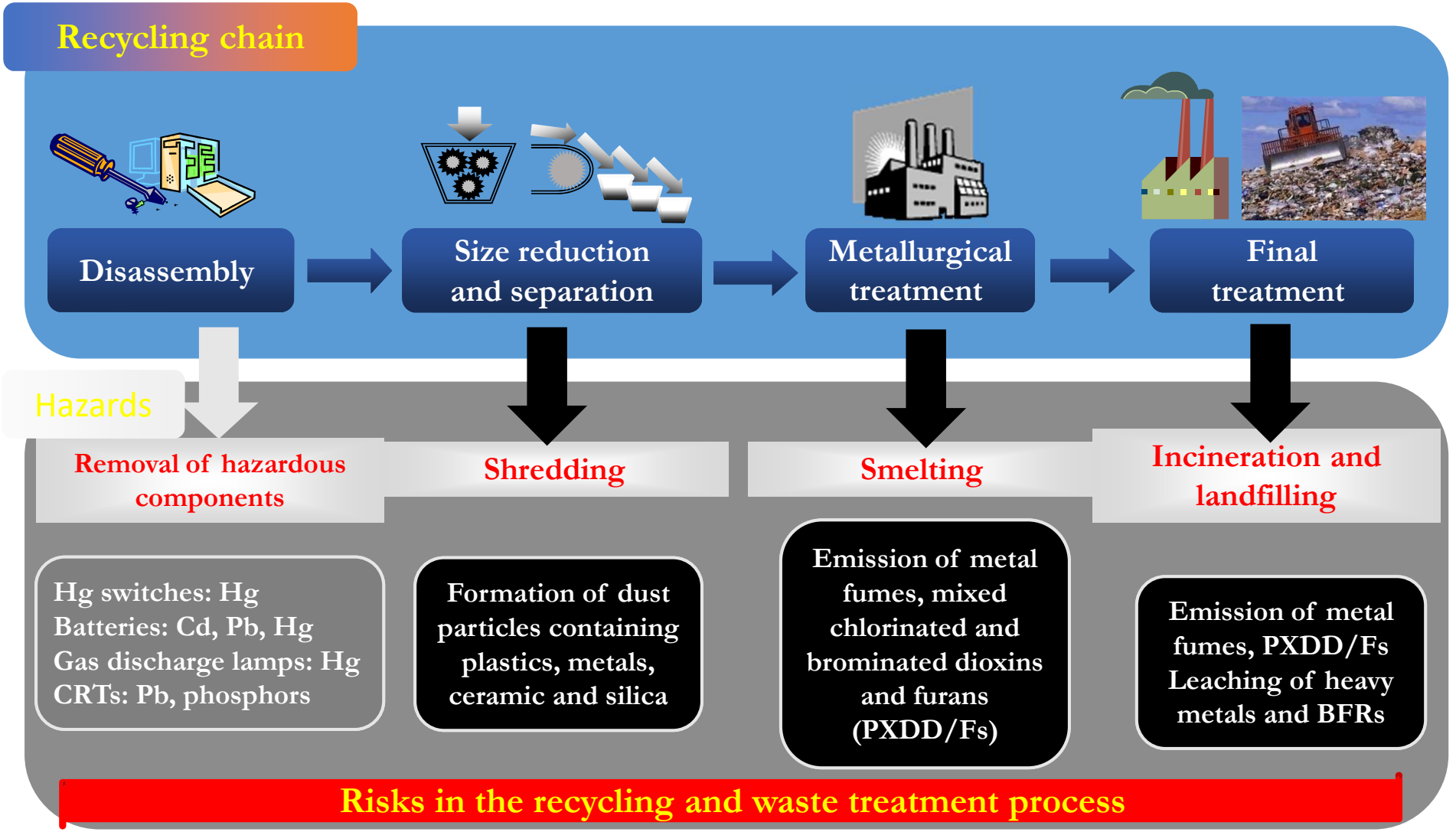
Yearly monitored parameters

- content of mineral nitrogen
- chosen microbiological and biochemical parameters
- contents of selected organic pollutants (persistent organochloric pesticides, PCBs, PAHs, PCDDs/Fs)
- contents of risk elements in plants on contaminated plots

Contents

- Sediment monitoring – active sampling
- Sediment monitoring – passive sampling
- Design of sediment sampling
- Biota monitoring – approaches, sampling
- Soil – sampling, monitoring
- Technical matrices – sampling, monitoring
- Plastics

Hazards associated with the recycling chain



E-waste

E-waste

The global impact of e-waste. Addressing the challenge

Karin Lundgren
SafeWork and Sector
International Labour Organization, Geneva 2012

Chemicals in products/articles
Analysis of chemicals in new products/articles

Samples:

- **Liquid crystal display (LCD) TV, Laptop PC, Power supply unit**
- **Wallpaper, Curtain, Heat insulation material**

Target compounds:

- Organobromine compounds (PBPhs, TBBPA, HBCDs, PBDEs)
- Phosphoester plasticizers and flame retardants (TMP, TEP, TPrP, TBP, TClPP, TCEP, TBEP, TDCPP, TOP, TPhP, TCP)

Analytical method:

- **Samples were pulverized by frost shattering using liquid N_2.**
- **HRGC/HRMS and LC/MS methods.**

Pulverized samples

Rear cover

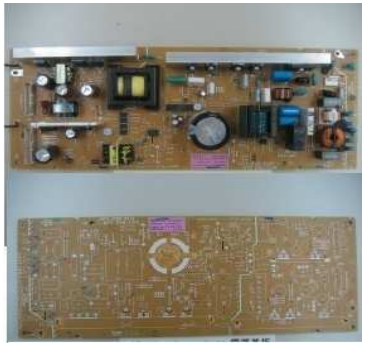
Front cover

Chemicals in products/articles

LCD panel

Printed circuit board

Printed circuit board (power supply unit)



Printed circuit board (LCD panel)

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Measurement of POPs in waste matrices - problem of matrix

Wastes:

- liquids ⇔ solids
- soil like ⇔ plastics
- homogeneous ⇔ complex mixture
- << interferences ⇔ >> interferences

- no fixed matrix “waste” from an analytical point of view

- no fixed method for matrix “waste” from an analytical point of view

- no fixed analytical sensitivity as no fixed matrix

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Analysis of POPs in waste matrices

Steps for analysis of POPs: (examples for available techniques)

1. Sampling/Transport
2. Pretreatment: grinding, centrifugation, filtration
3. Extraction: liquid / liquid
   shaking / ultrasonic
   soxhlet and ASE
4. Clean-up: gel permeation
   multi-layer silica
   carbon
   alumina
5. Measurement: GC/ECD
   MS/HRMS

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

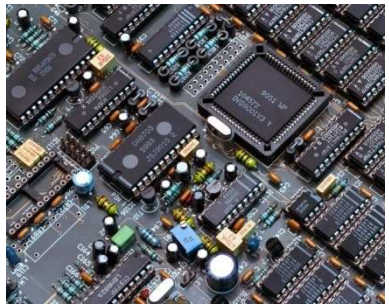
POPs measurement standards for waste

Harmonised European standards not yet available

- Recommendation for standard requirements
- Modular system including the different analytical techniques used and recommendation which technique can be used for which matrix
- Final decision on the methods used has to be taken by the analyst depending on individual matrix
- Minimum performance criteria have to be accomplished" (QA/QC)

→ Standards to be developed or under development by CEN/TC 292 (sampling, analysis, leaching)

Materials and consumer products



Collection of many kinds of materials and consumer products used as indoor equipment (plastic casing of electronic goods, tapestry, insulation foams, carpets, flooring, paints, pieces of furniture…) of various age totally about 60 individual samples (1st part)

Consumer products study

- collection of many kinds of materials and consumer products used as indoor equipment of various age
  - plastic casing of electronic goods
  - fabric, carpets, flooring, paints
  - pieces of furniture
  - insulation foams
- hypothesis of connection between high contaminated consumer products and indoor contamination



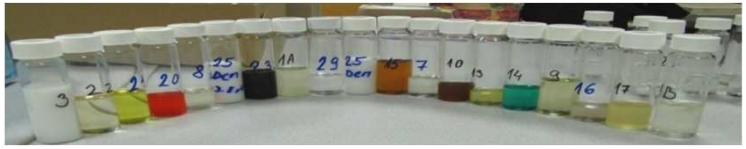


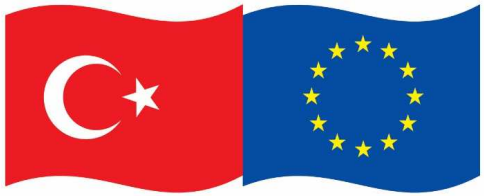




Consumer products study

- totally about 80 individual samples
- samples crushed and homogenized
- sample preparation for FOSAs and FOSEs
  - ½ samples - DCM Soxhlet extraction
  - cleaned-up on a activated silica column
- sample preparation for PFCAs and PFSAs
  - ½ samples - Soxhlet warm extraction with
  - MeOH with 5mM amonium acetate
  - filtration (0.25 mm nylon syringe filter)
- LC - MS/MS analysis of both groups (Agilent 1100 + Applied Biosystem QTRAP 5500)





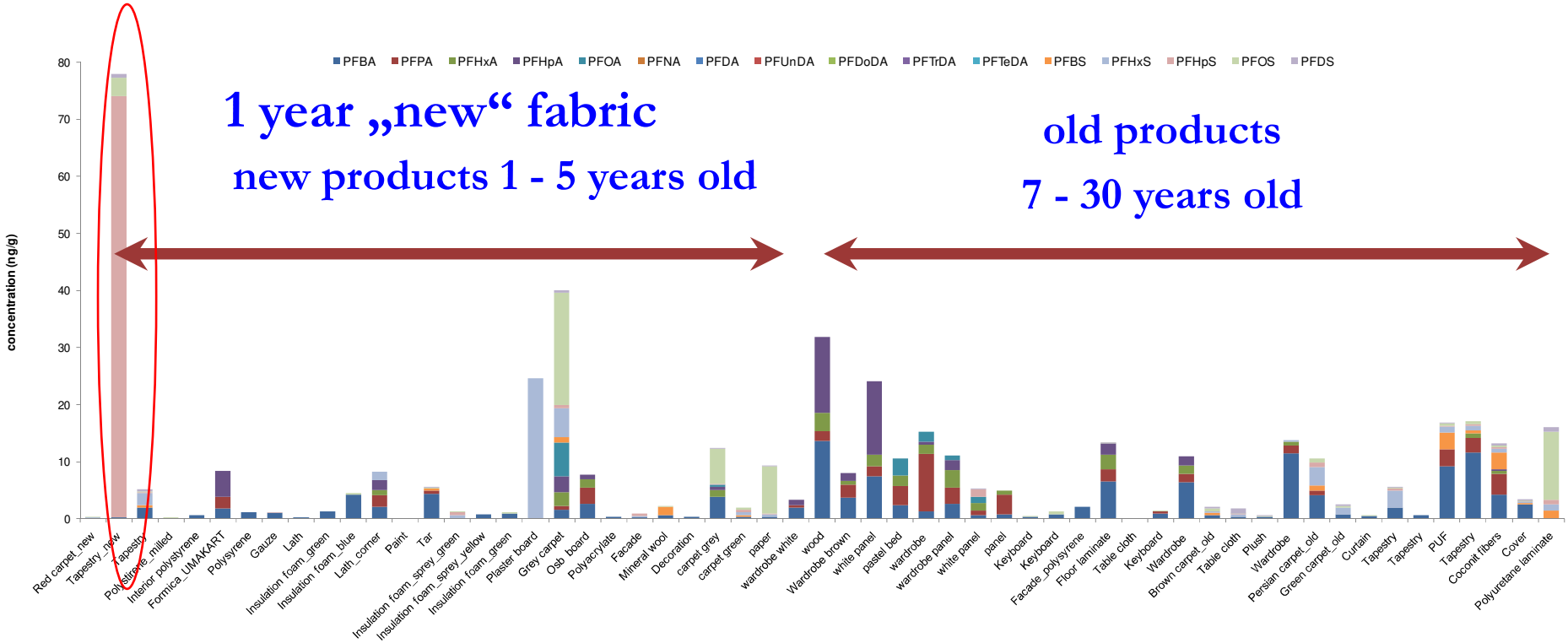
Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Materials analysis, sample homogenization

- plastic samples: chopping into small pieces, preferably after soaking the samples in liquid nitrogen (plastic become very fragile when deep frozen)
- textiles, foams: cutting into small pieces
- wood, furniture: drilling, taking the wood shavings
- polystyrene dissolves in DCM

Consumer products study

Used consumer products from sampled households - analysis of PFCAs and PFASs



Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

PBDEs and PFCs in e-wastes and car wastes in the Czech Republic

Klánová, J., Kukučka, P., Bečanová, J., Benešová, J. Bačová, B.: Polybromované difenylethery a perfluorované látky v elektronických odpadech a odpadech z automobilových vraků. Masarykova univerzita, RECETOX REPORT No. 401. Brno, březen 2011.

T.C. ÇEVRE VE ŞEHİRCİLİK BAKANLIĞI

Çevre ve İklim Eylemi Sektör Operasyonel Programı

Kalıcı Organik Kirleticiler

UNDP

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti
tarafından ortaklaşa finanse edilmektedir.

PBDEs and PFCs in e-wastes and car wastes in the Czech Republic

PBDEs

PUF – Škoda car – 489 017 ng g^{-1}
PUF – Hyundai – 76 ng g^{-1}

PFOS

PUF – Škoda car – 35.5 ng g^{-1}
PUF – Hyundai – 0.03 ng g^{-1}
Carpet Hyundai – 15.3 ng g^{-1}

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Çevre ve İklim Eylemi Sektör Operasyonel Programı
Kalıcı Organik Kirleticiler
UNDP

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PBDEs and PFCs in e-wastes and car wastes in the Czech Republic

PBDEs

Mixed plastic from small electrical equipments – 362 000 ng g^{-1}

Dust from the factory for waste treatment

362 000 ng g^{-1}

PFOS – 2,5 ng g^{-1}

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Çevre ve İklim Eylemi Sektör Operasyonel Programı

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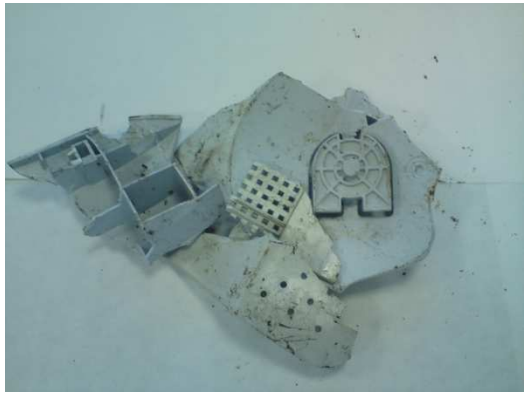
PBDEs and PFCs in e-wastes and car wastes in the Czech Republic



PBDEs

Monitors - 530 – 1 136 ng g^{-1}

TV plastics – 253 042 ng g^{-1}

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Çevre ve İklim Eylemi Sektör Operasyonel Programı
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UNDP

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

House dust - a pilot study

- hypothesis of high PFCs levels in dust from rooms with new equipment
- collect 40 house dust samples

- information about furniture, fabric, floor covering etc.
- different sampling techniques (depending on inhabitant)
  - vacuum cleaner
  - settled dust
- sieve (0.2 µm) and exclude samples with low content of dust
  - hair, food residues, etc.
- remain only 15 samples with ample amount of dust

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

House dust - a pilot study

Sample preparation

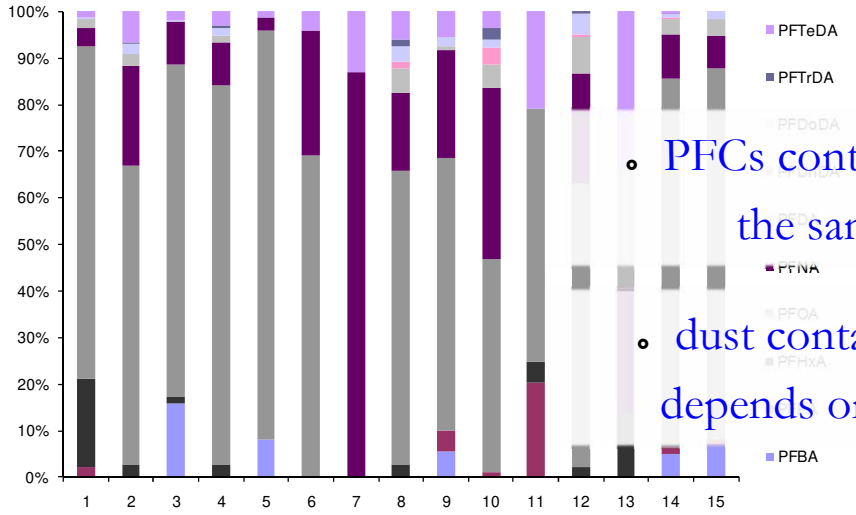
- Soxhlet warm extraction (standard procedures for extraction of POPs - PCBs, PAHs, BFRs) using MeOH with amonnium acetate
  - needs large amount of sample
  - precipation during extraction
- ion-par extraction (used for extraction of PFCs from biota, sediment[1], etc.)
  - needs low amount of sample
  - simple sample preparation (0.4 - 1.5 g of individual dust + 1 ml of terc-butyl ammonium hydrogen sulfate + 4 ml of Na_2CO_3 (pH = 10) ⇨ shaking + 5 ml of MTBE ⇨ 30 minutes shaking ⇨ centrifuged and MTBE aliquot

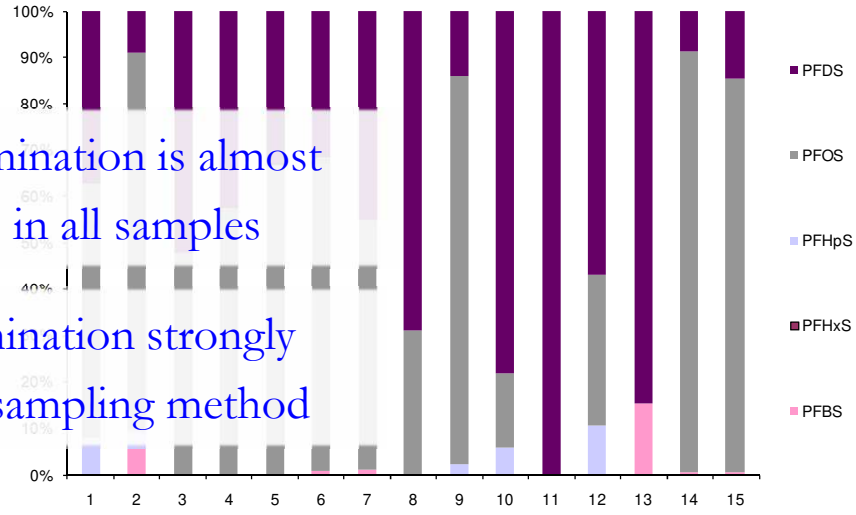
LC - MS/MS analysis of PFCAs, PFASs

(Agilent 1100 + Applied Biosystem QTRAP 5500)

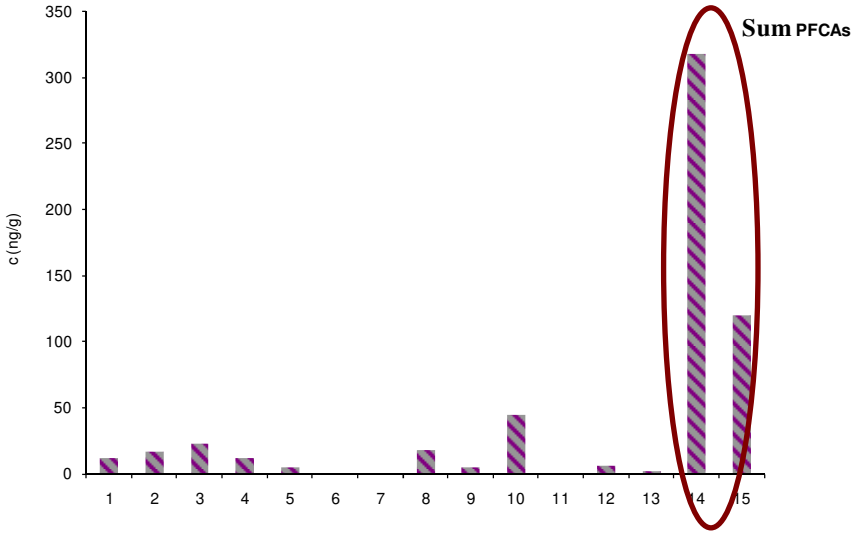
[1] D'Eon, J.C., et al., Environmental Science & Technology, 2009. 43(12): p. 4589-4594.

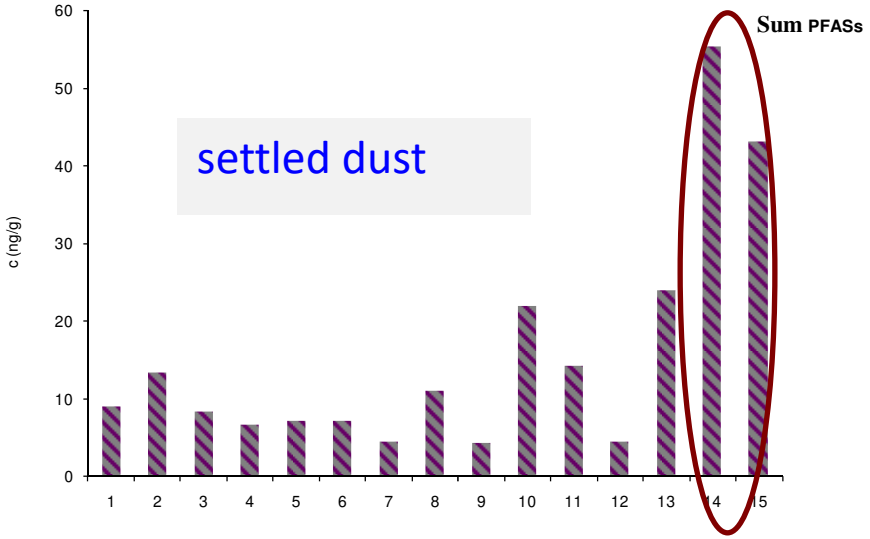
House dust - a pilot study





- PFCs contamination is almost the same in all samples
- dust contamination strongly depends on sampling method





Indoor/ambient air study

20 flats and houses in Brno and surrounding:

- non refurbished houses from 1920s
- non refurbished brick of prefabricated flats
- refurbished brick of prefabricated flats (both from 1940s to 1990s)
- newly built flats
- newly built houses of "common" construction
- low-energy houses with heat exchange ventilation units

Sampling in August 2010 (summer) and February 2011 (winter)

Hypothesis of increased PFCs levels in rooms with new equipment (generally indoor)

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Indoor/ambient air study

Passive sampling using PUF disks deployed for 28 days - simple tool, good „inhabitant acceptance factor“

Sample preparation:

- Soxhlet warm extraction with DCM, addition of ^{13}C labeled standards before extraction
- ¾ extract processed for BFRs and PCBs (not included in this presentation)
- ¼ extract cleaned-up on an activated silica column, sample volume reduction

LC - MS/MS analysis of FOSAs and FOSEs
(Agilent 1100 + Applied Biosystem QTRAP 5500)

T.C. ÇEVRE VE ŞEHİRCİLİK BAKANLIĞI

Çevre ve İklim Eylemi Sektör Operasyonel Programı

Kalıcı Organik Kirleticiler

UNDP

Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Indoor/ambient air study

- PFCs contamination in all flats/houses near to LOQ
- PFCs outdoor contamination bellow LOQ in both seasons
- indoor levels mostly higher in summer than in winter
- the highest levels found in the living rooms (than in bedrooms) with new equipment
  - newly built flats/houses
  - refurbished flats
- the lowest levels were observed in the oldest houses

  - (more than 60 years old) with low number of new devices

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Kalıcı Organik Kirleticiler

UNDP

Contents

- Sediment monitoring – active sampling
- Sediment monitoring – passive sampling
- Design of sediment sampling
- Biota monitoring – approaches, sampling
- Soil – sampling, monitoring
- Technical matrices – sampling, monitoring
- Plastics

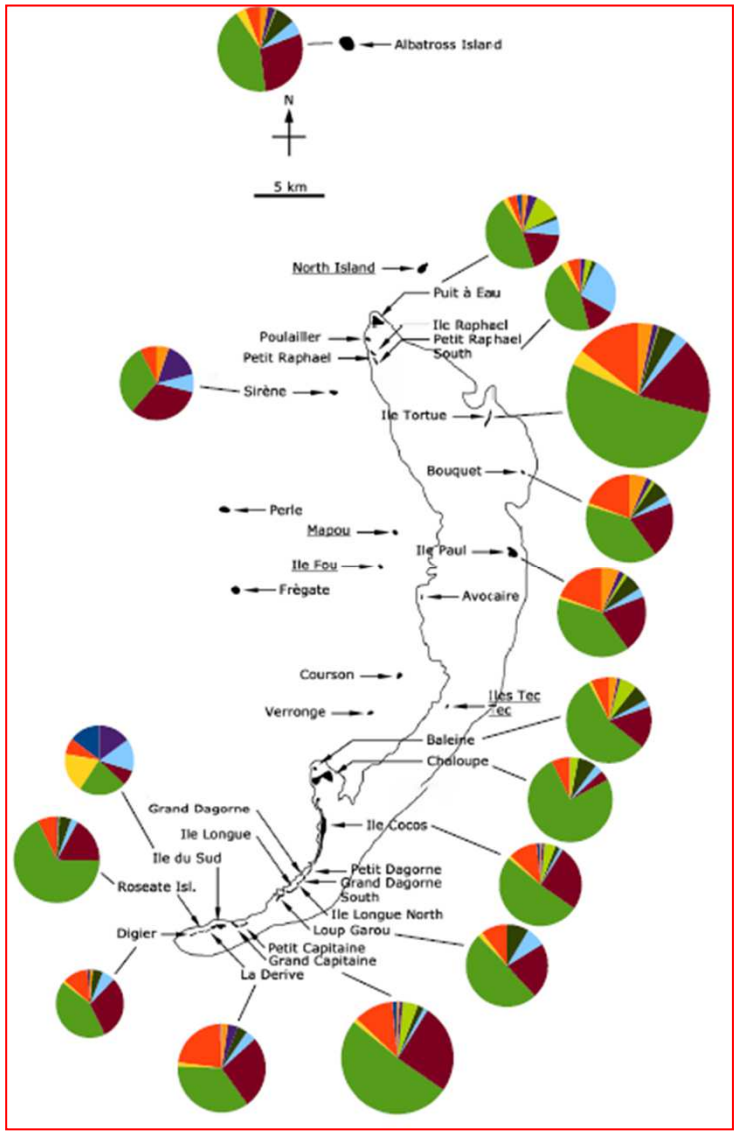
Plastics in aquatic environment

- One from the most import contaminants
- Long-term exposure
- Adsorption of PTS
- Floating solid phase
- Bioconcentration
- Bioaccumulation

Plastics in aquatic environment - inventory

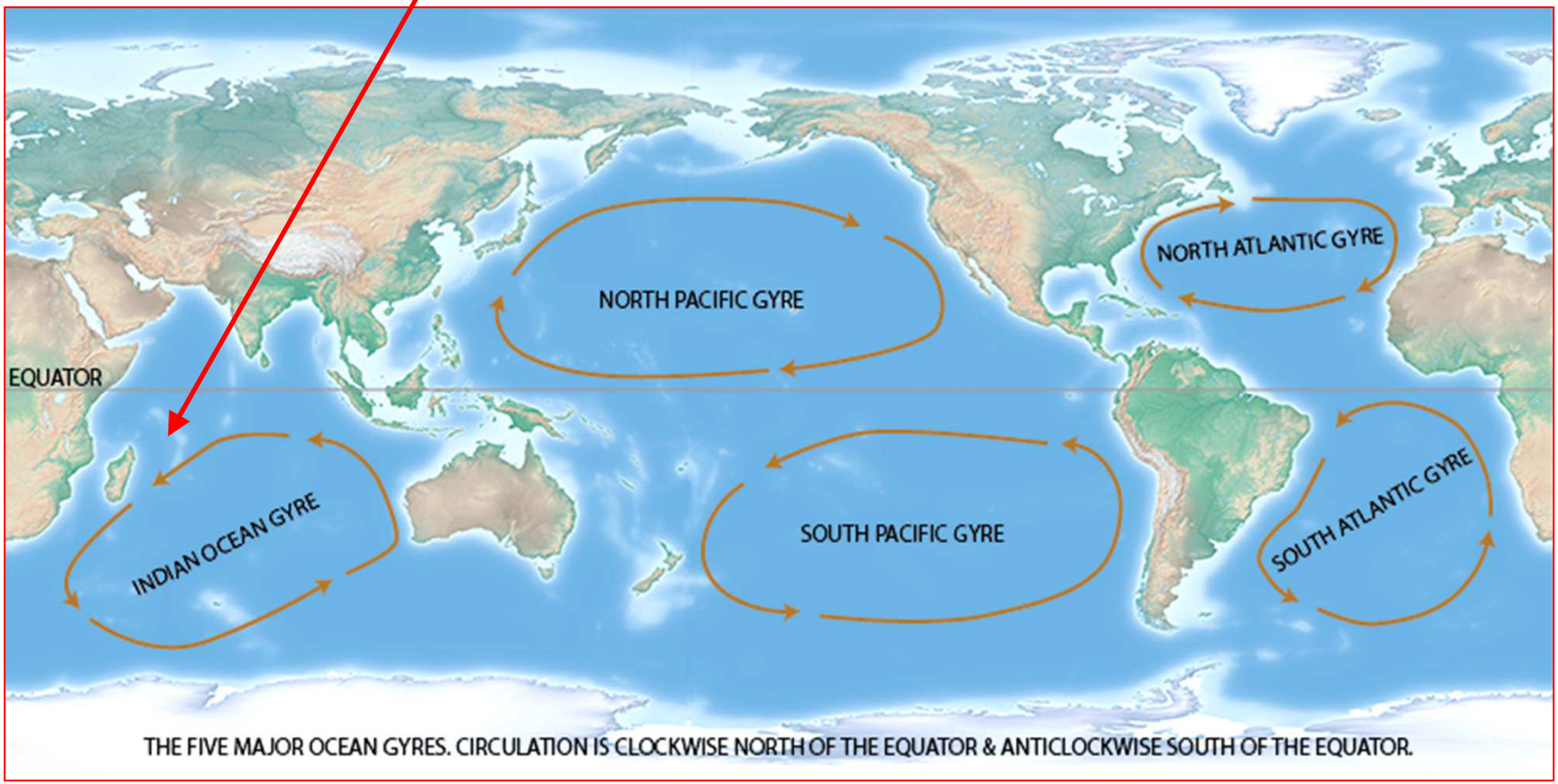
- Sampling October 2010
- Archipelago St. Brandon (31 islands, 50 inhabitants)
- Study of North-West University (JAR)
- Types and amounts of washed sea wastes

- % textilie
- % sklo
- % kov
- % plast a plastifikovaný papír
- % plážové boty (žabky)
- % rybářské sítě, nylonová a jutová lana, nylonové tašky
- % pryž
- % polyuretanová pěna
- % dřevo
- % polystyren
- % rybářské vlasce



Territory of sampling

Mauritius



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TEŞEKKÜR EDERİM…