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Design of monitoring of contaminated sites
1 – Air, water, sediments

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Target

Examples of different methods used for determination and monitoring of pollutants in air, waters and sediments

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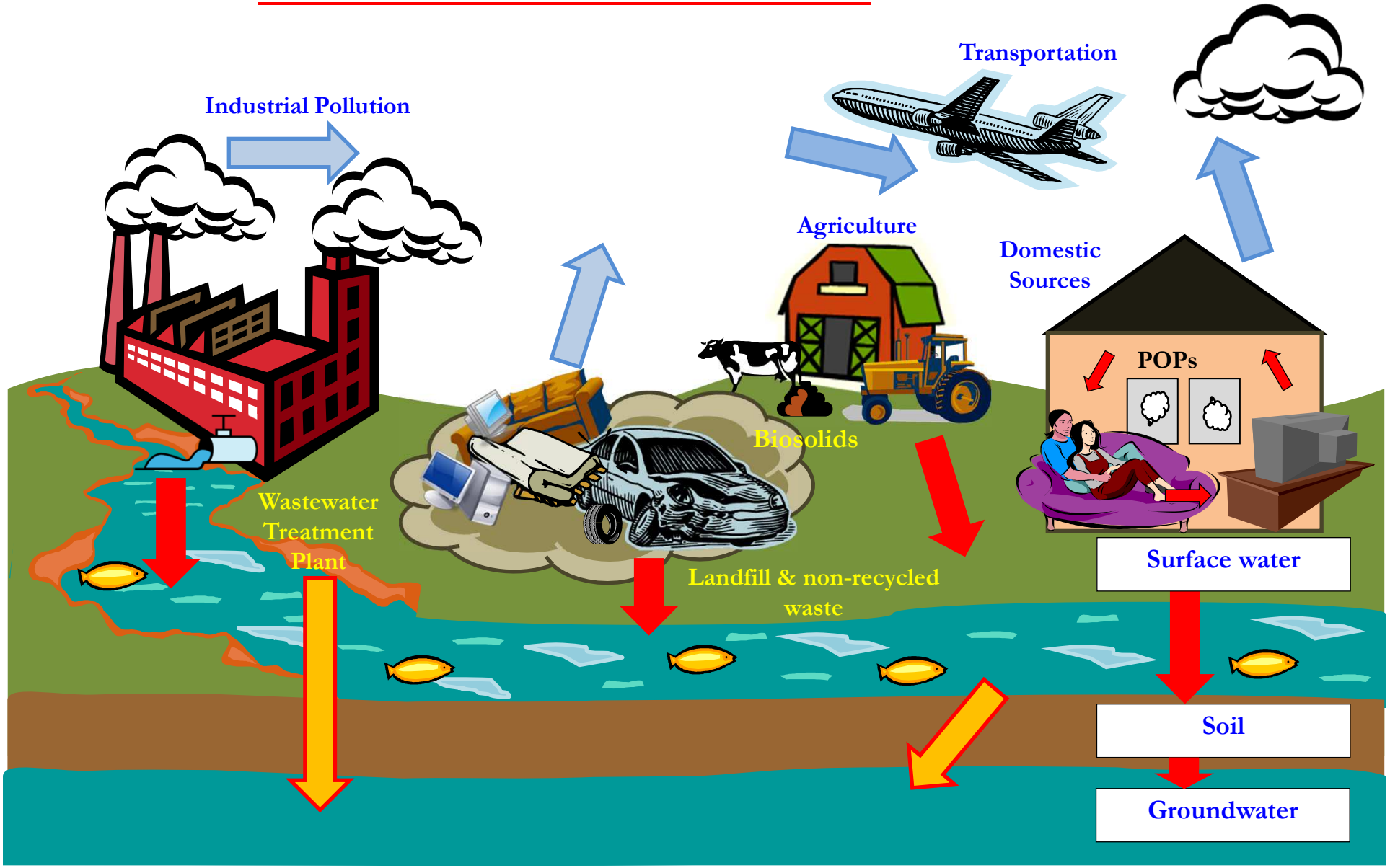
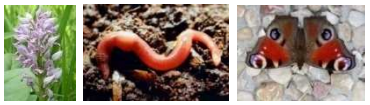
Contaminated sites

Routes of POPs contamination



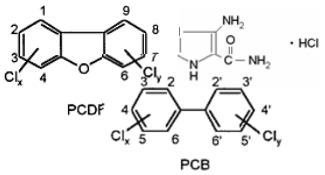
Problem definition = complex information survey

Environmental characteristics

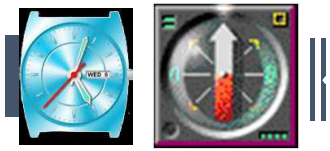


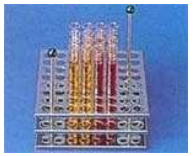
Source of contamination

Compounds of interest



EXPOSURE



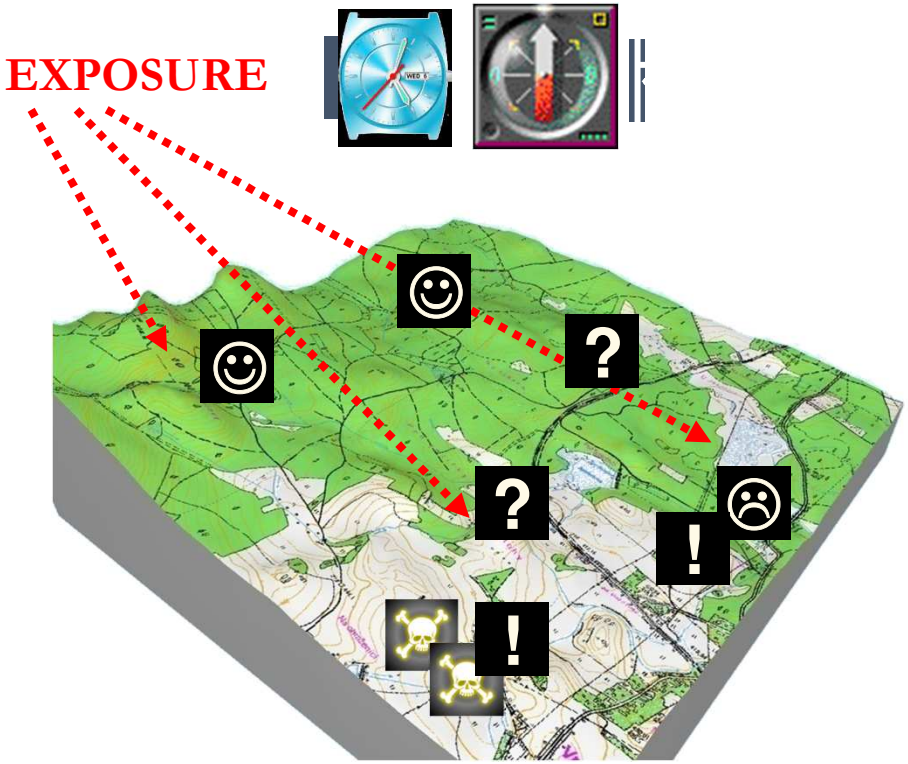


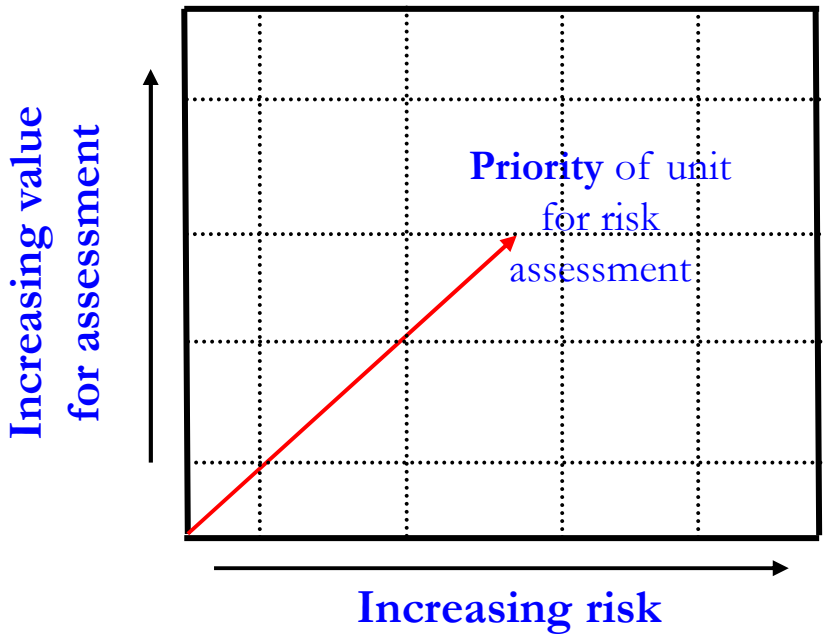
Pilot (screening) test

Development of exact situation plan integrates also exposure assessment (at least at screening level)

COMPARTMENTS ACCORDING TO POTENTIAL RISK

Problem definition generates comprehensive SITUATION PLAN ……





☺ Background sites with no influence of exposure

? Uncertain influence and/or uncertain assessment endpoint

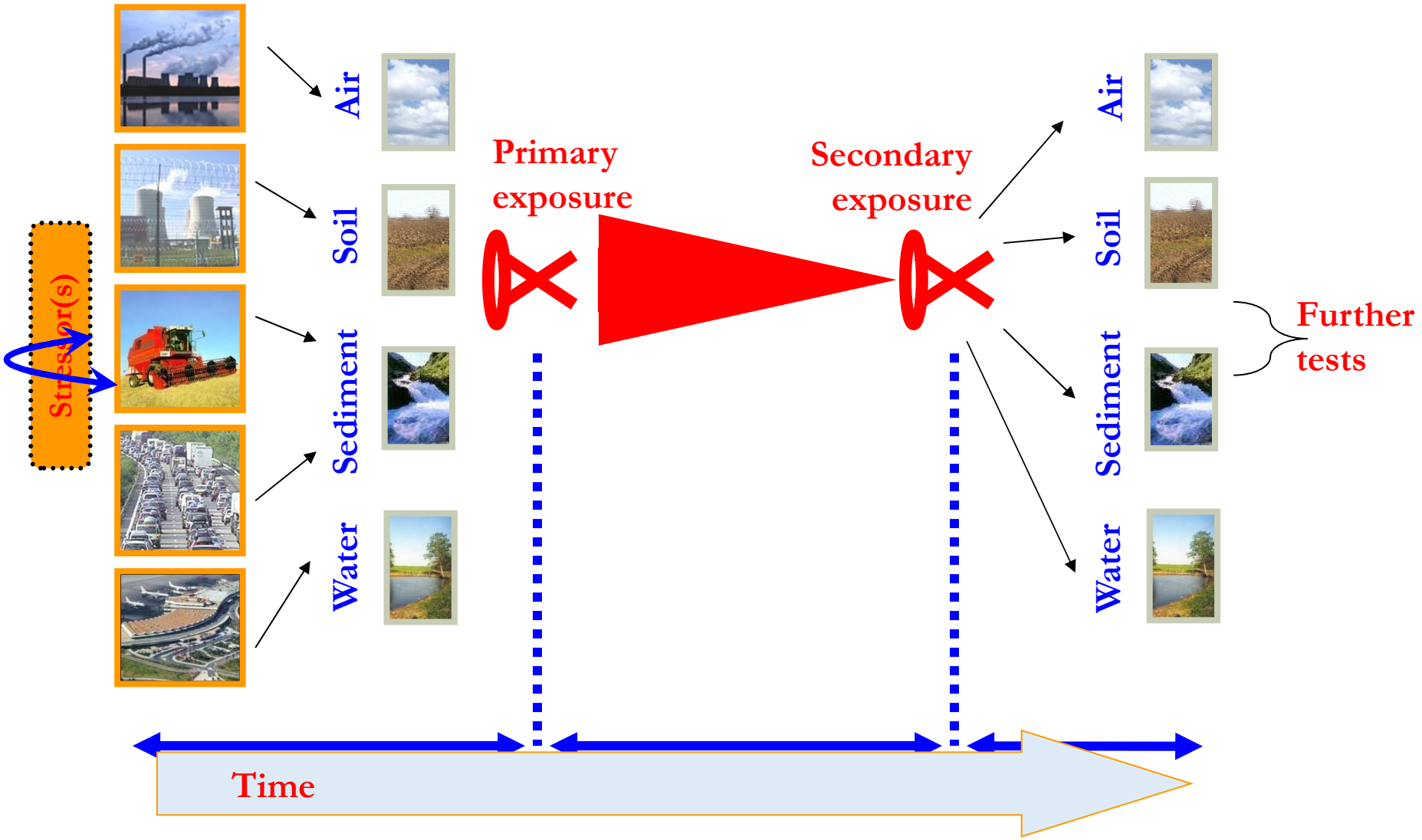
! Potentially affected sites, still clean or with negligible effect

☹ Area with probable and substantial toxic impact

☠ Already strongly affected area with remarkable effects

Assessment scenario and basic principle: „Where is the problem“

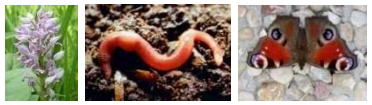
Scenario is in direct relation to estimated (predicted) exposure pathways: all further analyses follow from this starting point



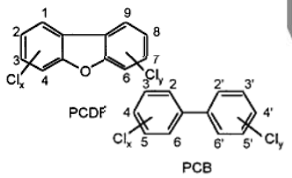
Scenario as milestone of the assessment process

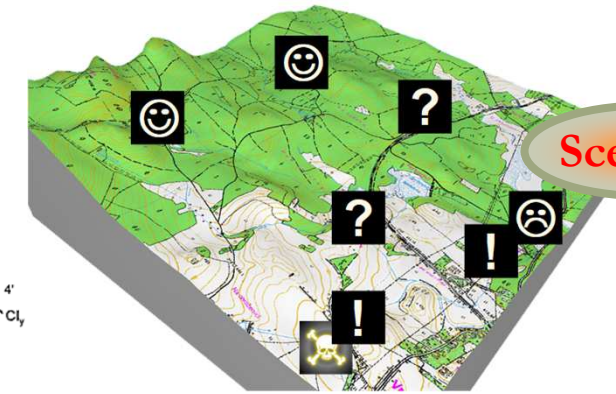
Source of contamination

Environmental characteristics

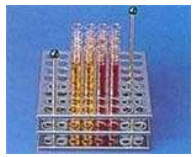


Compounds of interest





SITUATION PLAN



Pilot tests (screening)

Scenario

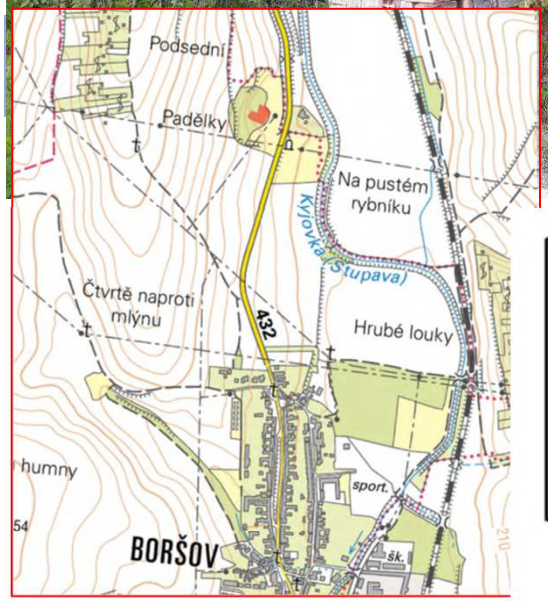
Primary exposure

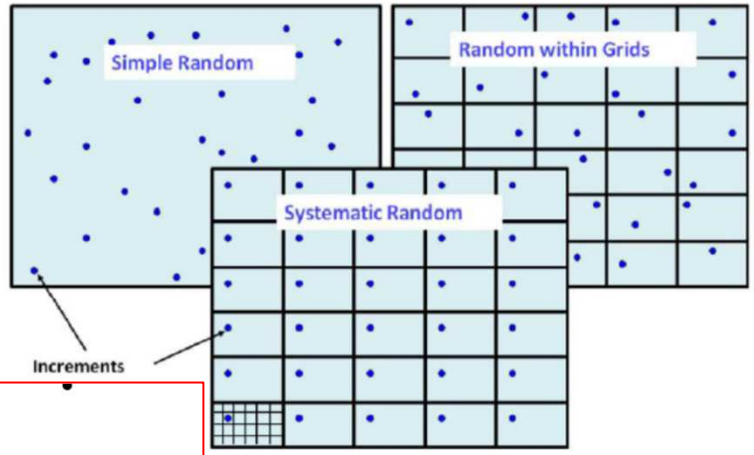
Secondary exposure

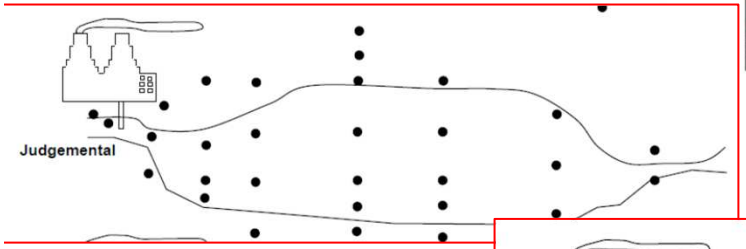
- Soil
- Sediment
- Water
- Air
- Biota

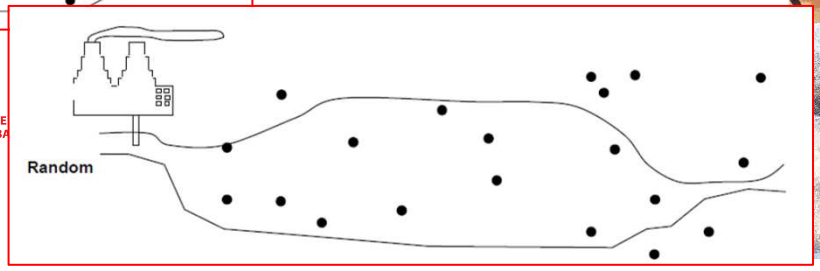
- Soil
- Sediment
- Water
- Air
- Biota

Further tests









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Monitoring

Data reach, information poor

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Monitoring

- is a long-term consistent observation or measurement of precisely defined indicators well described in the space and time
- is performed in the monitoring network representative for the region
- consists of the observations and measurements, evaluation of the current status, changes as well as future perspectives.

Environmental monitoring is at the very beginning of the environmental information chain:

- it is the basis of environmental data collection,
- environmental reporting and environmental research,
- the basis of understanding of environmental problems and trends.

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Monitoring programmes should

- be developed in accordance with the demands of the current legislature
- monitor the efficiency of the strategic documents such as international conventions and protocols or national measures with respect to the environment

Monitoring outputs

- evaluation of the exposure within systems
- evaluation of the human and ecological risks
- support of decisions
- changes in economic practises, legislature, operation and strategies
- determination of efficiency of accepted measures, funding, etc.

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Environmental monitoring

Environmental monitoring is therefore a powerful tool for

- **supporting a decision-making**
- **enforcing policy decisions**
- **assessing compliance with policy regulations and objectives.**

These programmes are essential in identifying subsequent measures.

The crucial elements in the development of the monitoring program are the measurement methods and standards.

Is monitoring only a rutine procedure or it can be used as a tool for study of environmental processes ?

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Measurement of substances in the environment

Screening - is it possible to detect the substance in environmental samples?

Survey - how big is the problem?

Monitoring - long-term measurements of the temporal trends
and/or - large scale measurements of the spatial distribution

Modelling - where is the substance ?

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Local monitoring programmes

- State/county programmes
- Community programmes
- Emission control/surveillance
- It is important to co-ordinate these as far as possible to produce comparable results

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Monitoring of contaminated sites

- Preliminary phase – collection of basic information
- Phase 2 – collection of detailled information
- Monitoring during remediation process
- Post-remediation monitoring

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Harmonization of the monitoring programmes

It is important that data from different sources are comparable for the same parameter

Intercalibration of the analytical laboratories requires significant efforts

Various approaches of existing monitoring programmes represent currently the major problem (various matrices, frequency, sampling procedures, etc.)

Local – national – regional – global levels

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Specific problems of environmental analysis

- low homogenity of samples (soil, wastes)
- low stability of samples (biota)
- various matrices (methods for extraction of analytes from matrices)
- wide range of analytes (method development)
- wide range of concentration (robust methods)
- monitoring on the levels close to the detection limits (high deviations)
- risk of secondary contamination
- price of ultra-trace analysis (instrumentation, chemicals, standards)

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What is sampling about?

Definitions (Oxford dictionary):

- **To sample:** Take a sample or samples of (something) for analysis
  - Example: one hair on a jacket, orange in a supermarket

- **A sample:** A small part or quantity intended to show what the whole is like

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Why do we need to sample?

- To know levels of pollution prior to take specific measures
- To understand emissions of specific pollutants or from specific sectors
- To understand time trends (diurnal, weekly, seasonal variations?)
- To understand specific processes (e.g. air-surface exchange)
- To support legislation

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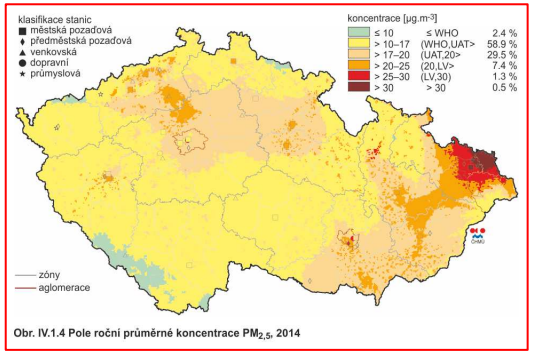
Why do we need to sample?
To know levels of pollution



Obr. IV.1.4 Pole roční průměrné koncentrace $PM_{2,5}$, 2014

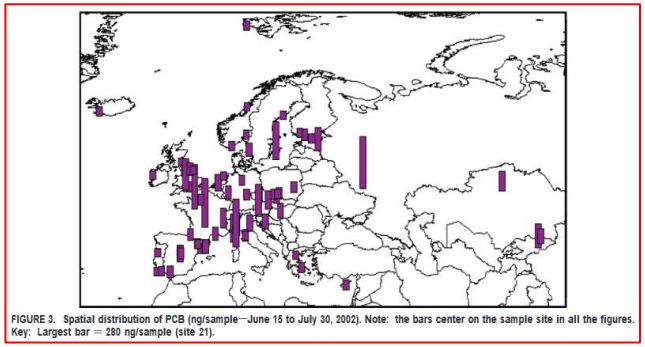


FIGURE 3. Spatial distribution of PCB (ng/sample—June 15 to July 30, 2002). Note: the bars center on the sample site in all the figures. Key: Largest bar = 280 ng/sample (site 21).

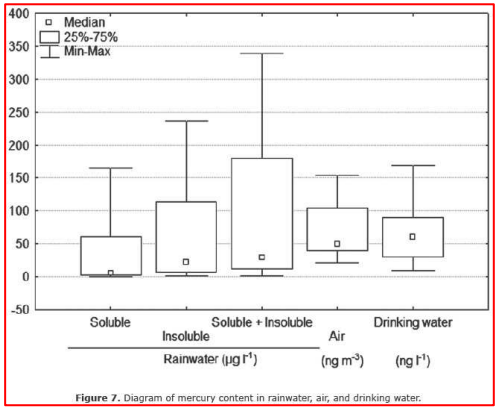


Figure 7. Diagram of mercury content in rainwater, air, and drinking water.

At a national level

At a European level

Prior to take actions
(e.g. drinking water)

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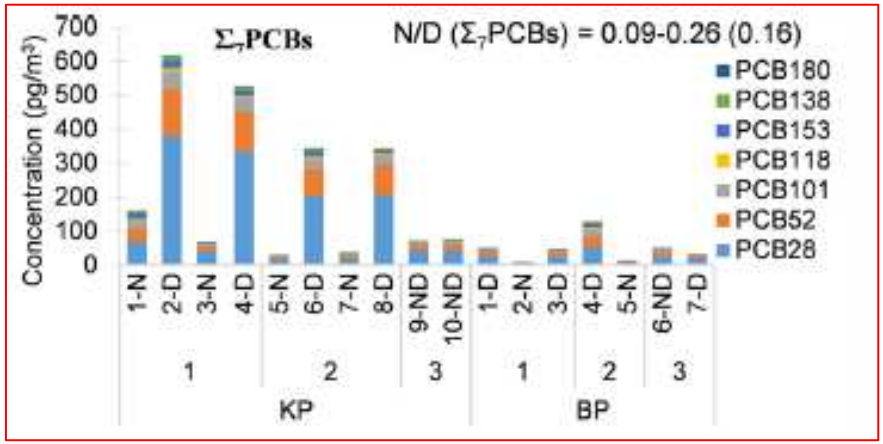
Why do we need to sample?
To understand emissions

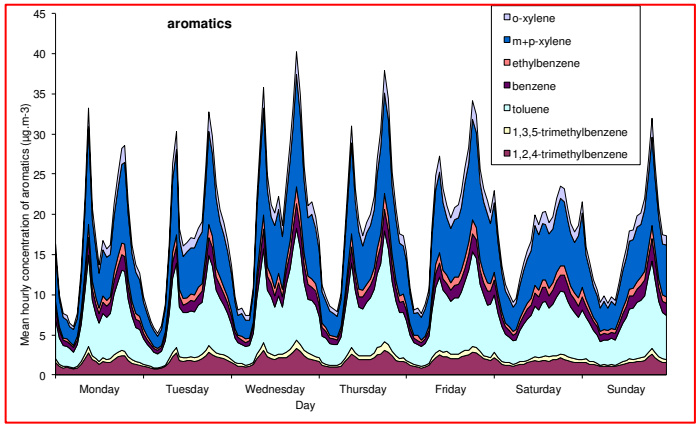
Emissions from specific sector e.g. traffic

Emissions of specific pollutants from consumer products e.g. flame retardants in computers

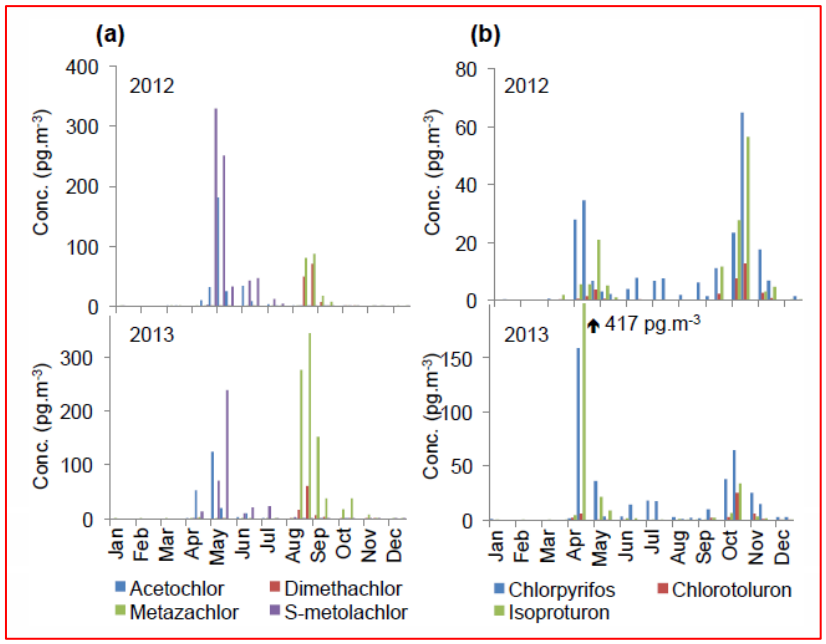
Why do we need to sample?
To understand time trends



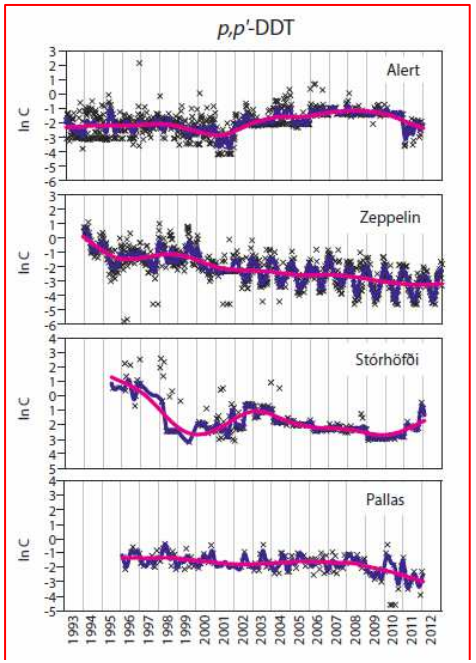
Daily



Weekly

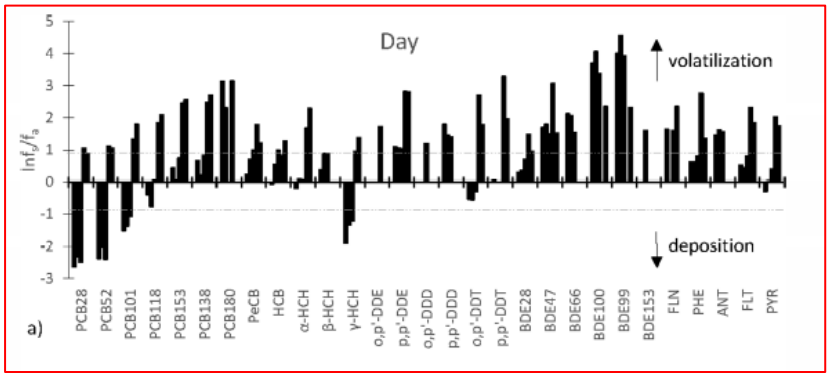


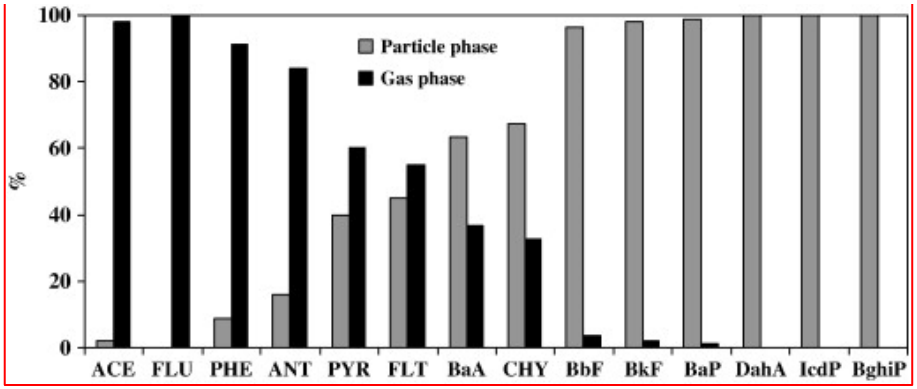
Seasonally



Long-term

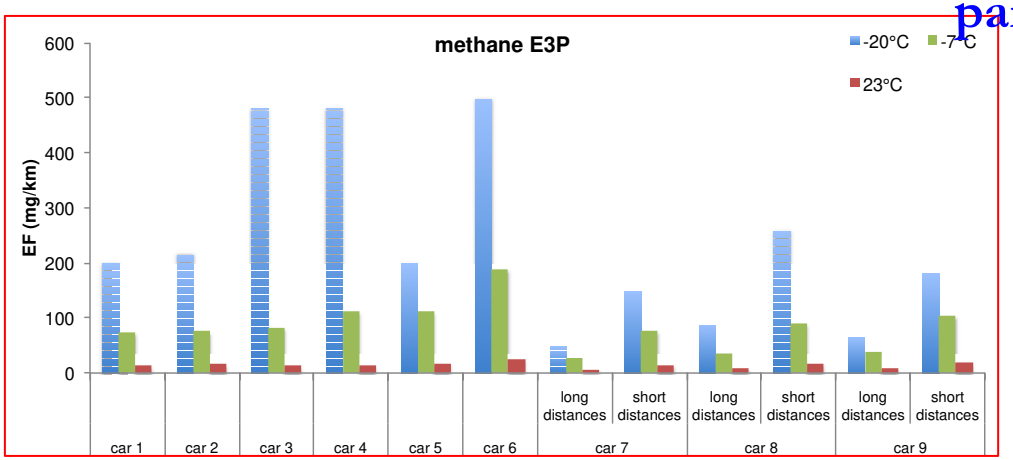
Why do we need to sample?
To understand specific processes





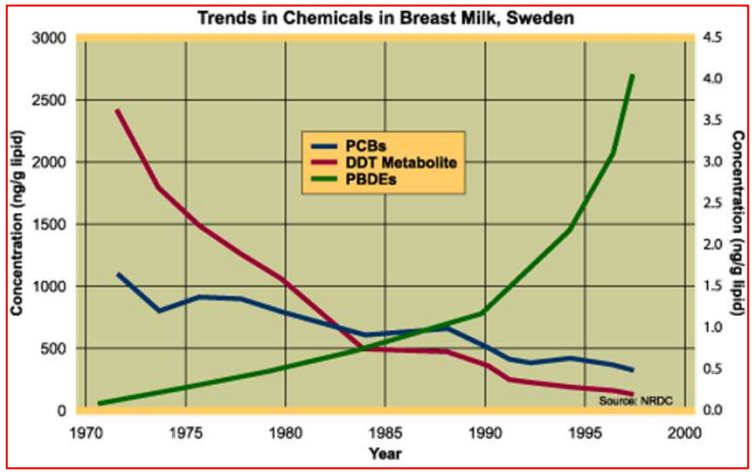
Air-soil exchange

Gas-particle partitioning



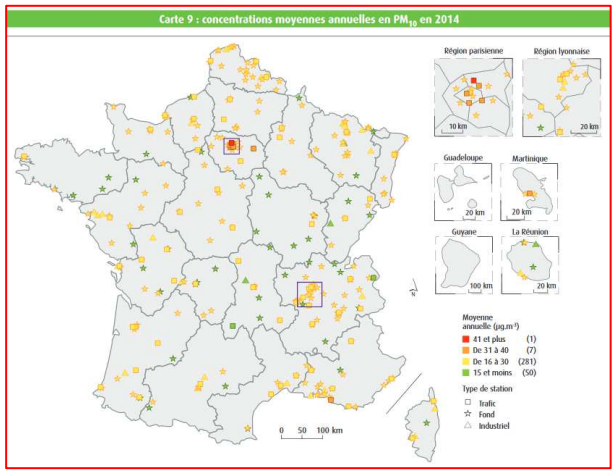
Influence of temperature on cold start emissions from passenger vehicles

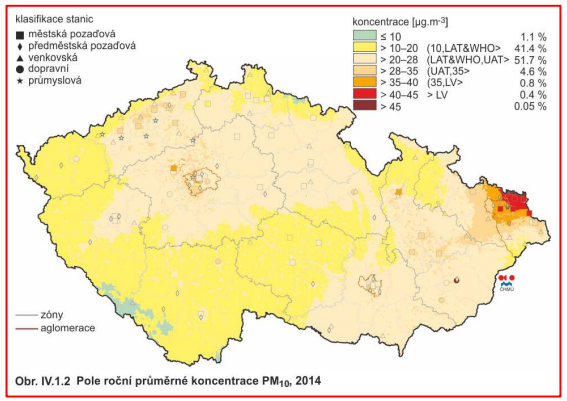
Why do we need to sample?
To support legislation



E.g. Stockholm Convention on Persistent Organic Pollutants

E.g. European regulation about air quality





Obr. IV.1.2 Pole roční průměrné koncentrace PM_{10}, 2014

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How to sample?

Air

Syringes for gas sampling, 1-100 mL

- Ideal for corrosive, radioactive, or sterile materials
- Removable handle minimizes heat transfer from hand to sample, making the syringe easy to hc
- Accurate and reproducible to +/- 1%
- Field repairable

Soil

Water

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How to sample?

- This should be connected to the answer of why do we need to sample?
- E.g. No need for the same type of air sampler if one wants to understand long term trends vs. specific processes

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Importance of field blanks

- **Field blank** = sample that goes through the same procedure as "real samples" that can assess the extent of contamination during sampling and transport to the laboratory
- To know that what you are measuring is from the environment and not just a contamination (example of pesticide application)
- How to do it with air? With water? With soil?
- They should be treated as a sample

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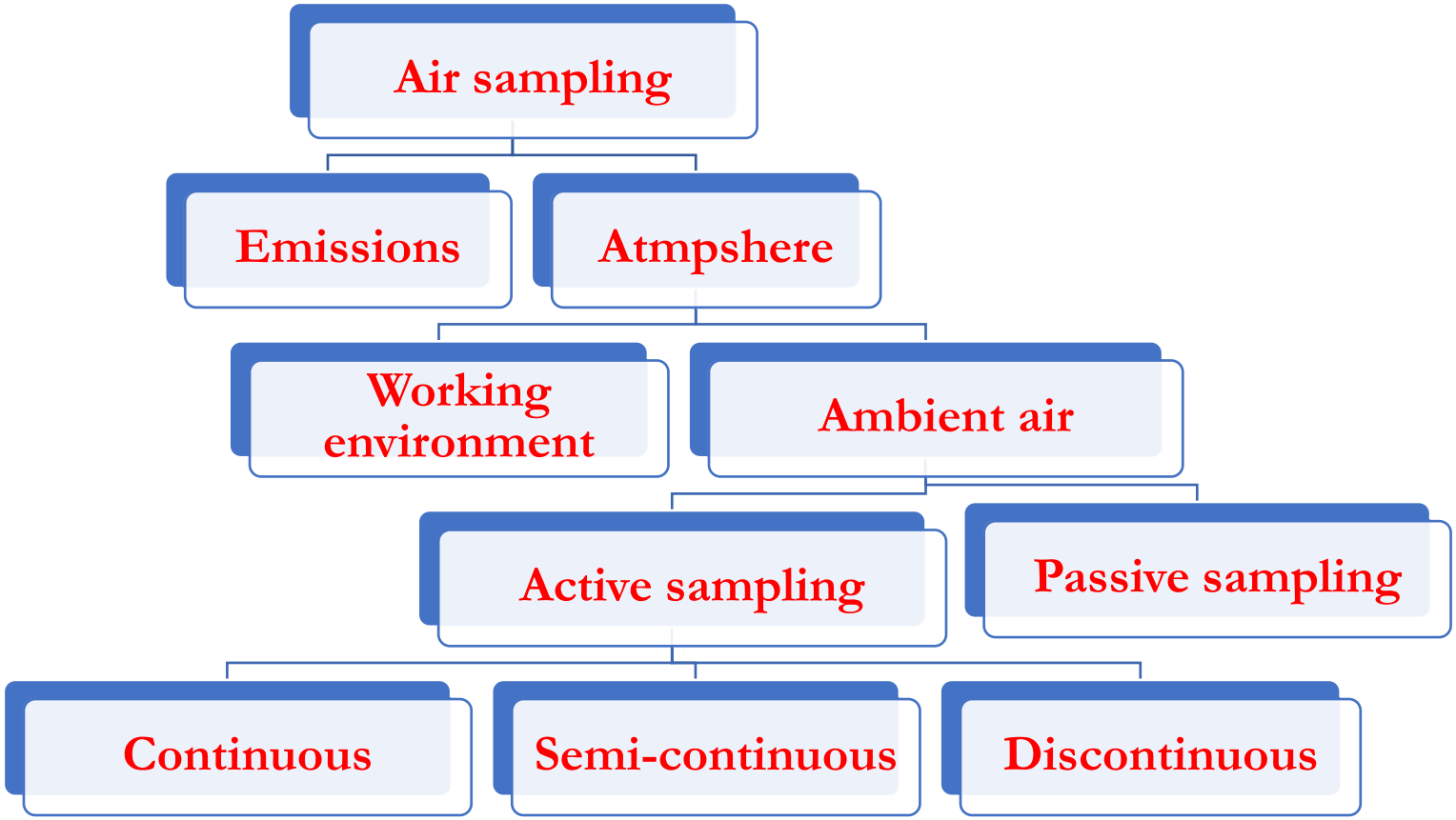
Distribution of sampling



Air sampling

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- **Emissions (chimneys, ventilations,...) continuously**

- **Ambient air (environmental levels) sampling points, time period**

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Distribution of sampling

Sampling of emissions

- High pollutants concentrations
- High agresivity of sampling air
- Isokinetic sampling
- Sampling using the condense or dilution method

Sampling of immissions

- Sampling of working environment
- Sampling of ambient air

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Sampling methods

- Isokinetic sampling in accordance with ISO 9096 or EN 13284-1

+

- filter/condenser method
- dilution method
- cooled probe method

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Estimate of uncertainties of measurement

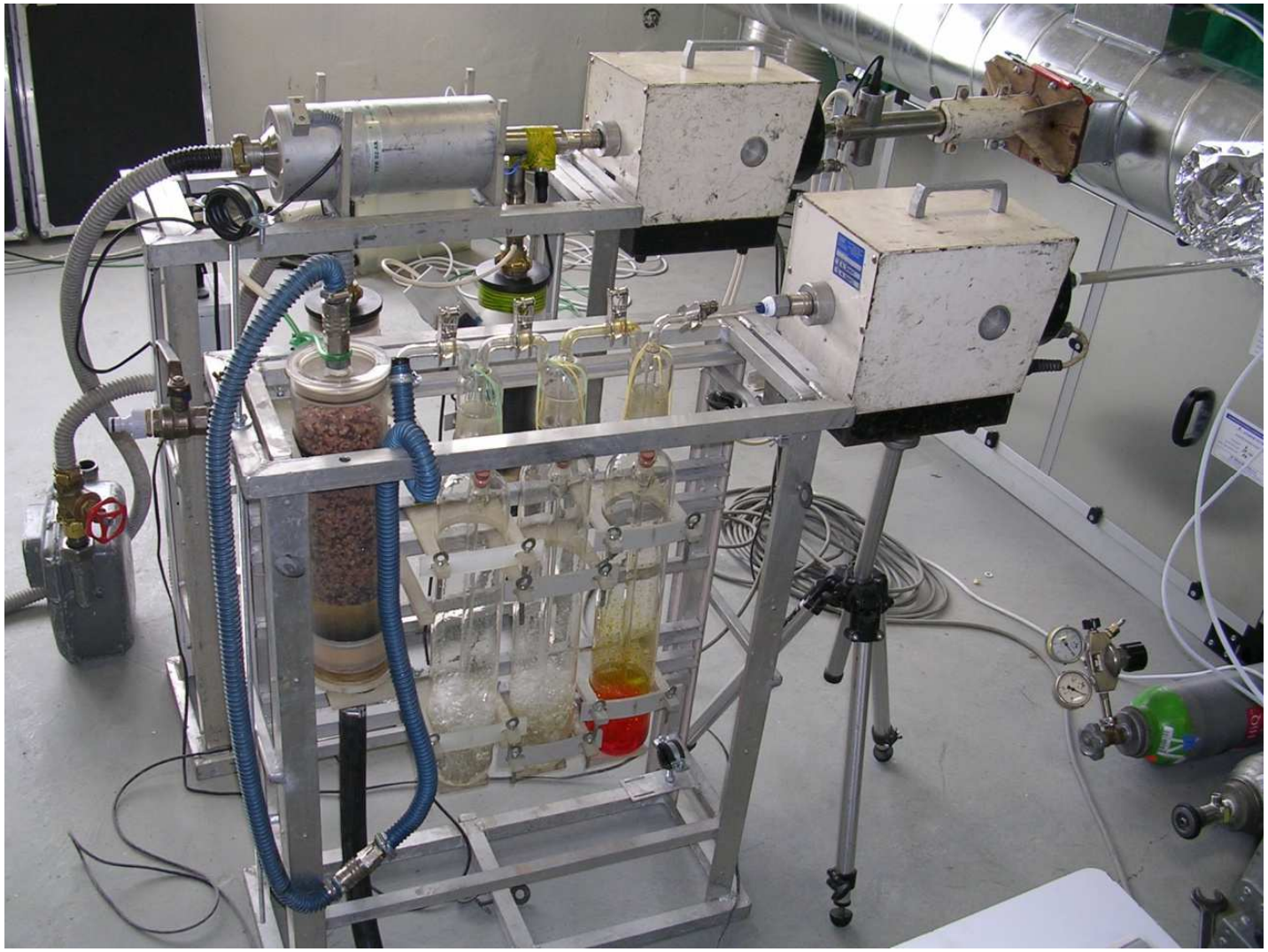
- **Total data variability** = combined uncertainty of measurement and matrices unhomogeneity

- **Uncertainty of measurement** = uncertainty of sampling + uncertainty of analyses

- **Uncertainty of unhomogeneity** = uncertainty of unhomogeneity in time (matrices change in the sampling course) + uncertainty of unhomogeneity in location (matrices change in the sampling spot)

Sampling of occupational environment air for PCDDs/Fs, HMs and VOCs determination

One-off sampling of emissions for PCDDs/Fs and Hg determination



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Air

- Air a key medium - responds quickly to sources
- Air concentrations fluctuate widely in space and time
- Different phases and different concentrations - compromises over sample time/volumes
- Short-term sampling/bulking etc ?
- Learn from existing national/regional programmes (e.g. IADN in Canada/US; EMEP)

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Ambient air sampling

Aim air sampling:

Qualitative and quantitative detection of the presence and concentration of pollutants or groups of pollutants in the atmosphere at a given location

Specifics of air sampling:

- Low concentrations of pollutants
- Heterogenity of the sampled matrix
- Pollutants present in multiple forms

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Sampling methodology

Factors affecting selection of sampling methods:

- Phase distribution of pollutants
- Stability of pollutants
- Time resolution considerations
- Analytical considerations
- Other physical-chemical properties of pollutants:
  - Termic stability
  - Volatility
  - Polarity
  - Ionic character
  - Chemical composition
  - Environmental-chemical properties

Meteo – measurement of meteorological parameters

WV – wind velocity

WD – wind direction

p – atmospheric pressure

h – relative air humidity

RAIN – sum of precipitation

GLRD – sun irradiation

T – temperature (not specified)

T2m – temperature 2 m above terrain

T10m – temperature 10 m a

Sampling methods

Two main types of methods:

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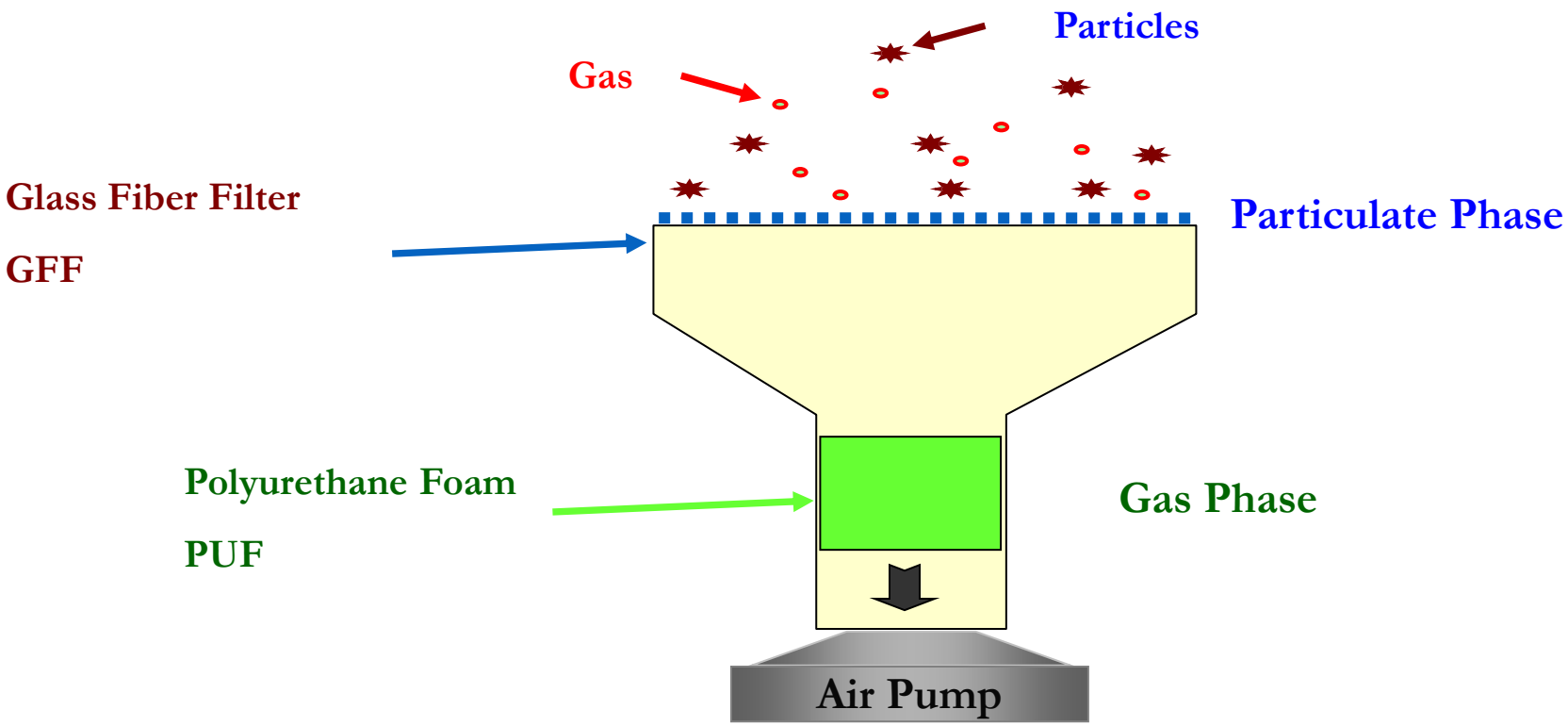
Active sampling

- Active sampling – cost, training, power, supporting meteo data
- Establish regional 'super stations'?

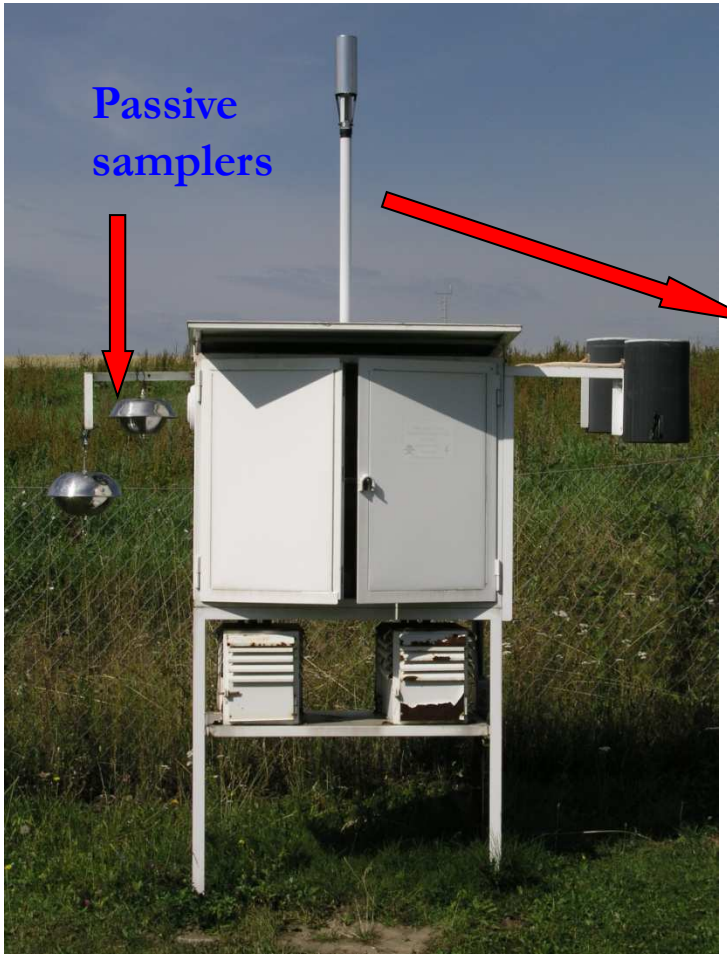
Active sampling techniques

✓ **AEROSOLS**



High-Volume sampler

Combine samplers for sampling of POPs



Active sampling

Dust aerosols samplers

Active samplers

Active samplers

PM-10 (Thermo Andersen, USA) flow more than 1 m^3 per minutes (1 500 m^3/24 hrs.)

PS-1 (Thermo Andersen, USA) flow more than 280 l per minutes (400 m^3/24 hrs.)

Active samplers

Leckel – sampling had - bio, PM1, PM2,5, PM10, PM+PUF, ozon denuder, TSP







Changes in levels over time

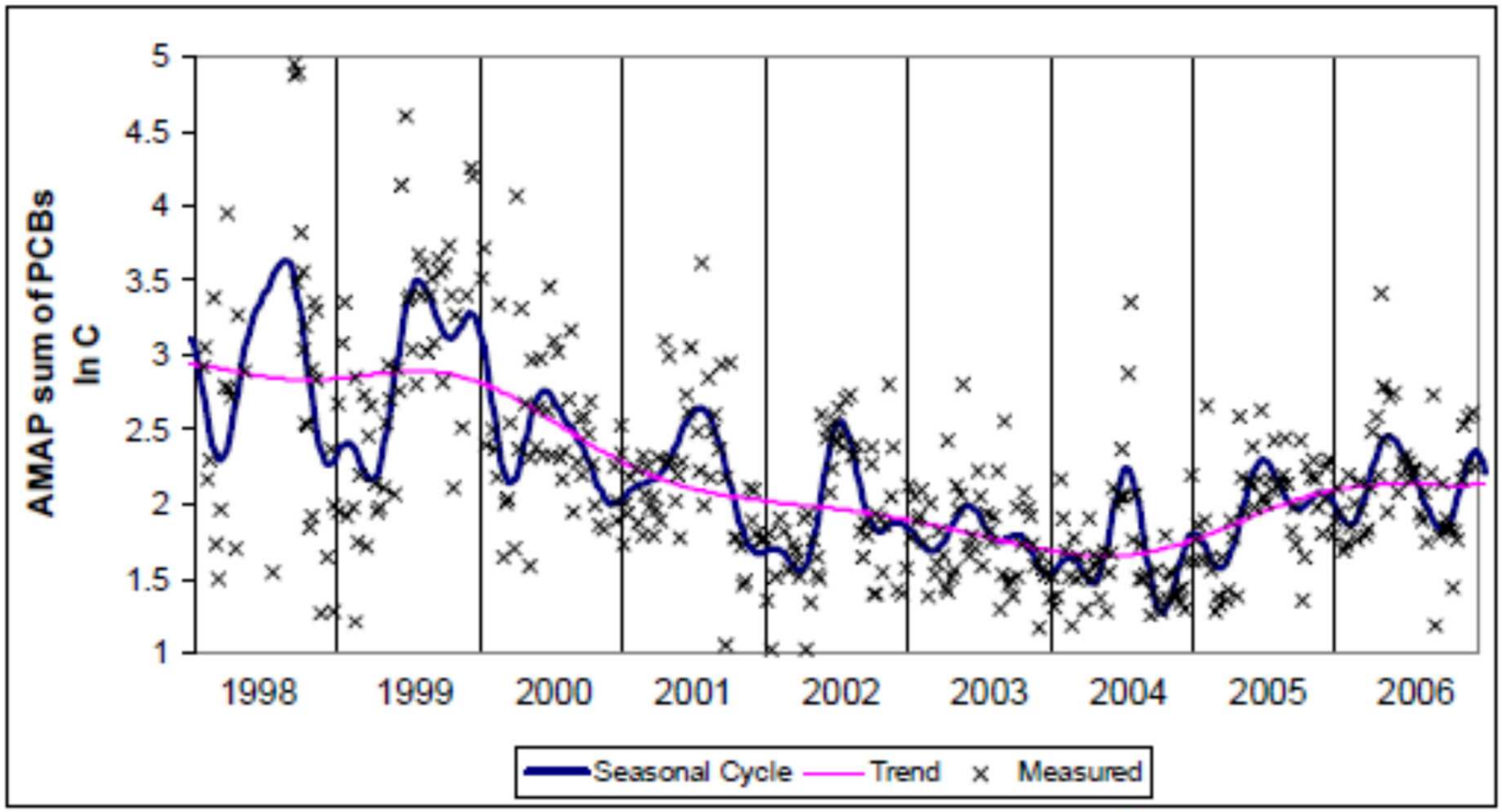
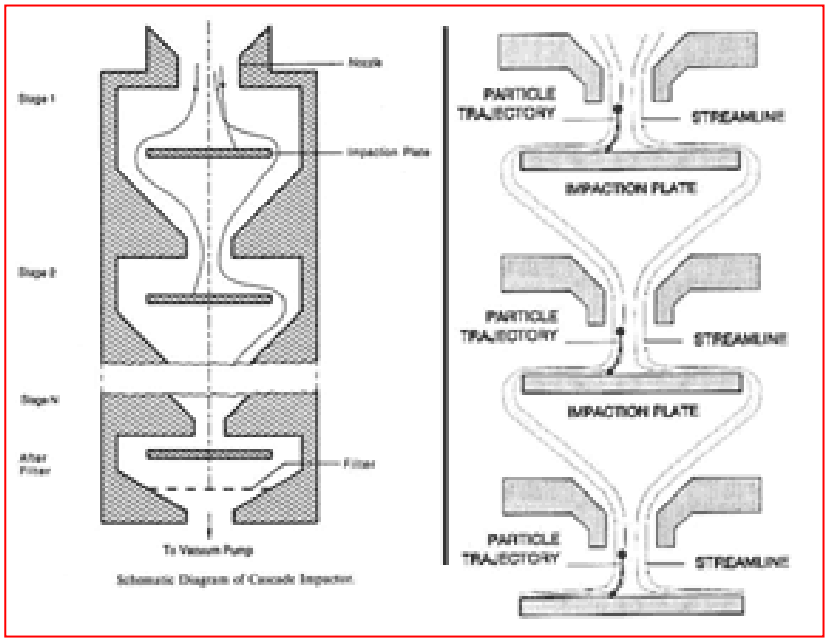


Figure 4: DF analysis of PCB levels in Zeppelin air [pg/m^3] from 1993 – 2006. Measured data, seasonal cycles and trend line is presented.

Fractionation of PM



Dust aerosols samplers

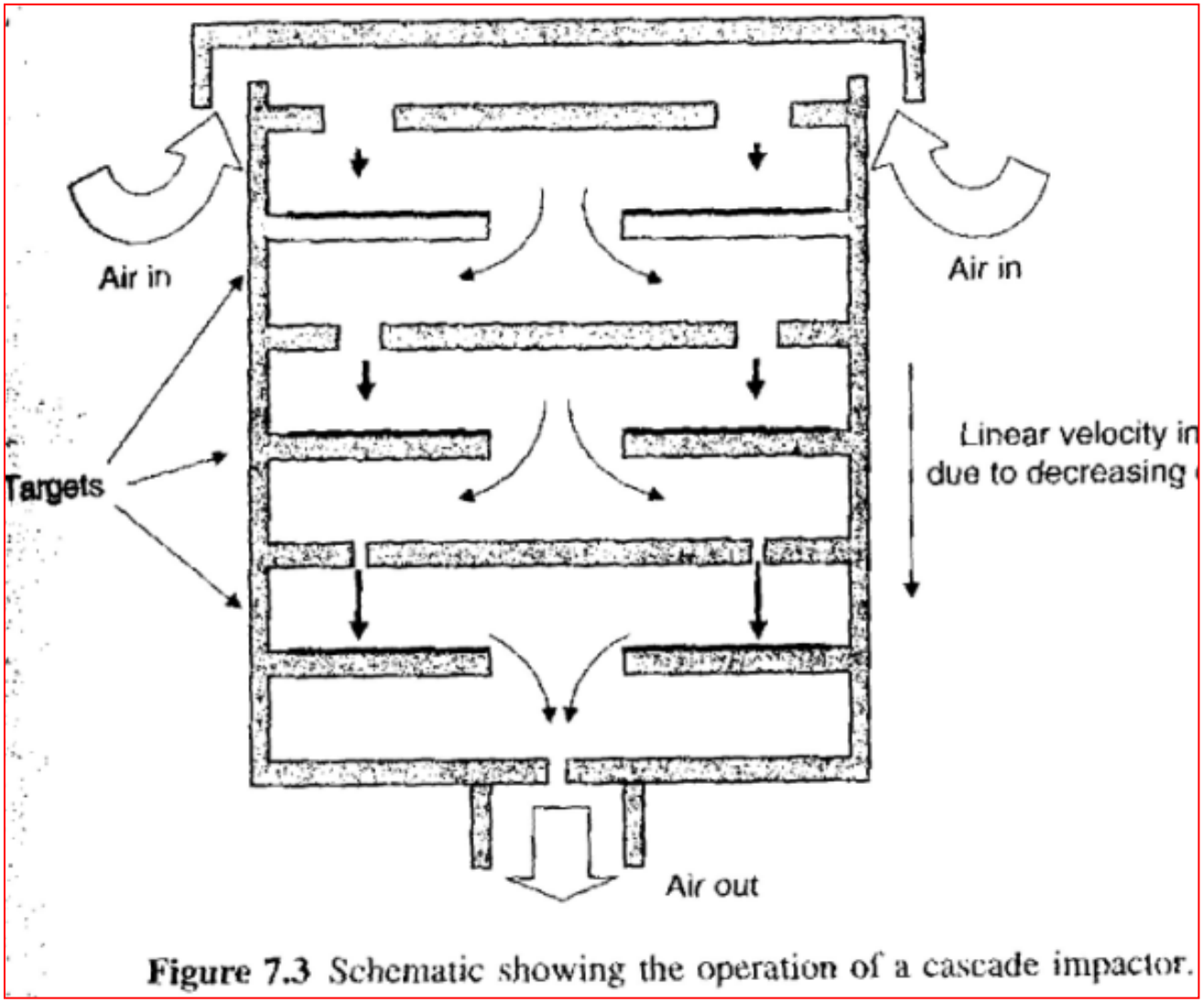
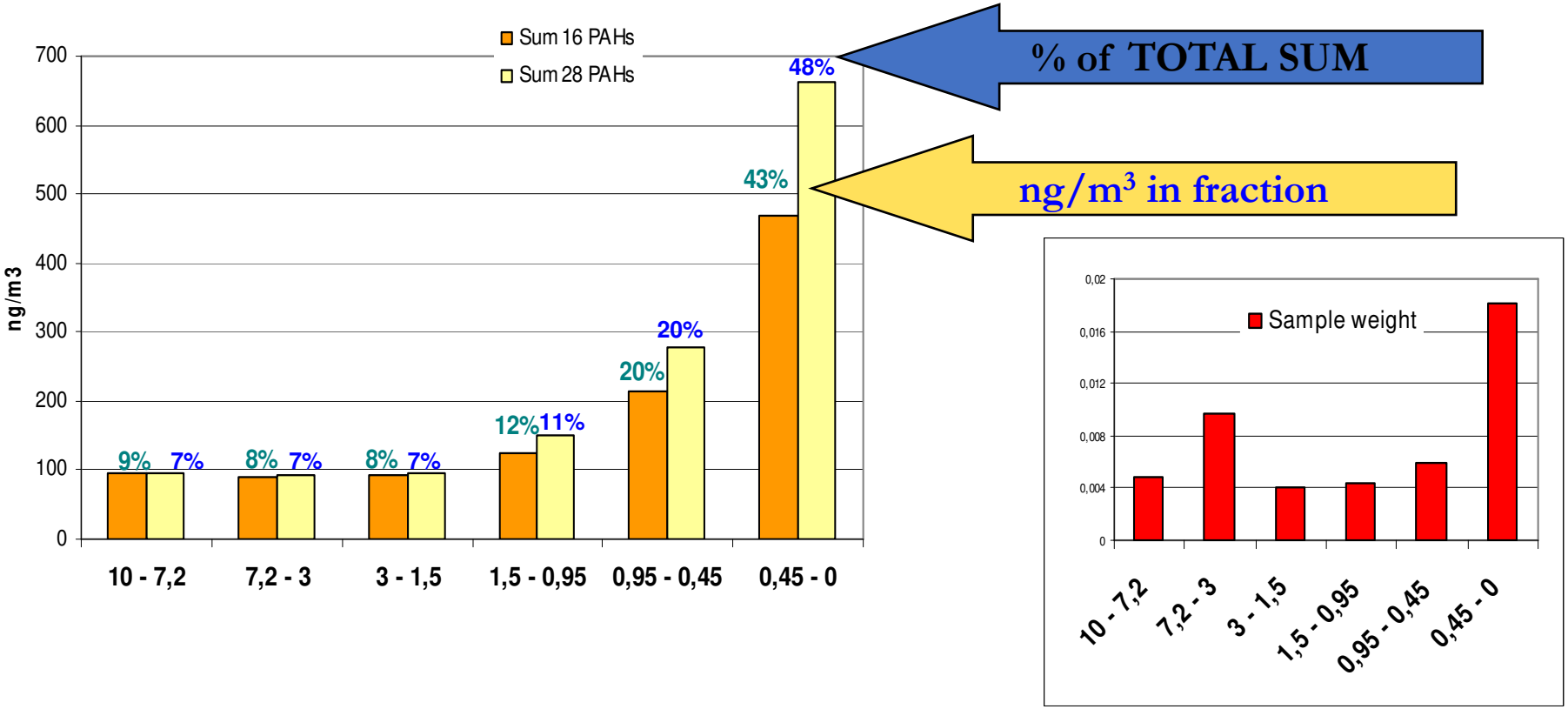


Figure 7.3 Schematic showing the operation of a cascade impactor.

Fractionation of PM



A comparison between the umu assay based B[a]P equivalencies and equivalences determined using chemical analysis showed that in the particle phase only 10% of chemicals were identified and less than 1% in the gas phase (Bartkow et al., 2008).

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Contents

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

Passive sampling

- Such techniques provide a cheap and powerful tool for obtaining detailed spatially resolved and time trend data relatively cheaply and efficiently.
- A number of exciting developments have been made in this field in recent years; the utility of passive samplers has been demonstrated for local, national and regional scale monitoring.

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Passive sampling

The advantages/opportunities of passive air samplers are as follows:

- Low cost
- Excellent opportunities for high spatial and temporal sampling resolution data
- No power supply needed, easy deployment and little operator training required

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Passive sampling

Their disadvantages/constraints are:

- Current techniques are still 'semi-quantitative', requiring knowledge of the sampling rate (m^3 air sampled/day) and the effects of temperature
- Optimisation of sampling requires further study, of the effects of wind speed, temperature
- Sampling is efficient for the gas phase component, but generally poorer for the particulate phase
- The time to reach gas phase-sampler equilibrium varies widely between POPs

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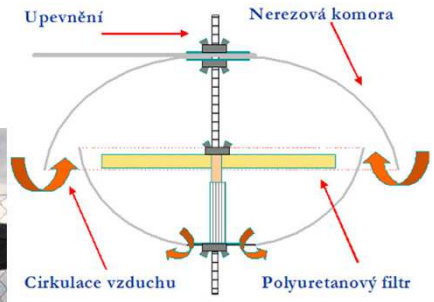
Passive sampling

- **No pump – sampling air flows round exposed filter, membrane or other media (sorbents), which trapped determined pollutant(s)**
- **Mechanism of separation is based on the difference between pollutant concentration in air and sorbent**
- **Time of sampling is driven by time, which is necessary to establish the equilibrium state (saturation adsorption capacity)**
- **Samplers are less sensitive to random extreme changes in the actual concentration of pollutants - provide information on the long-term level of contamination**

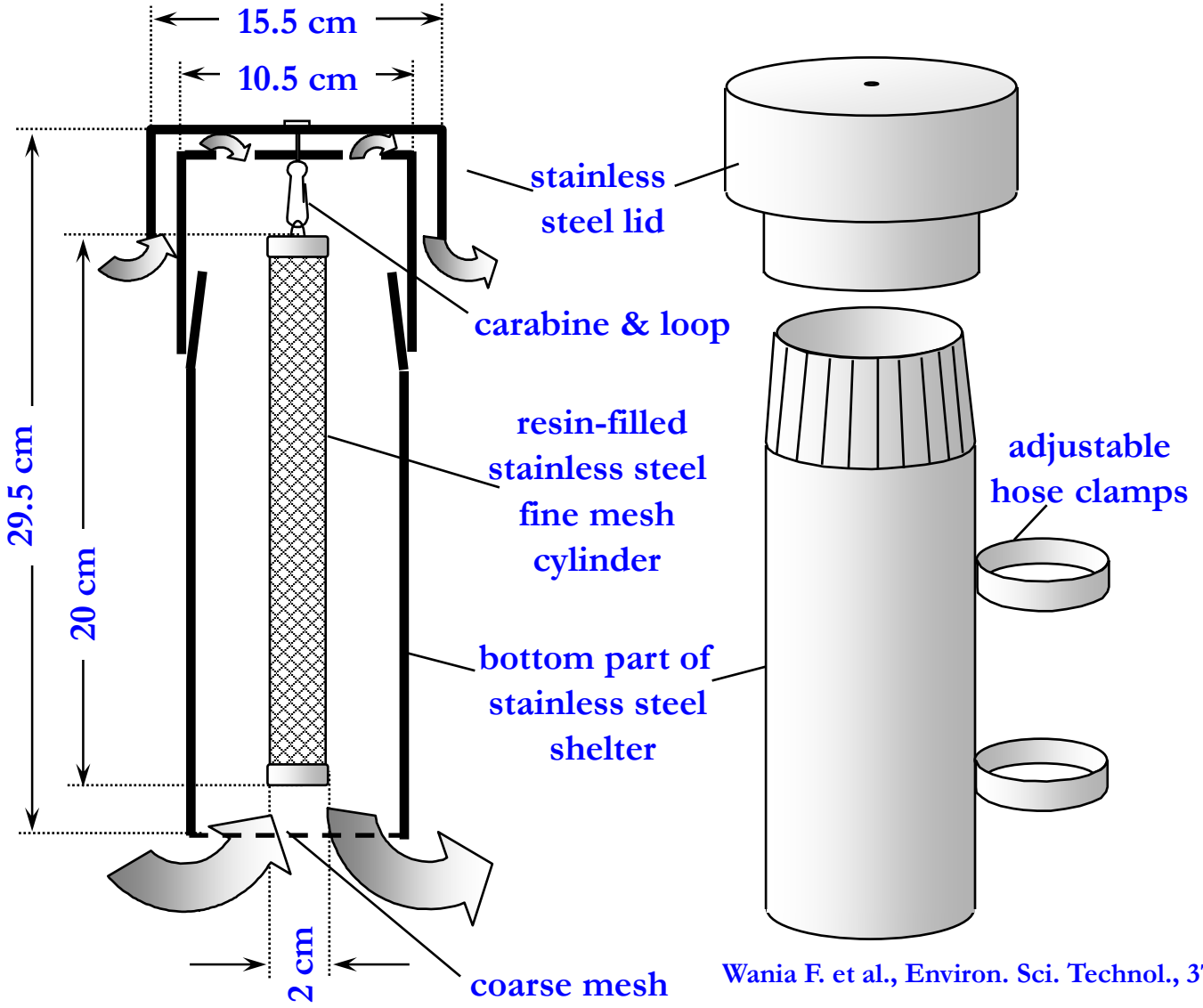
Passive sampling

Sorbents

- Biotic – mosses, needles, lichens
- Abiotic - SPMD, PUF, amberlit, ….

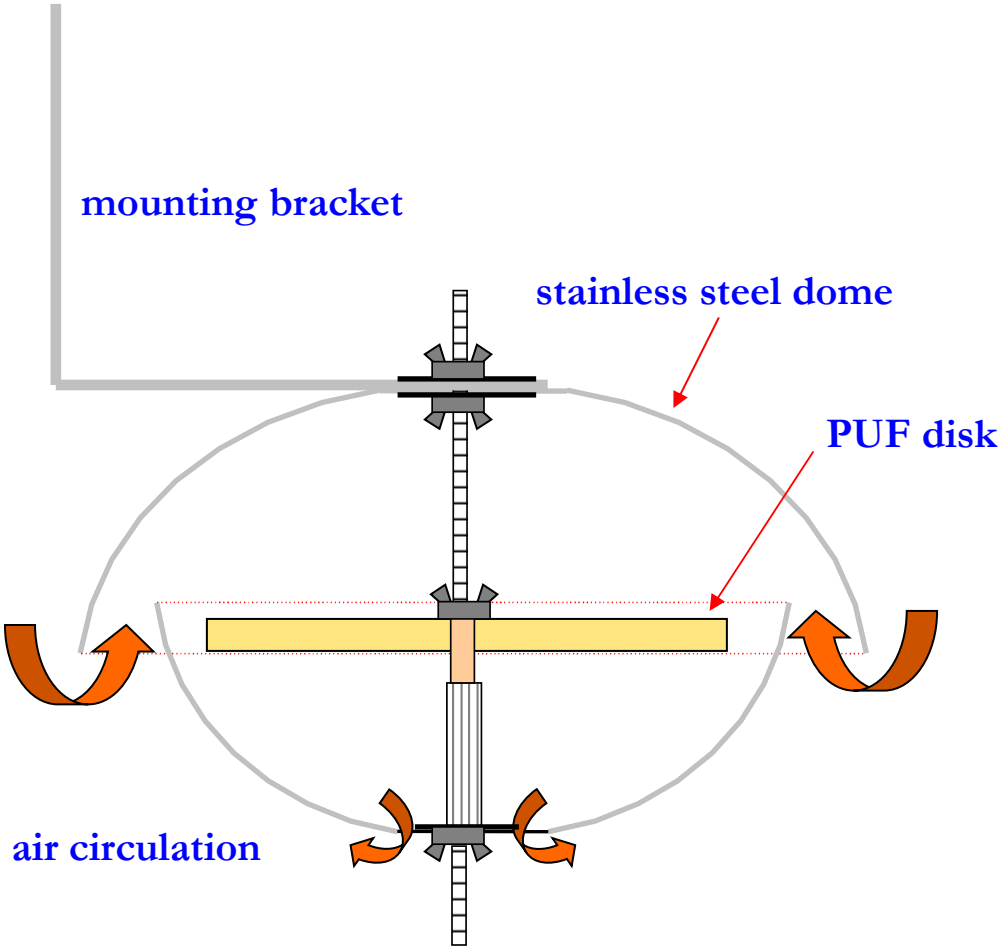


XAD-Resin Based Passive Air Sampling System for POPs

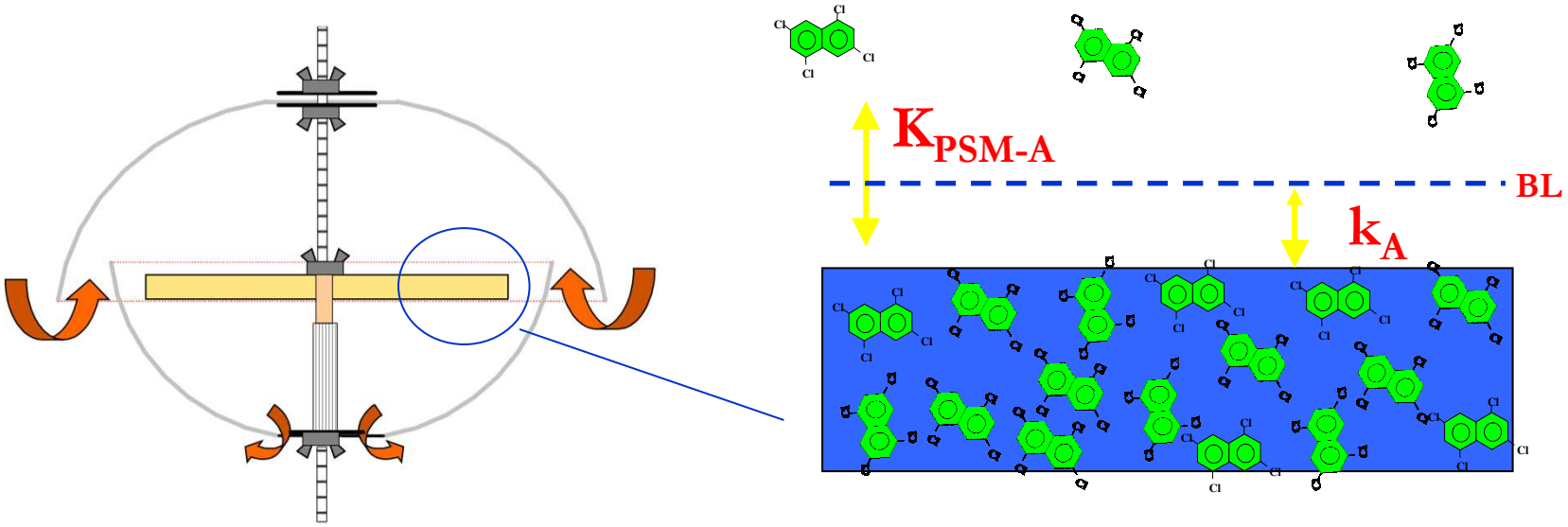


Wania F. et al., Environ. Sci. Technol., 37,1352-1359 (2003)

Passive samplers for POPs sampling



How do they work?



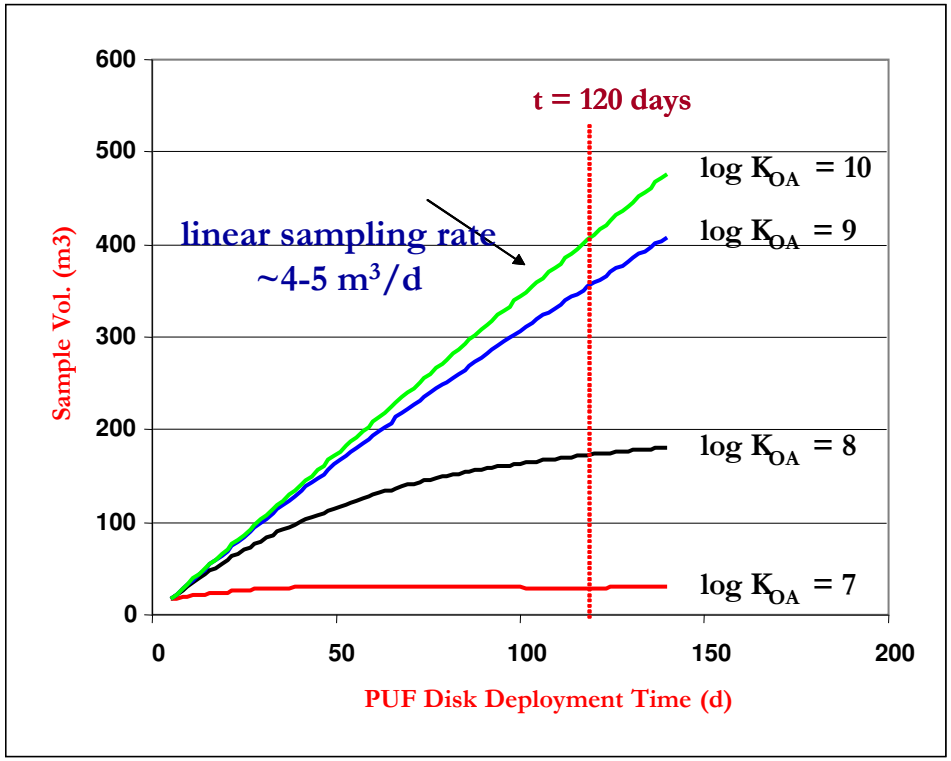
Uptake Parameters – Calibration:

- K_{PSM-A} - passive sampler-air partition coefficient (K_{PSM-A} is similar to K_{OA}, the octanol-air coefficient; foam density & mass)
- k_A - air-side mass transfer coefficient

PUF-Disk Uptake Profiles/Rates

Option 1: No DCs – rely on 'average' sampling rate of ~4 m^3/d

Option 2: Use DCs – site specific rates (esp. for windy sites)



- Depuration compounds (DCs) added to provide site specific average air sampling rates
- DCs should cover a range of known Koa values.

DCs:

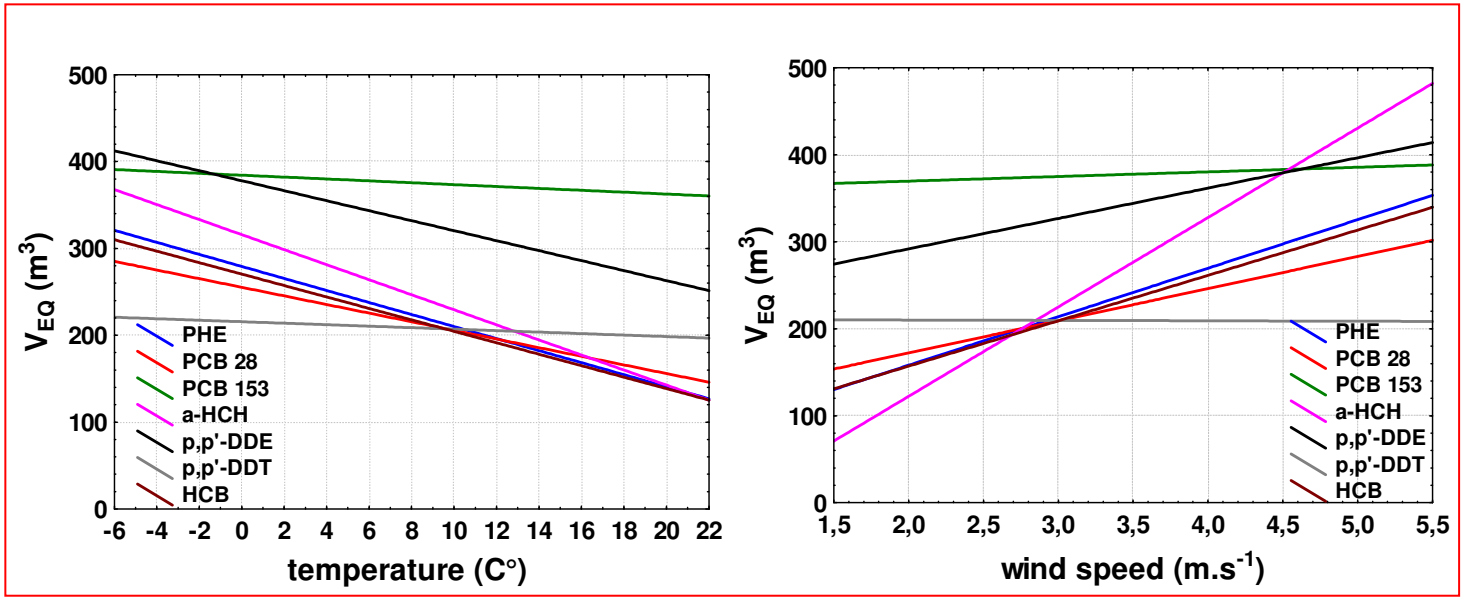
- d_6-γ-HCH
- PCBs - 3, 9, 15, 30, 107, 198

$$V_{PSM} (dC_{PSM}/dt) = k_A A_{PSM} (C_A - C_{PSM}/K_{PSM-A})$$

Passive samplers for POPs sampling – influence of environmental variables

- Effects of meteorological conditions ??

Correlation of VEQ (m^3) with temperature (°C) and wind speed ($m\ s^{-1}$) for various gas phase associated compounds.



Klanova, J., Čupr, P., Kohoutek, J., Harner, T., 2008. Assessing the influence of meteorological parameters on the performance of polyurethane foam-based passive air samplers. ES&T 42, 550-555.

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Passive sampling

Can environmental concentrations of pollutants be calculated from the analyte levels accumulated in an integrative passive sampler?

- **Calibration conditions should approximate field conditions**
- **Performance Reference Compounds**

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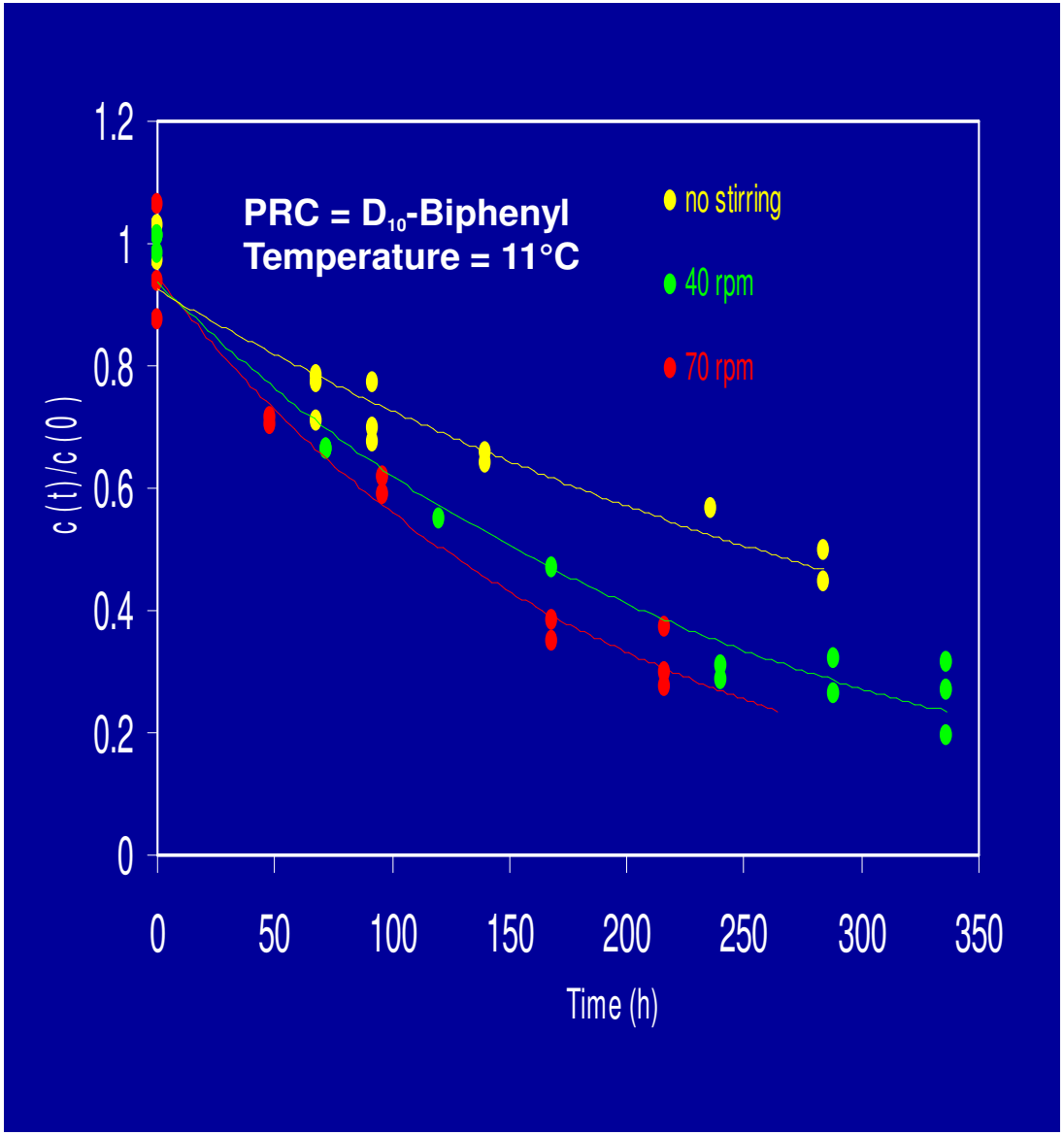
Performance reference compounds

PRCs are non-interfering compounds added to the sampler prior to exposure.

They are used for in situ calibration approach, where the rate of PRC loss during an exposure is related to the target compound uptake.

This is accomplished by measuring PRC loss rates during calibration studies and field exposures.

Use of performance reference compounds



B. Vrana, R. Greenwood, G. Mills

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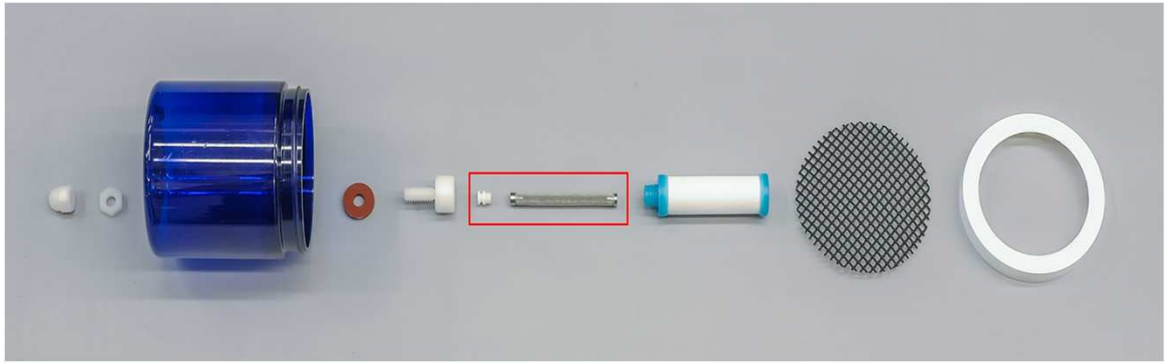
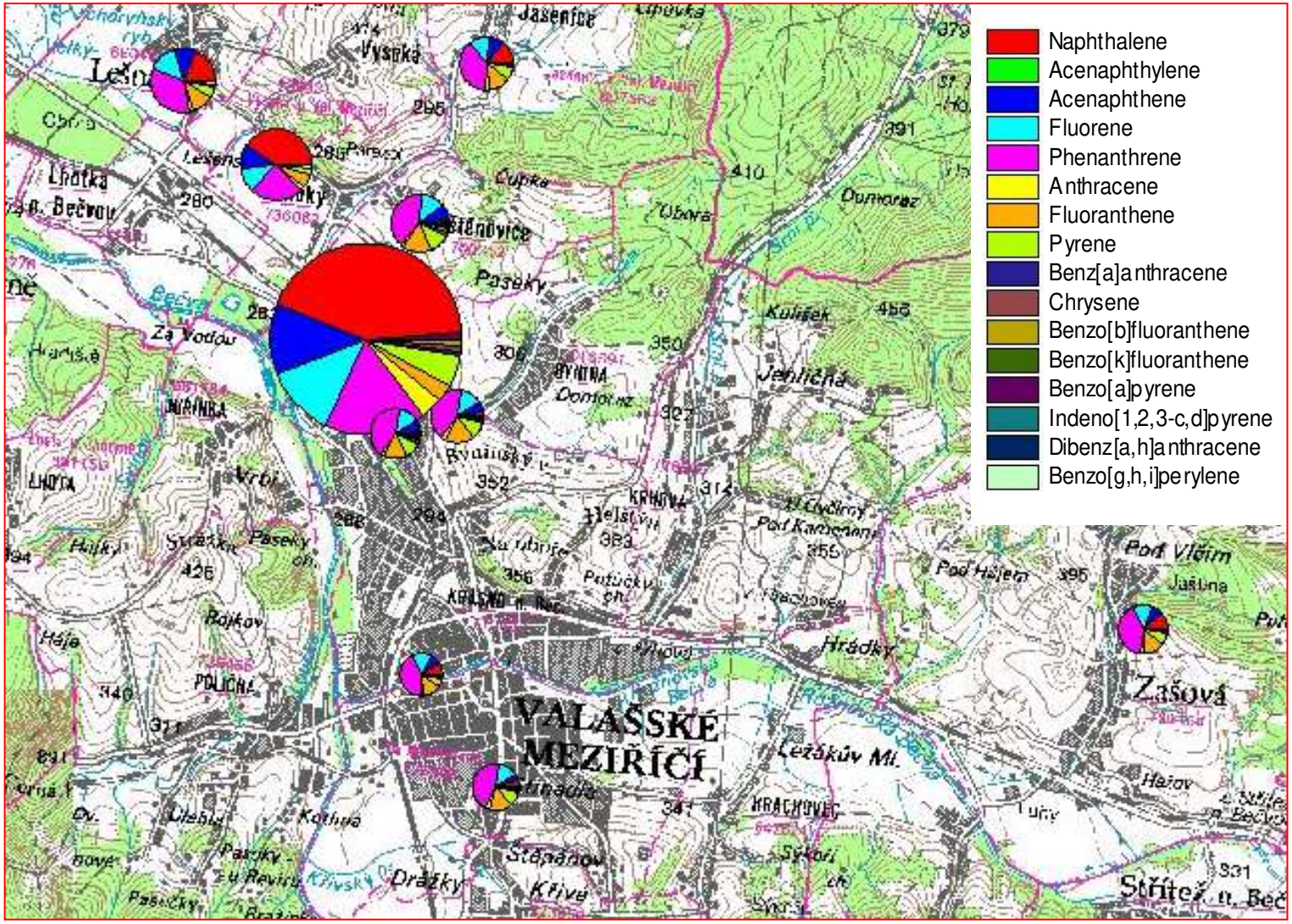
tarafından ortaklaşa finanse edilmektedir.

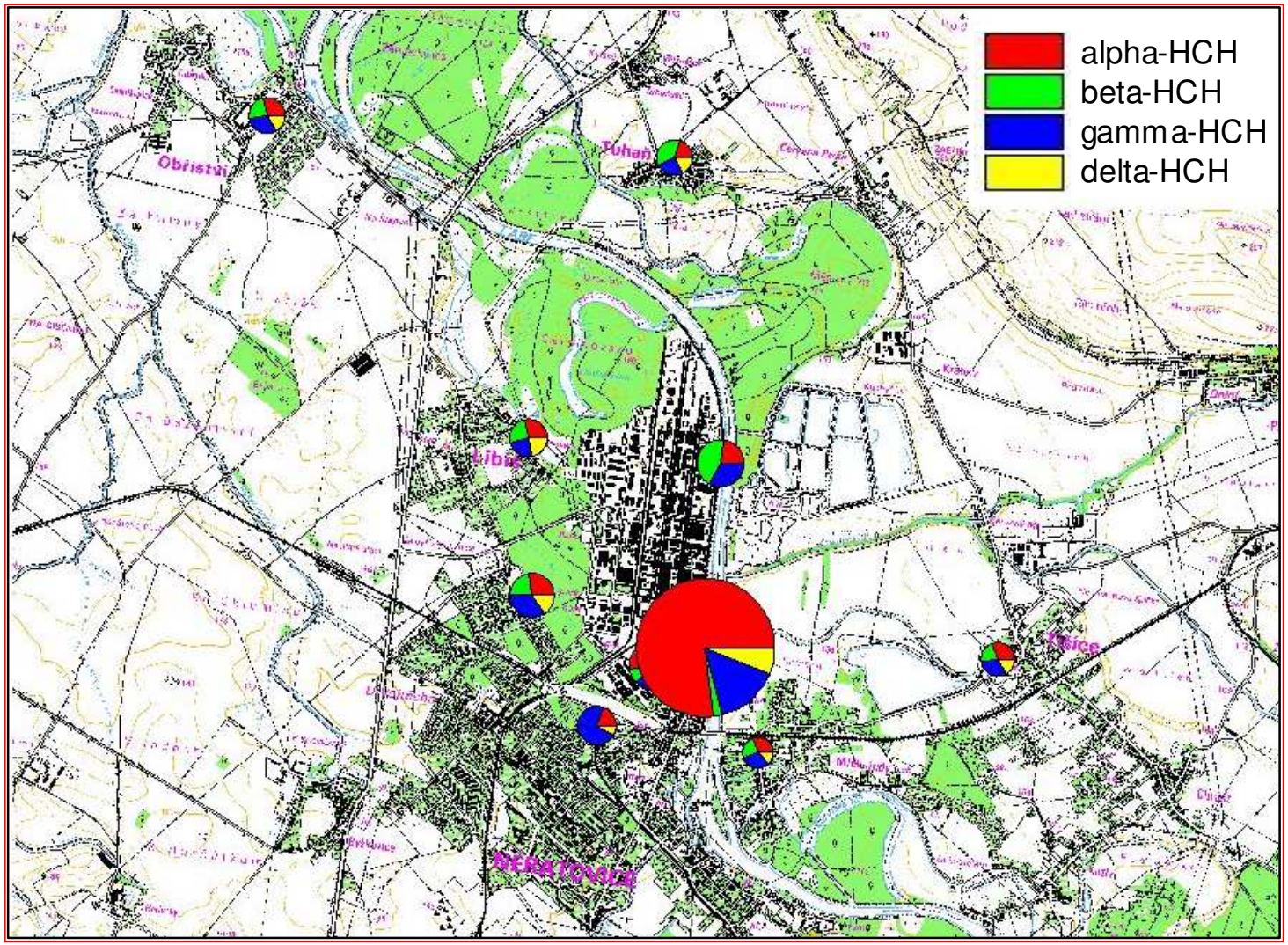
MerPAS campaign



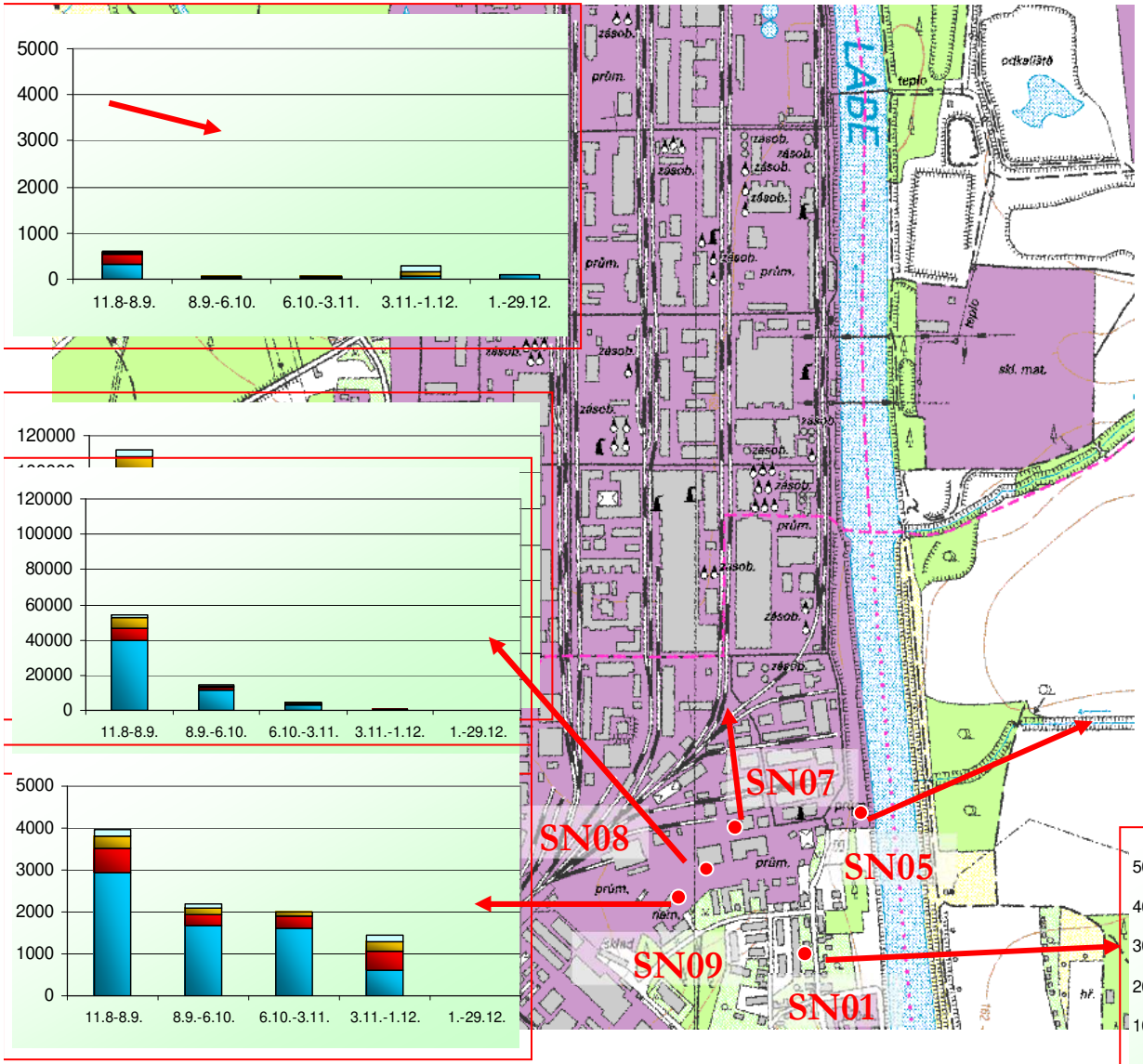
Distribution of PAHs in DEZA vicinity, 19/03-16/04/2004 - Biggest circle represents the total amount of 0.533 mg of PAHs sequestered on the filter



Distribution of HCHs in SPOLANA vicinity, 16/02-15/03/2004
Biggest circle represents the total amount of 445 ng of HCHs (a sum of α, β, γ, δ-HCH) sequestered on the filter

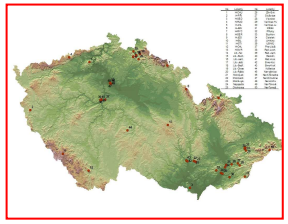


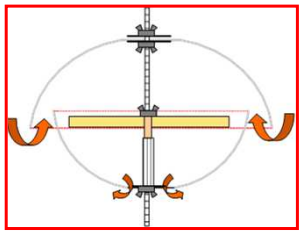
Monitoring of remediation – case of Spolana Neratovice – relative levels of HCHs in ambient air (ng/filtr)



Global/national POPs monitoring - MONET

RECETOX Monitoring Network





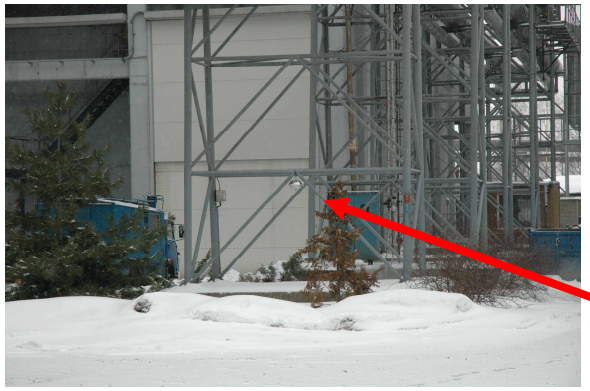
MONET = MOnitoring NETwork

MONET-CZ = Czech Republic	MONET-PIs = Pacific islands - Fiji
MONET-CEECs = 20 CEE countries + 2 CA countries	MONET-Africa = 17 African countries

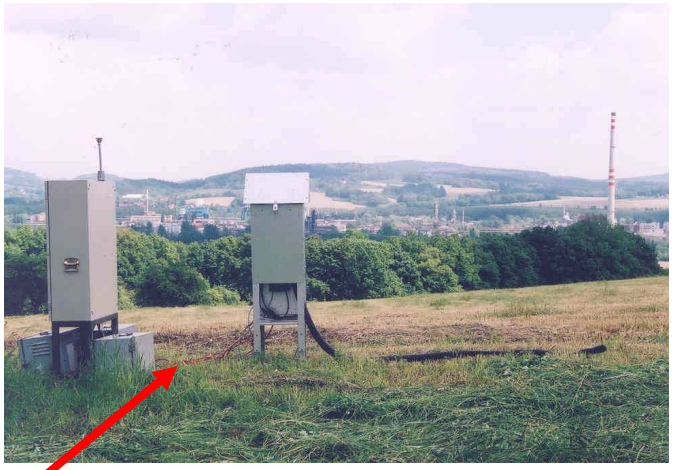
MONET-EUROPE – 55 sampling sites round whole Europe

monet

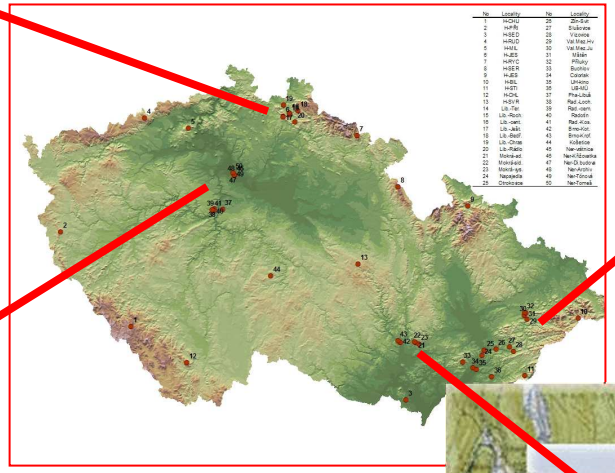
POPs Monitoring in ambient air – selected POPs sources



Municipal waste incinerator

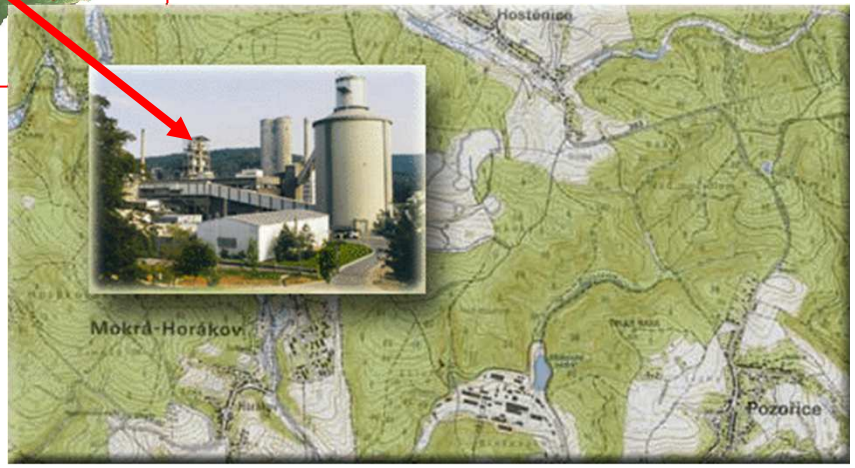


DEZA Valaššské Meziříčí



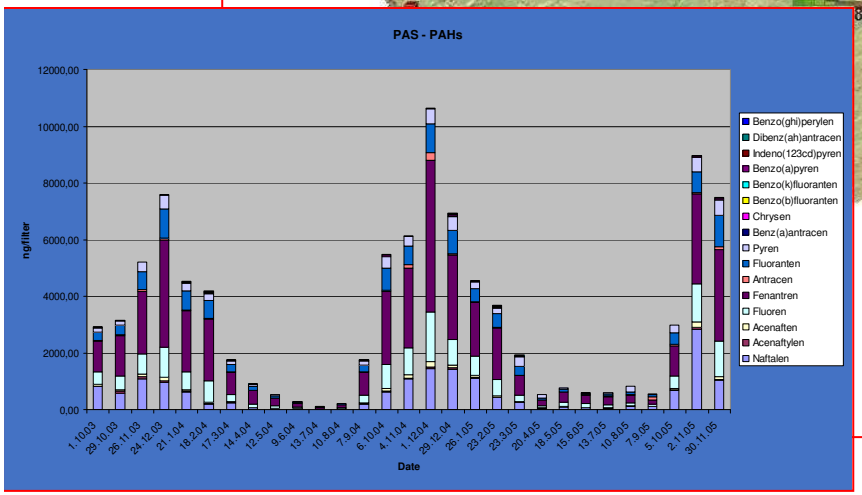
Spolana Neratovice

CMC a.s.



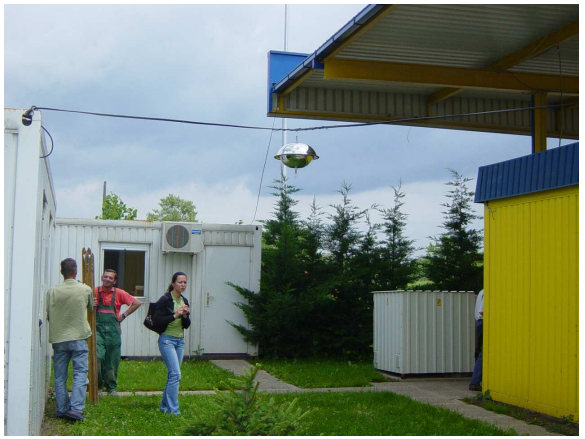
MONET-CZ - Monitoring of POPs in ambient air – passive sampling - Σ 16 PAHs [ng/filtr], January - December 2006

+PCBs, DDTs, HCHs, HCB, PeCB



EU Project APOPSBAL

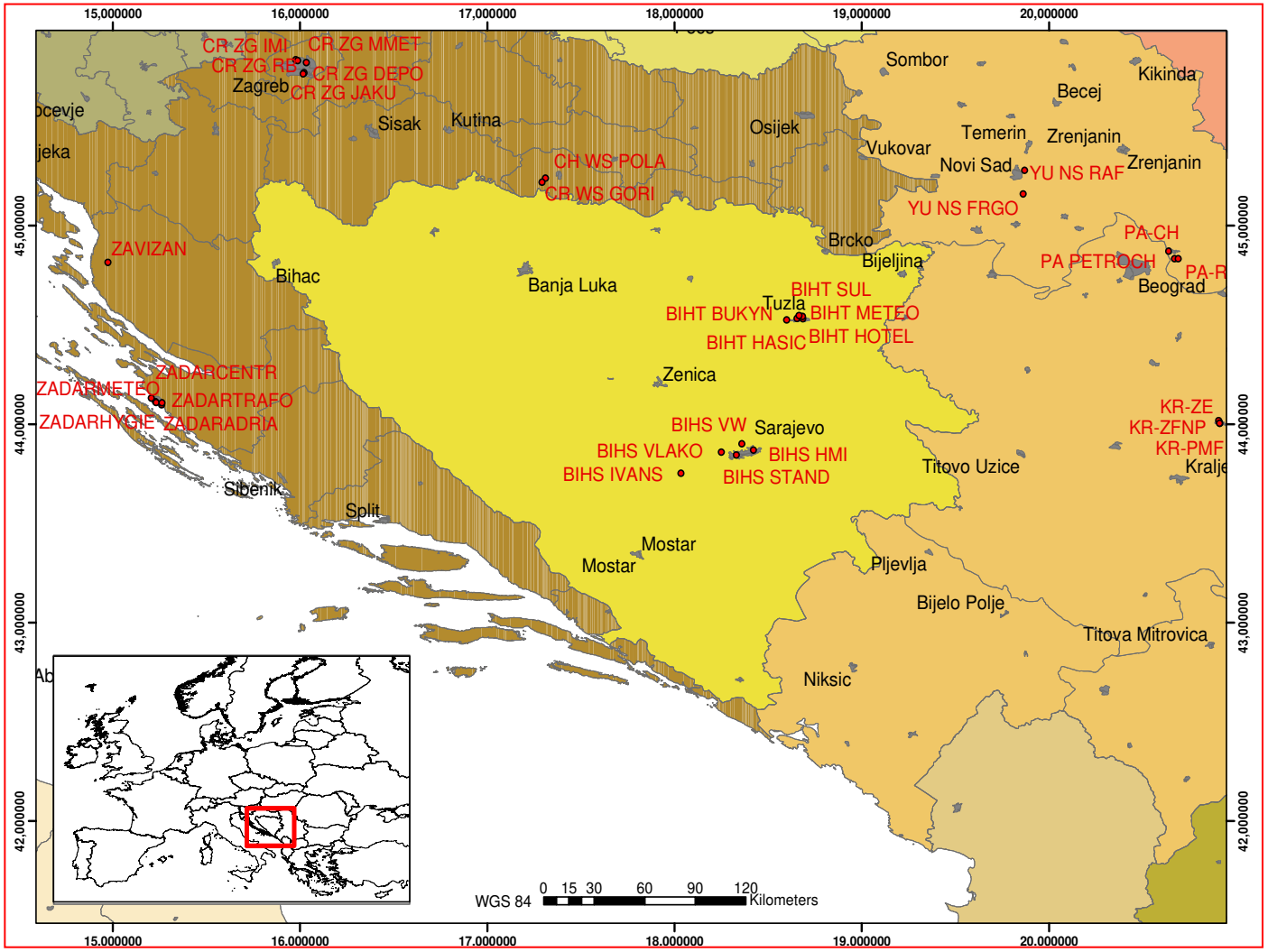
Assessment of the selected POPs (PCBs, PCDDs/Fs, OCPs) in the atmosphere and water ecosystems from waste materials generated by warfare in former Yugoslavia



Klanova, J.; Kohoutek, J.; Cupr, P.; Holoubek, I. Are the residents of former Yugoslavia still exposed to elevated PCB levels due to the Balkan wars? Part 2: Passive air sampling network. Environ. Int. 2007, 33, 727-735

Skarek, M.; Cupr, P.; Bartos, T.; Kohoutek, J.; Klanova, J.; Holoubek, I. A combined approach to the evaluation of organic air pollution - a case study of urban air in Sarajevo and Tuzla (Bosna and Hercegovina). Sci. Tot. Environ. 2007, 384, 182-193

APOPSBAL – RECETOX sampling sites



APOPSBAL – passive sampling – identification of hot spots

Klanova, J.; Kohoutek, J.; Cupr, P.; Holoubek, I. Are the residents of former Yugoslavia still exposed to elevated PCB levels due to the Balkan wars? Part 2: Passive air sampling network. Environ. Int. 2007, 33, 727-735

Skarek, M.; Cupr, P.; Bartos, T.; Kohoutek, J.; Klanova, J.; Holoubek, I. A combined approach to the evaluation of organic air pollution - a case study of urban air in Sarajevo and Tuzla (Bosna and Hercegovina). Sci. Tot. Environ. 2007, 384, 182-193

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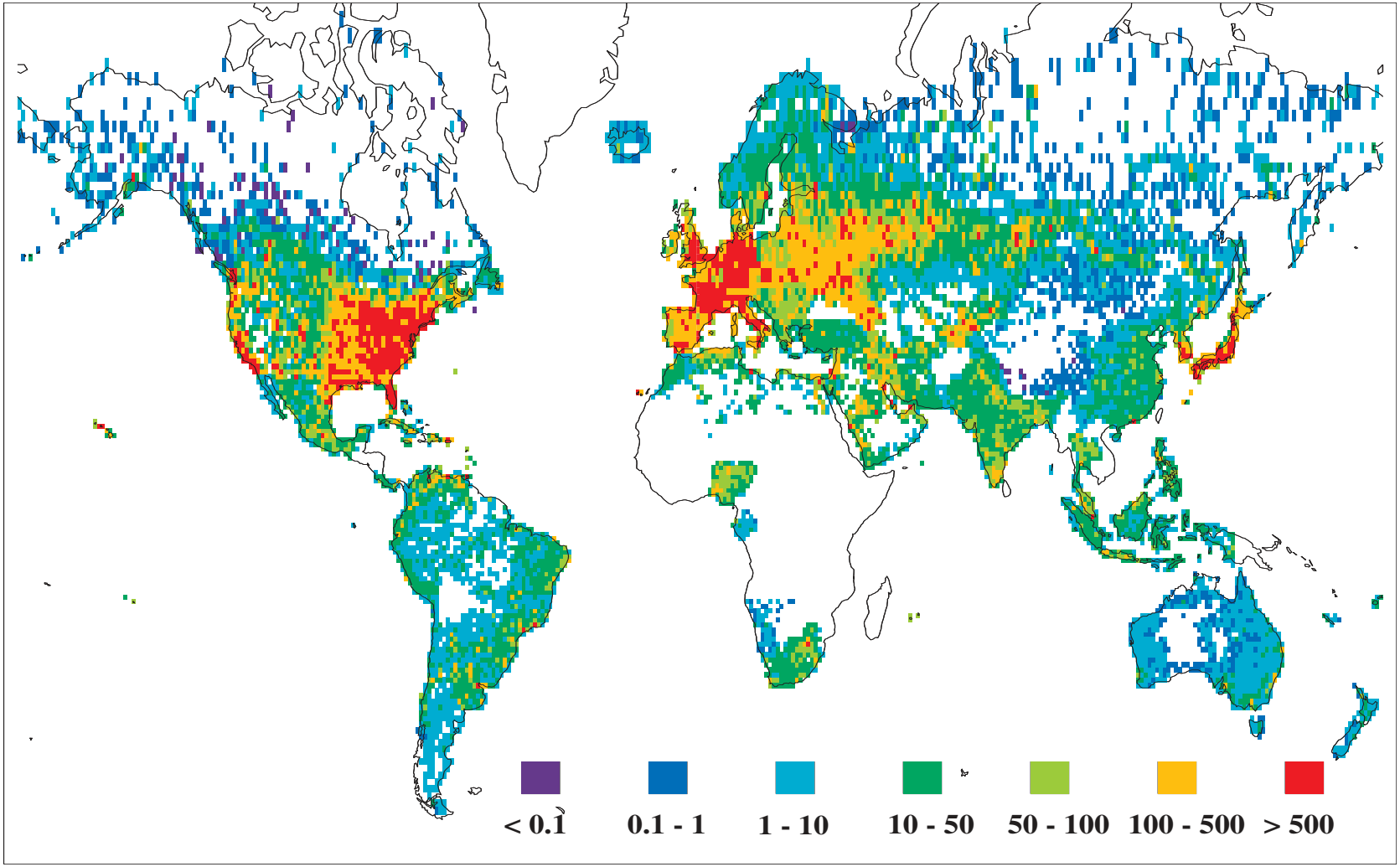
Temporal trends

- What timescales are envisaged (months – years – decades) ?
- Different media will have different response times to a reduction in use/emission
- There is much short-term variation e.g. seasonally; 'noise' in biological data
- What are the implications for sample frequency ?
- Should sampling be concurrent at different locations regionally/globally, and for different media? e.g. air in the north and south hemisphere

Spatial trends

Global PCB Emission



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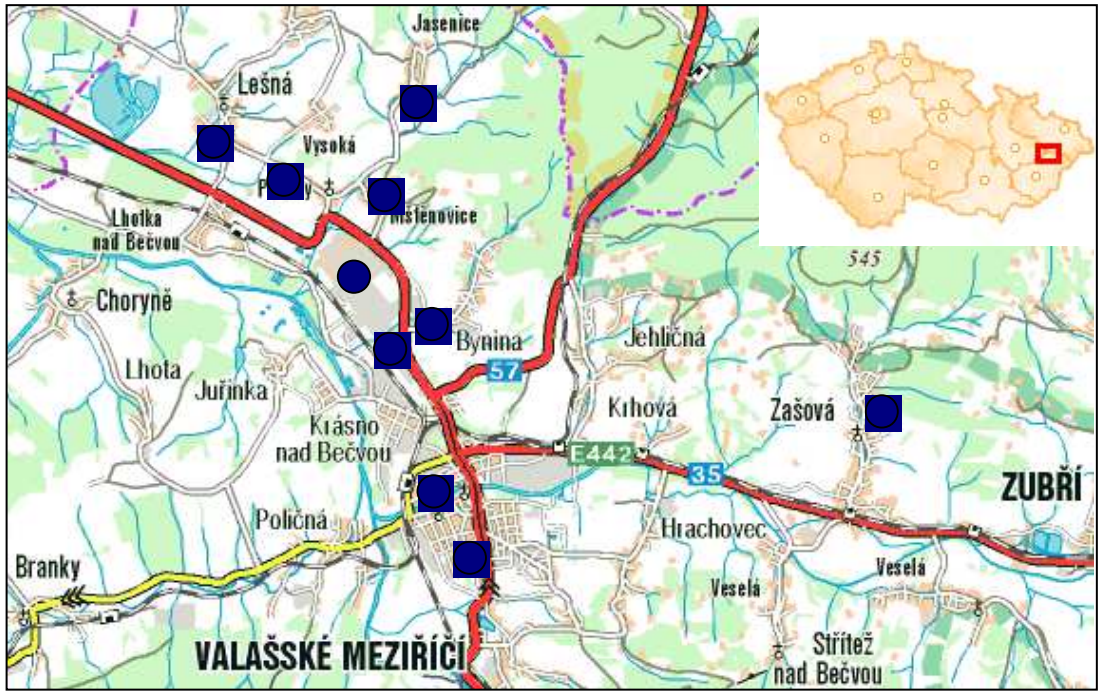
Sampling – documentation required

- **Sampling plan** (a goal, selection of sampling sites, analytes, sampling method, number of samples, sampling period and frequency, safety procedures), seeks the balance between the value of data and its price
- **Standard operational procedure** for sampling various matrices (sampling devices, steps involved in collecting of representative sample - homogenous, of reasonable size and stability, quality of transport and storage)
- **Sampling protocols** (name and number of the sample, sampling site, matrix, date of sampling, local conditions and measurements, methods, sample size, responsible person)

Sampling documentation

Sampling site 1. DEZA



GPS: 49°29'48"; 17°57'14"; 245 m

Local conditions: surroundings, near potential sources of contamination, terrain orography, fundamental meteorological condition

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Sampling site location

Requirements of sampling locality:

- location must ensure maximum representativeness of sampling relative to the reference object or situation
- must ensure complete coverage of the space and the phenomenon both in terms of space, and time

Influence the choice of location:

- locating stationary and mobile sources around the site
- transport characteristics of pollutants from these sources and the effects of meteorological and geographical conditions of these characteristics
- suitability of the site in terms of location of the sampler

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Sampling site location

The placement of sampling points in macroscale

- **Sampling aimed at protecting human health - must provide details of locations in areas with poor air quality in agglomerations, which leads to the occurrence of the highest concentrations, which are representative of the exposure of the population**
- **Must be excluded in measuring very small micro-environments and in their immediate area measurements shall be representative of air quality in the vicinity of at least 200 m^2 at traffic-oriented sites and several square kilometers at urban sites; should represent a model similar locations**
- **Sampling focused on the protection of ecosystems or vegetation - more than 20 km from agglomerations or more than 5 km from other built-up areas, industrial installations or motorways should represent the air quality in the vicinity of at least 1 000 km^2**

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Sampling site location

The placement of sampling points in the microscale:

- air flow around the inlet sampling equipment must not be barriers to entry (must be a distance of several meters from buildings, balconies, trees and other obstacles)
- the inlet of the sampling device should be at 1.5 m (the breathing zone) up to 4 m above the ground; Higher positions (up to 8 m) are necessary if the data should be representative for larger areas (long-range transport of pollutants)
- the inlet probe should not be positioned in the immediate vicinity of sources of air pollution, to avoid direct emissions sampling undiluted by mixing with ambient air
- sampling device-oriented transport should be at least 25 m from the edge of major junctions and at least 4 m from the center of the nearest traffic lane

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tarafından ortaklaşa finanse edilmektedir.

Sampling site location

Additional requirements for the location of sampling points

It must be taken into account the following factors:

- interfering sources of air pollution
- sampling equipment operator safety
- accessibility to the sampling device
- the availability of electricity
- visibility of the site in relation to its surroundings
  public safety
- requirements for co-location of different sampling devices
- planning requirements

It must be taken documentation and photographic sampling point.

Must be entered in the map the exact location (GPS coordinates).

Must be inspected at regular intervals of selection criteria.

Selection of sampling sites

What we must evaluate ?

- **Potential source of contamination – direct effect – nearby sources, transport via air, near to village, town, road**
- **Terrain configuration – inversion valley, lowland, hills, top of hills**
- **Localization of sampling sites**

  – height (1.5 -2 m – respiratory zone – more focused on the local sources; 6-10 m – for evaluation of long-range transport

  - surroundings of sampling site – natural or anthropogennic barriers – hills, trees, buildings – traps for dust, „immission shadow“

  - surface in the sampling site – grassland – optimal; sandy, asphalt or other surface – source of contamination

Selection of sampling sites

- **Meteorological conditions before and during sampling:**

  - wet deposition (rain, snow,..) – washing effects
  - wind speed – low – mainly effects of local sources, high – emissions are dispersed, effect of long-range transport
  - wind direction – important for localization of sampling site
  - temperature – higher – evaporation of highly volatile compounds from soils and surfaces in the given locality; lower – effect of local heating systems; pressure – effect to meteorological conditions
  - humidity – low – higher contents of dust, particles and increasing sorption of contaminants; higher – effect of humidity on the process of sampling

- **Other – nature of dust – size etc.**

Meteorological conditions

Meteorological conditions affect the transport, dilution rate and the stability of the monitored pollutants.

- **wind speed and direction**
- **temperature**
- **atmospheric pressure**
- **intensity of solar radiation**
- **precipitation (type and intensity)**

These parameters should be monitored during each sampling directly at the sampling site. can be used and data from the meteorological station near the site.

Meteo – measurement of meteorological parameters

WV – wind velocity

WD – wind direction

p – atmospheric pressure

h – relative air humidity

RAIN – sum of precipitation

GLRD – sun irradiation

T – temperature (not specified)

T2m – temperature 2 m above terrain

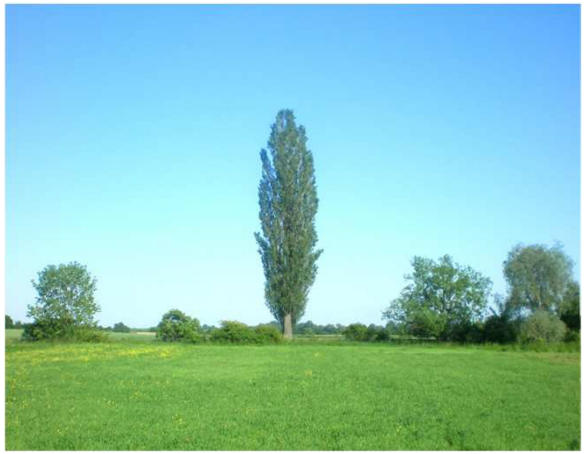
T10m – temperature 10 m above terrain

Meteorological conditions

In terms of repeatability air sampling should be distinguished:

- type of weather (eg winter inversion)
- Type of weather occurs repeatedly (albeit irregularly), therefore samples for the same type of weather are repeatable.
- Case weather conditions (the sum of all its parameters)

Case the weather is quite unique, so the subscription is for exactly the same conditions (for the same case weather) unrepeatable.



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Sampling plan, QA/QC

- Developing the sampling plan is the first step in the preparation of sampling
- Sampling plan must contain all the time, and local information on upcoming sampling, the number of samples and the method of handling
- Must specify the sampling technique (used types of samplers and sorbent)
- An integral part of each sample is also planned system assurance and quality control QA / QC (Quality Assurance / Quality Control)
- Operation of the sampling device is specified standard operating procedures (SOP) for each type of sampling

Sampling plan, QA/QC

Atmospheric Station, CzechGlobe

Atmospheric Station, CzechGlobe

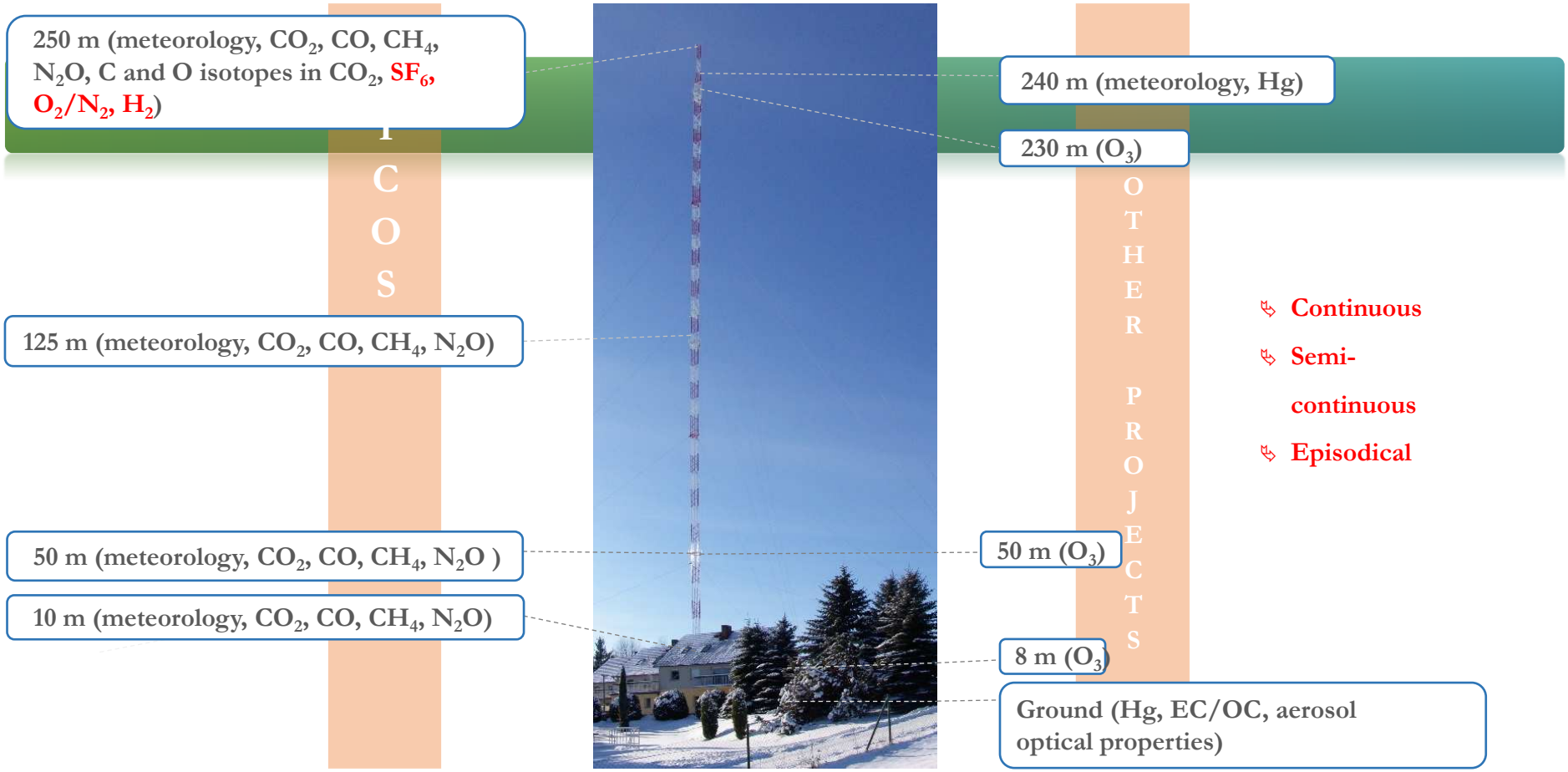
- **250 m tall atmospheric tower**
- **ground based technological containers**
- **technological container at 230 m**
- **air-conditioned cabinets at 8, 50, 125 m**
- **elevator (230 m)**

AS is focused on the investigation of the background temporal trends, vertical concentration gradient and long-range transport of GHGs and selected atmospheric pollutants.

This is complemented by the monitoring of basic meteorological characteristics.

Atmospheric station Křešín u Pacova

Exploiting the potential of high tower Measurements



NAOK – existing instrumentation

240-250 m: Meteo, CO_2, CO, CH_4, NO_2, O_3, isotopic CO_2, Hg …

230 m: Meteo, O_3, Aerosols

230 m height platform

125 m: Meteo, CO_2, CO, CH_4, NO_2

80 m: Meteo, CO_2, CO, CH_4, NO_2 , fluxes

50 m: Meteo, CO_2, CO, CH_4, NO_2, O_3

8-10 m: Meteo, CO2, CO, CH4, NO2

Ground containers

4 m: Meteo, Hg, Aerosols

ECOC by **Sunset Laboratory Inc., USA**

Ground container (4 m agl), from 2013.

230 m agl, from 2019.

BC and its light-absorption coefficient by aethaometer **AE31, AE33 (Magee Scientific)**

4 m agl, from 2012

230 m agl, upcoming.

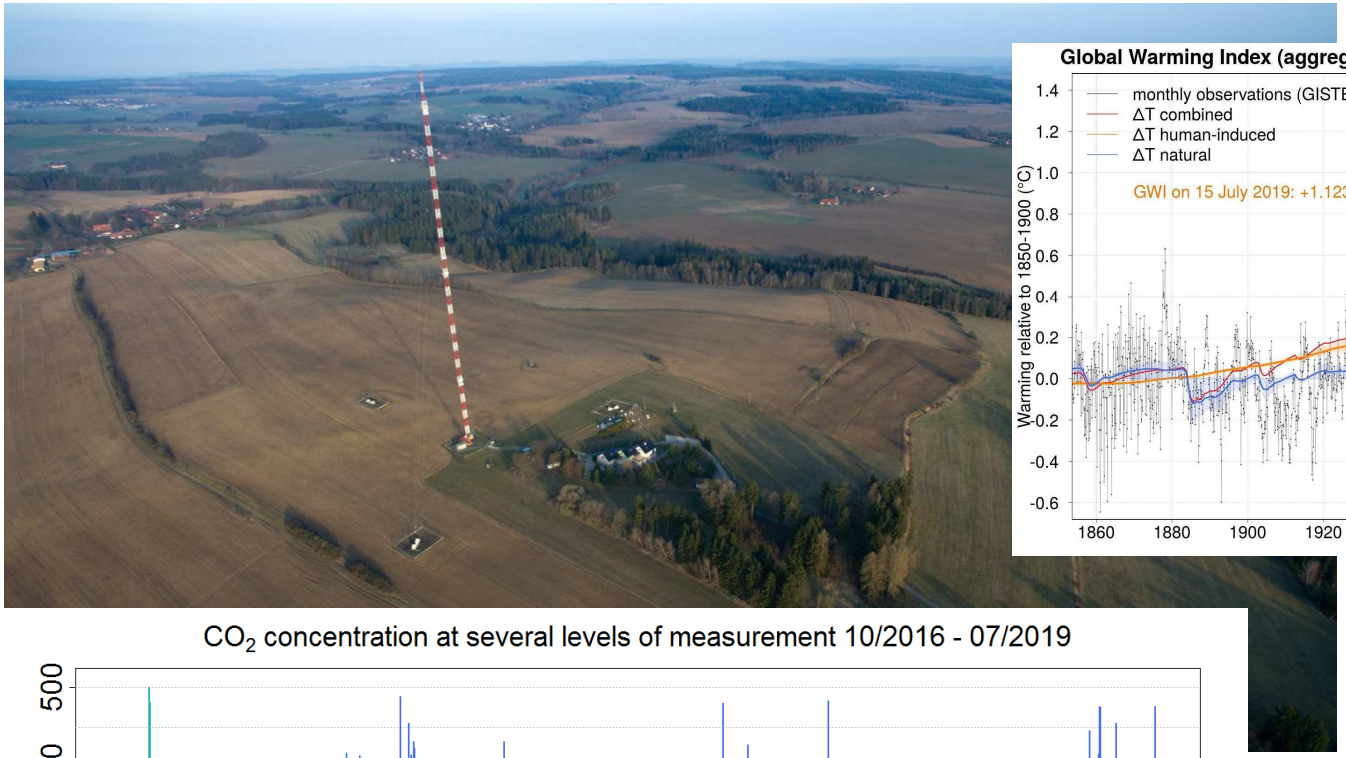
Light-scattering coefficient by Nephelometer

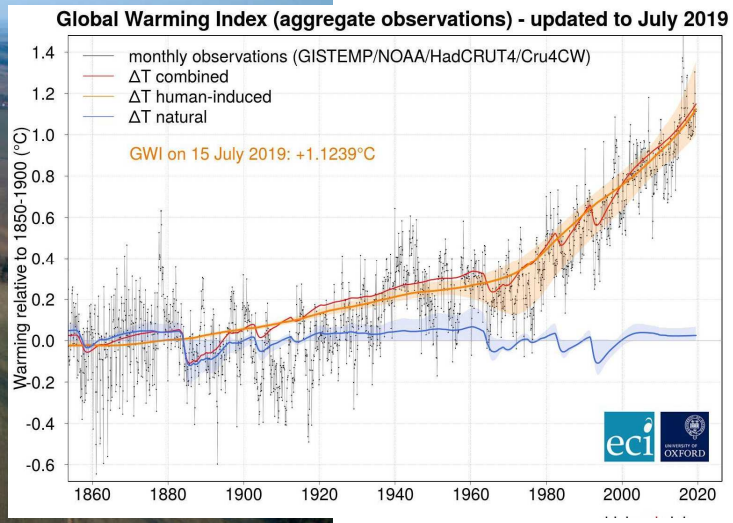
TSI 3563, Ecotech Aurora 3000

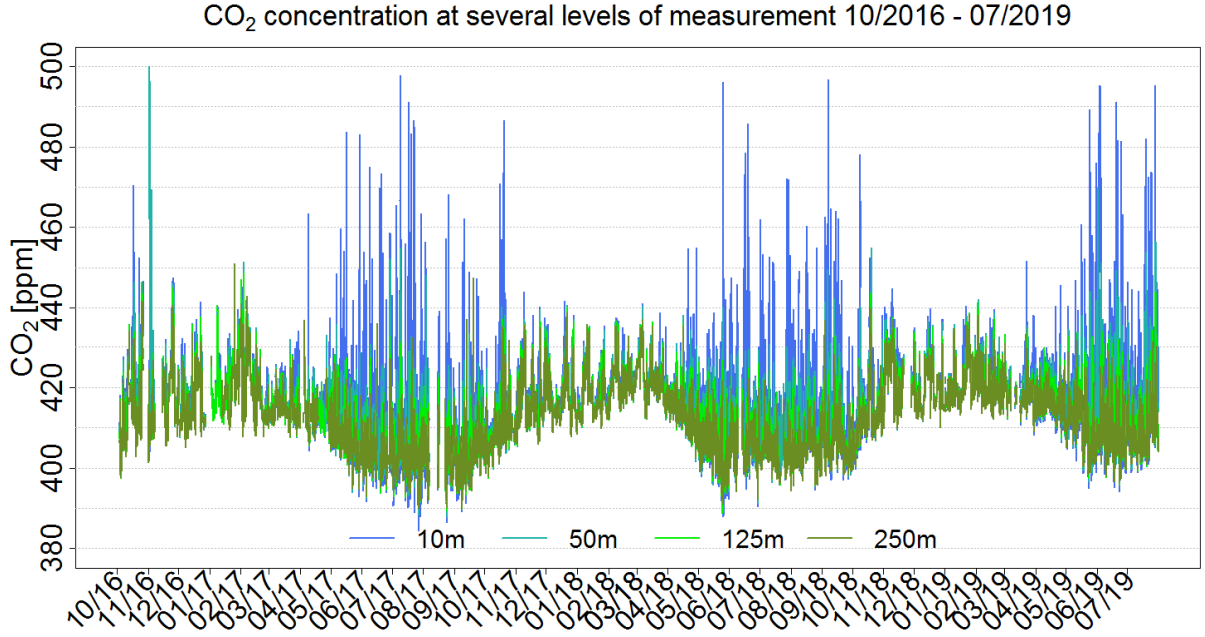
230 m agl, upcoming.

4 m agl, from 2012

National Atmospheric Observatory (NAO) Košetice



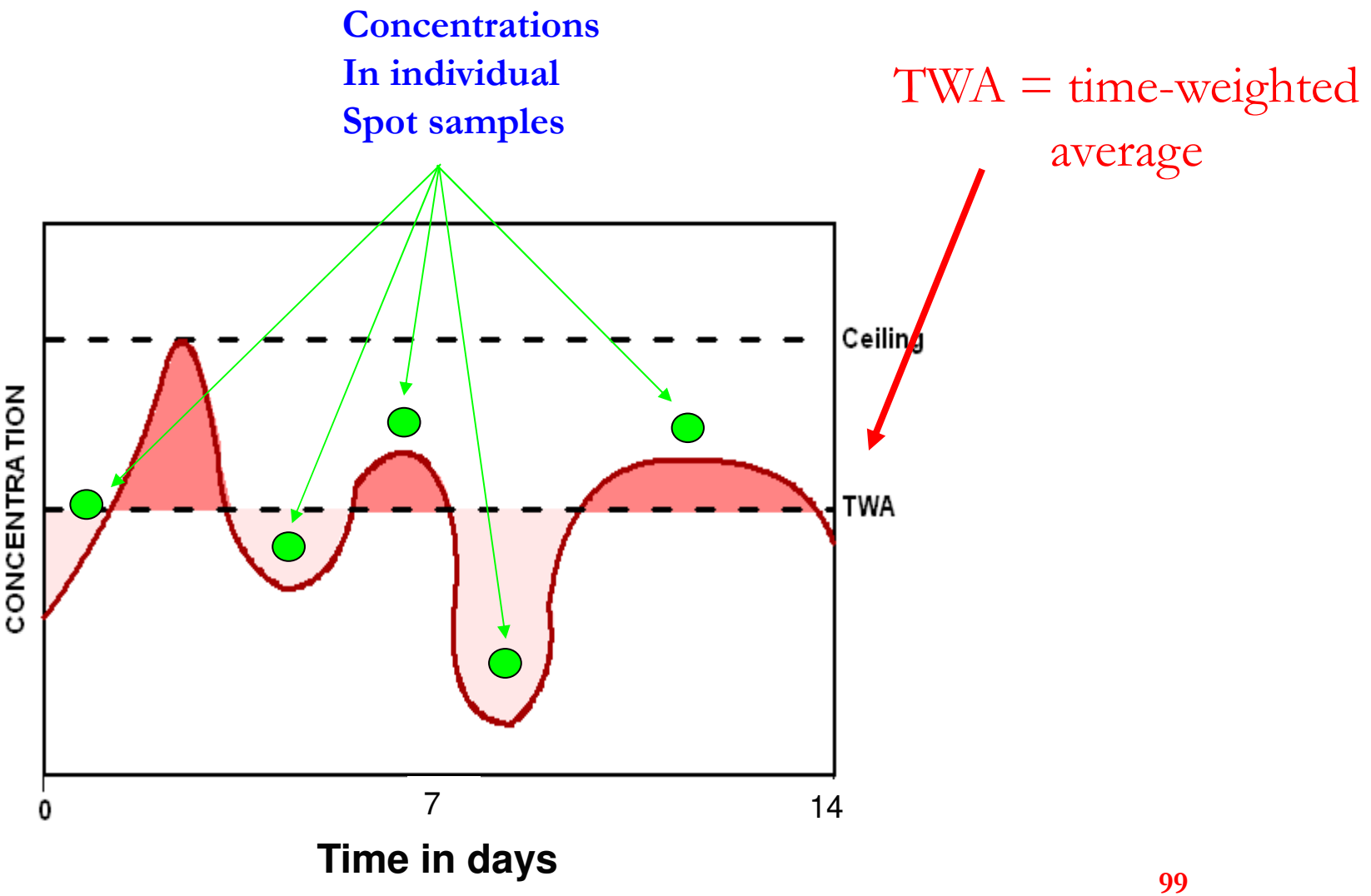




Bu Proje, Avrupa Birliği ve Türkiye Cumhuriyeti tarafından ortaklaşa finanse edilmektedir.

Contents

Variation in pollution over time



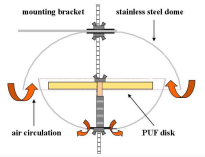
Air sampling: Active vs. passive sampling

Active sampling

- ☺ Accurate (?)
- ☺ Ideal for understanding processes
- ☺ High temporal resolution (e.g. pesticides application)

- ☹ Expensive, "hard" to use, large size
- ☹ Source of electricity needed
- ☹ High request on personal involvement, maintenance and support
- ☹ Subject to sampling artefacts
- ☹ Noise

Passive sampling



- ☺ Cheap, easy to use, small size
- ☺ No electricity needed (remote areas)
- ☺ Low need on personal involvement
- ☺ No noise (working environment, bedrooms)

- ☹/ ☺ Long term studies
- ☹ Uncertainty with assessment of concentration (a factor of 2-3 of the "true" air concentrations)
- ☹ Strong influence of meteorological parameters
- ☹ Subject to sampling artefacts
- ☹ Collect mainly the gas phase (less efficient for particle-bound compounds)

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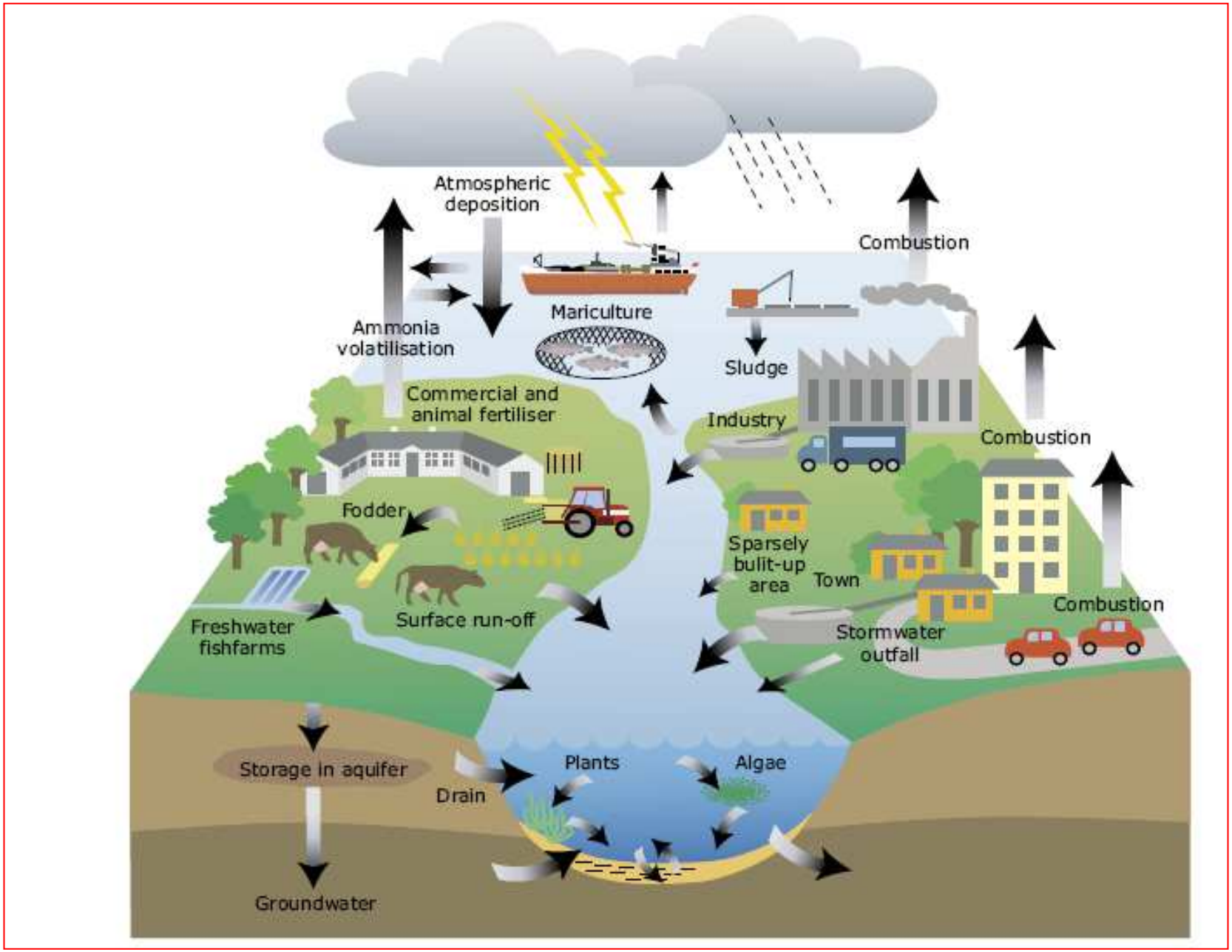
Surface water pollution by chemicals



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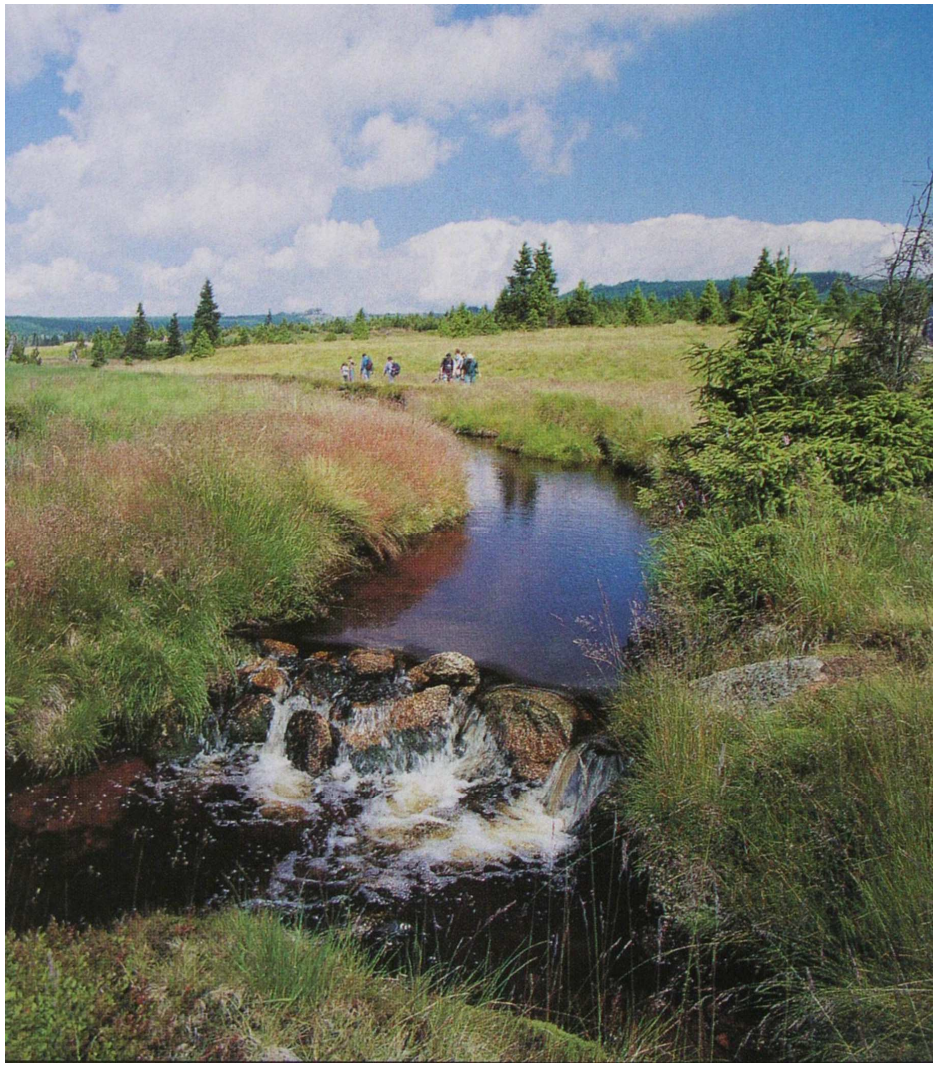
Water pollution - organics

Primary – by substances from waste water, deposition, leaching etc.

- **Natural (humic substances, phenols,..)**
- **Anthropogenic (oil pollution, pesticides, detergents, PCBs, HCX..)**

Natural „pollution“



Industrial waste waters

Agricultural pollution

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Types of waters

Occurrence:

a) atmospheric (deposition)
b) surface
c) subsurface
d) groundwater

Use:

a) drinking
b) household
c) technological
d) waste

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Water sampling: Where to sample?

- Depends on the aim of the study, but it is important to have a representative sample (e.g. close to waste water treatment plan if we want to assess its efficiency in removing some chemicals). Otherwise far from industrial or municipal waste water discharges or polluted tributaries
- Easy of access by a bridge or by boat.
- Sampling at the surface or deep?
- Potential of vandalism or robbery of the samplers






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Water sampling: grab vs. passive?

Grab

Representative only of a specific time (may be different few hours later) and specific location (maybe different few meters away)

Passive

- Easy to deploy and no maintenance needed
- They provide time-weighted average concentrations rather than a snapshot

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Sampling of natural waters

Goals:

Surface waters

- quality assessment
- determination of pollution level
- study of living conditions

Soil water

- agricultural, forestry, relationships to other types of waters

Ground waters

- prognosis of composition (relationships to environmental factors)
- nature of waters
- flows
- using and protection of waters

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Çevre ve İklim Eylemi Sektör Operasyonel Programı
Kalıcı Organik Kirleticiler
UNDP

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Available monitoring methods

- Equipment for measuring physical and physico-chemical variables
  - dissolved organic carbon (DOC)
  - pH
  - temperature
  - turbidity
- Biological assessment techniques
  - biomarkers
  - bioassays
  - biosensors
  - biological early warning systems (BEWS)
- Chemical analytical methods
  - sensors
  - test kits
  - passive sampling devices
  - immunoassays

Environmental monitoring methods

Biomonitoring

- **Chemical Monitoring:** by measuring levels of a selected set of well-known contaminants in abiotic environmental compartments (water, sediment)
- **Bioaccumulation Monitoring:** exposure assessment by measuring contaminant levels in biota or determining the critical dose at a critical site (bioaccumulation)
- **Biological Effect Monitoring:** exposure and effect assessment by determining the early adverse alterations that are partly or fully reversible (biomarkers)
- **Health Monitoring:** effect assessment by examining the occurrence of irreversible diseases or tissue damage in organisms
- **Ecosystem Monitoring:** assessment of the integrity of an ecosystem by making an inventory of species composition, density and diversity

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Water Framework Directive

- Water Framework Directive (WFD) 2000/60/EC
- Environmental Quality Standards (EQS) Directive 2008/105/EC
- Directive 2013/39/EU amending Directives 2000/60/EC and 2008/105/EC as regards priority substances in the field of water policy
- Directive 2009/90/EC on technical specifications for chemical analysis and monitoring of water status (QA/QC)

Sampling of water from rivers and lakes

When samples are collected from a river or stream, observed results may vary with:

- depth
- stream flow
- distance from each shore.

Selection of the number and distribution of sites at which samples should be collected depends on:

- study objectives,
- stream characteristics
- available equipment etc.
- other factors

If equipment is available, take an integrated sample from top to bottom in the middle of the main channel of the stream or from side to side at mid-depth.

If only grab or catch samples can be collected, preferably take them at various points of equal distance across the stream; if only one sample can be collected, take it in the middle of the main channel of the stream and at-mid-depth.

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Sample handling and storage

A unique sample identification, i.e. a number or code.

All details about the sample should be recorded:

- storage conditions
- documented transfer from person to person
- details of the container and closures
- the appearance of the sample on receipt
- the length of storage

Properties of the analyte:

- volatility
- sensitivity to light
- thermal stability
- chemical reactivity
- potential hazard to laboratory staff

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Sample handling and storage

- The integrity of the sample must be preserved
- No risk of contamination or 'cross-contamination', i.e. no material should enter or leave the sample container.
- Extremes of environmental conditions should be avoided.
- Sample storage in a separate area away from analytical calibrants or any other material which may contain a high concentration of the analyte.
- Take precautions to avoid cross-contamination between sample storage areas and other laboratory areas
- Use a maximum/minimum thermometer to check for temperature fluctuations during storage
- The samples must also be stored under appropriate conditions during the time interval between sampling and arrival at the laboratory for analysis

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Sample containers

Sample containers must not cause:

- Sample contamination
- Surface adsorption, absorption or evaporation of analytes
- Leaching of interfering compounds to the sample
- Special sampling vessels for certain types of analytes

"Blank" sample for testing correct selection of sample containers and their cleaning

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Sources of error during sampling

- **Contamination** – materials of sampling tools, sample transport and storage containers, cross-contamination of samples, preservation agents, improper storage and transport
- **Sample stability** - unsuitable sampling tools, sample containers, transport and storage
- **Sample conservation** materials of containers, preservation agents
- **Sampling procedure** deviation from standard operation procedure, improper sampling technique
- **Transport and manipulation**

Insufficient or absenting communication with the analytical laboratory

Storage conditions for laboratory samples

Storage condition	Appropriate sample types	Inappropriate sample types
Deep freeze (−18°C)	Samples with high enzymatic activity Perishable goods/products Less stable analytes	Samples which liquefy on thawing Aqueous samples
Refrigerator (4°C)	Soils Fresh fruit and vegetables Aqueous samples	Samples with possible enzymatic activity
Room temperature (in the dark)	Dry powders and granules Minerals Stable analytes	Fresh foods
Desiccator	Hygroscopic samples	Samples which are more hygroscopic than the desiccant

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tarafından ortaklaşa finanse edilmektedir.

Sampling according to ISO/IEC 17025

A defined procedure whereby a part of a substance, material or product is taken to provide for testing or calibration a representative sample of the whole.

Sampling may also be required by the appropriate specification for which the substance, material or product is to be tested or calibrated.

Quality assurance (QA) of sampling and sample treatment

- Sampling - the process of selecting a portion of material, in some manner, to represent or provide information about a larger body of material
- The purpose of sampling must be clearly defined. It will affect both the sampling plan and the choice of analytical method
- These will depend on the acceptable level of uncertainty in the final result the analytical result may depend on the method used for the analysis, it will always depend on the type of sampling plan used
- Sampling uncertainties cannot be evaluated or controlled using standards or reference materials
- Sampling should adhere to SOPs or international norms

Small sampling error => serious error in analytical results

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

Operations in a sampling scheme and the analysis

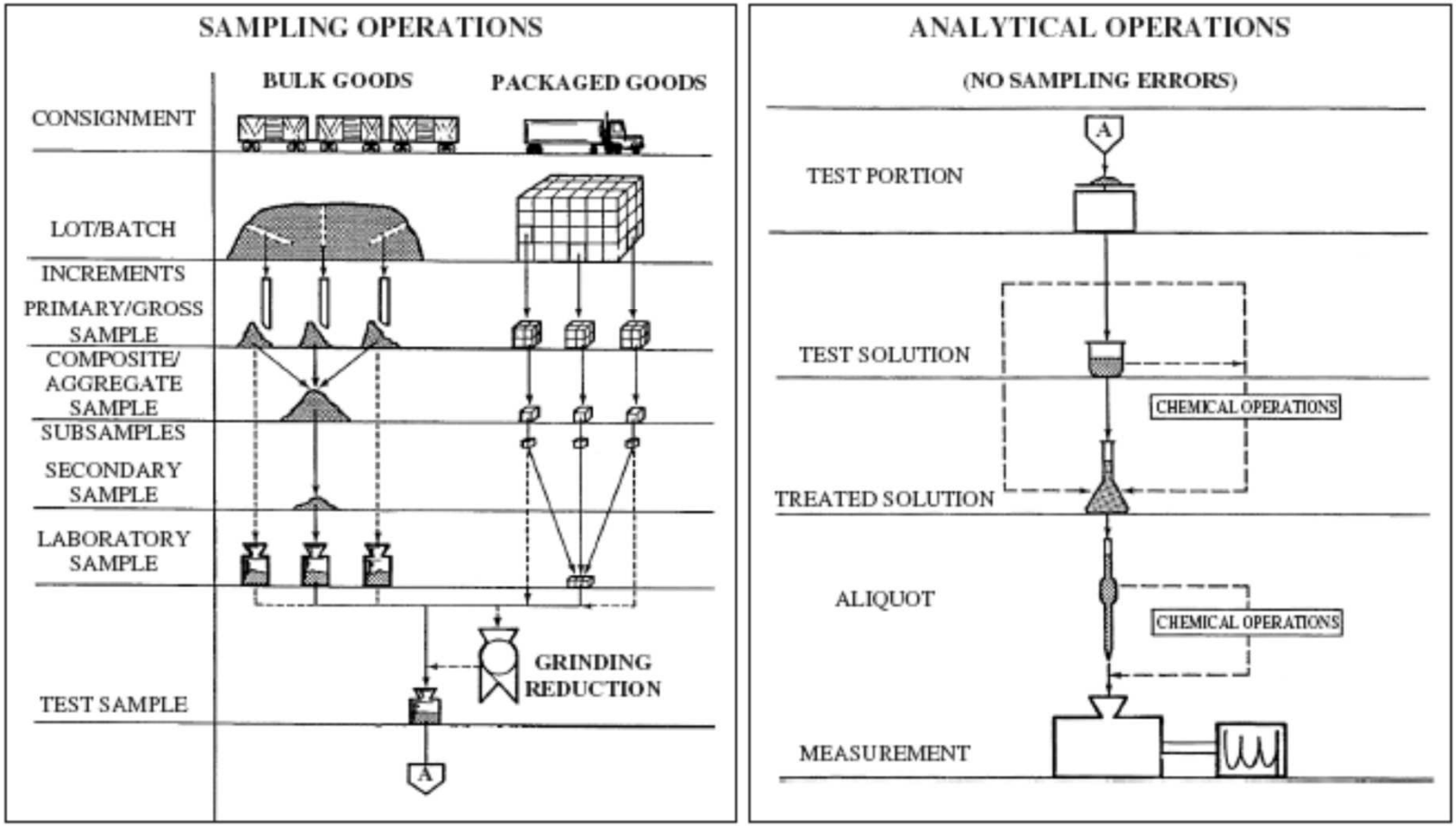

Figure 3.1 Schematic of sampling and analytical operations. Note: the lower "A" of the sampling operations continues with the upper "A" of the analytical operations [1]. Reproduced by permission of the International Union of Pure and Applied Chemistry, from Horwitz, W., *Pure Appl. Chem.*, **62**, 1193–1208 (1990).

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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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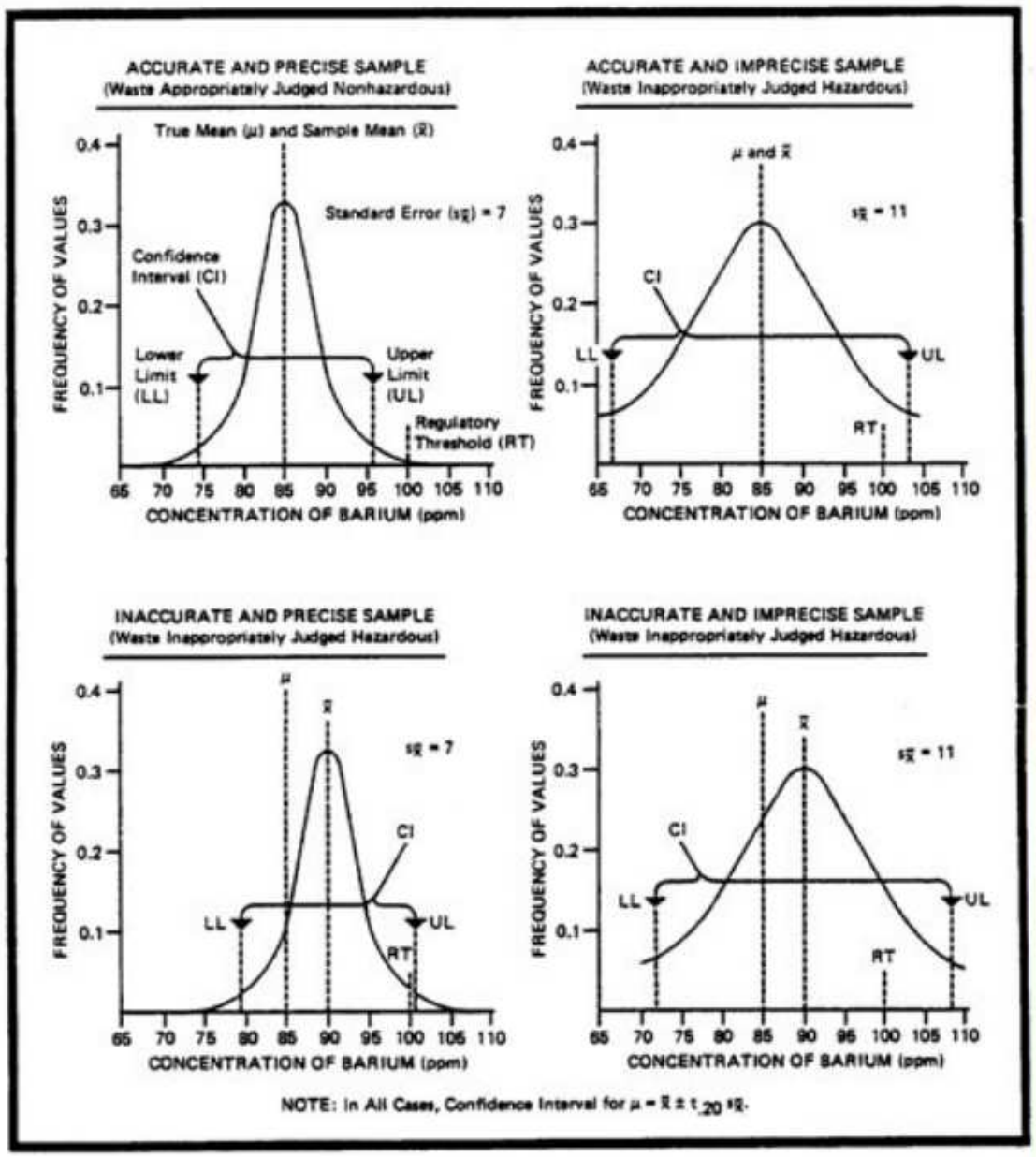
tarafından ortaklaşa finanse edilmektedir.

Representative sample

How do we get an accurate sample?

It must be one that accurately represents our material

Accurate sample



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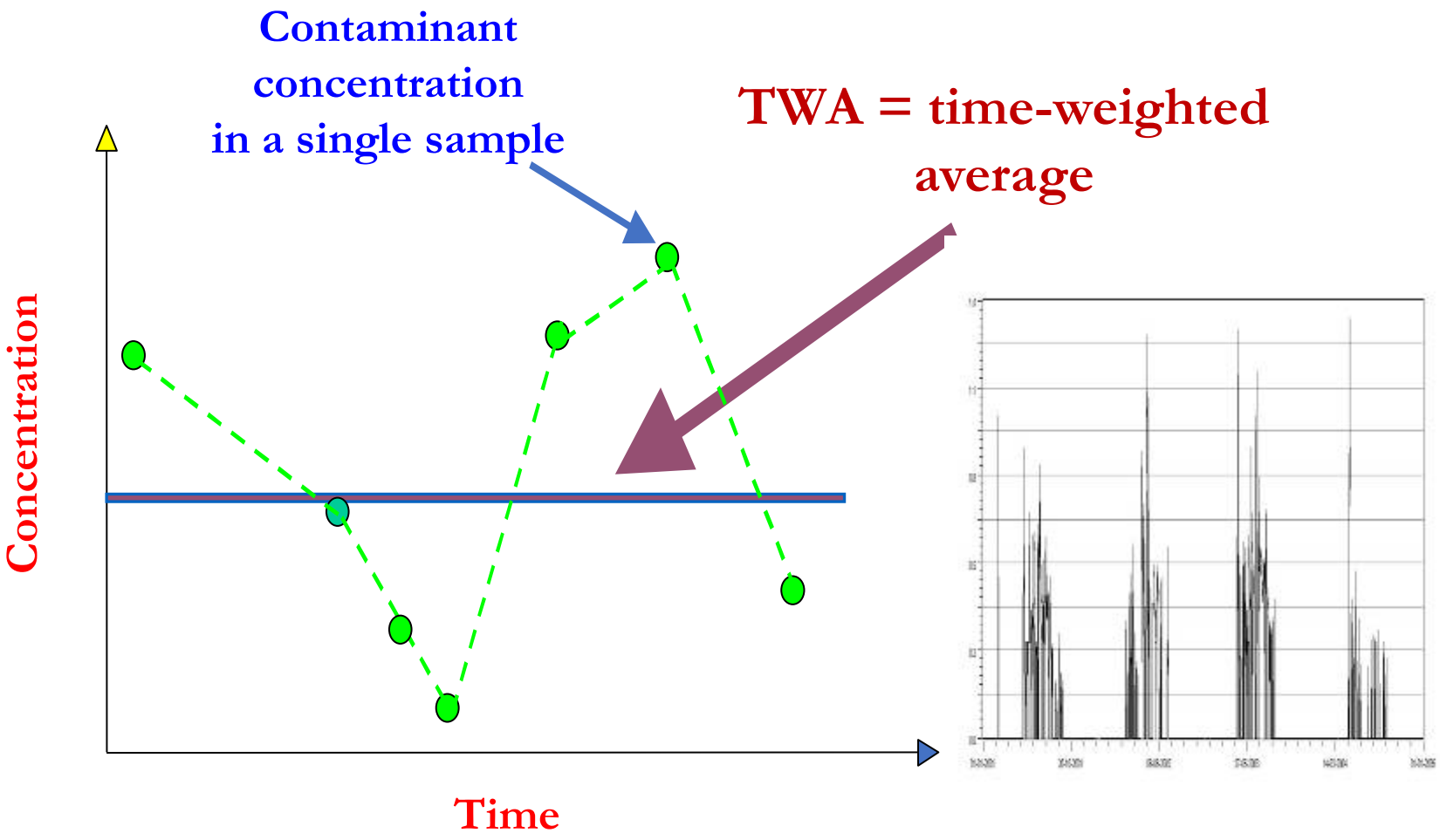
Representative sample

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

Dynamic system: contaminant in a river



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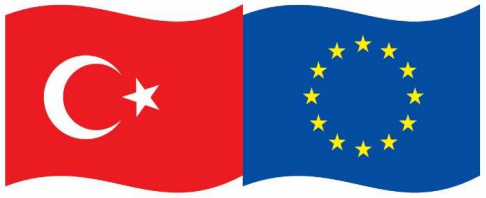
Selective Sample

- 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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Random Sample

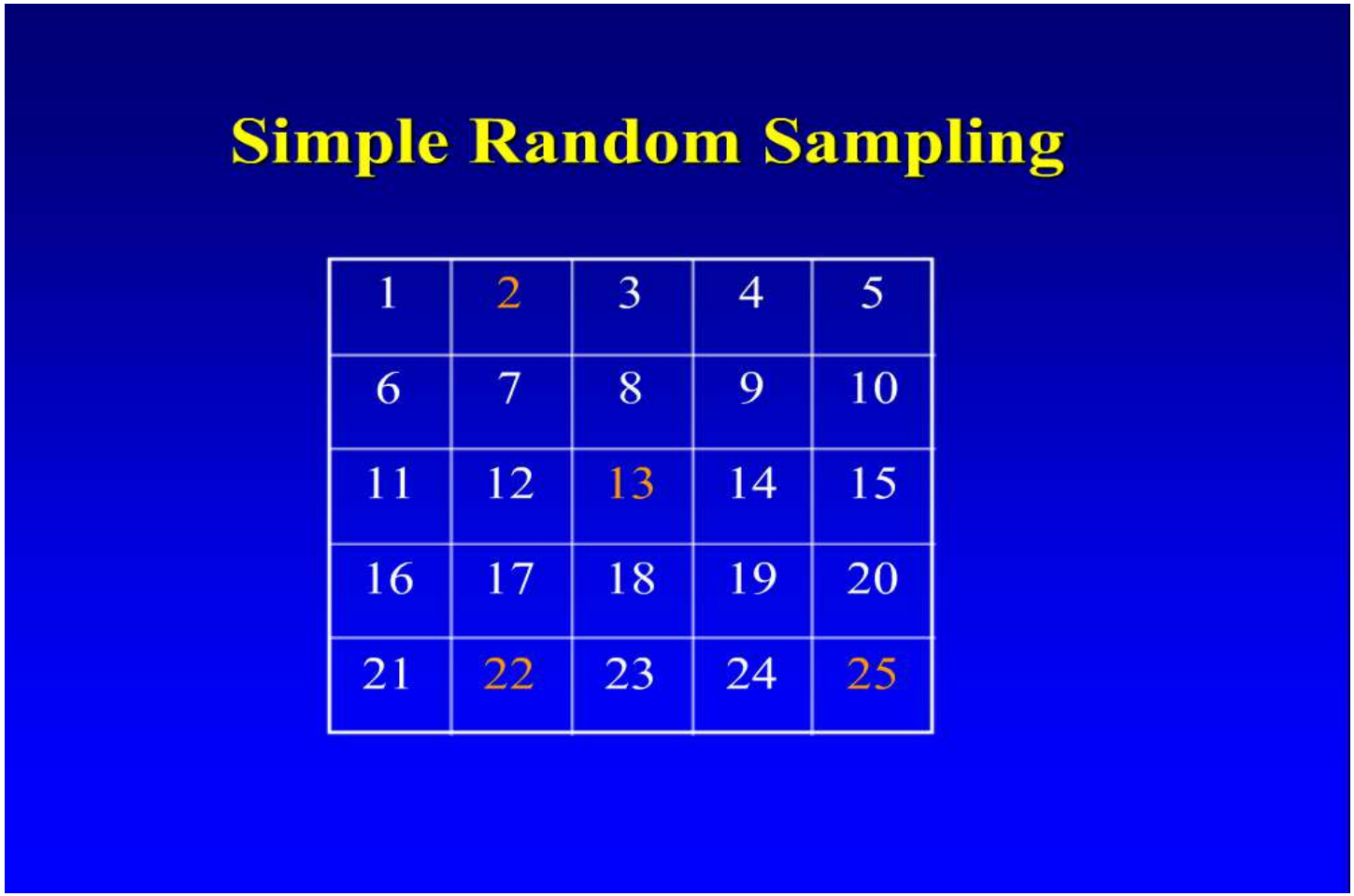
A sample is selected by a random process to eliminate problems of bias in selection and/or to provide a basis for statistical interpretation of measurement data.

There are three sampling processes which give rise to different types of random sample:

- Simple random sampling - any sample has an equal chance of selection.
- Stratified random sampling - he lot is subdivided/stratified and a simple random sample selected from each stratum.
- Systematic sampling - the first sample is selected at random and then the subsequent samples are taken according to a previously arranged interval, e.g. every 5th, 10th or whatever is appropriate

Random Sample

Sampling accuracy is usually obtained using a random sampling technique



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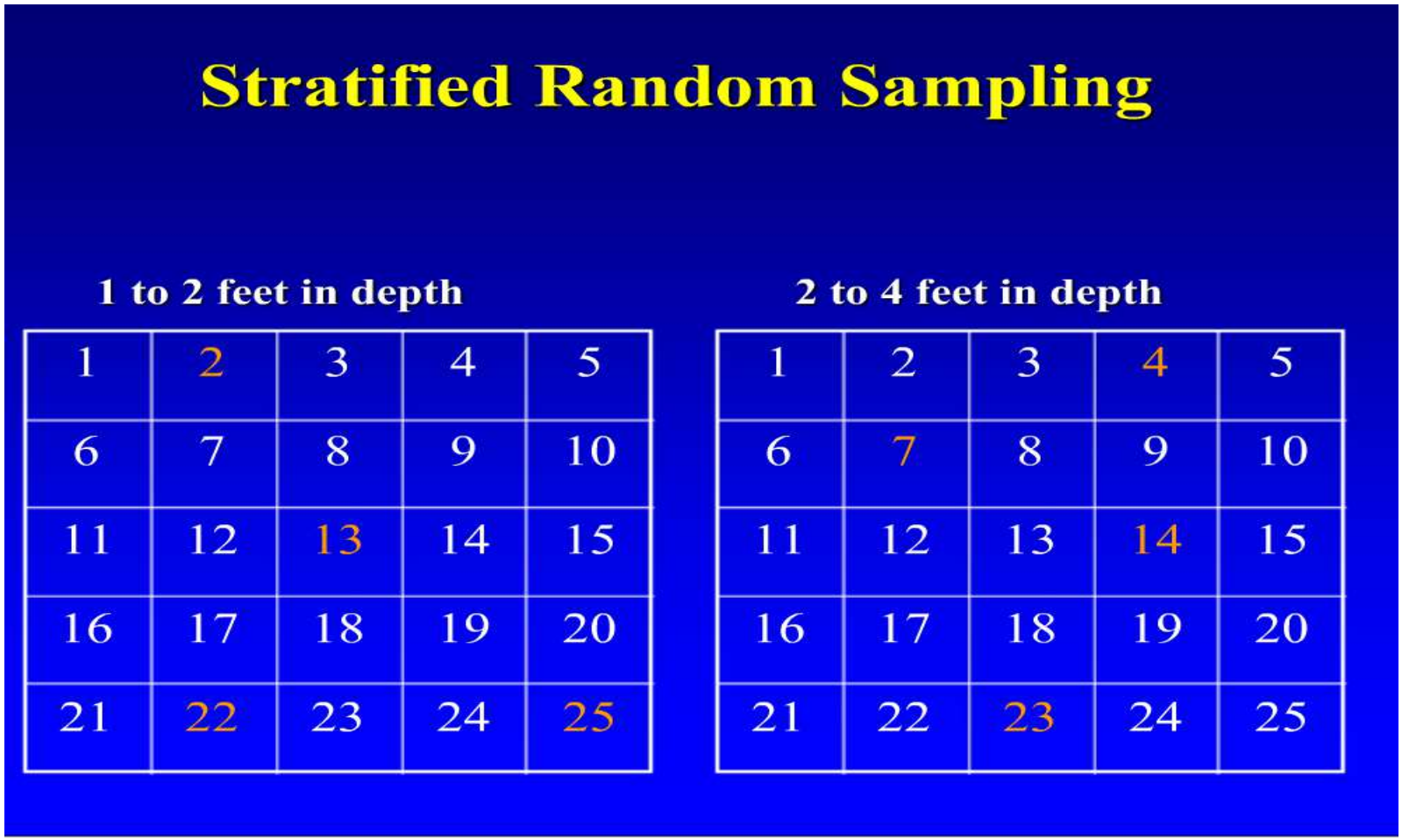

Random Sample

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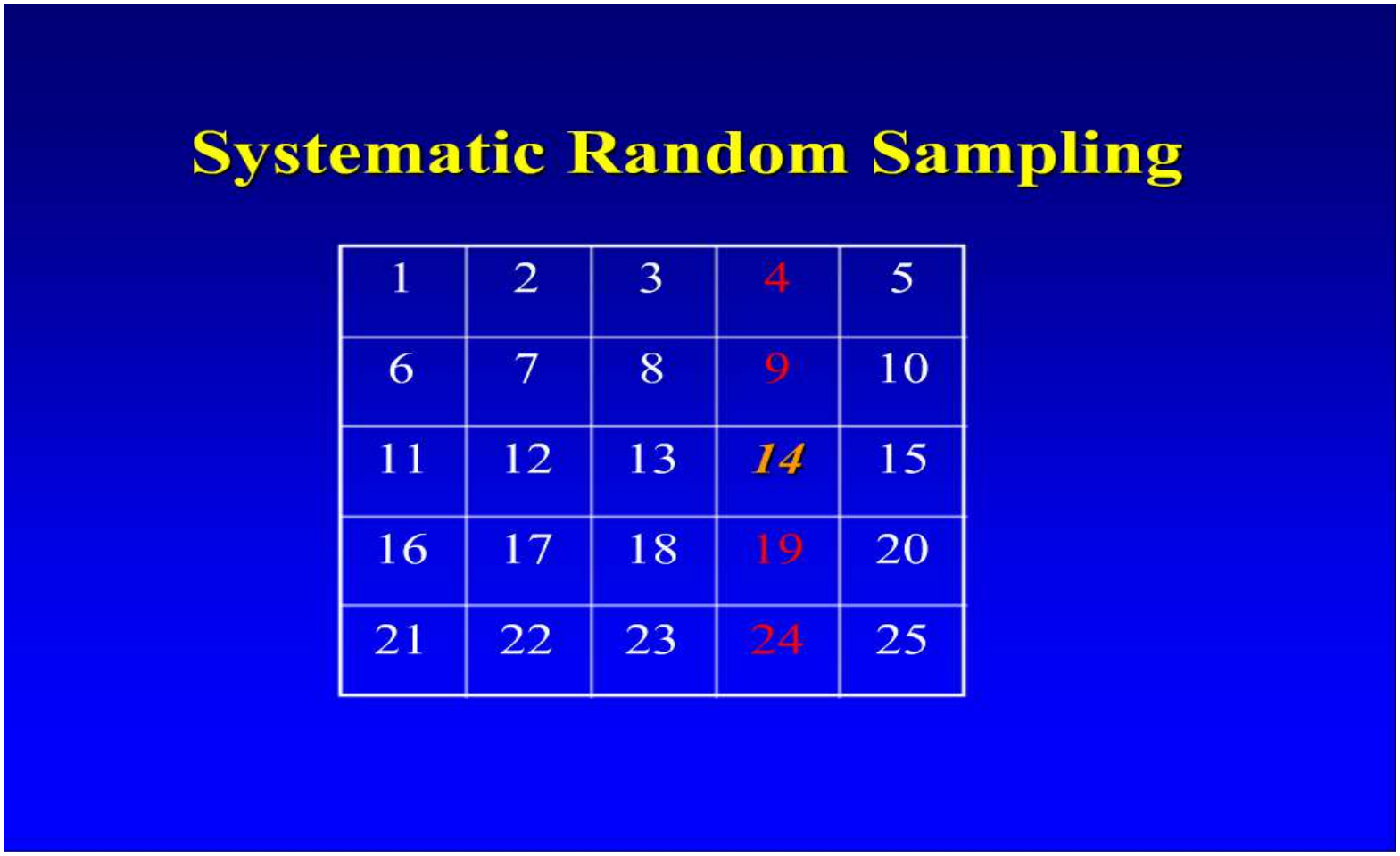

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Composite Sample

- 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 inproportion to the amount of the material that they represent

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tarafından ortaklaşa finanse edilmektedir.

Composite Sample

Water Autosampler

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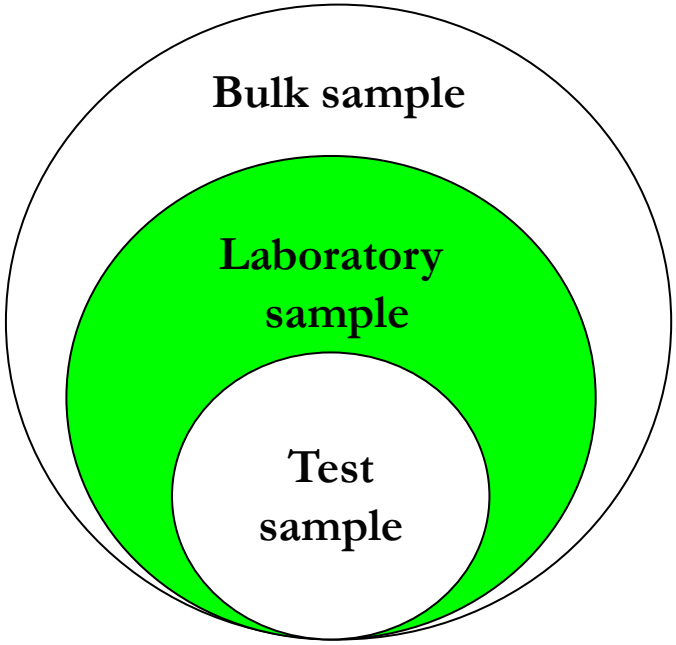
Subsampling

- 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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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

Subsampling liquid materials

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- Less of a problem than solid samples. However, this is only the case when the volume of liquid to be sampled is small enough that it can be homogenized by shaking, and the liquid consists of only one phase.
- The presence of suspended material can affect the determination of the concentration of the analyte.
- The suspended material may adsorb the analyte and so it is important to check whether filtration, if used, has a significant effect on the analytical result
- In some cases, the analyte may be in suspension rather than in solution in the test sample.
- Liquids may settle in layers on standing
- It is important that there is sufficient 'headspace' in the container for adequate shaking (not in case of sampling volatile compounds!!!)
- When material is prone to rapid sedimentation, the samples need to be taken during the mixing process as the material will immediately start to separate once the mixing is stopped

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tarafından ortaklaşa finanse edilmektedir.

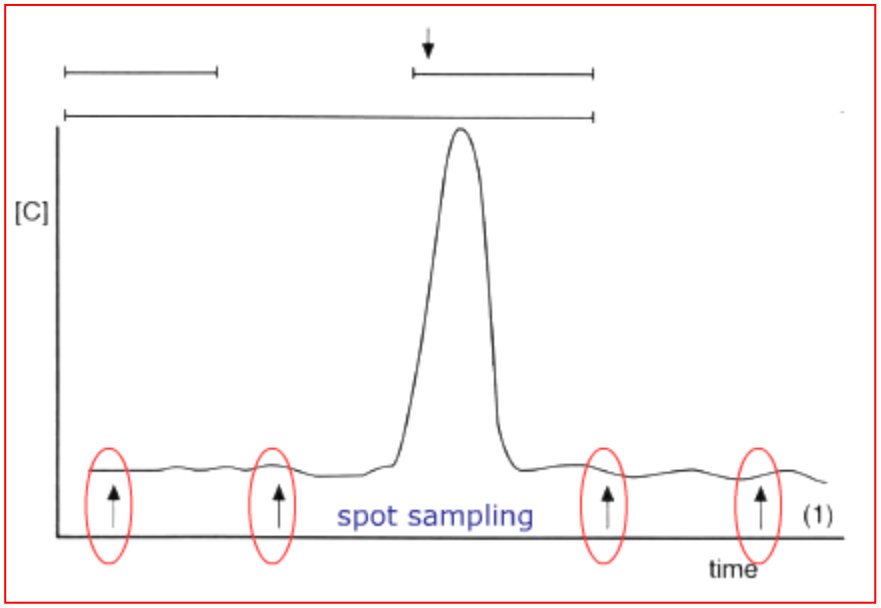
Conventional Water Sampling

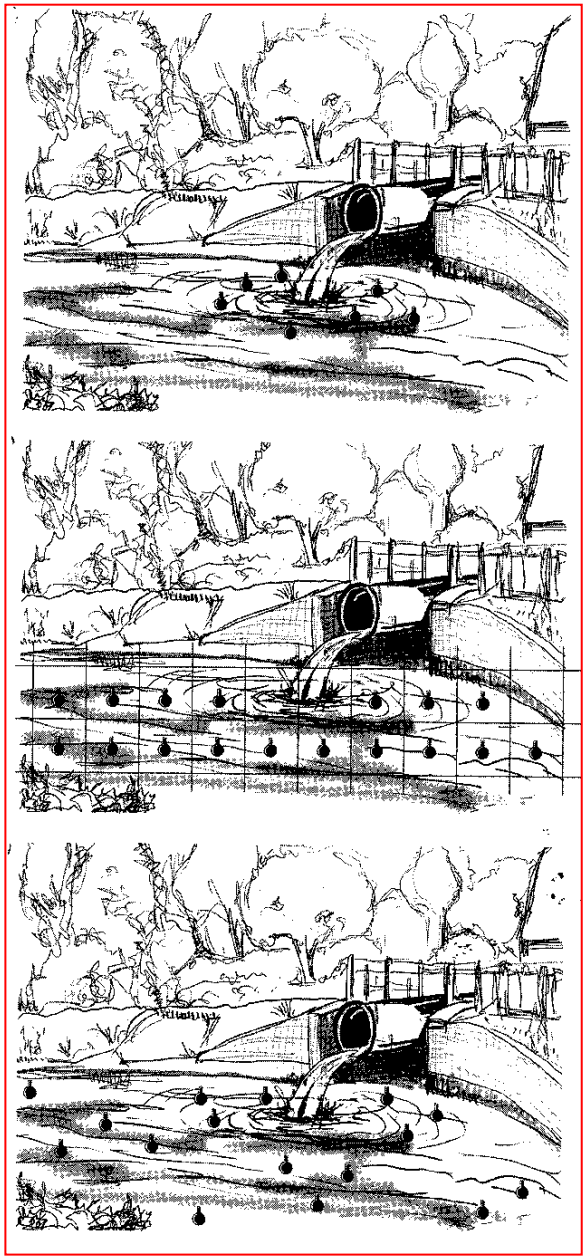
- Currently based low volume spot sampling
- A lot of experience
- Analysis of samples by accredited methods
- Legislation
- Environmental Quality Standards (EQS)
- Compliance monitoring

One-off point sampling

Sampling:

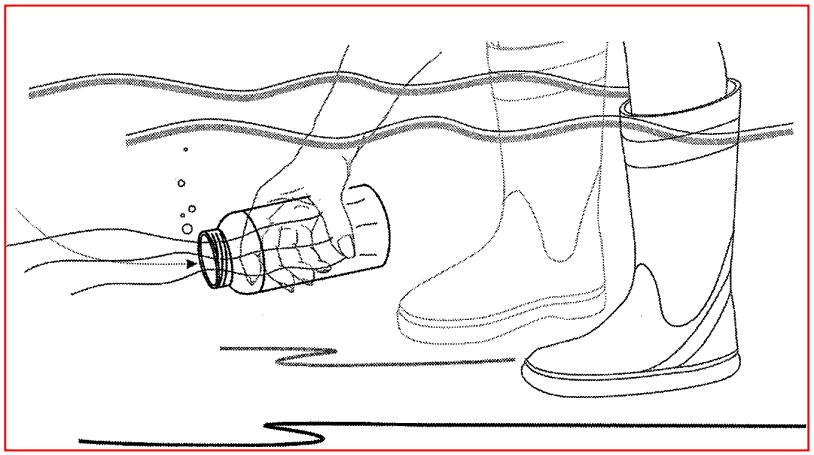
- **In one site**
- **At time**

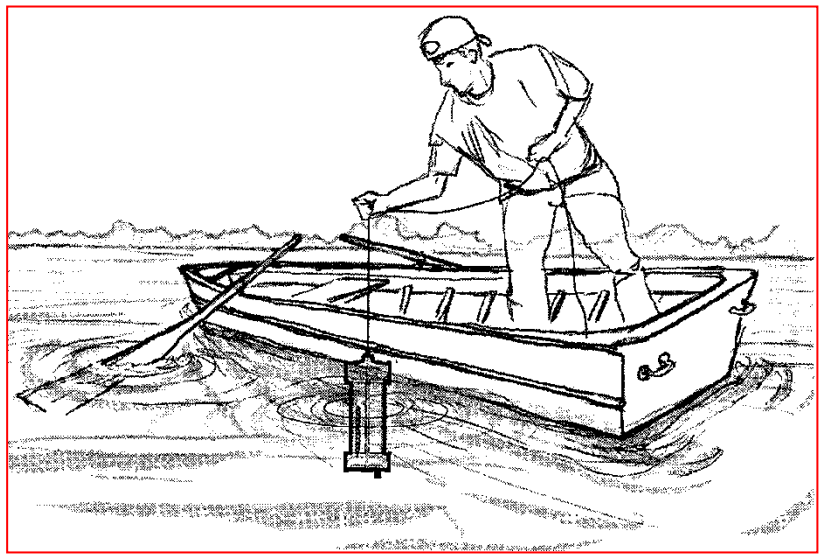


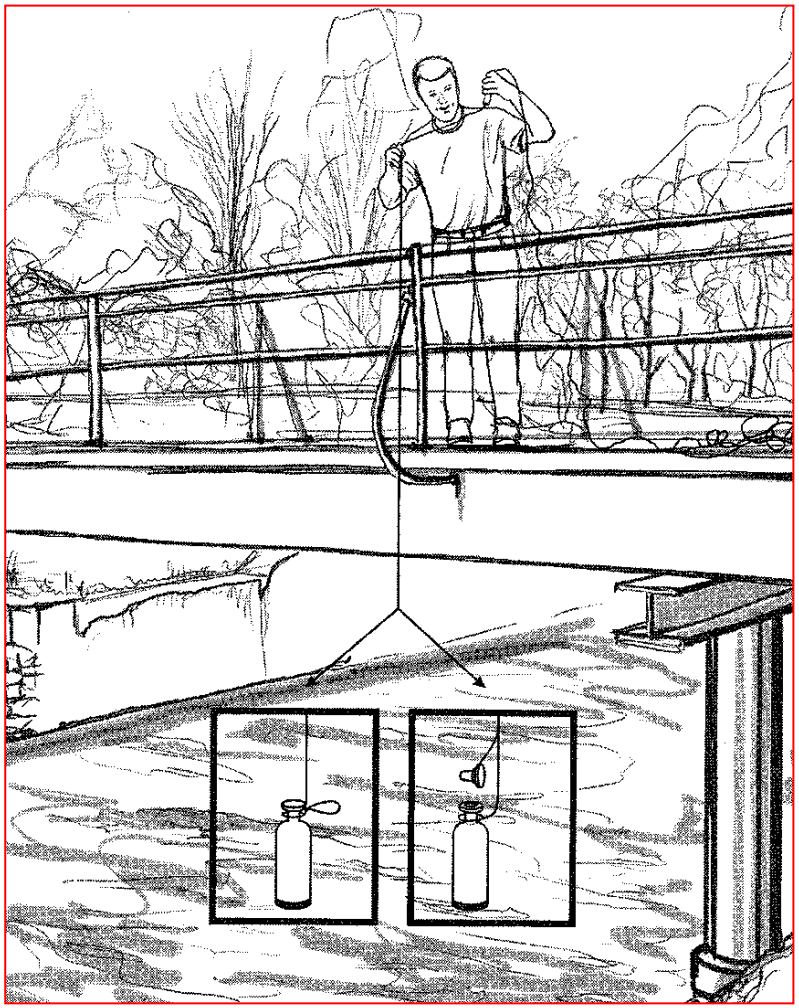


One-off point sampling

Types of sampling







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Conventional Water Sampling

- **Advantages of spot sampling**
  - Laboratory analysis accredited
  - Historical data can be used as an environmental archive
- **Disadvantages**
  - Snapshot only (reflect residue composition only at the moment of sampling and may fail to detect episodic contamination)
    - Fluctuations in time
    - Variation between regions (different environmental pressures)
- **Limitations**
  - Quality control and physical difficulties - large volumes of water necessary for quantifying and assessing trace organic contaminants
  - Concentrations of truly dissolved contaminants are not accurately measured by most conventional approaches
  - Standard low volume (< 5 l) techniques often fail to detect trace levels of contaminants

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Conventional Water Sampling

- Precise picture at moment and site of sampling – may not be representative (fluctuations may be fir instance seasonal not randomly distributed in time), and may not detect trends
- Error could be large
- Large risk if used as basis of risk assessment
- Wrong decisions could be costly
- Repeated sampling expensive (transport and labour not just extra analysis)
- Roose, P. and Brinkman, U. A. Th. (2005) TRAC

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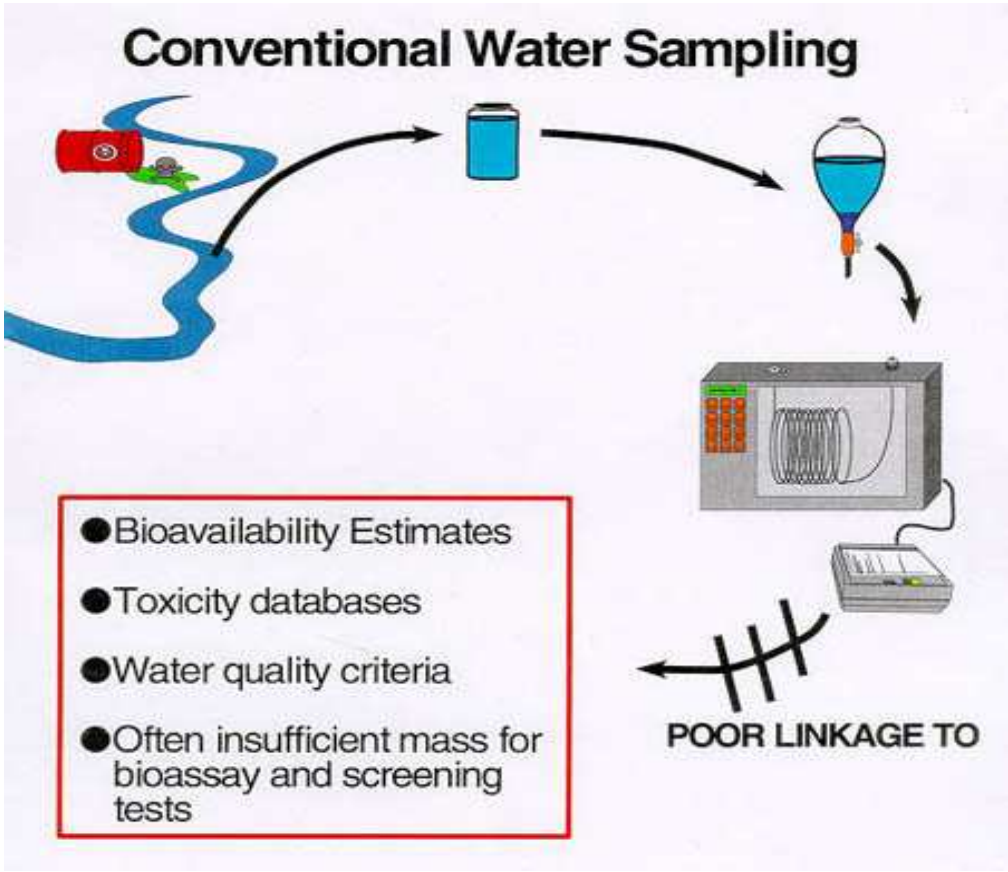
Limitations of Conventional Water Sampling



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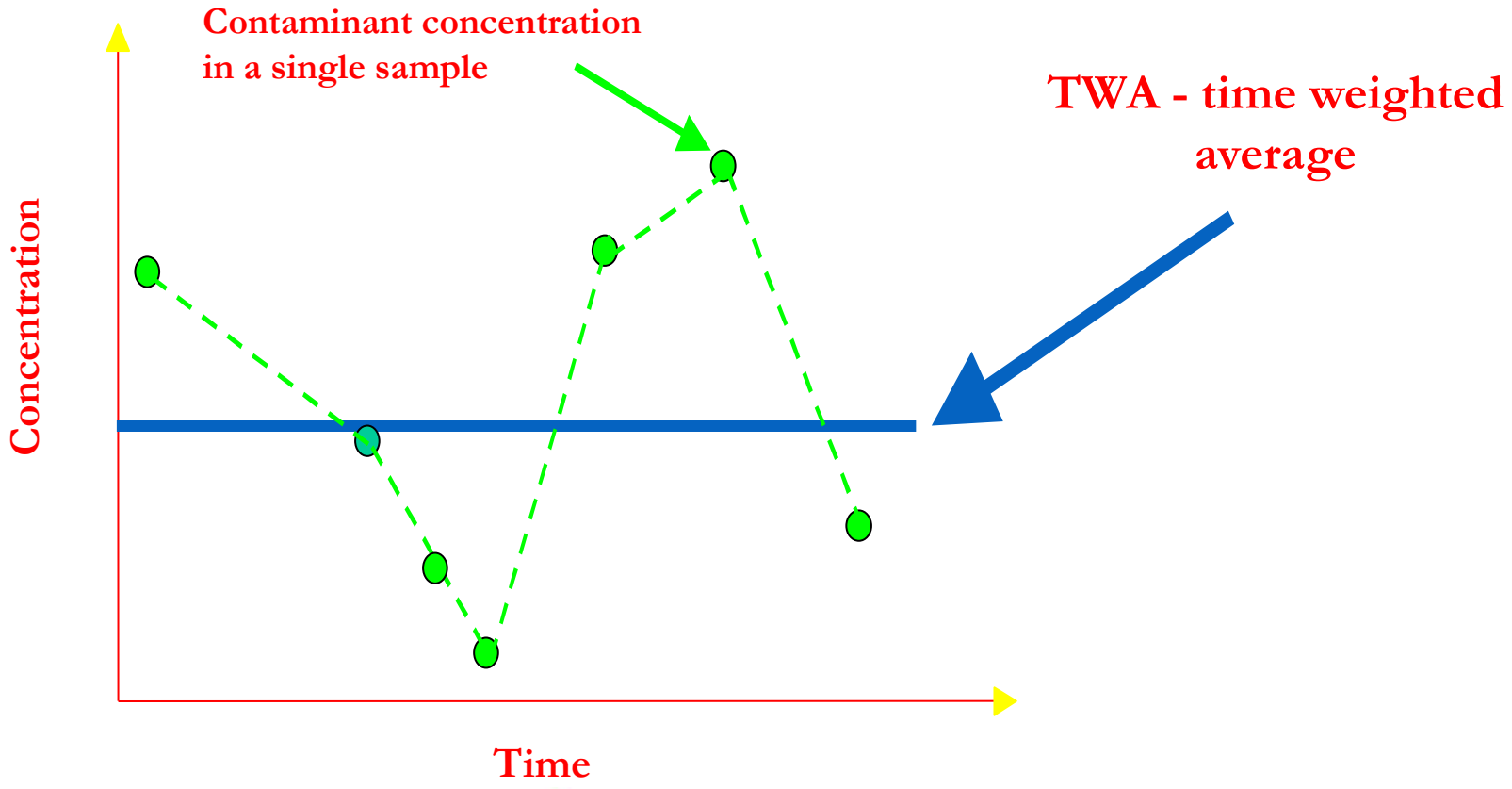
tarafından ortaklaşa finanse edilmektedir.

Necessity for Time Integrative Sampling



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Bucket on a rope

- Simple
- Sampling from bridges, piers
- Use a bucket made of an inert material
- Does not allow sampling in deep water
- No exact depth
- Contamination chance

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- Only where direct access to water
- Only to water depth cca 1.5 m
- Excellent for sampling trace contaminants

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Bottle on a pole

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Errors Associated with Bottle Sampling

- Changes occur
  - as soon as sample taken and there can be further changes during transport and storage (e.g. by binding to the bottle walls)
  - during preparation (e.g. by filtration) for analysis
- These cause systematic errors (bias) rather than random errors, and these errors can be large compared with those associated with the analytical stages
- Ackerman, A.H. and Hurtbusise, R.J. (2000)

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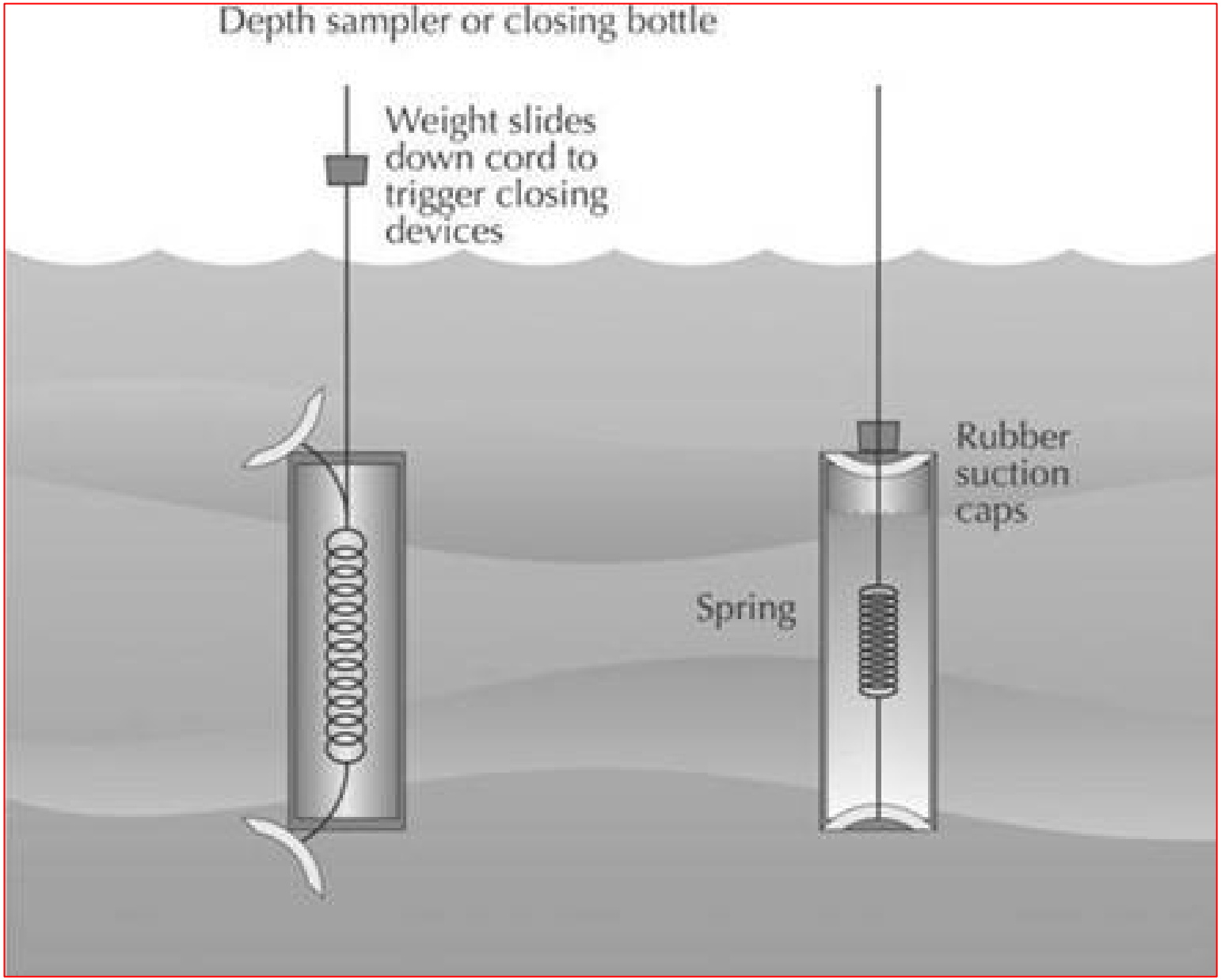
Water sampling - Van Dorn water sampler

- Acrylic sample tube, contents 2.2 litres.
- With messenger, 30 m synthetic line and carrying case (suitable for trace metal sampling).

- Main users
  - Sea and inland lake researchers
  - Harbour authorities
- Remarks:
  - Suits sampling for most micro-parameters

Water sampling



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Kemmerer water sampler

- With transparent acrylic sample tube, contents 1.2 litres. Complete with messenger, 30 m synthetic line and carrying case.
- Main users
  - Sea and inland lake researchers
  - Harbour authorities
- Remarks:
  - Does NOT suit sampling for certain micro-parameters

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Rosette sampler

- Samples collected at different depths
- Niskin bottles fill with water and close at desired depth
- Stainless steel vessels
- Application in oceanography

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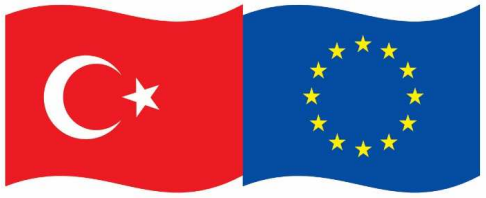

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More Representative Methods for Monitoring

More representative picture of water quality can be obtained using a number of approaches:

- **Frequent sampling**
- **Automatic sequential sampling to provide composite samples over a period of time (usually 24 hours)**
- **Continuous, on-line monitoring systems (e.g. the SAMOS system, some sensors, BEWS)**
- **Biomonitoring**
- **Passive samplers**

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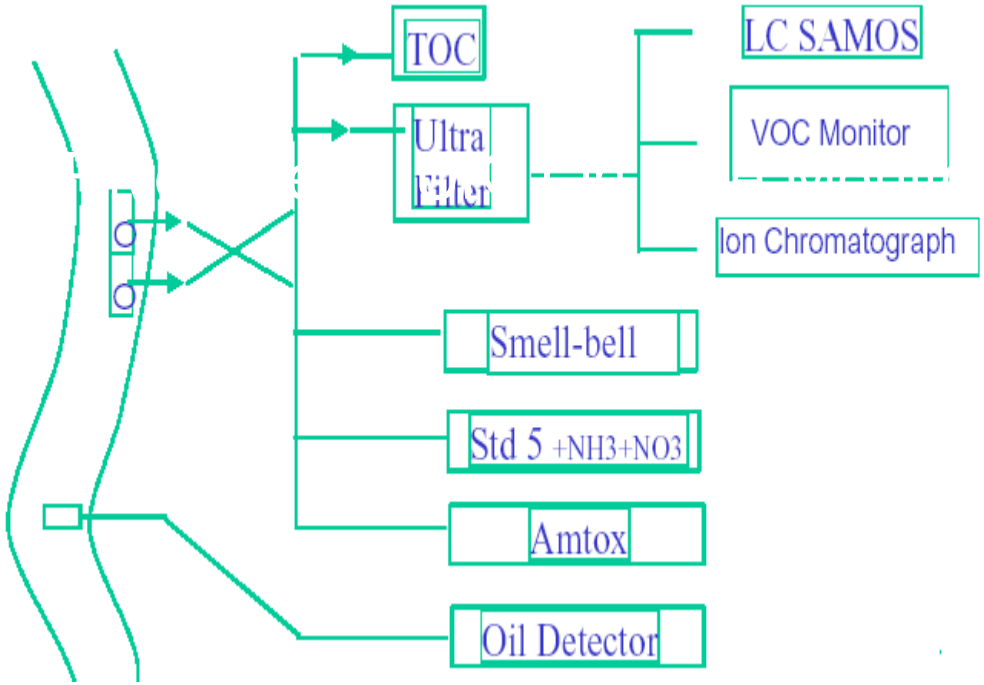
On-line, Automated Analytical Systems

- Used in investigative monitoring, which might also include alarm or early warning monitoring
- Very suitable for protected areas (e.g. drinking water intake sites) for the protection against accidental pollution
- At some sites with a power supply, and a high level of security, continuous monitoring systems (e.g. SAMOS) can be deployed
- Unsuitable for wide deployment over a catchment area, expensive and requires maintenance

On-line, Automated Analytical Systems



On-site laboratory

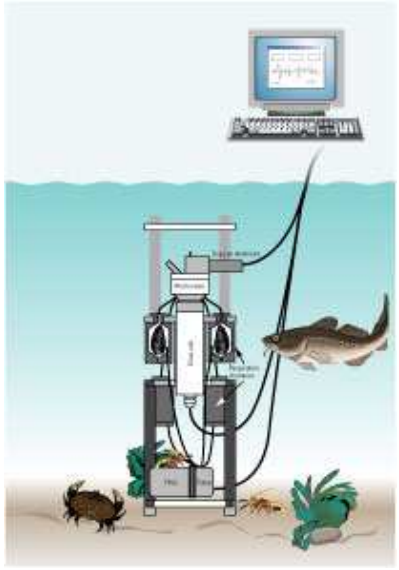
Herbicide measurements

Automatically generates alarm

Biological Early warning Systems (BEWS)

Biological early warning system: on-line (*in-situ*) whole-organism bioassays using fish (freshwater), *Daphnia*, freshwater and marine mussels, algae or combinations - e.g. Musselmonitor®, Truitel™, ToxAlarm…

Response to toxicants: Swimming behaviour, valve movement response, O_2 consumption/production, Chl *a* fluorescence, ventilatory activity…Response may trigger an alarm, sampler…

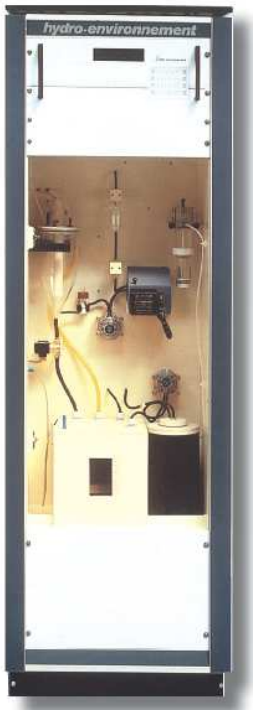


Online systems

- Online systems = online monitoring of water quality (WWTP, water companies, industry, water authorities)
- Total concentration of hydrocarbons (extraction with tetrachloroethylene, spectorfotometric determination)

→ TOC (selektive electrode, UV, peroxosulphate)

→ Analyser of ammonium ions, nitrates, phosphates (selective electroedes)



Online systems

Online systems

→No need to filtrate water

→Low maintenance costs – automatic cleaning

→UV spectrometry, no need of chemicals í

→detection: nitrates, ammonium, chromium Cr VI- COD, phosphates, PAH, chlorophyll, rhodamine, pH, conductivity, turbidity, dissolved O_2, nondissolved substances

Automatic samplers

Portable automatic samplers

→Thermostat

→Battery operated or 220 V

→Automatic washing procedure

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Detectors

→ Detection of oil spills (petrol 4 s., crude oil 2 min., petroleum 26 s., oil 2 min.)

→ alarm of oil leaks (optical sensor down to 5 m below water level

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New (Bio)sensor Technologies for Potential Use in Early Warning Systems

- Screen-printed, disposable biosensors – enzymatic systems to detect and identify specific pollutants
- Lateral-flow devices with electrochemical detectors – detection and measurement of specific microbial pathogens
- Automated optical immunosensors with multi-analyte determination of selected compounds, depending on monitoring situation (Automated Water Analyser Computer Supported System (AWACSS); RIANA; FIAA)
- Miniaturised optical (bio)sensors employing microspectrophotometers – specific pollutants
- Image and pattern recognition – cellular pathogens
- Protein microarray technology – complex mixtures of pollutants or toxins
- Polymerase chain reaction (PCR) technology – identification of organisms by their DNA

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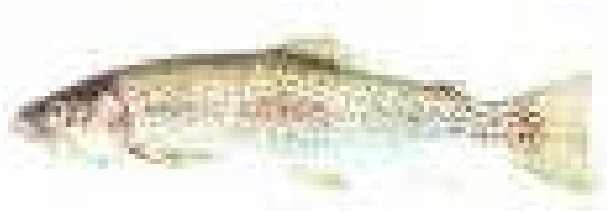
tarafından ortaklaşa finanse edilmektedir.

Bioaccumulation Monitoring

- Sentinel organisms – accumulation of pollutants from the surrounding environment or from food
- In situ-expose sessile or caged organisms, or wild organisms
- Exposure assessment by measuring contaminant levels in biota
- Difficulties: biotransformation
- Identity and nature of sampled pollutant fraction



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Water Sample Types

- Spot samples
- Integrated samples
  - Composite samples
  - Passive samples
- Proportional samples

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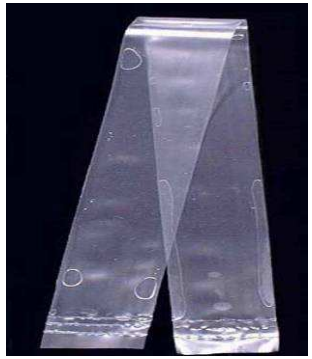
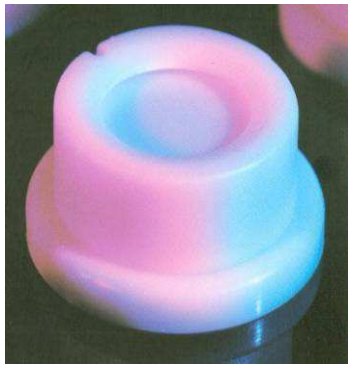
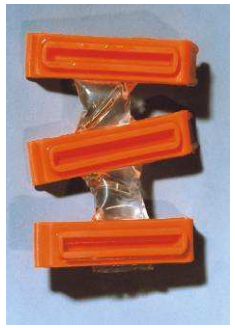
Contents

Passive Samplers




- Biomimetic tools – assessment of bioaccumulation
- Short term (less than a day) and long term (weeks) monitoring
- Time-weighted-average (TWA) concentrations of organic pollutants and metals
- Sensors
- Immunochemical methods
- Bioassays

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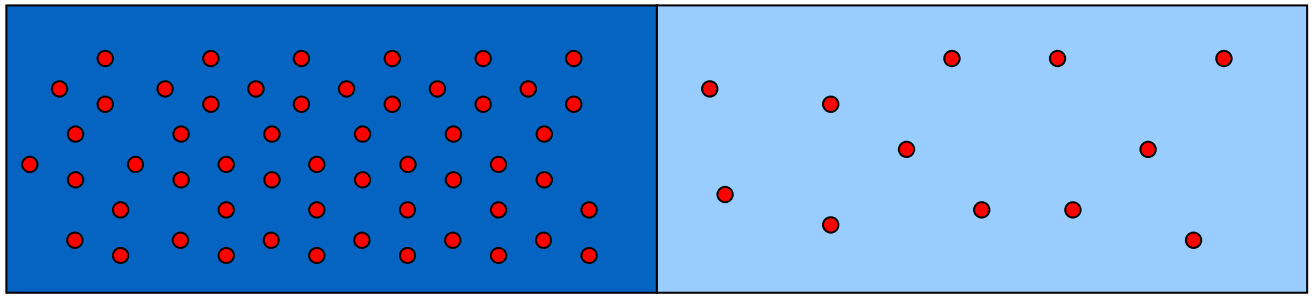
What is partitioning

- Distribution of a compound over two phases following a compound specific ratio – partition coefficient – in thermodynamic equilibrium.

- This ratio is based on the difference in solubility of the compound in those two phases



Phase left high solubility, right low solubility

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Partition coefficients

K_{ow} - octanol-water partition coefficient

$$K_{OW} = \frac{C_{OW}}{C_W} \quad \text{unit} \quad \frac{L}{L}$$

K_{pw} - polymer-water or sampler-water partition coefficient

$$K_{PW} = \frac{C_P}{C_W} \quad \text{unit} \quad \frac{L}{kg}$$

K_{SED} - sediment-water partition coefficient

$$K_{SED} = \frac{C_{SED}}{C_W} \quad \text{unit} \quad \frac{L}{kg}$$

K_{oc} - sediment organic carbon-water partition coefficient

$$K_{OC} = \frac{C_{OC}}{C_W} \quad \text{unit} \quad \frac{L}{kg}$$

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Few other related to biota

BCF - bioconcentration factor

thermodynamic equilibrium

$$BCF = \frac{C_{ORGANISM}}{C_W} \quad \text{unit} \quad \frac{L}{kg}$$

C_W supposed to be freely dissolved

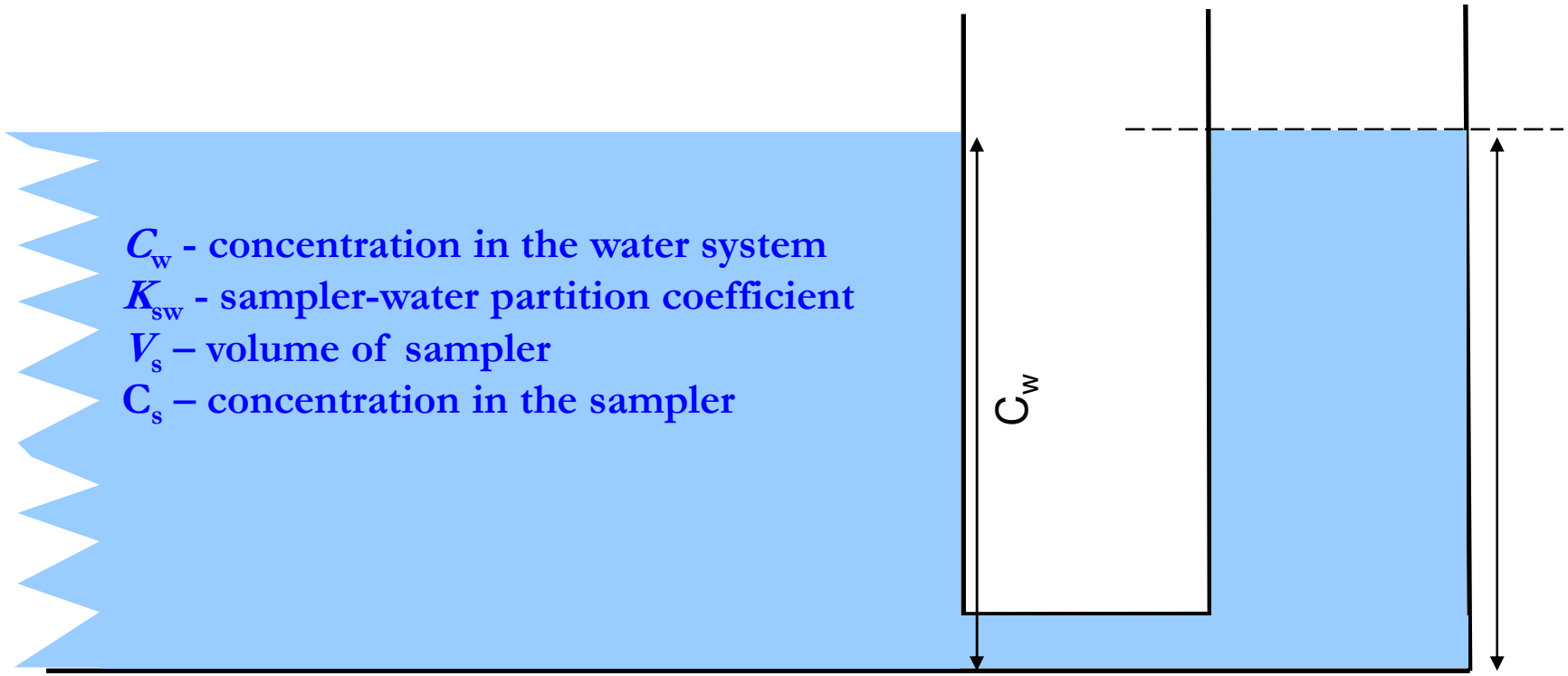
Be aware that $C_{ORGANISM}$ can be on

- **wet-weight basis**
- **dry-weight basis**
- **or lipid basis**

BAF – bio-accumulation factor

as BCF but food route is considered and C_W is not strictly the freely dissolved

Principle of a hydrophobic passive sampler - sampler as a communicating vessel



V_w = infinite

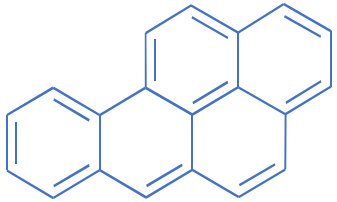
Sampling rate - R_s $V_s K_{sw}$

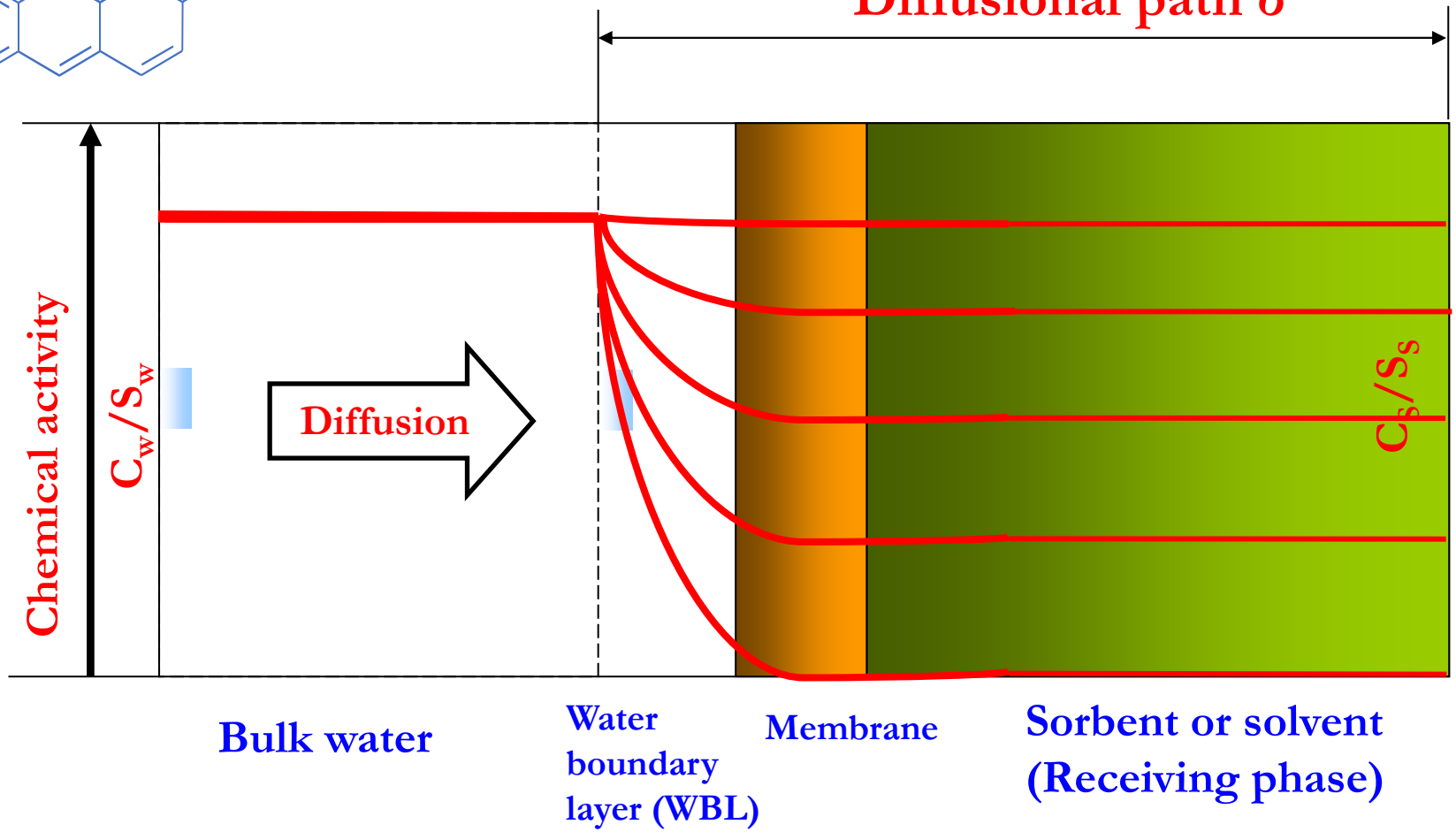
equivalent volume of water extracted per unit of time [L/d]

sampler capacity - $V_s K_{sw}$

maximum volume of water extracted [L]

Principle of a passive sampler - uptake process from water

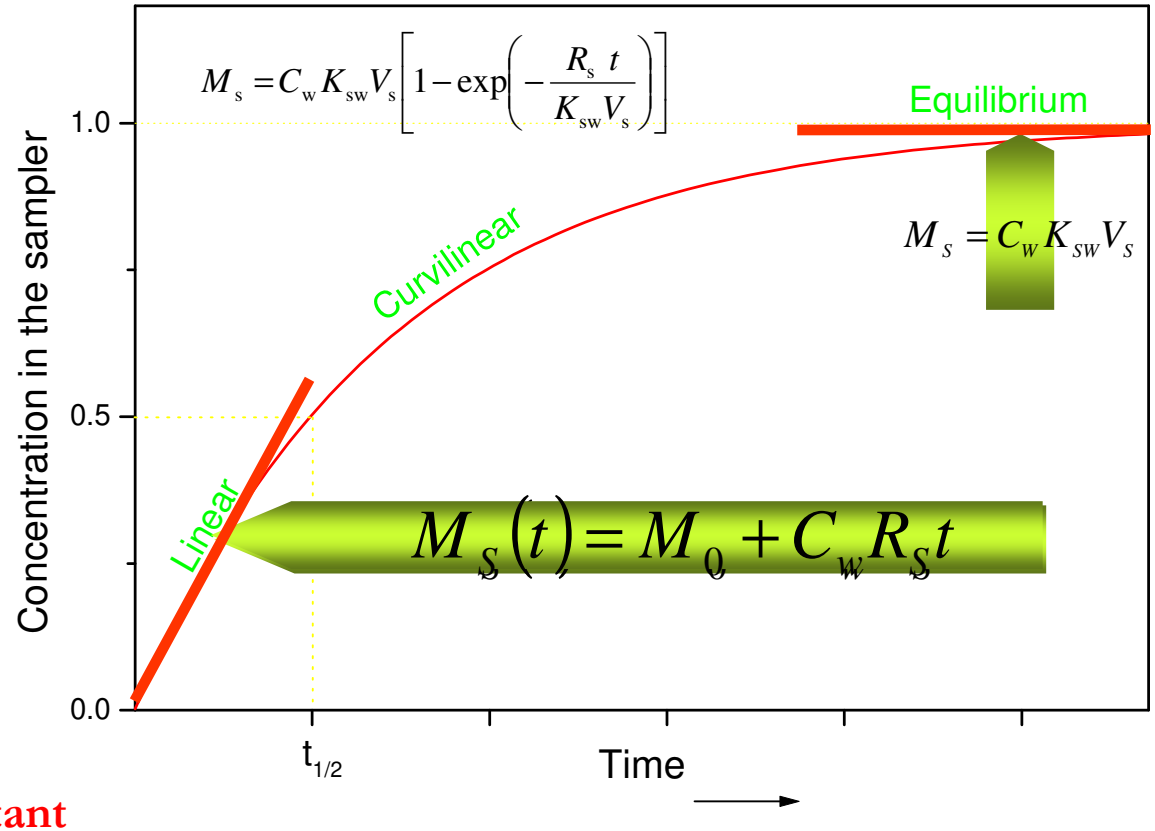




Mass transfer coefficients:

$$\frac{D_W}{\delta_W} \qquad \frac{D_M K_{MW}}{\delta_W} \qquad \frac{D_S K_{SW}}{\delta_S}$$

Uptake of a chemical by a passive sampler



$$\frac{dM_S}{dt} \sim C_W$$

C_W = constant

R_S = substance specific sampling rate [L d^{-1}]

Sampler selectivity

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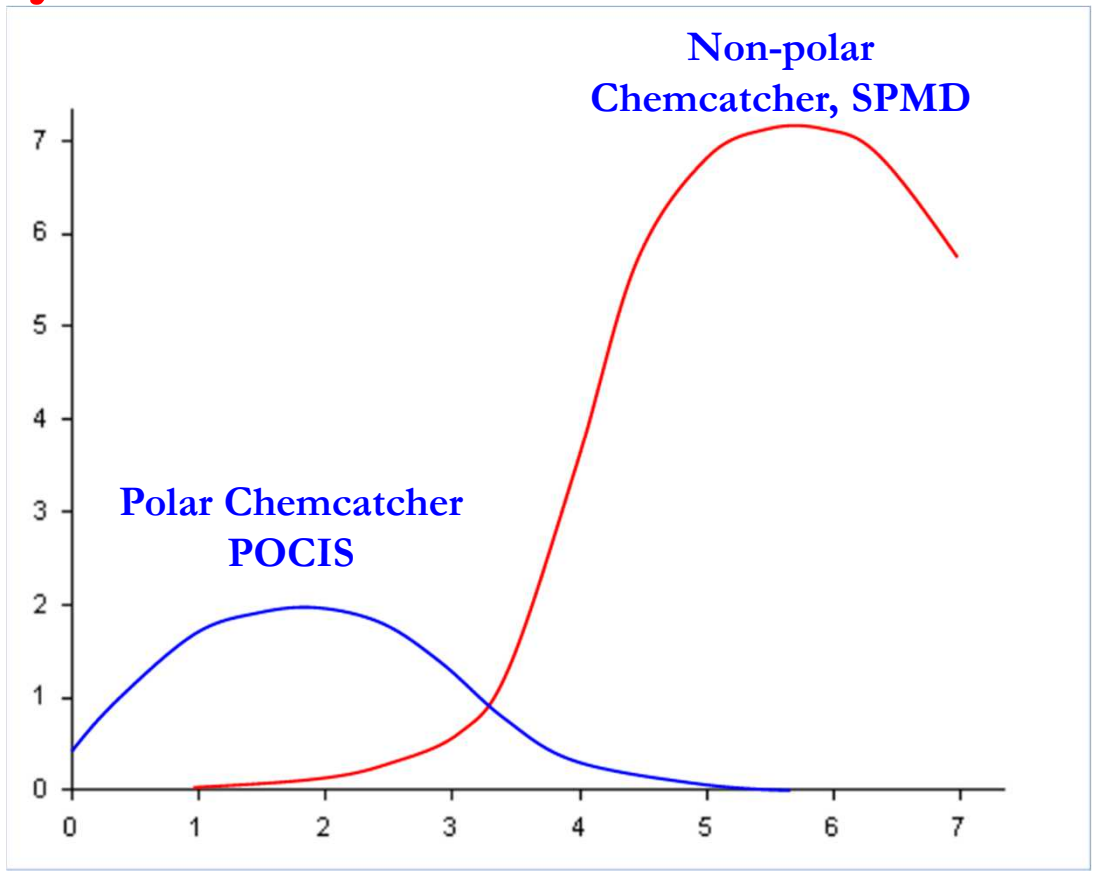
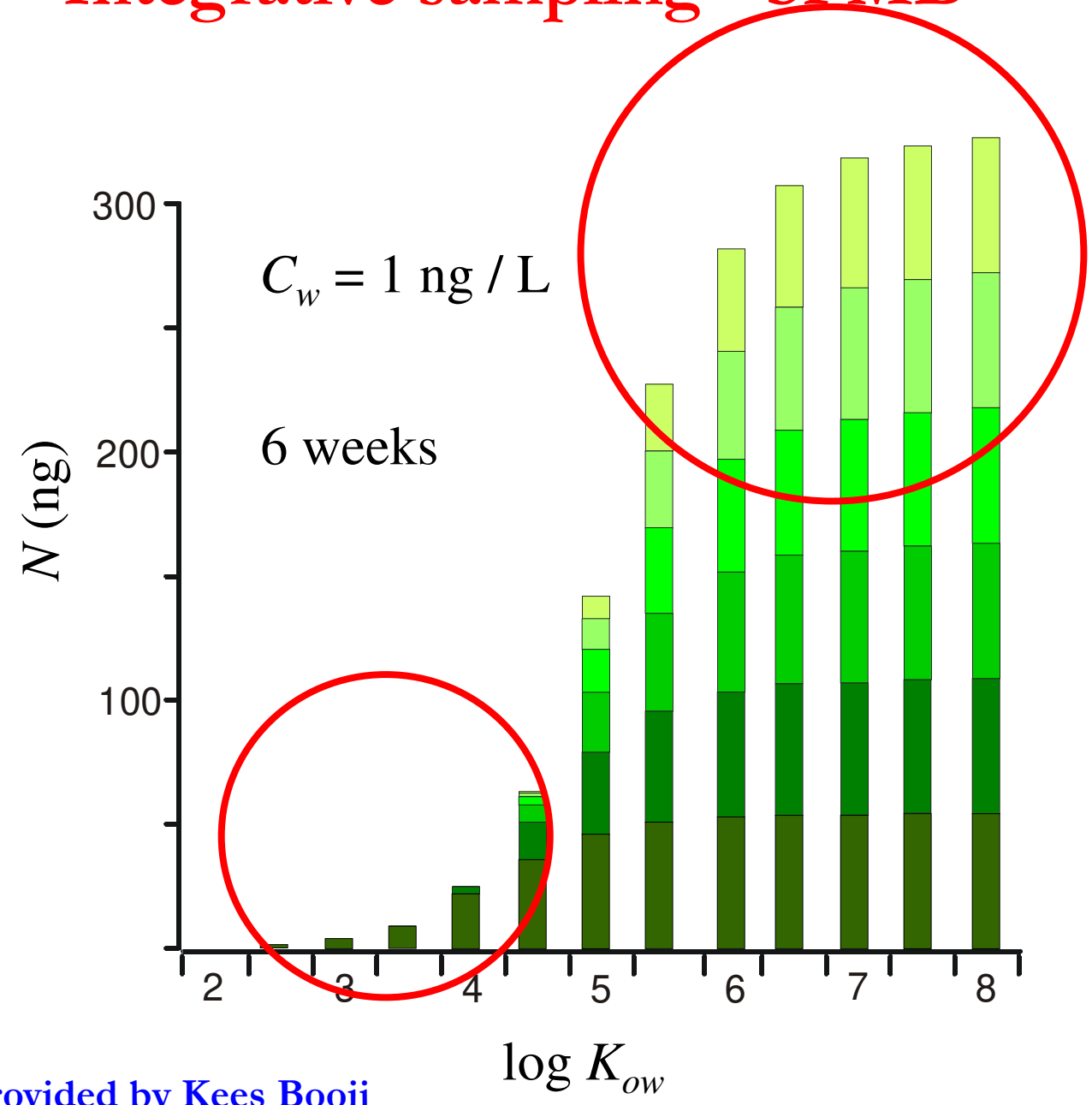
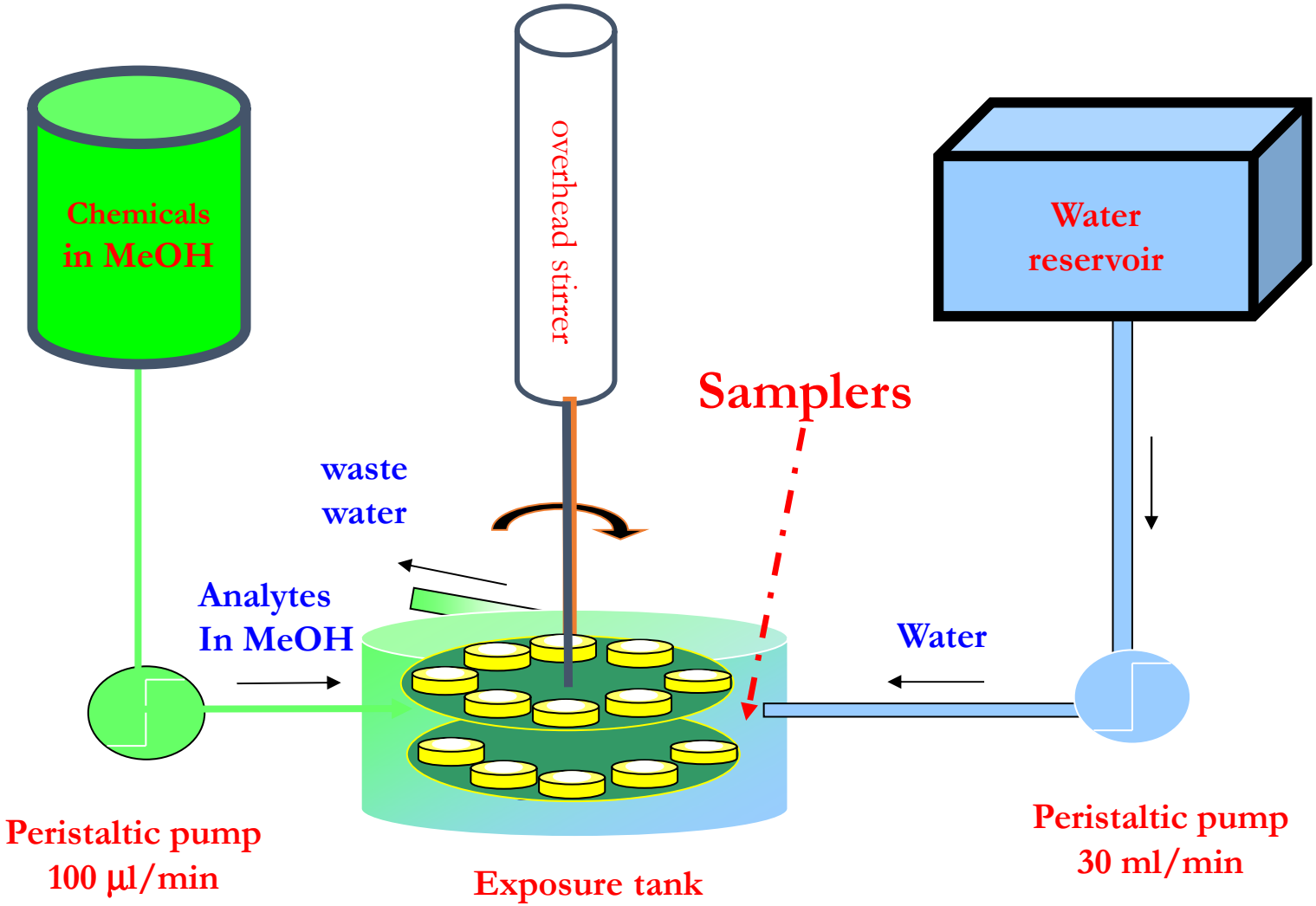


Integrative sampling - SPMD



Data provided by Kees Booij

Calibration of Chemcatcher in a flow-through system

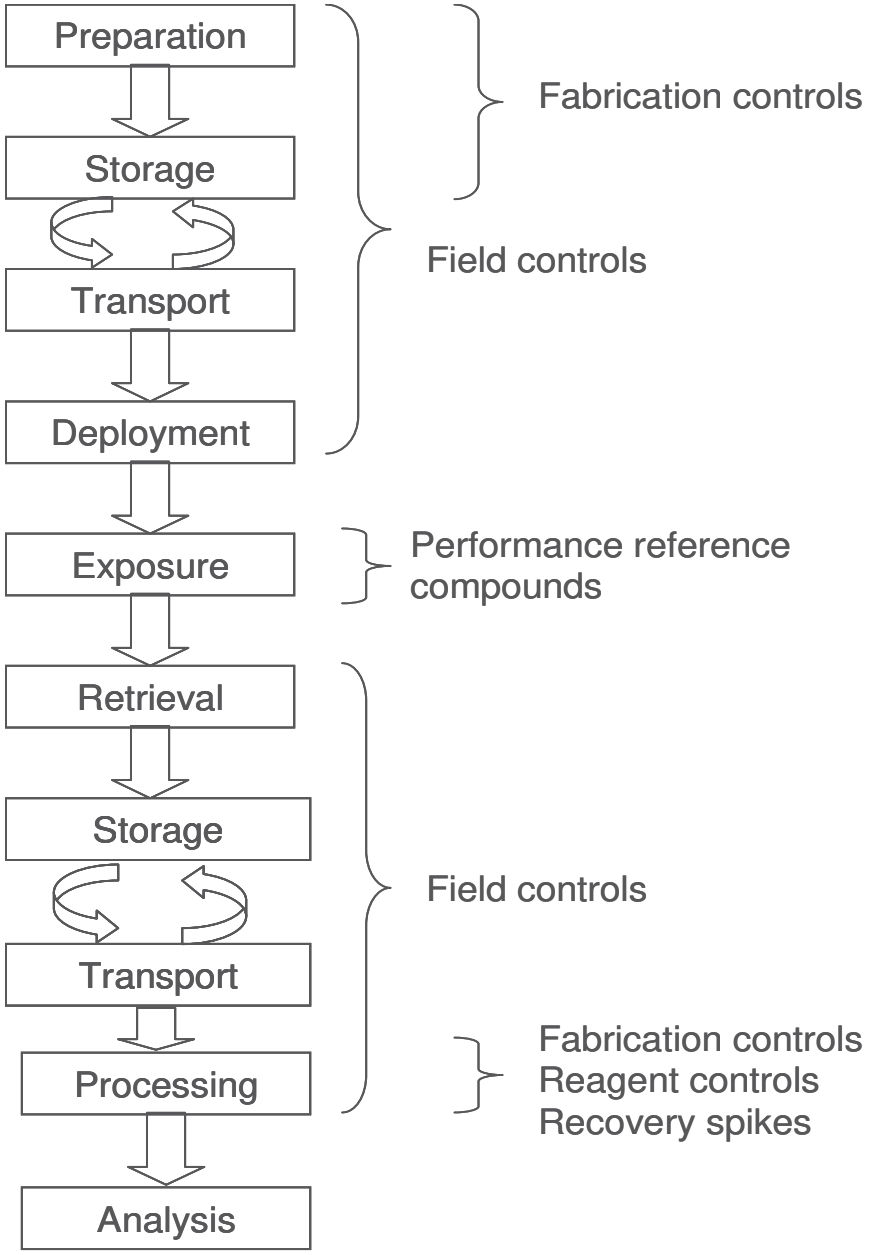


Standardisation of passive sampling

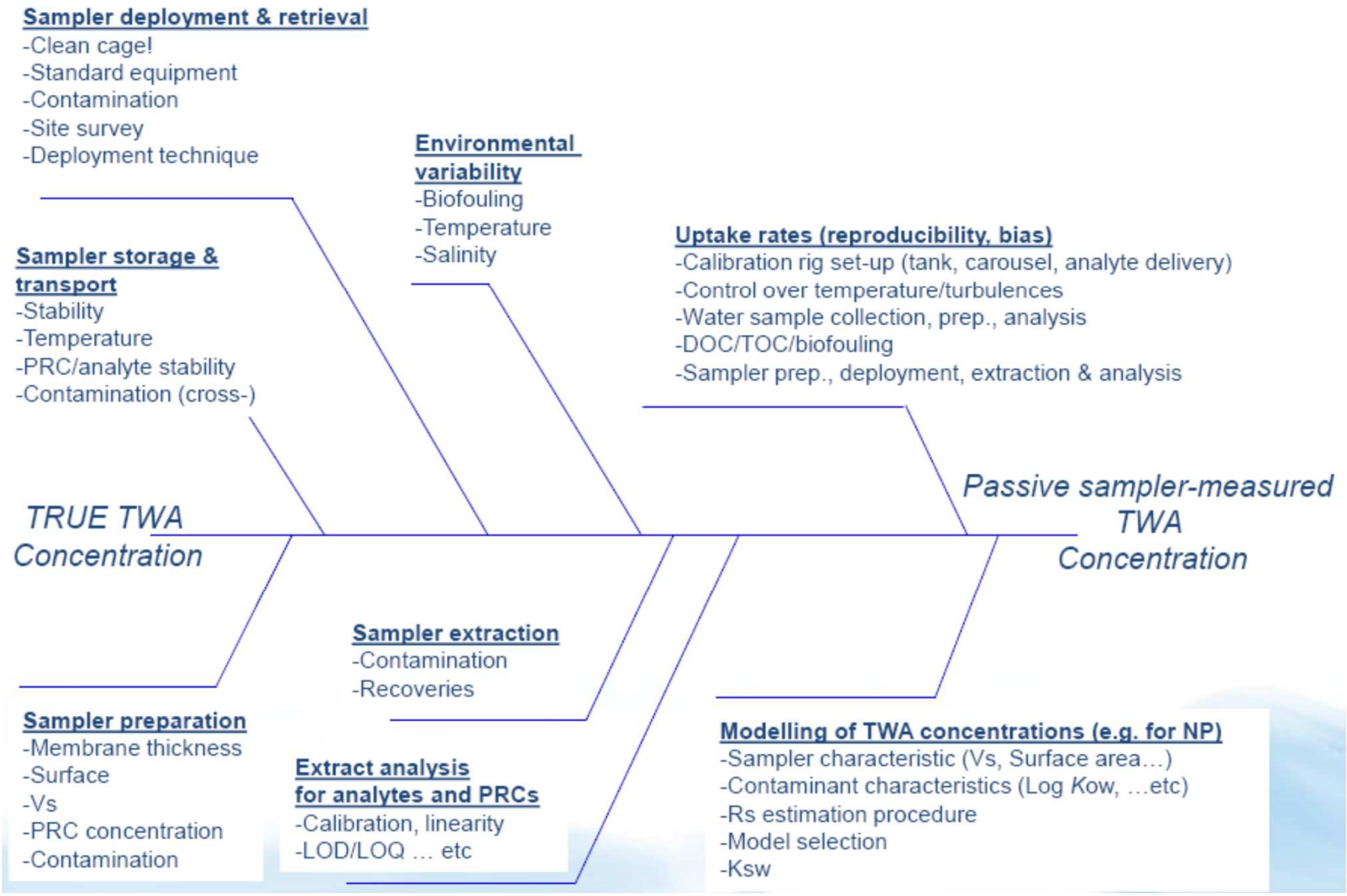
BSI PAS 61

ISO 5667-23

Water quality — Sampling — Part 23: Determination of priority pollutants in surface water using passive sampling



QA/QC in passive sampling



Current monitoring practice

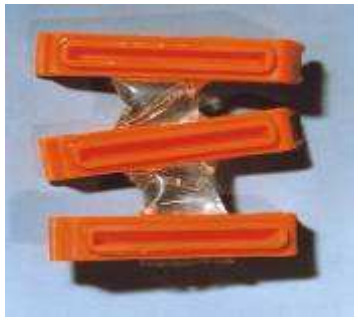
Currently the method used for measuring chemical pollutants in water is spot (*bottle/grab*) sampling and laboratory analysis

Disadvantages:

- costly (manpower/transport)
- provides only a 'snapshot' of pollution at the instant of sampling
- may not be representative where levels of pollutants fluctuate
- required sensitivity often not achieved

Alternative monitoring methods needed to overcome these problems

Passive sampling



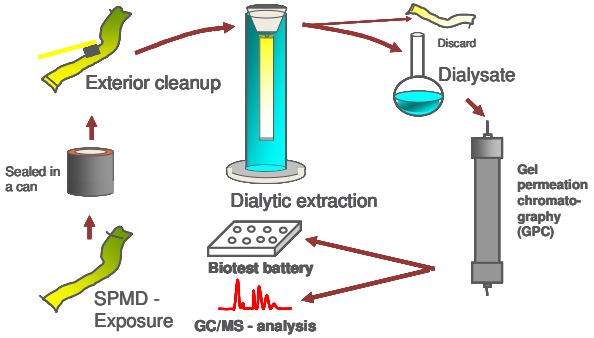
Passive sampling

Sampler selection

Sampler deployment

Exposure for days-weeks

Sampler retrieval



Extraction and analysis

Calibration Parameters

(sampling rates R_s, partition coefficients K_{sw})

Models

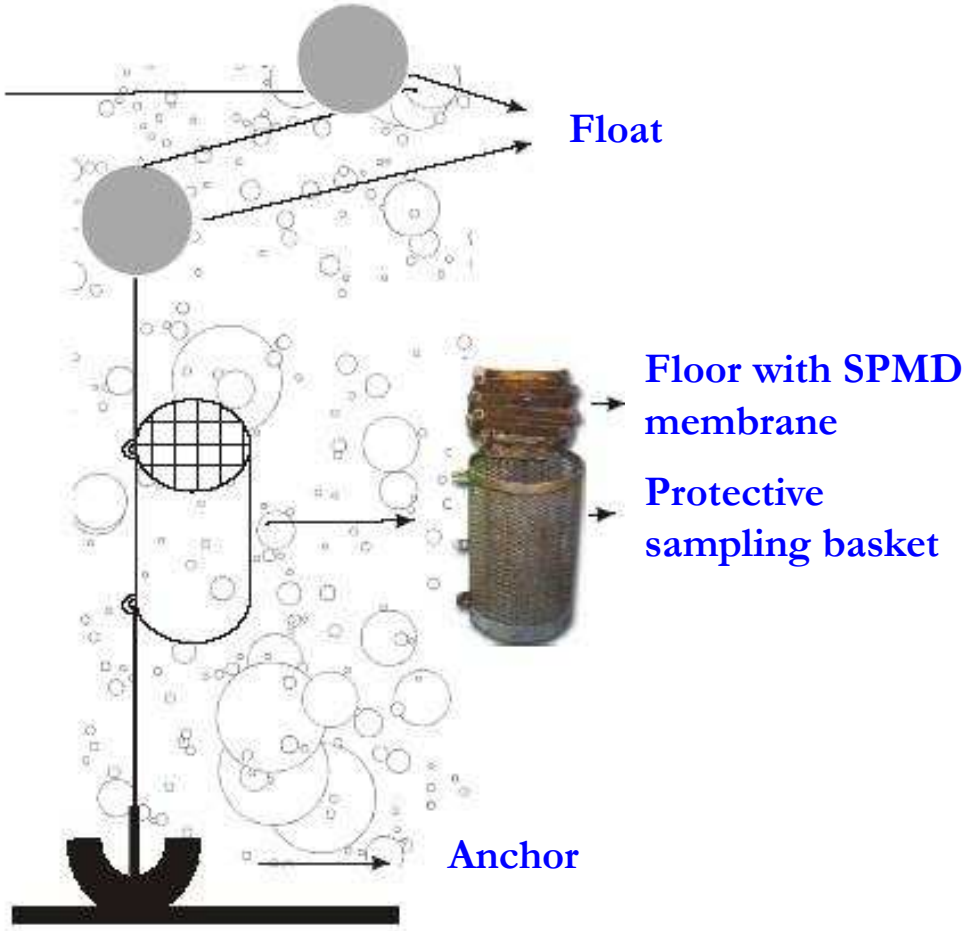
Calculation of water concentration C_w

Sampler selection: Available technology

Sampler	Construction	Compounds
SPMD 	Semi-permeable membrane devices; flat tube of LDPE filled with triolein	Hydrophobic semivolatile organic compoundswith $K_{ow} > 3$
POCIS 	Solid sorbent material enclosed in a polyethersulphone membrane	Polar pesticides and Pharmaceuticals with log $K_{ow} < 3$
MESCO 	PDMS rod enclosed in a membrane made of regenerated cellulose or LDPE	Hydrophobic semivolatile organic compounds with log $K_{ow} > 3$
Ceramic Dosimeter 	Ceramic tube filled with a solid-phase sorbent material, closed with PTFE lids	Groundwater contaminants with a broad range of physico-chemical properties
DGT 	Two layers of acrylamide gel mounted in a holder device	Metallic elements including the common heavy metals, phospho-rous, sulphide, ^{99}Tc
Chemcatcher 	A housing made of inert plastic, containing a disk of solid sorbent and a disk of diffusion membrane.	Many taylor-made versions; polar and nonpolar organics, metals, organometallic compounds
Silicone rubber 	Sheets from poly-dimethylsiloxane (PDMS)	Hydrophobic organic compounds, organometallic compounds….

…and many more…

Passive sampling - location



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Sampler deployment

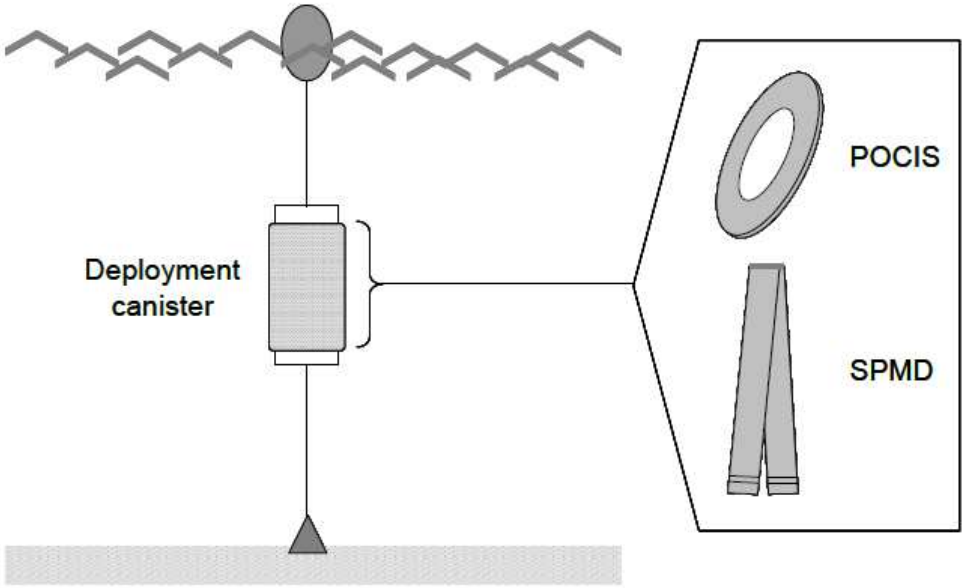
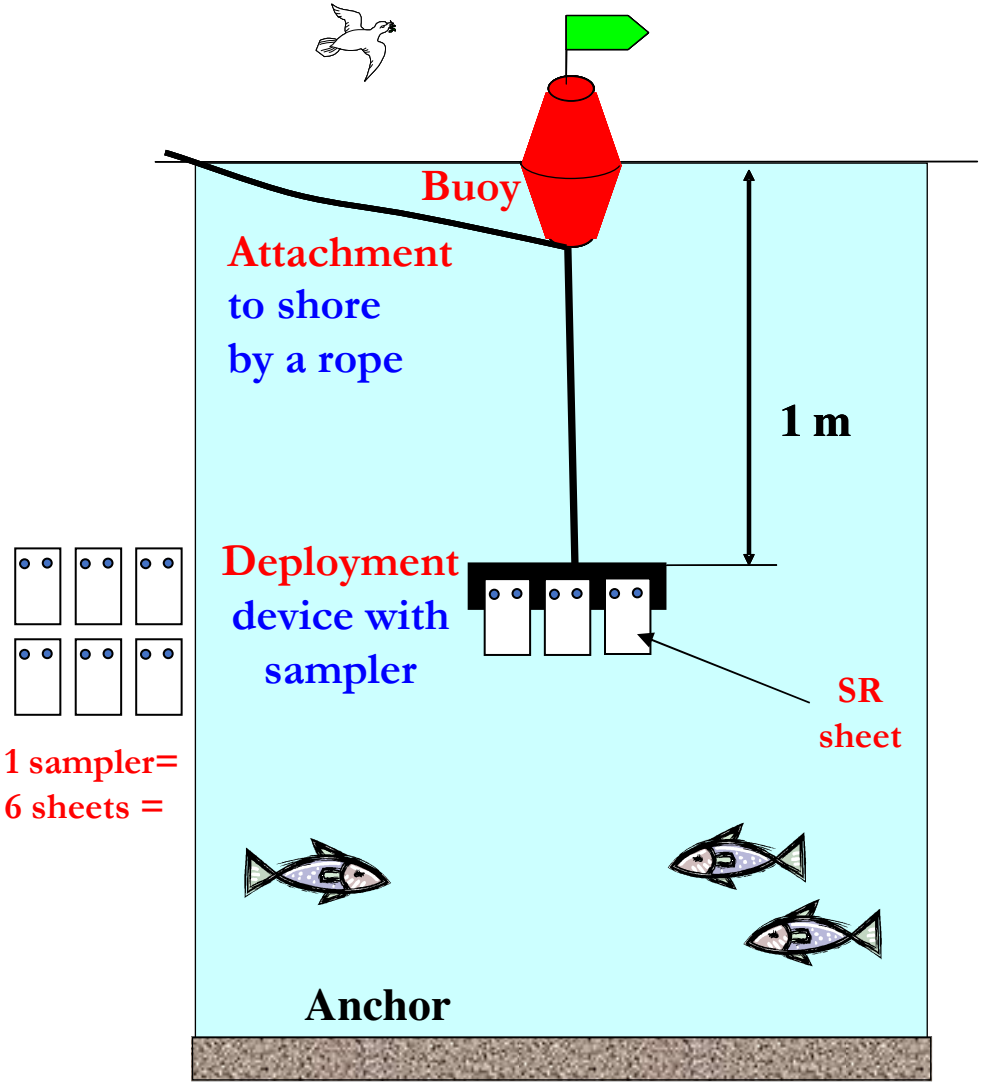
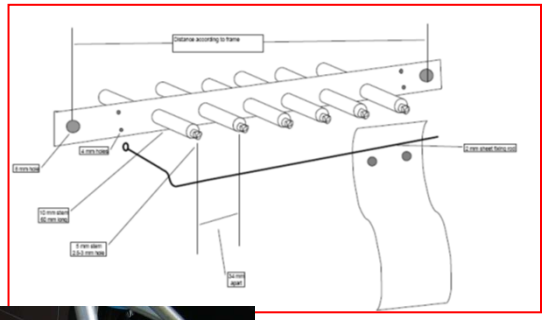


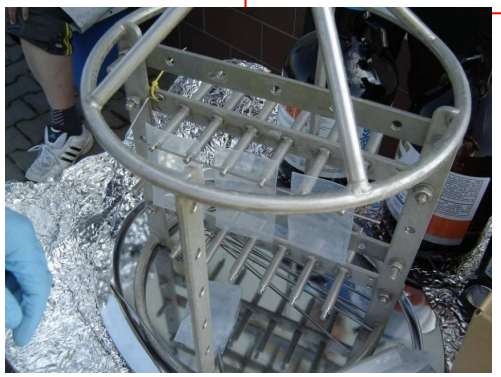
Figure 11.2 A typical apparatus for suspending passive samplers in the water column. Polar organic chemical integrative samplers (POCIS) and semipermeable membrane devices (SPMDs) are commonly housed in this type of protective canister.

Sampler deployment

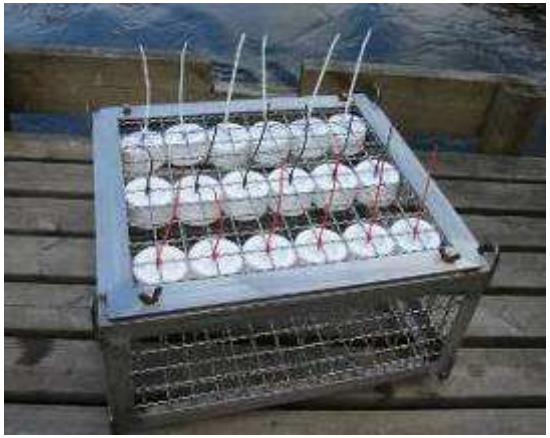


Sampler holder





Sampler deployment



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Sampler recovery

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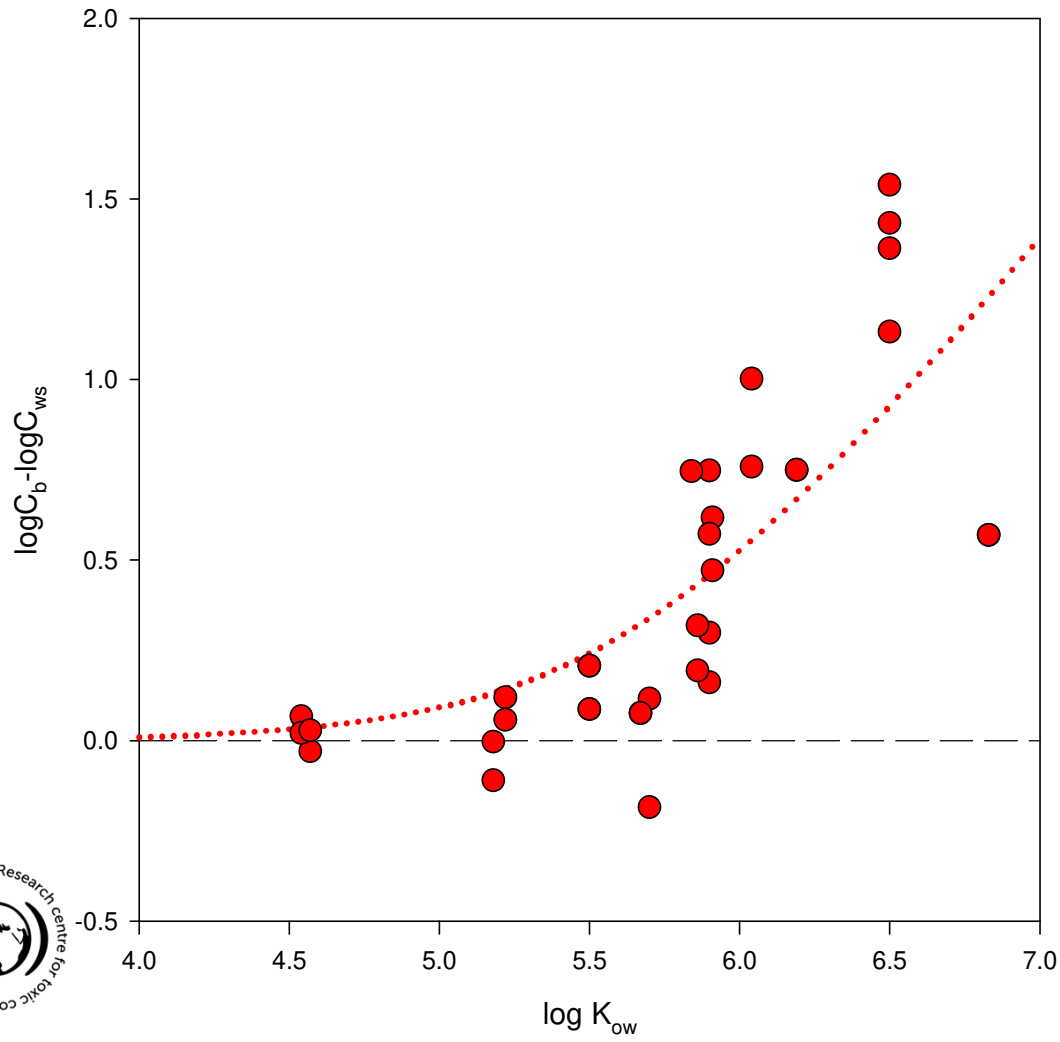
Advantages of passive sampling

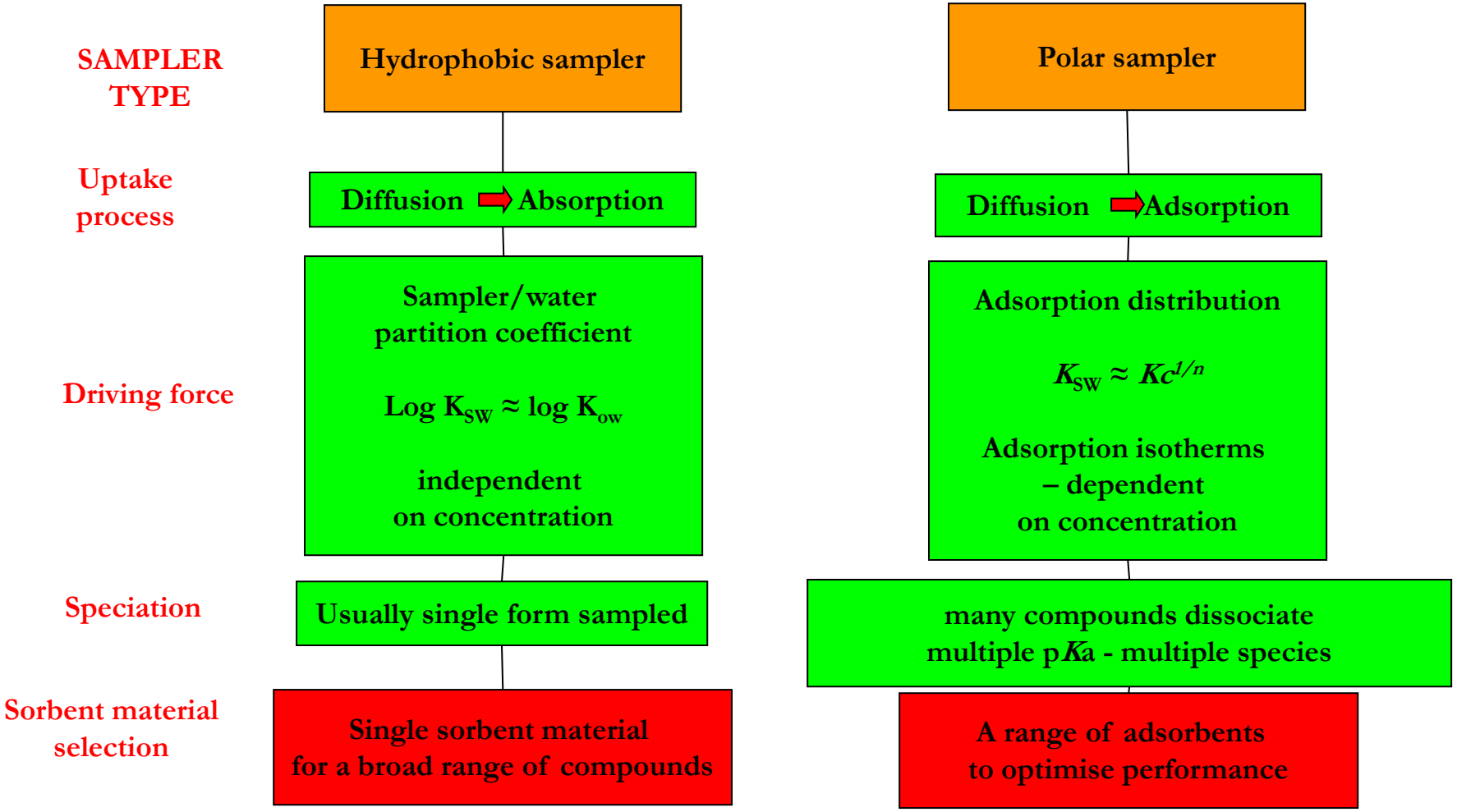
- Measurement of a freely dissolved concentration of contaminants in water
- Continuous sampling – measurement of TWA concentrations
- Extremely low limits of detection (low pg/l level)

Typical relation between dissolved concentrations in water determined using silicone rubber passive samplers and those from GF/F filtered samples of water



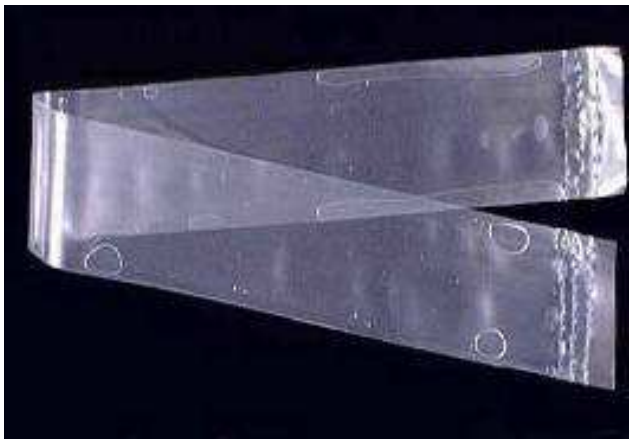
C_b – filtered water
C_{ws}- free dissolved concentration

Two types of passive sampler

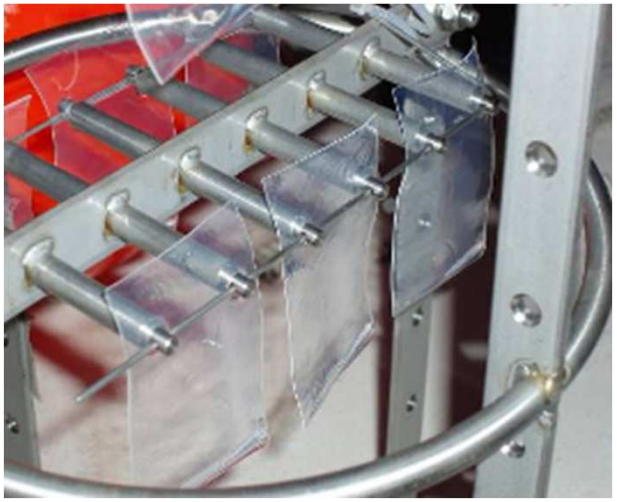


Hydrophobic samplers

- Semipermeable membrane devices – SPMD



- Silicone rubber sheets

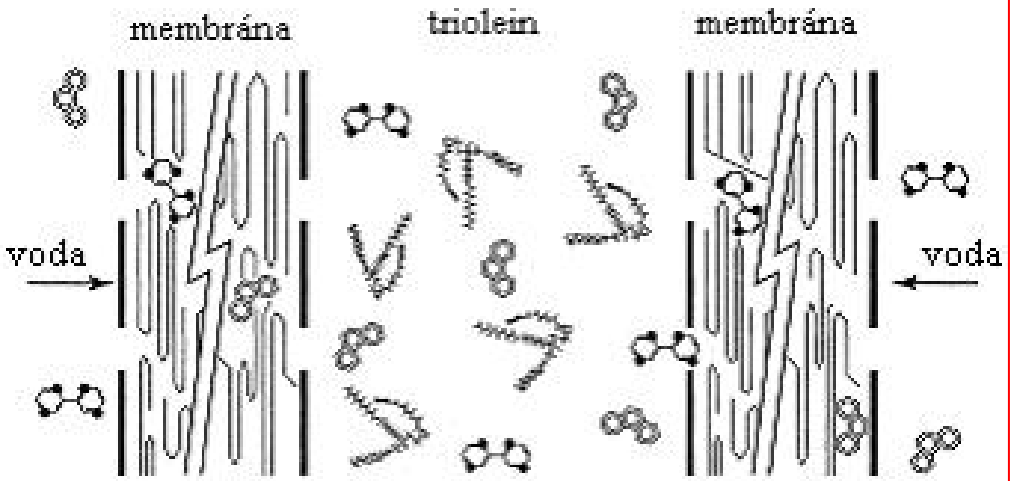


Integrative sampling up to several months

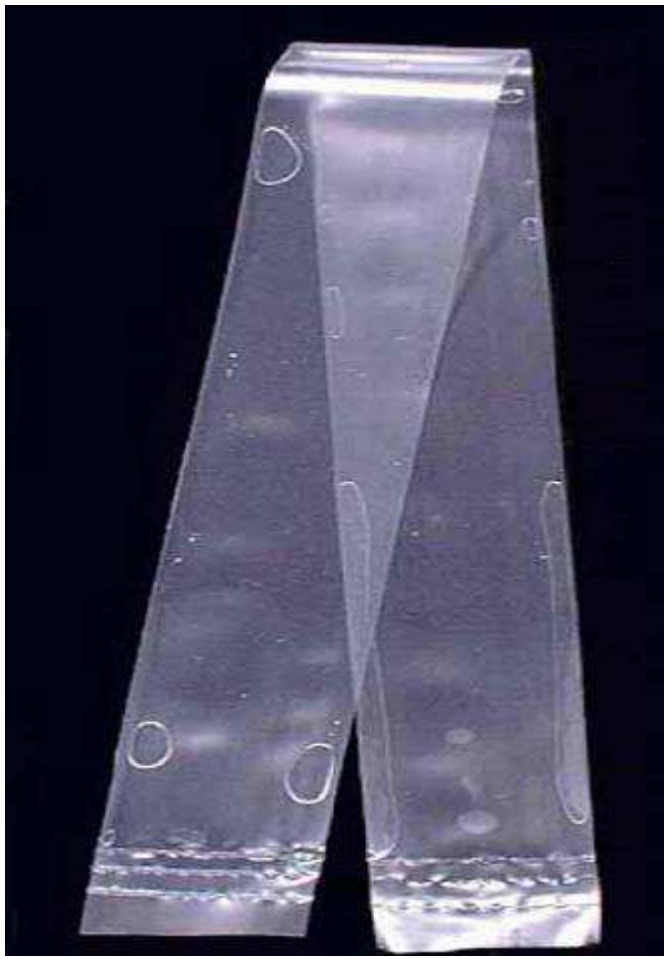
Application range: semivolatile hydrophobic organic compounds

Semipermeable membrane device - SPMD

- Lipid-filled low density polyethylene sheet
- Integrative sampling up to one month
- Application range: semivolatile hydrophobic organic compounds



sloučenina	vzorec	rozměry (nm) délka	průměr
triolein		2.7	2.8
fenantren		1.2	0.8
2,2´,5.5´-tetrachlorbifenyl		1.2	0.9



Membrene (LDPE): 94 x 2.5 cm, thick 75-95 µm, pores 1.10^{-9} m

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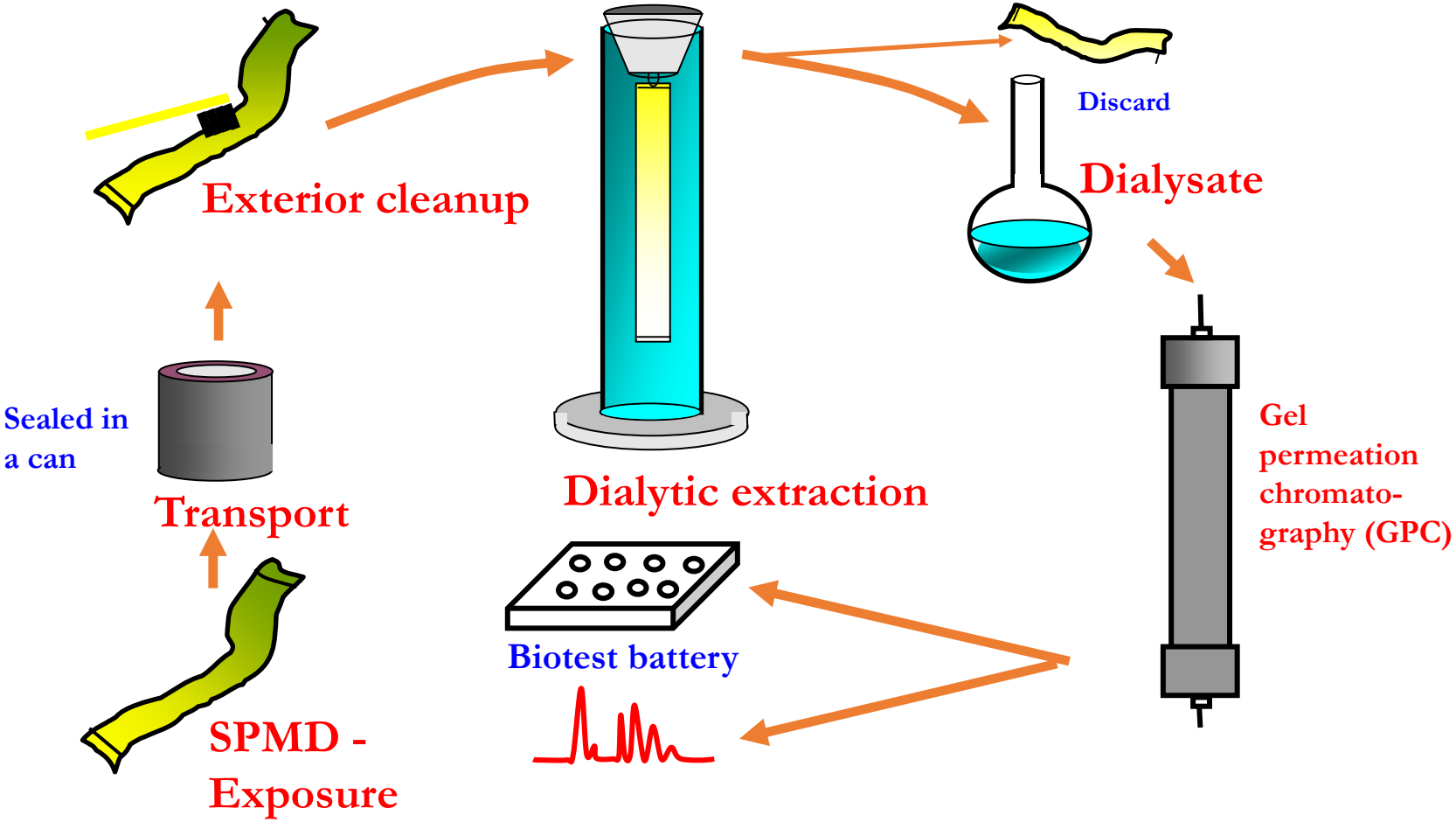
Passive sampling - SPMD

- SPMD is able to simulate the process of bioconcentration
- Exposure SPMD membranes in flow provides information about the quantity and periodically occurring pollutants
- Use SPMD well simulates the process of diffusion through biomembranes
- SPMD is made of synthetic materials (greater uniformity and reproducibility).
- Captures metabolized chemicals
- The mathematical model
- Exposure to natural or treated water, sediments, and in the air
- To determine the toxicological provide relevant mixture of pollutants present in the environment
- Detection of accidental releases of chemicals
- Concentration in relative terms (difficult quantification), required calibration study
- Patented technology
- Problematic deployment
- Leakage of triolein

SPMD processing



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Passive sampling - PDMS

- Structurally simple and inexpensive device
- Sampling difference of chemical potentials (the diffusion of molecules of the analyte)
- No diffusion barrier (membrane solvent)
- Easy to install
- Faster analysis
- Possibility of further research
- Calibration studies

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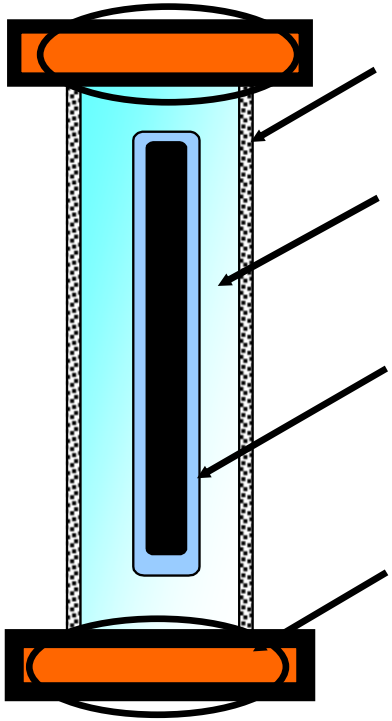
Membrane Enclosed Sorptive COating (MESCO)

- solventless preconcentration of organic contaminants in PDMS
- thermal desorption of sequestered analytes on-line with a capillary GC-MS system
- miniaturisation



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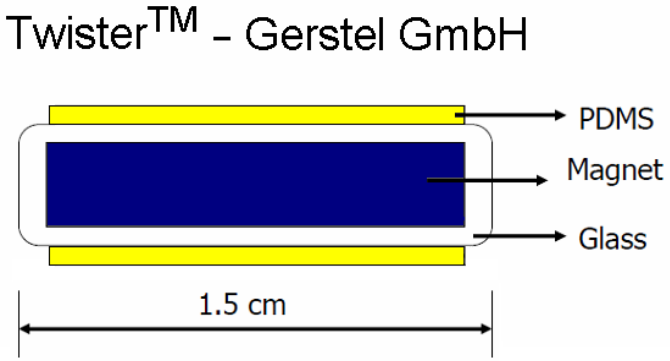
Sorptive extraction techniques

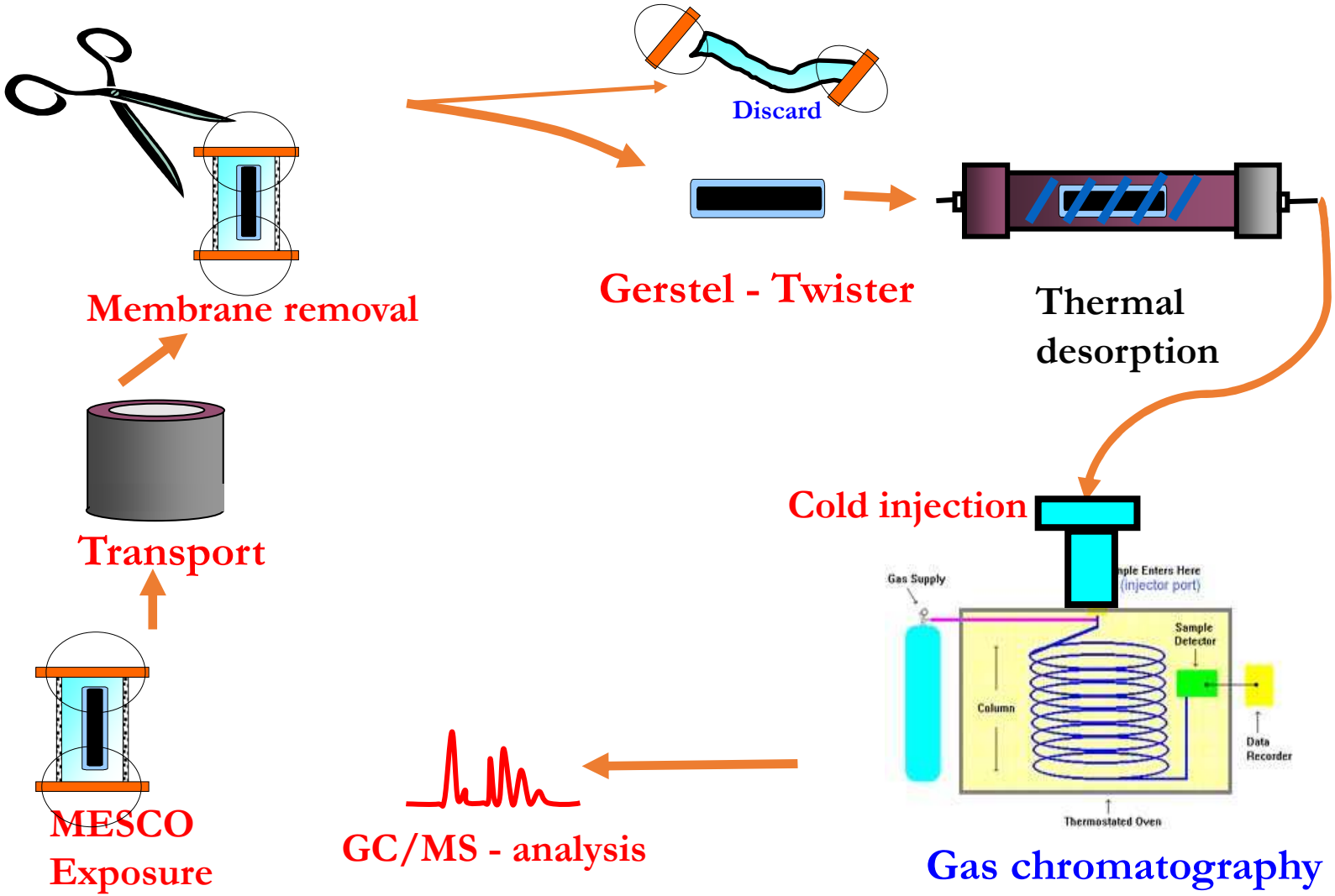
Stir-bar sorptive extraction (SBSE)

In SBSE stir-bars (so called "Twisters") are coated with a PDMS layer (typically 0.5-1.0 mm thick):



The stir-bars are commercially produced by Gerstel in Germany under the trade name Twister.

MESCO processing



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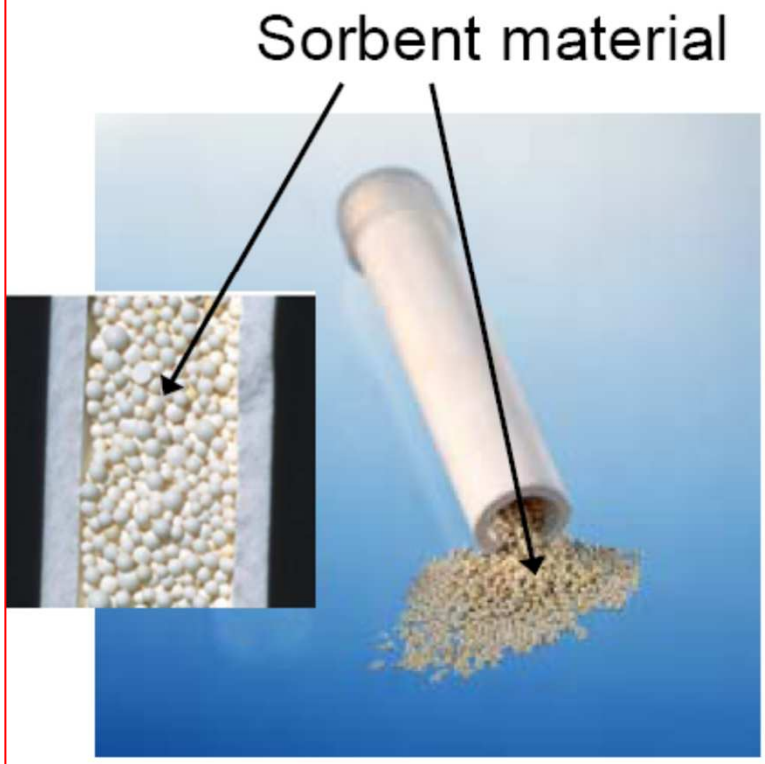
Ceramic dosimeter

- Sorbent receiving phase
- Ceramic permeation barrier
- Integrative sampling up to one year
- Low sampling rate and long response
- Application range: wide range of organic compounds

Ceramic dosimeter

Example: Ceramic Dosimeter – TWA Sampler



- **solid sorbent beads**
  - high affinity & capacity
    -> steep concentration gradient betwe
    the exterior and interior of sampler
    => continuos diffusion and linear upta
  - easily wetted by water
  - no swelling
  - easy to extract, e.g. simple solvent extraction
  - high recovery rates

- BTEX, PAHs, CHCs: Dowex Optipore L-493 (Supelco)
  Martin et al., 2003, ES&T
- PAHs: Amberlite IRA-743 (Sigma-Aldrich)
  Bopp et al., 2005, J. of Chromatography
- PAHs & Toxicity: Biosilon (Nunc); *Bopp, 2004, Dissertation*

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

Polar Organic Chemical Integrative Sampler (POCIS)

Adsorbent + PES membrane

Polar Chemcatcher

Empore disk +/- PES membrane

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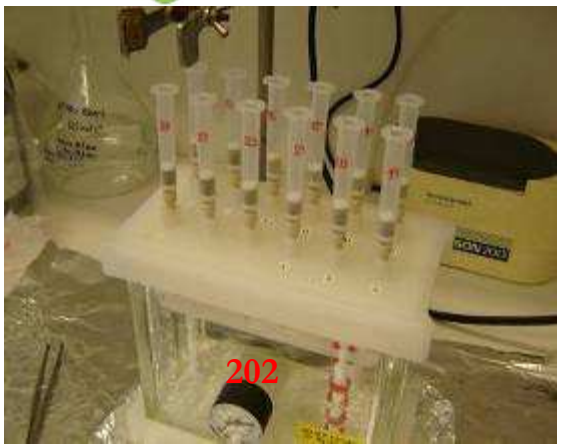
Polar Organic Chemical Integrative Sampler (POCIS)

- Sorbent receiving phase
- Polyethersulphone membrane
- Integrative sampling up to several weeks
- Application range: polar organic compounds

Laboratory preparation of POCIS samplers



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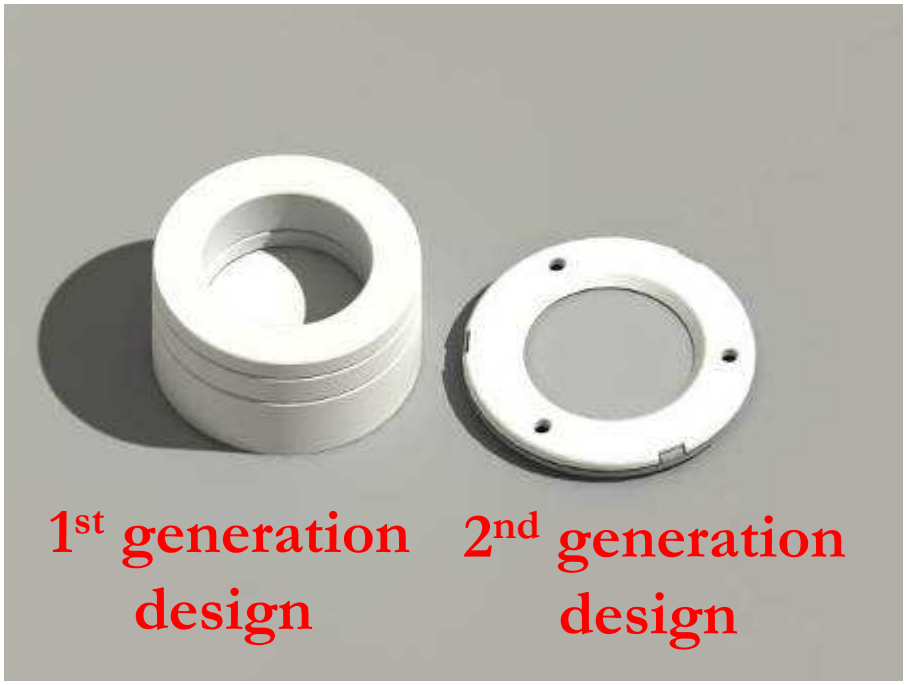
CHEMCATCHER

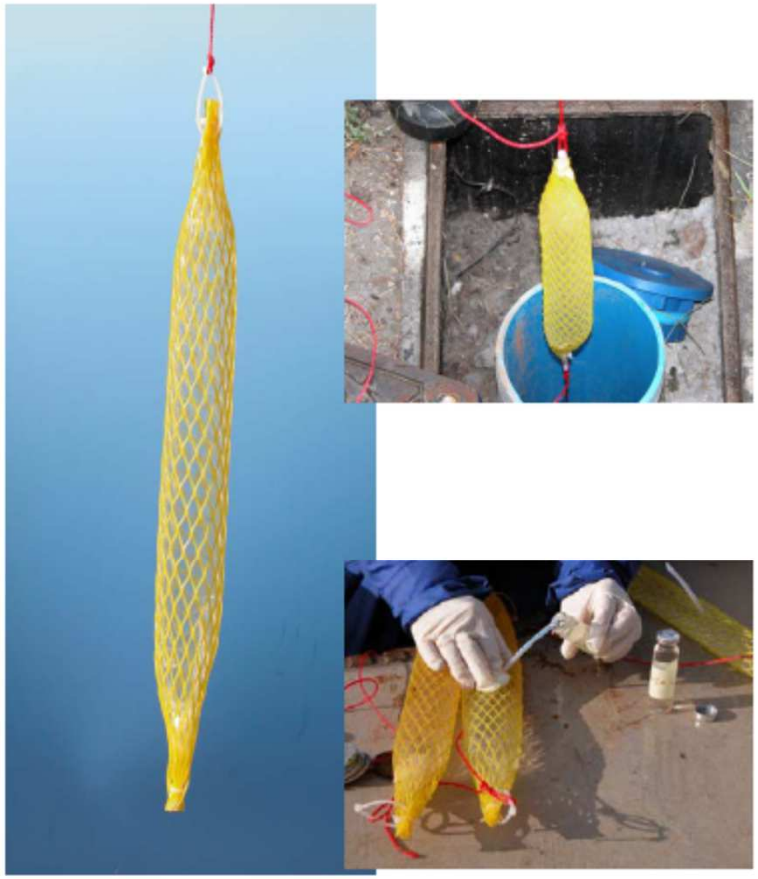
- The sampler consists of
  - Sampler body
  - Sorbent disk
  - Diffusion membrane
- Many particular sampler configurations
  - Non-polar organic
  - Polar organic
  - Metal
  - Organometallic
  - Mercury
- Two prototypes
  - 1st generation – reusable
  - 2nd generation – disposable



Samplers of volatile compounds

Example: PDB – Equilibrium Sampler

(Polyethylene-Diffusion-Bag-Sampler)



Design

PE-membrane, Ø: 3-5 cm x L: 30-50 cm, filled with deinonized water (ca. 300 ml)

Exposure time

minimum 2 weeks

Substances

High volatile organic compounds VOCs: BTEX, CHCs

Analysis

Conventional water analysis

Standard sampler since 2001 in the US (US-EPA)

http://diffusionssampler.itrcweb.org

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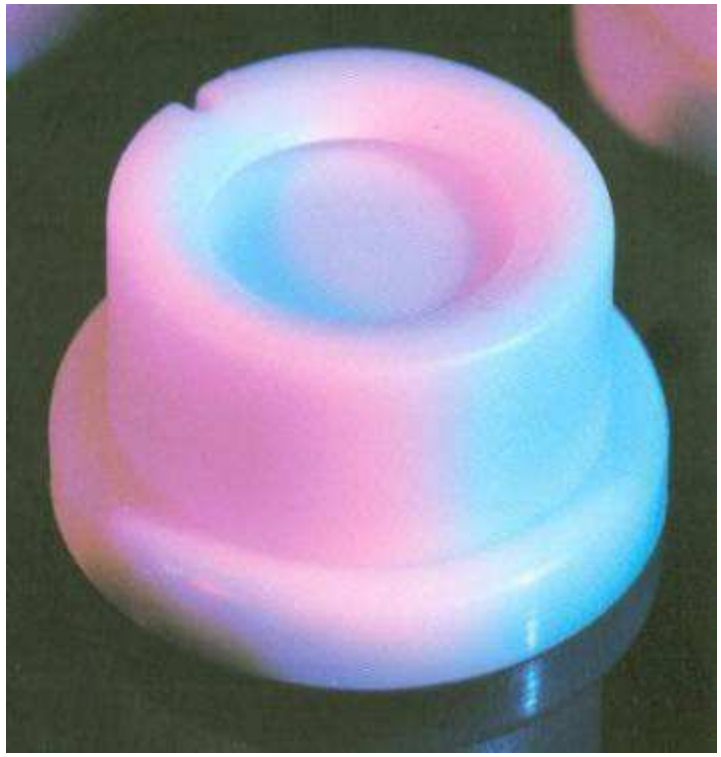
Diffusive gradients in thin films (DGT)

- A layer of binding agent impregnated in hydrogel to accumulate the solutes (a resin)
- A diffusive layer of hydrogel and a filter
- Application: metals, phosphate, sulphide, radionuclides
- If diffusion coefficients are known, no need for calibration



Properties of samplers

Sampler ID	Surface area (cm^2)	Surface/Vol (cm^{-1})	Analysis (PAH & PCBs)
 Chemcatcher	17.4	29	GC/MS
 SPMD	460	93	GC/MS
 Silicone strip	321	41	GC/MS or ECD
 LDPE membrane	325	183	GC/MS or ECD
 MESCO I	10	637	TD-GC/MS
 MESCO II (new)	12	255	TD-GC/MS
 Silicone rod	0.66	21	TD-GC/MS

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Concluding remarks

- Passive samplers can effectively be used as a tool in regulatory monitoring as the obtained freely dissolved concentration is a strong indicator for exposure to aquatic organisms
- They are suitable for trend monitoring because they integrate concentration fluctuations in time in a specific water body and long-term comparisons can be made with lower sampling frequency at the required sensitivity and statistical power to detect temporal or spatial trends
- Further research is needed for improving the accuracy of passive samplers for polar organic compounds
- Validation and QA/QC should focus more at the sampling than at the instrumental analysis.

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

Biofouling

- A potential source of systematic error is biofouling that
  - develops with increasing deployment time
  - reduces sampling rate in a time-dependent manner
- PRCs provide some correction for effects of biofouling
- Biofouling is the main factor setting the upper limit to deployment time
- Some on-line sensors are also affected by biofouling in a time-dependent manner

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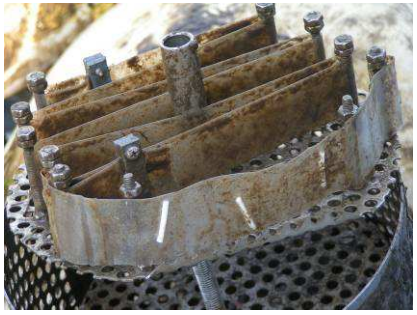
Biofouling

Semi-permeable membrane devices



Chemcatcher LDPE/Si_strip

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