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Trace analytical laboratories RECETOX

Petr Kukučka, RECETOX, Masaryk University, Brno, Czech Republic

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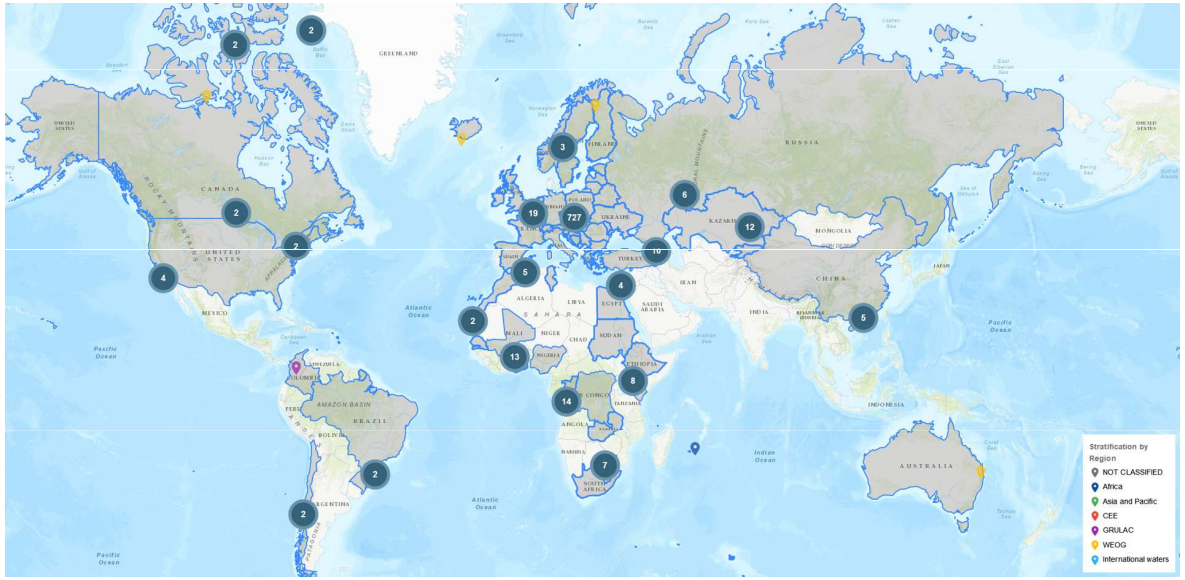
D29, 2NP, akreditace, omezený vstup

Petr Kukučka – laboratoř stopové analýzy

Overview of the POPs monitoring activities

POPs (persistent organic pollutants) are among the **most stable organic compounds** and have been **detected in all components of the environment**. The main reason for their monitoring is the proven **wide spectrum of toxic and genotoxic effects** of these substances. Concentrations of POPs have started to rise since the industrial revolution, mainly due to the **increasing use of combustion and thermal industrial processes** using fossil fuels and the **increased use of pesticides across a range of industries**. Their concentration depends on the proximity of point sources, but they are also **found in remote areas**, where they reach by **long-distance transport**. In general, POPs are dangerous in the environment because they are **strongly resistant to degradation** (both chemical and biological) and **strongly accumulate** in fatty tissues, thus causing **strong bioenrichment at increasing trophic levels**.



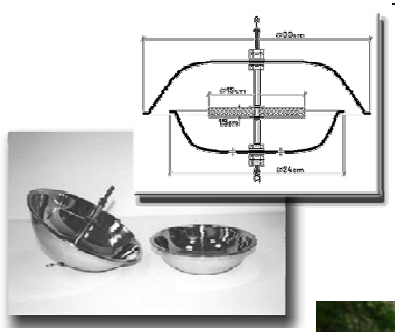
RECETOX –
experience since 1988

LSA major activities

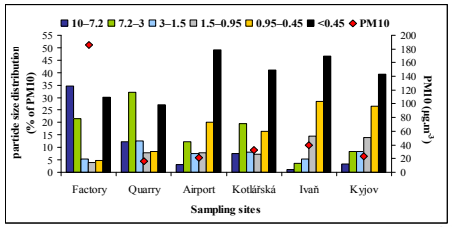
- Development of samplers for non-polar and polar compounds contamination and development of analytical methods for selected chemicals, pollutants and their metabolites in the human exposure studies
- Laboratory and field studies, programs of integrated monitoring

natural disasters

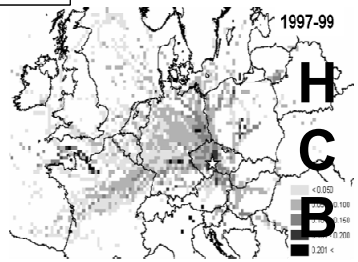
passive samplers



atmospheric particles

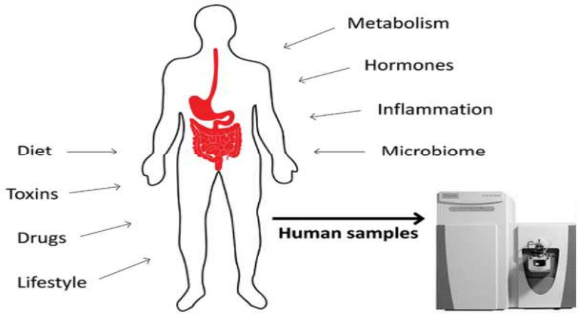


primary and secondary sources



long-range transport

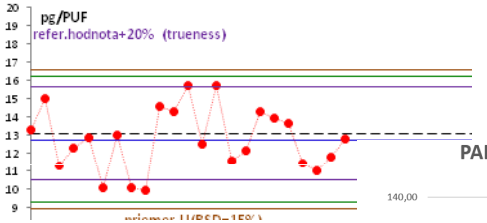
Human exposition

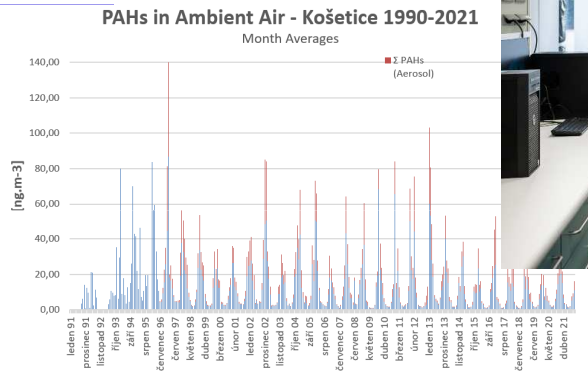


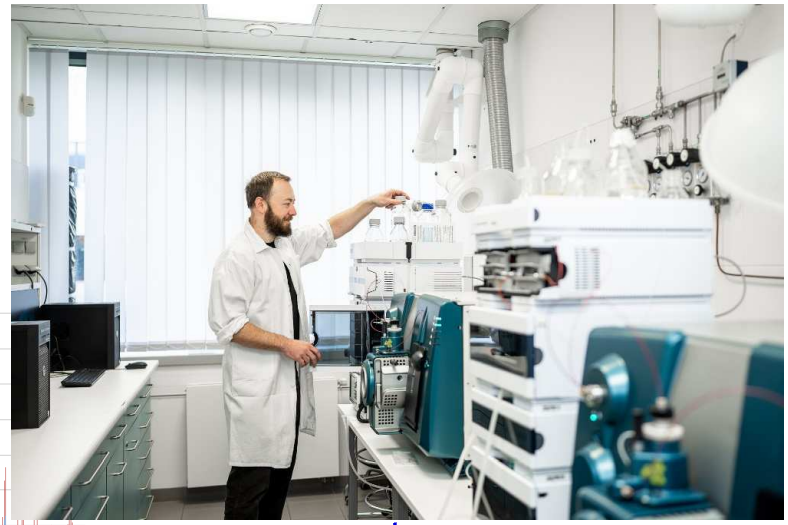
• RECETOX Laboratory services

- Sampling

- Samples preparation
- Analysis and data evaluation
- QA/QC system







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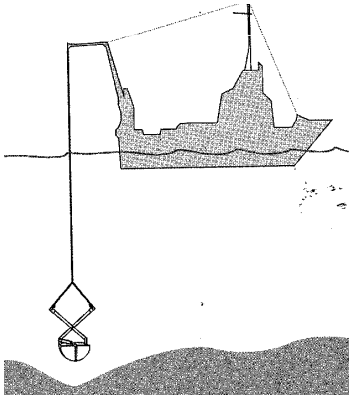
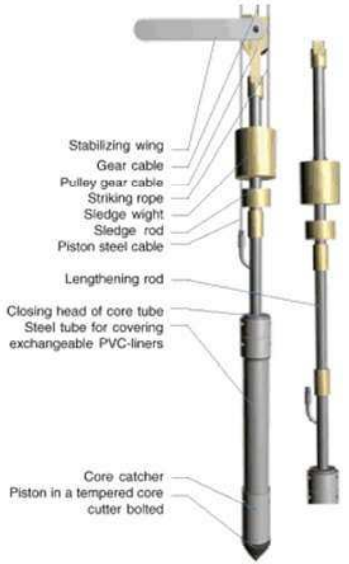
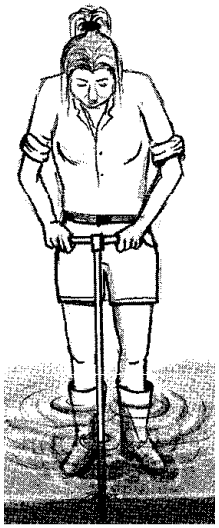
Sampling - services

- Water active and passive
- Sediment sampling
- Soil sampling
- Biota sampling
- Atmospheric deposition







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AIR MONITORING

Passive air sampling

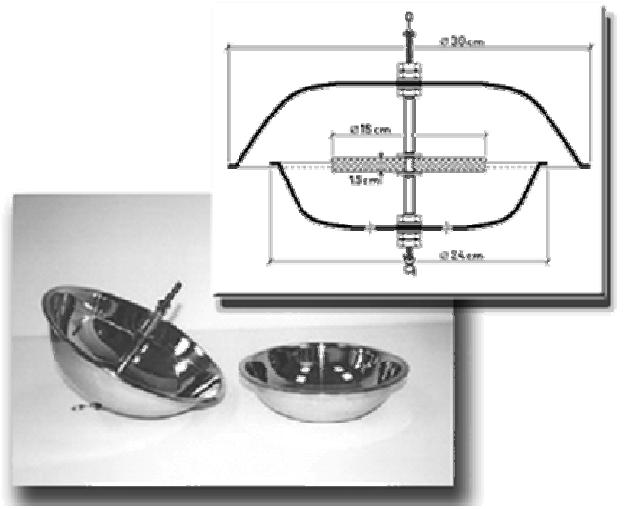
Advantages:

- low equipment price and low operating costs
- low requirements for installation and technical maintenance without the need to connect to a power source
- they provide information on the long-term level of contamination

Disadvantages:

- lower sensitivity and higher detection limit
- impossibility of exact determination of the air volume passed

(calculations depending on the duration of exposure, sorption rate and other parameters or empirically obtained coefficients are used)



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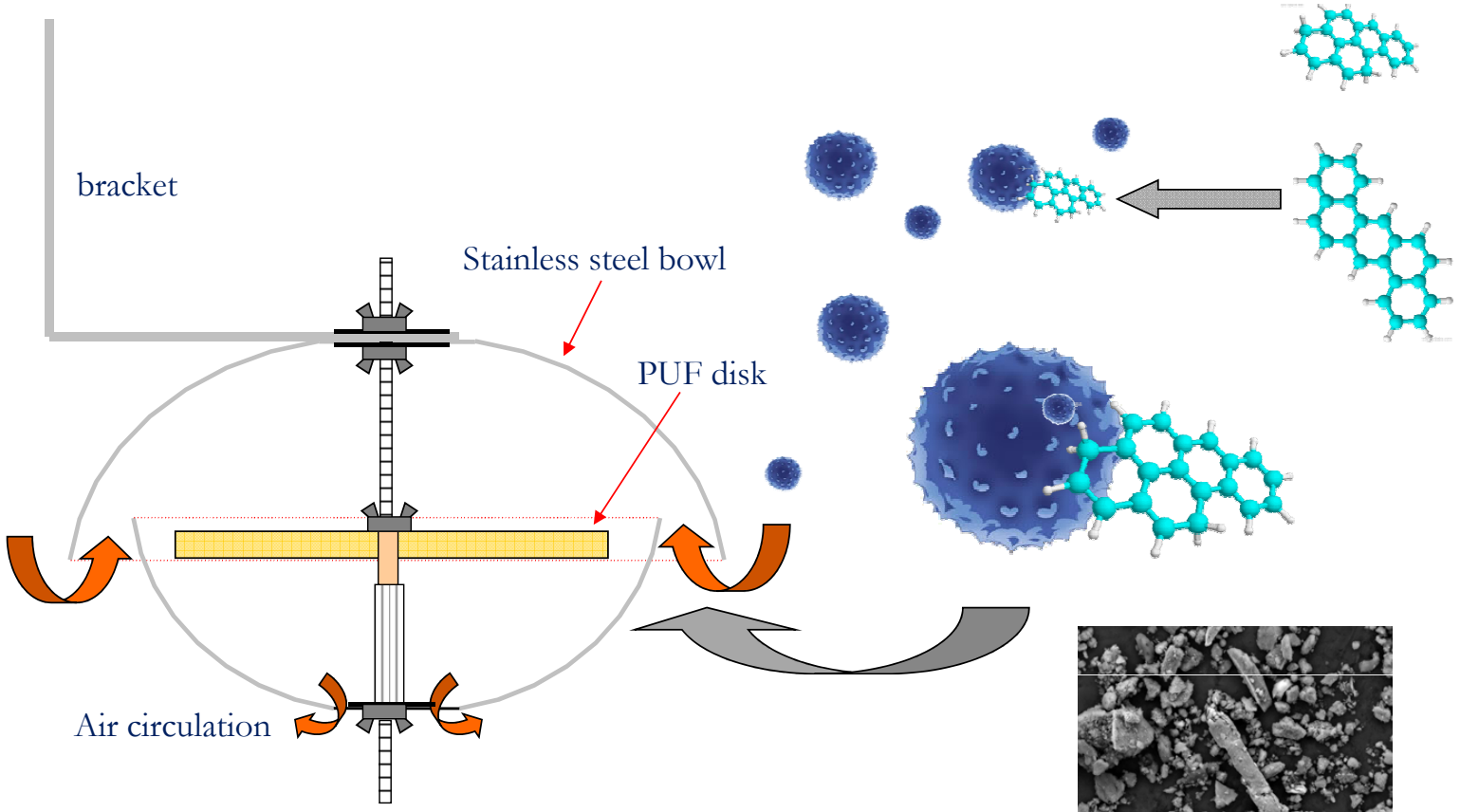
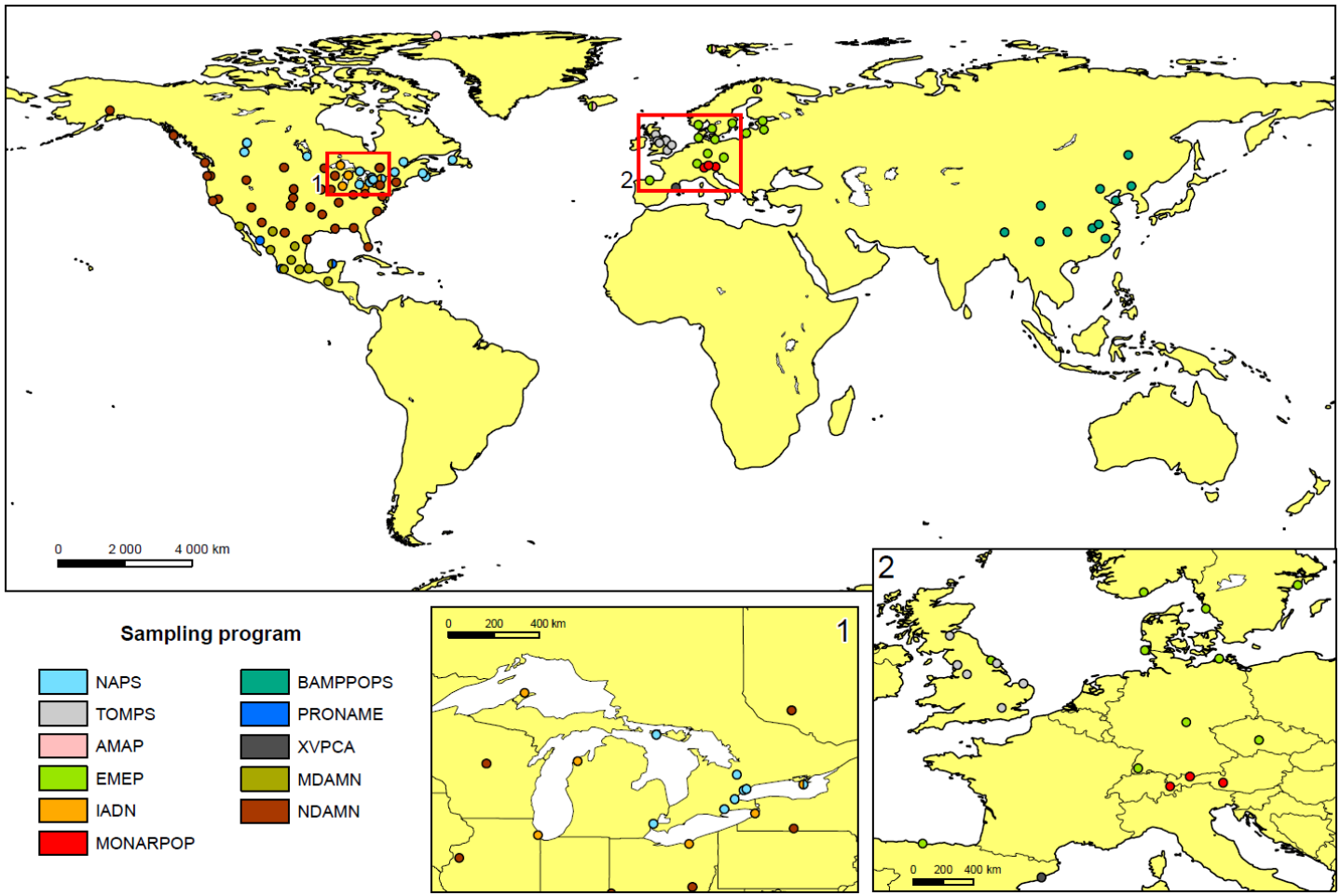
Passive air sampler



1) Gaseous phase sampling
2) Particle phase sampling with fixed pollutants

Air monitoring using active air samplers (2008)



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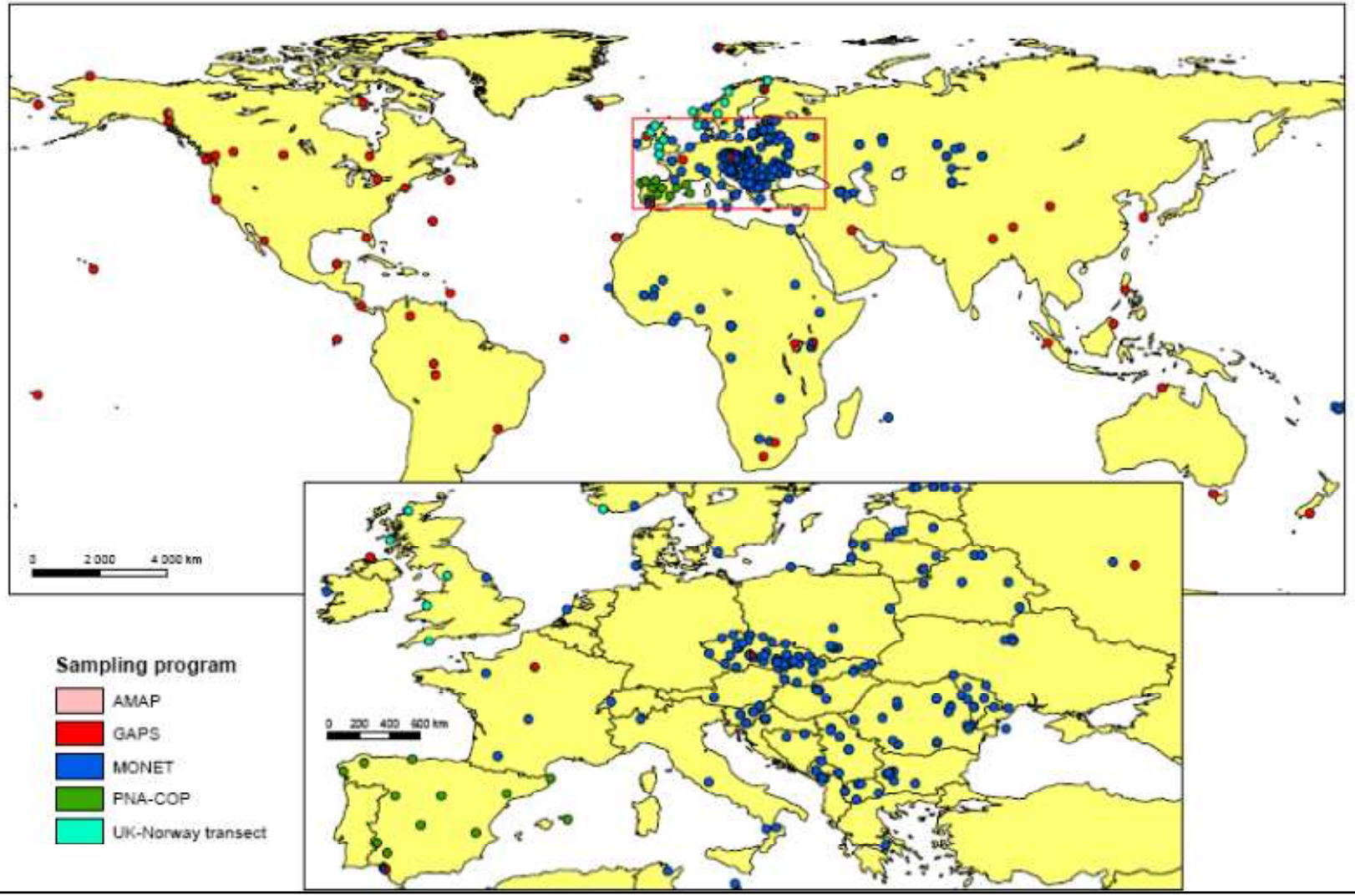
Passive air monitoring (2020)



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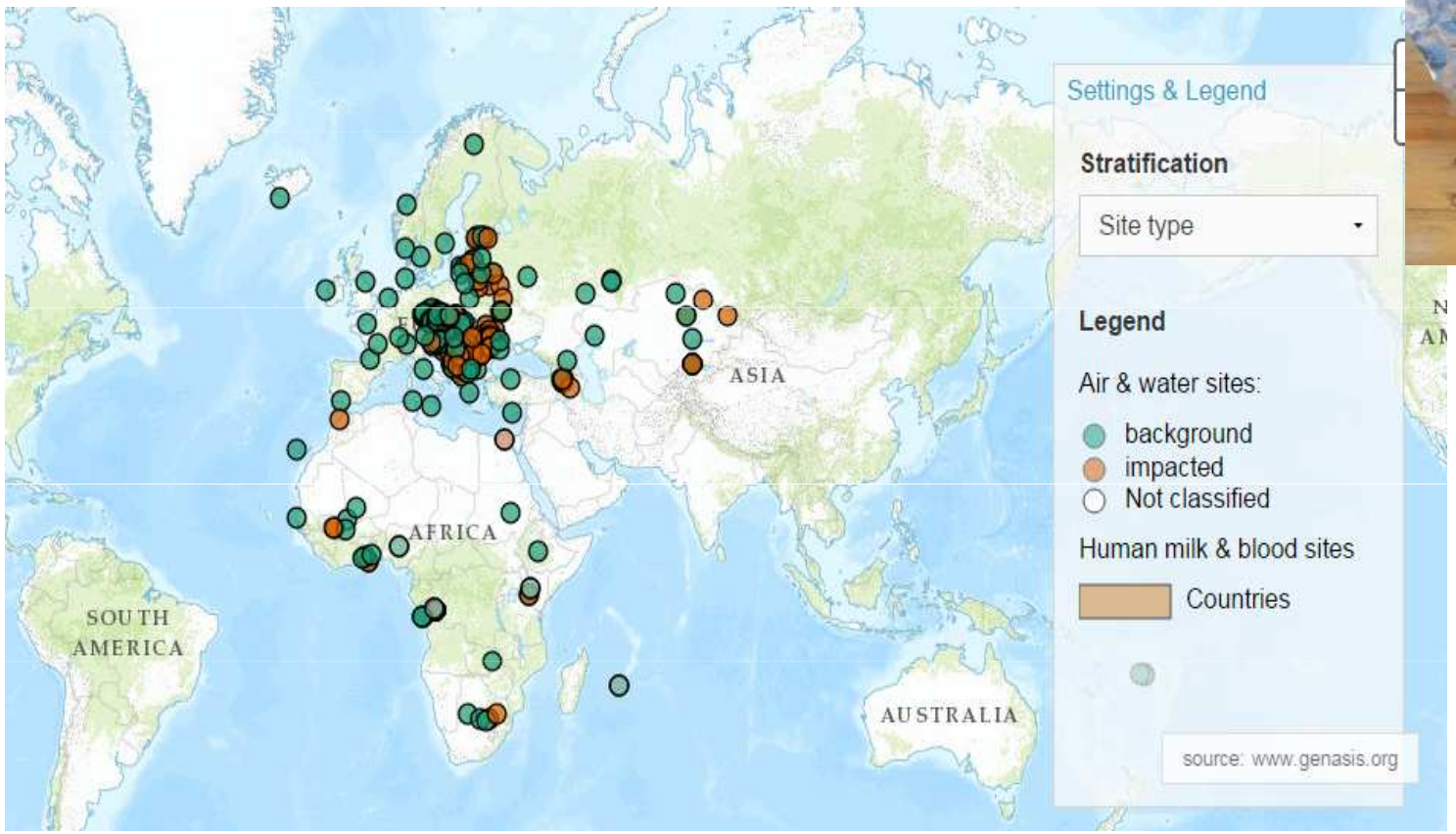
MONET (MOnitoring NETwork) projects

- MONET CZ – since 2003 (7000 samples)
- MONET EU – since 2006 (2000 samples)
- MONET Africa – since 2008 (650 samples)



Analysis (PAHs, subst. PAHs, PCBs (ind.+dl-PCBs, OCPs, cyclodiene pesticides, BFRs, PCDDs/Fs

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Passive air sampling in the Czech Republic (MONET CZ)

Passive air samplers on the base of PUF – since 2003

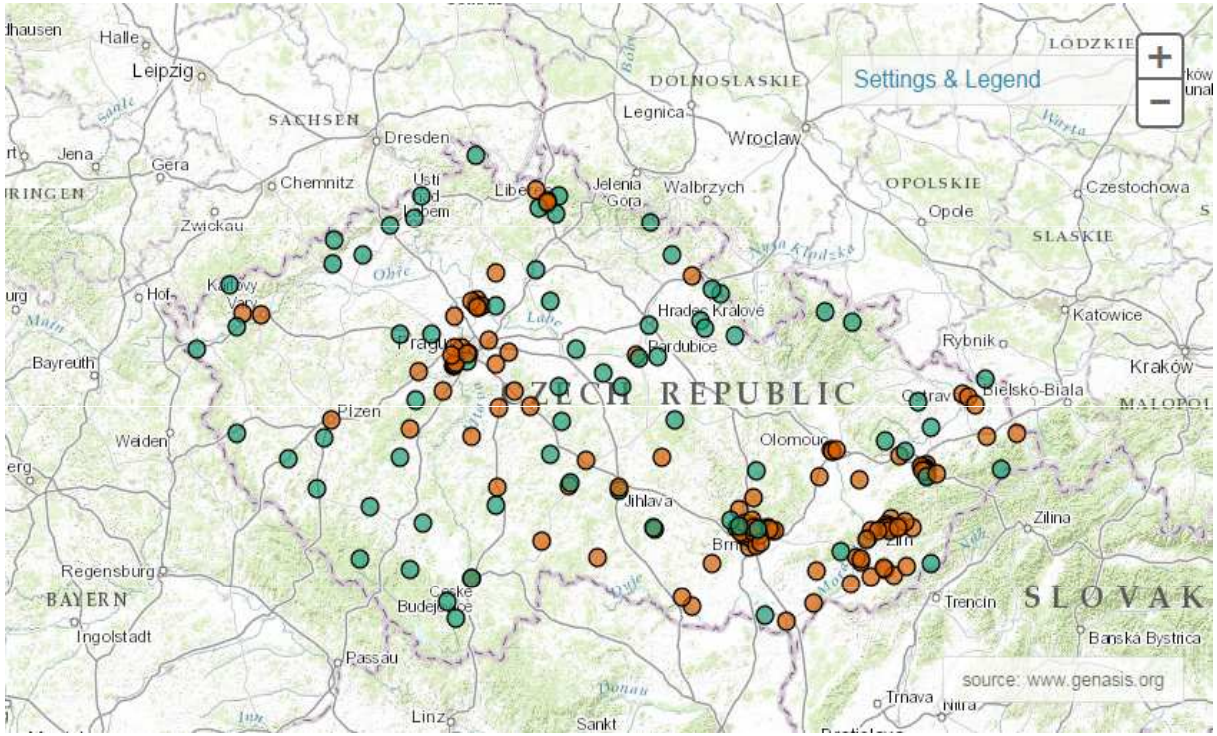
In 2003 – 2013 – continuously monitoring on 46 sampling sites (sampling period 28 days)

Optimization of the monitoring network in 2014 – nb of sampling sites were reduced to 30

Sites belongs to MONET_CZ MONET_EU:

Košetice
Libuš
Svratouch
Churáňov

(sampling period 84 days)



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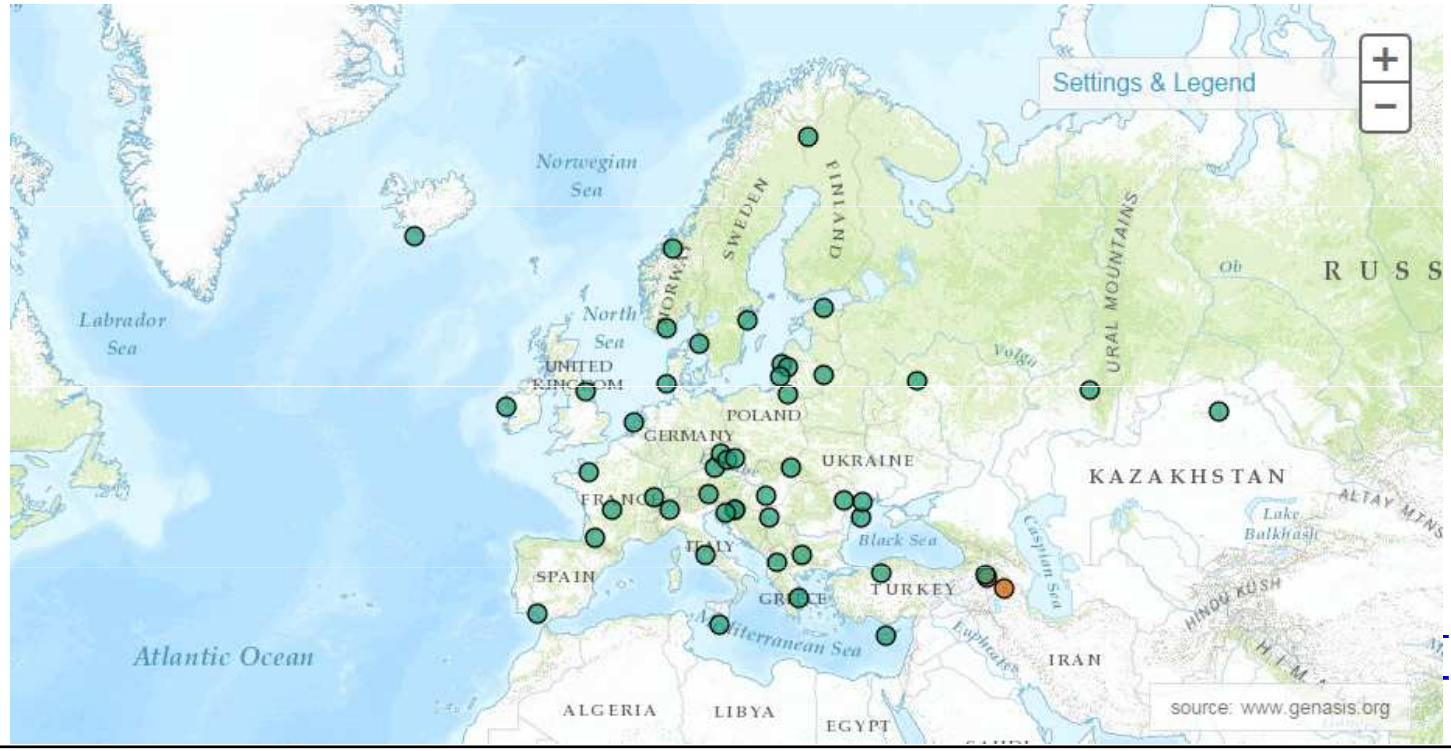
Passive air sampling in European Network (MONET EU)

After optimization of monitoring network – 30 background sampling sites

Wide range of analytes (PCDDs/Fs, BFRs, cyklodiene, organochlorine and polar pesticides and perfluorinated compounds) are monitoring in 9 sampling sites

Calibration study (active x passive samplers) in Spitzbergen sampling site (NILU)



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Passive air sampling in African Network (MONET Africa)

Wide range of analytes (PCDDs/Fs, BFRs, cyklodien, organochlorine and polar pesticides and perfluorinated compounds) are monitoring since 2008

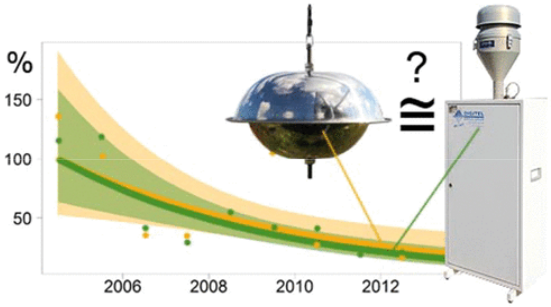
2014 – 15 Two calibration studies (active x passive samplers) in Kenya and Ghana

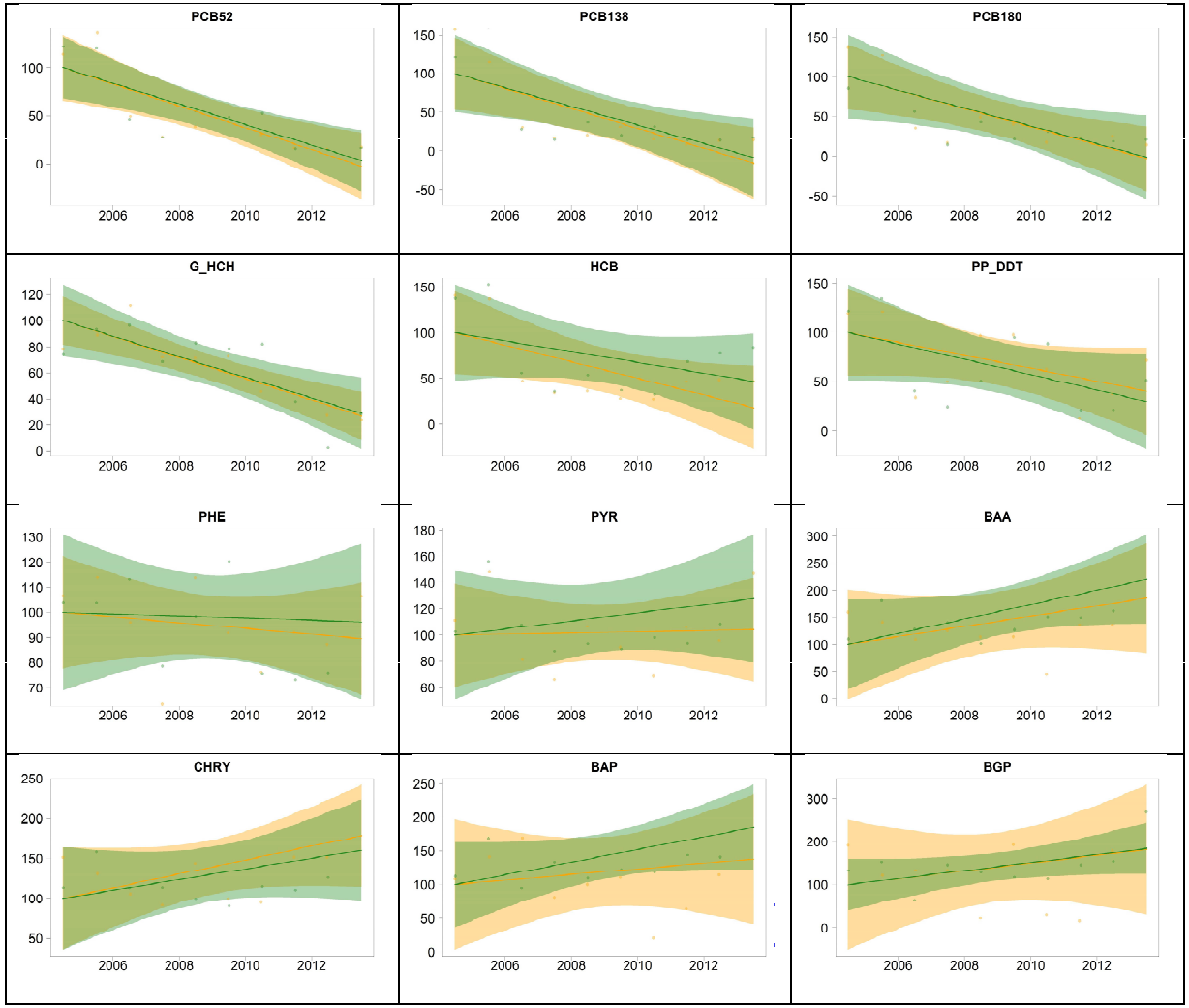
2015 – 16 The first passive water sampling campaign

Calibration studies – active *vs*. passive sampling

MONET CZ –Košetice, EMEP station, Czech Republic
MONET EU – Svalbard, Zeppelin, EMEP station, Spitzbergen, Norway
MONET Africa – University in Nairobi, Keňa; Ghana

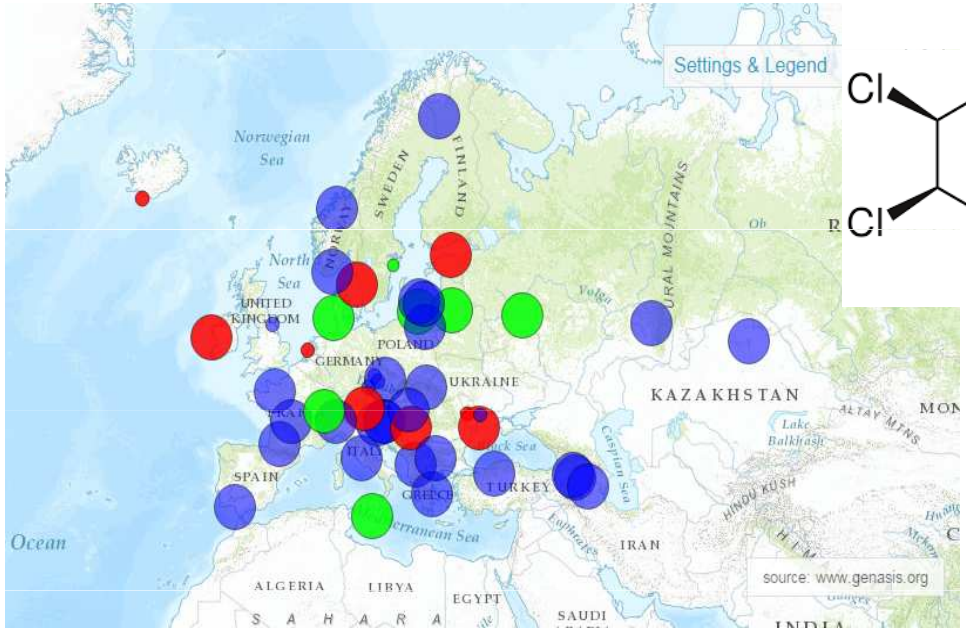
Calibration studies – active *vs*. passive sampling

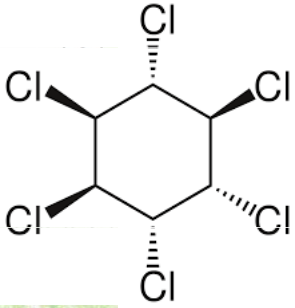




Passive Air Samplers As a Tool for Assessing Long-Term Trends in Atmospheric Concentrations of Semivolatile Organic Compounds, Kalina et al., *Environ. Sci. Technol.* 2017, 51, 12, 7047–7054

Concentration trend of ∑HCHs and ∑PCBs in the Europe (since 2006)



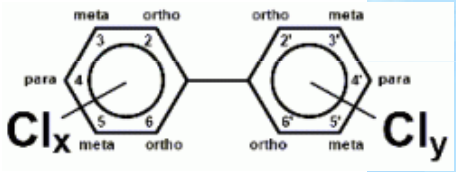


Size of symbol – Concentration:

- 0.8 ng sample-1
- 27.98 ng sample-1

Color – Trend type:

- Increasing
- Decreasing
- Statistically non-significant
- Not available

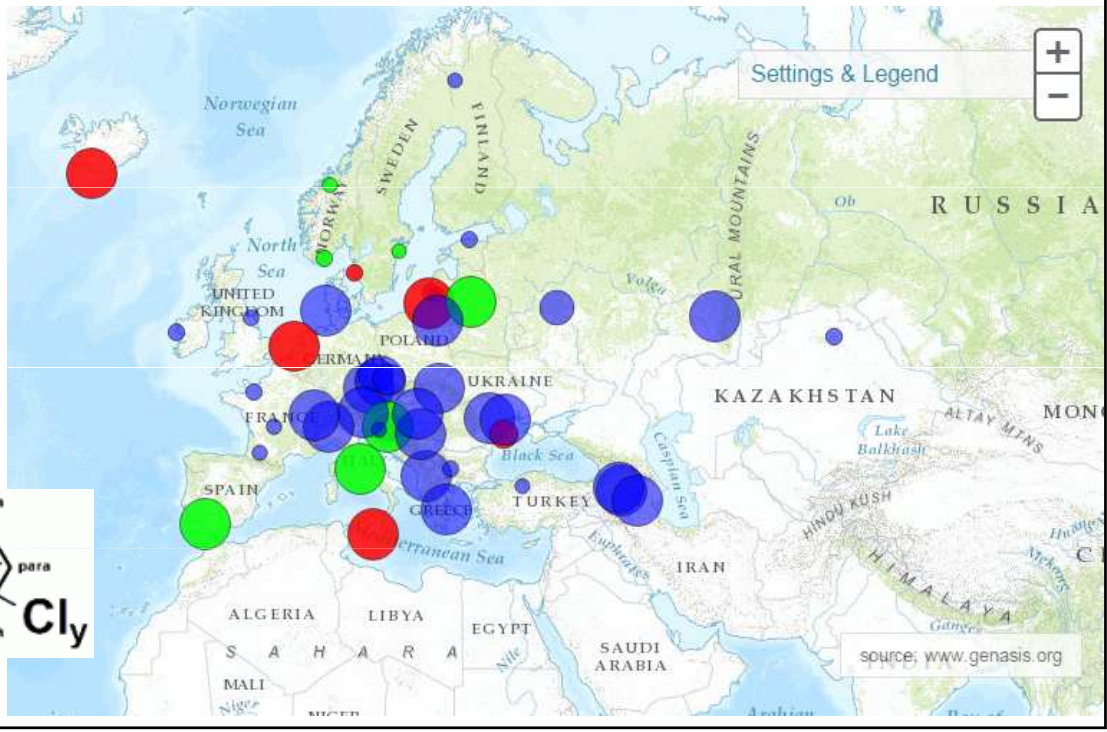


Size of symbol – Concentration:

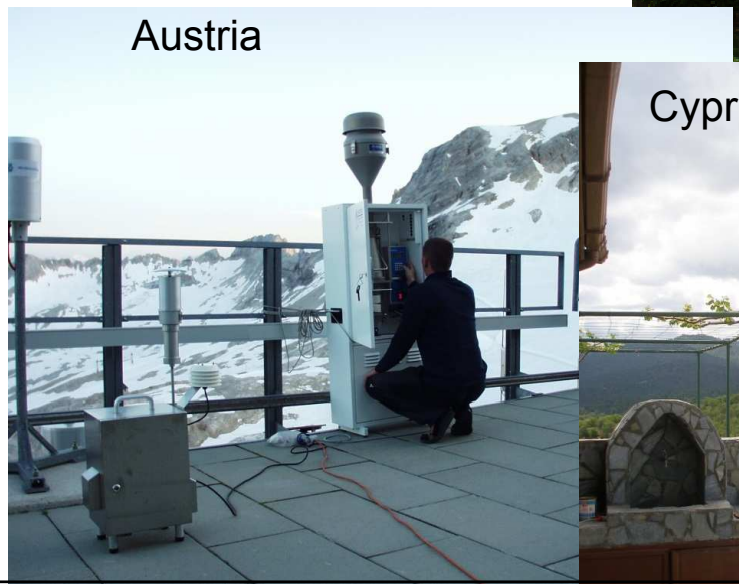
- 1.9 ng sample-1
- 33.252 ng sample-1

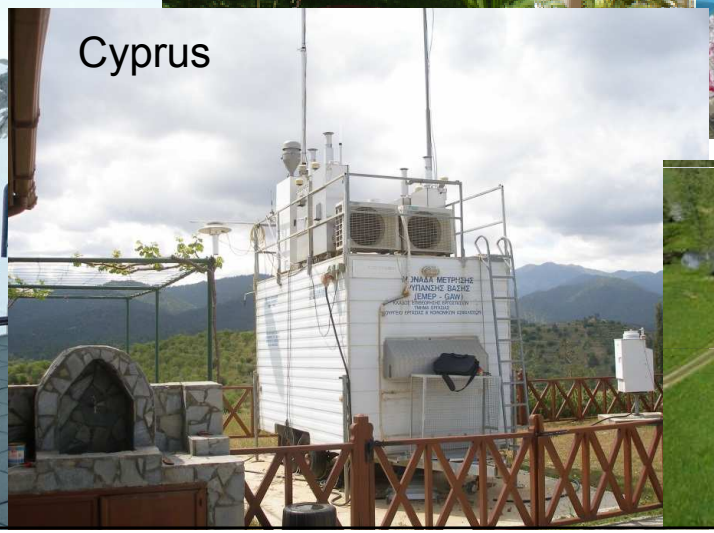
Color – Trend type:

- Increasing
- Decreasing
- Statistically non-significant
- Not available



Mauritius





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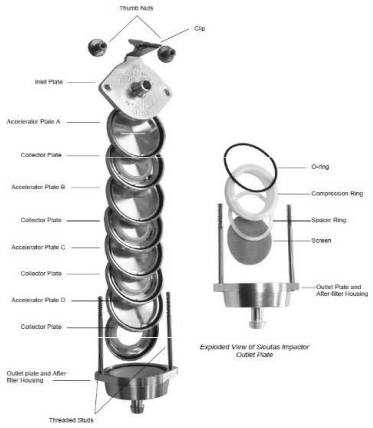
AIR MONITORING

Air sampling

- **Active air samplers** Low/High volumes including particles

  (PM distribution since 0,5 μm – 10 μm)

- **Multi-directional active air samplers**
- Sampling media – compounds dependent: PUF, XAD, or sandwitch (PUF/XAD/PUF), QFF



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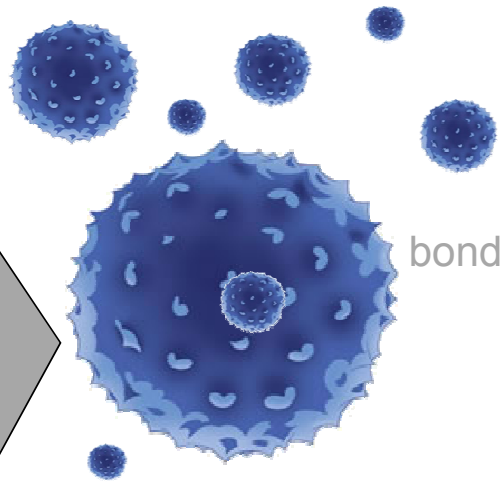
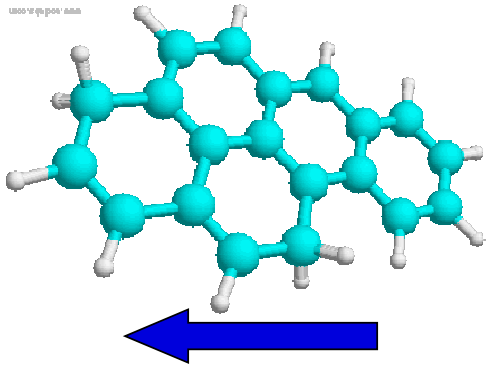
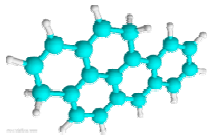
Atmospheric particles

PM 10
PM 2.5
PM 1.0
.....
....



bond





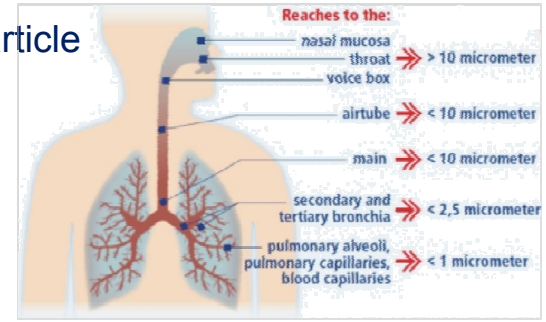
A large number of resources toxic substances

A large number of particle sources

1. Local heatings (source of particles and bound chemicals)
2. Secondary sources (contaminated soils, landfills – volatilization
3. The decisive parameters are the size of the particle surface, material, quantity,...

Immission limits: PM10 – 50 μg/m^3 (24 h)
PM10 – 40 μg/m^3 (calendar year)

PM 2.5 – 25 μg/m^3 (calendar year)k

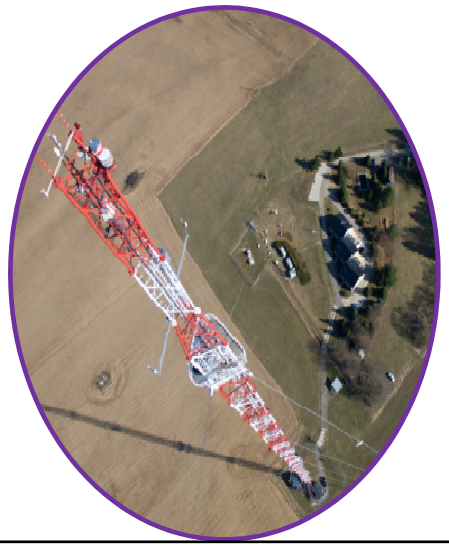


It is therefore important to monitor both the particles and the chemical substances bound to them.

Active air samplers

- **RECETOX has experiences with long term air monitoring since 1988 in NAO Košetice**
- Sampling media – compounds dependent: PUF + QFF (PM10) for POPs and PAHs; since 2015 sandwich (PUF/XAD/PUF) QFF for the polar compounds PFAS, CUPs

ACTRIS - Vertical gradients of POPs and PACs in near-ground central European background air – also open – access Research Infrastructure



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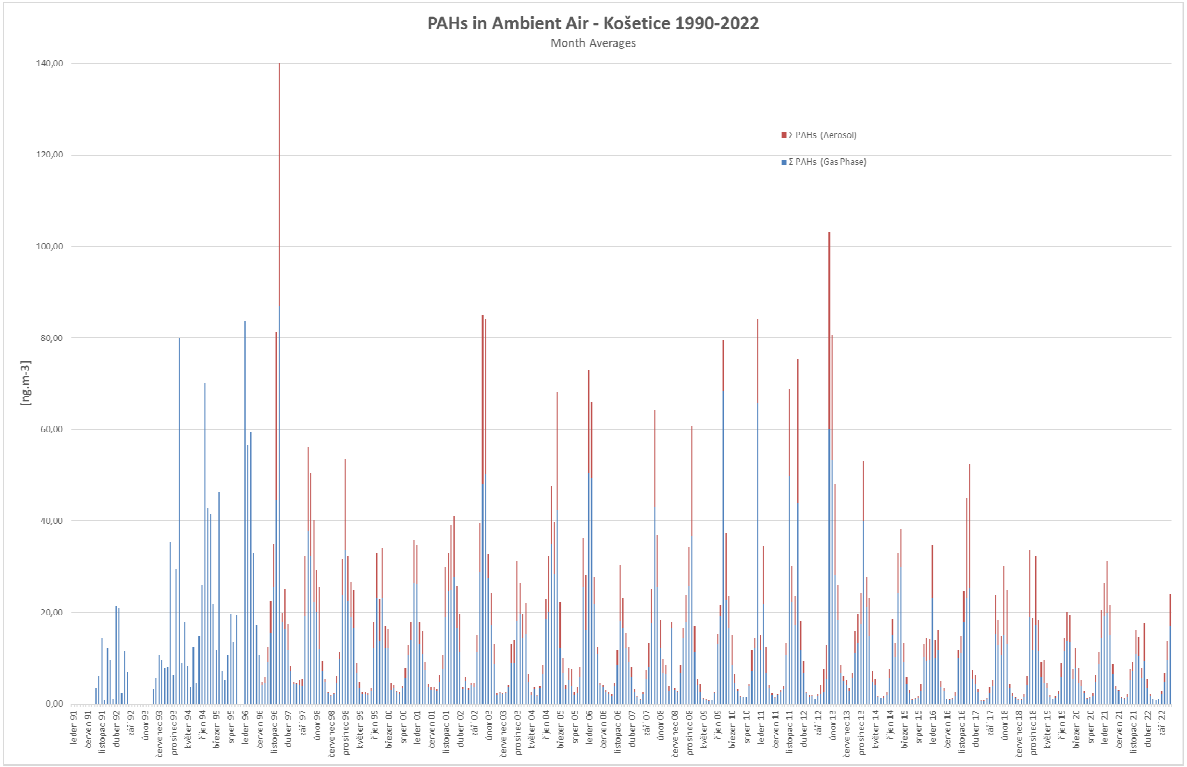
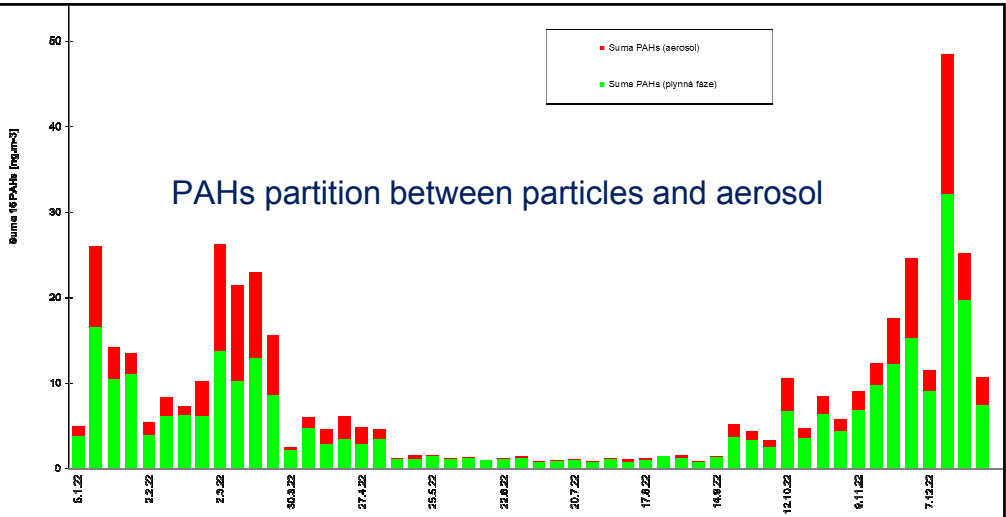
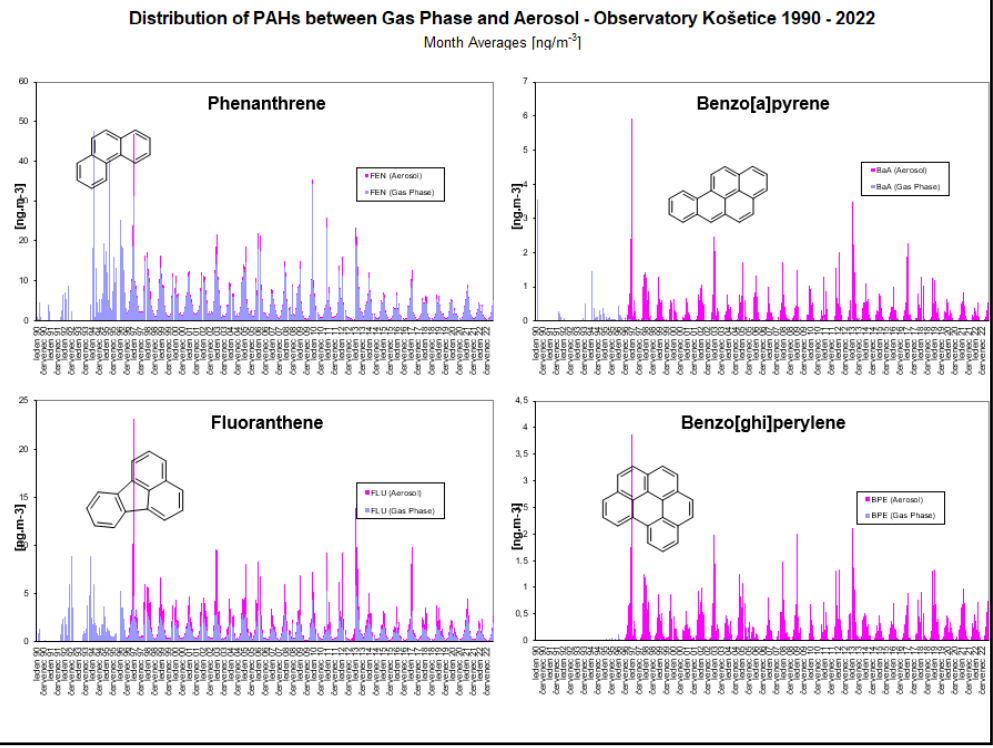
Active air samplers

Trends in PAHs concentrations

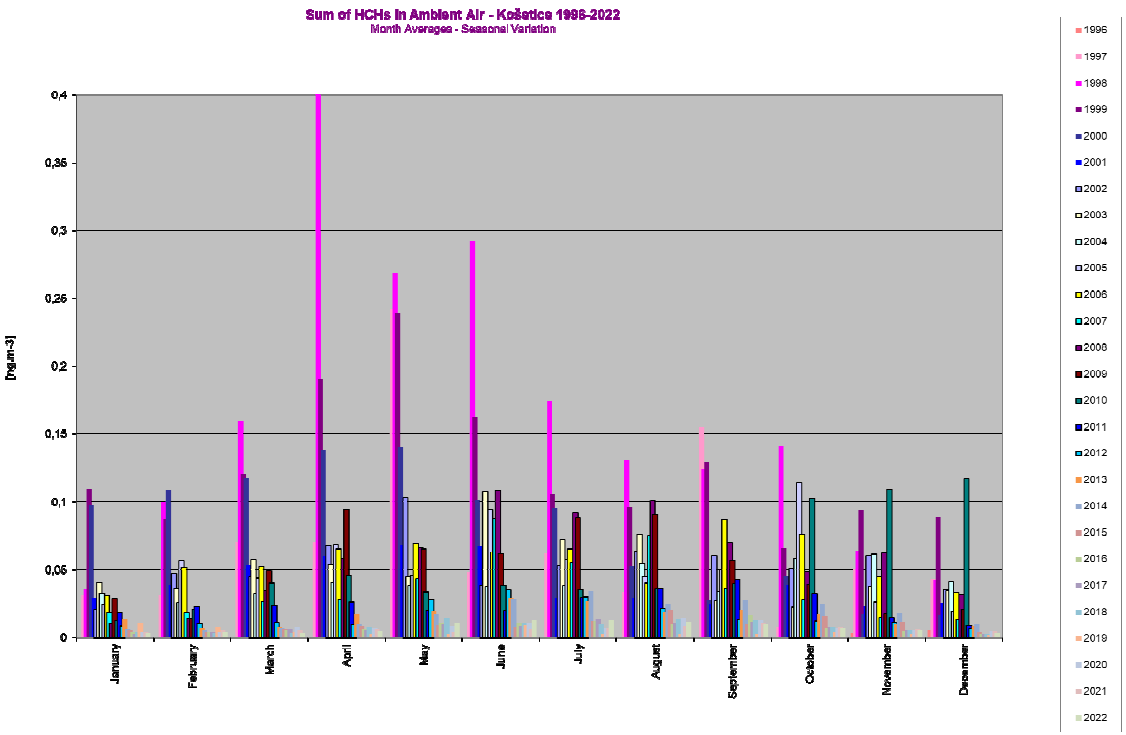




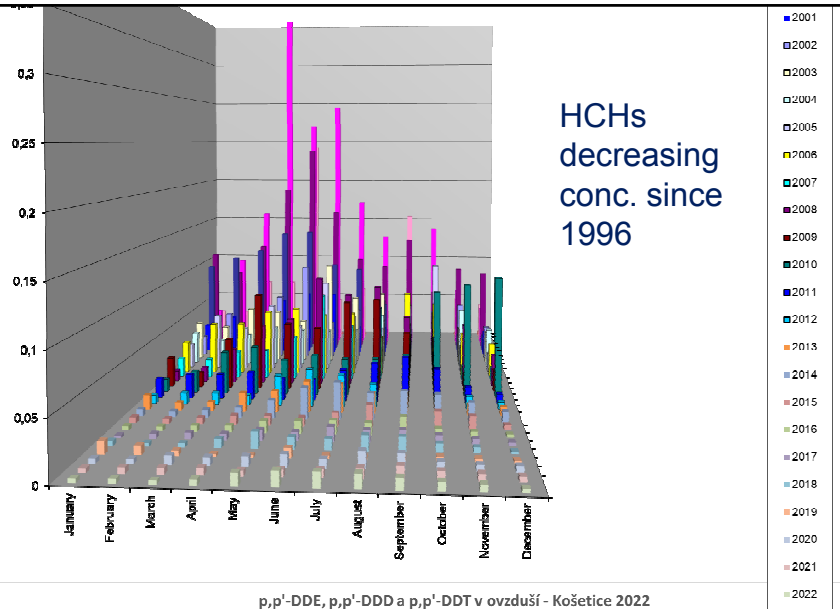


Active air samplers

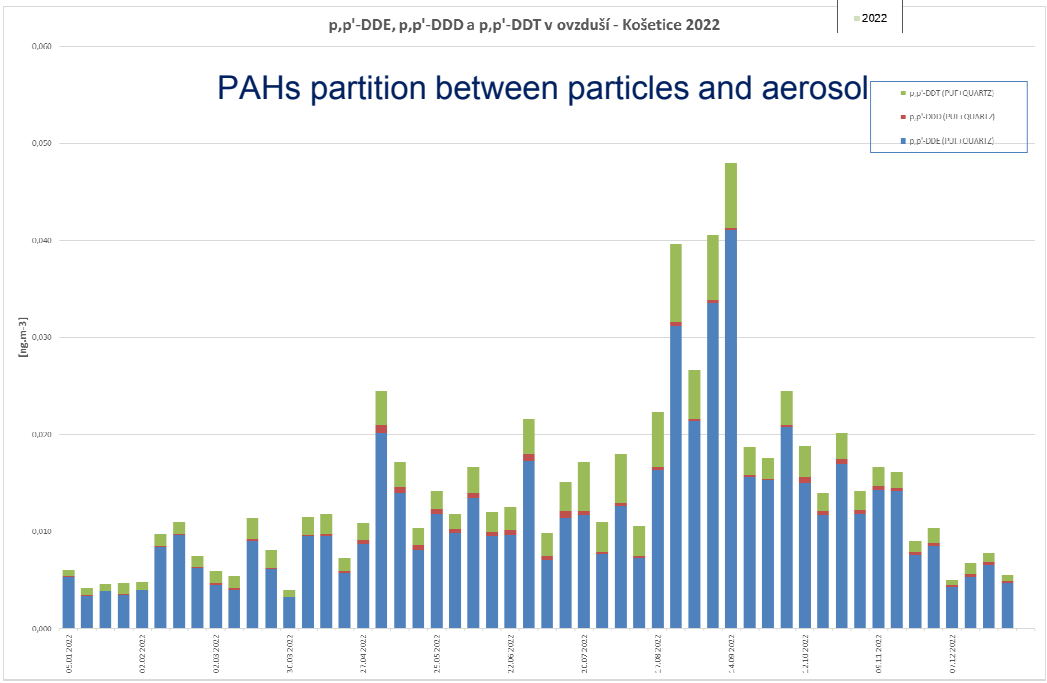
Trends in Pesticides concentrations



HCHs decreasing conc. since 1996 and seasonal variation typical for organochlorine pollutants mainly from the secondary sources



HCHs decreasing conc. since 1996

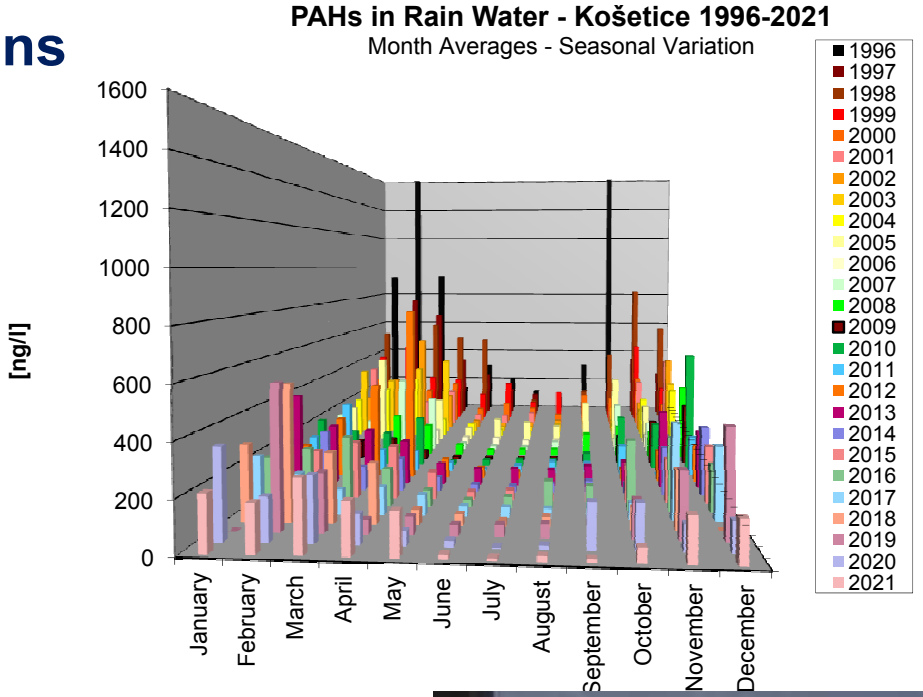


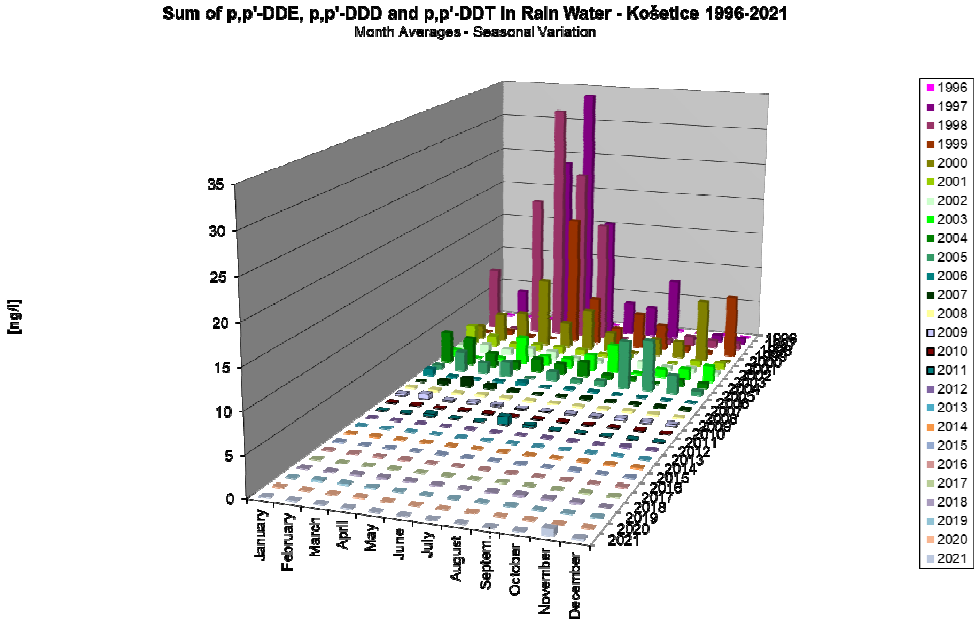
PAHs partition between particles and aerosol

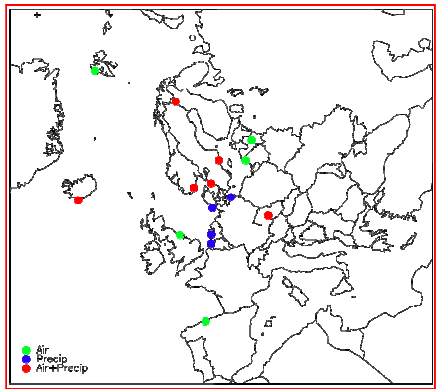
Atmospheric wet deposition

Trends in Pesticides and PAHs concentrations

- Continuously sampling
- *Analysis (PAHs, PCBs, OCPs)*
- Data used for EMEP





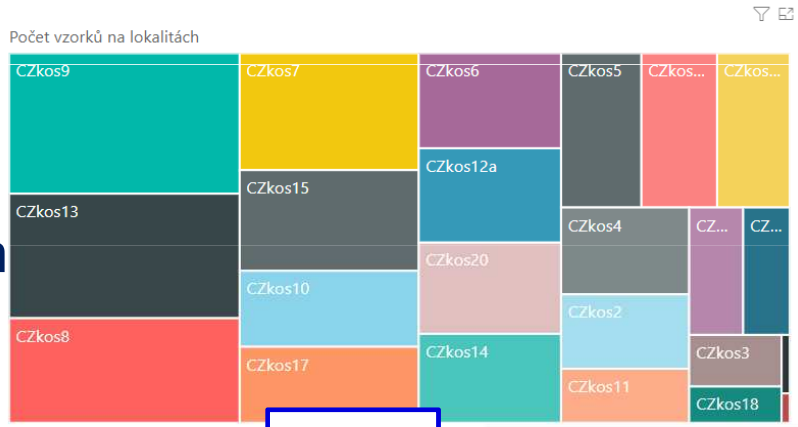


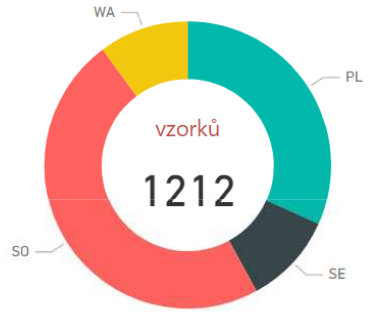
Biota monitoring like Bio-Passive air samplers

- Pine, spruce needles, moss
- Analysis (PAHs, PCBs, OCPs)
- Problem with the variability

of vegetation and data interpretation

2022





2018

Data usage

- **Air Data** are used in monitoring programs:

EMEP (European Monitoring Environmental programme,

UNEP (UN Environmental program with GMP like Global Monitoring Plan),

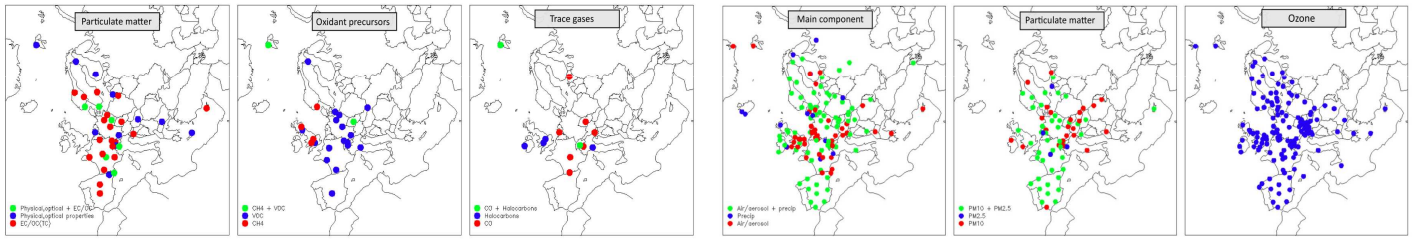
UNECE (UN Economic Commission for Europe with LRTAP),

MONET_CZ, MONET_EU, MONET_Africa (Monitoring Networks established by RECETOX)

ACTRIS (The Aerosol, Clouds and Trace Gases Research Infrastructure)

- **Water Data** are used in monitoring programs:

Norman water quality monitoring



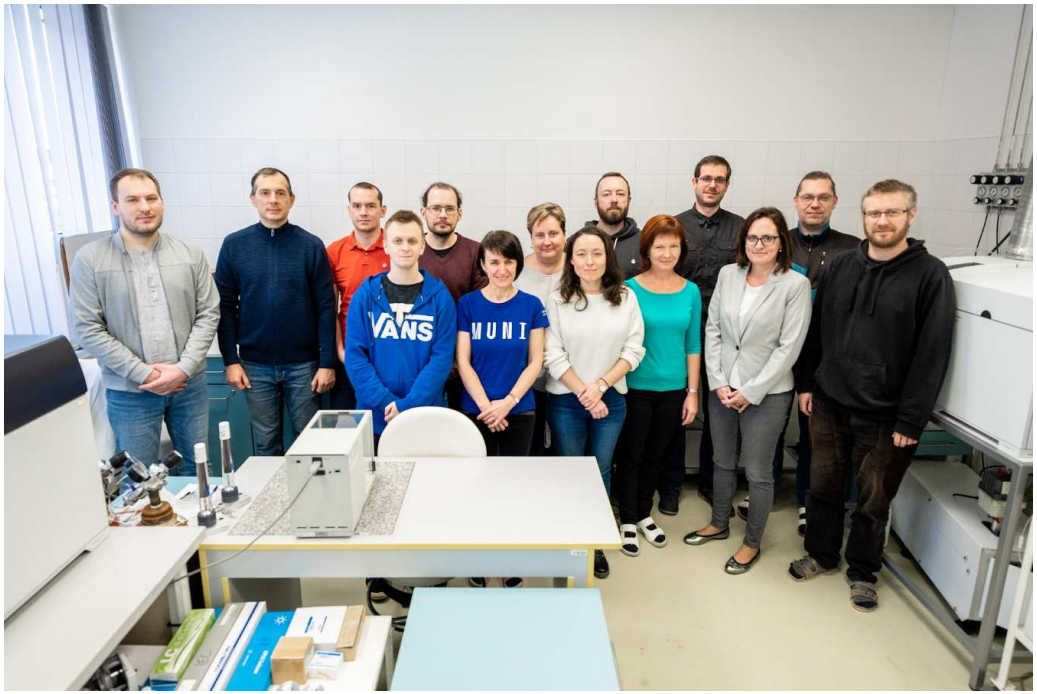
Stockholm and Basel Conventions Regional Centres - UNEP

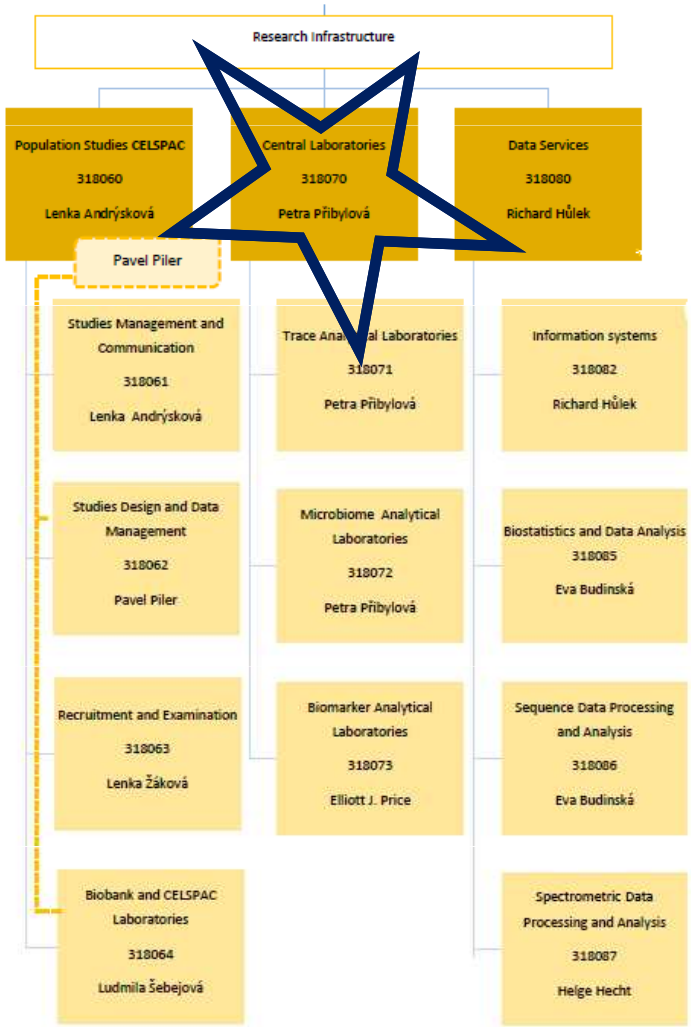
Technology transfer + capacity building;

RECETOX: SC Regional centre in the Central and Eastern European (CEE) region with the activities far beyond the region

Sample analysis – Central Laboratories

Trace Analytical Laboratories team



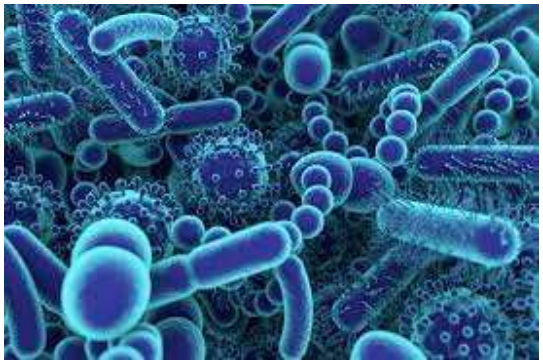


RECETOX Central Laboratories

- currently **the busiest part** of the RECETOX Research infrastructure
- **accredited according to European standards** for environmental sampling and a wide range of chemical analysis
- consist of three units

•**Trace Analytical Laboratories**
•Microbiome analysis
•Biomarker Analytical Laboratories

Accredited laboratories provide the analytical background for the research programmes of the RECETOX centre and provide the advanced analytical services to our research teams, as well as to external clients. The laboratories also realize long-term monitoring programs of various matrices (MONET EU, MONET Africa).



Microbiome laboratory provides the bacterial and mycobial profiles in various samples using the 16S rRNA gene and ITS gene sequencing. It also performs whole metagenome sequencing, in which all DNA in the sample is sequenced.

The biomarker analysis laboratory focuses on methods of targeted and non-targeted analyses of biologically important molecules (metabolites, proteins, lipids) as potential biomarkers of effects associated with chemical exposure.

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Trace Analytical Laboratories - accredited

L 1666

Equipped by: GC-MS/MS, GC-HRMS, LC-MS/MS, (LC)-ICP-MS/MS,

www.cai.cz

ilac-MRA

NÁRODNÍ AKREDITAČNÍ ORGÁN

EA MLA Signatory
Český institut pro akreditaci, o.p.s.
Olšanská 54/3, 130 00 Praha 3

issues

according to section 16 of Act No. 22/1997 Coll., on technical requirements for products, as amended

CERTIFICATE OF ACCREDITATION

No. 149/2022

Masarykova univerzita
with registered office Žerotínovo náměstí 617/9, 602 00 Brno, Company Registration No. 00216224

to the Testing Laboratory No. 1666
RECETOX: Trace Analytical Laboratories

Scope of accreditation:

Special trace and ultratrace analysis of organic compounds and elements in environmental samples, biotic samples, food and feed; sampling of air, surface water, soils, and bottom sediments to the extent as specified in the appendix to this Certificate.

This Certificate of Accreditation is a proof of Accreditation issued on the basis of assessment of fulfillment of the accreditation criteria in accordance with

ČSN EN ISO/IEC 17025:2018

In its activities performed within the scope and for the period of validity of this Certificate, the Body is entitled to refer to this Certificate, provided that the accreditation is not suspended and the Body meets the specified accreditation requirements in accordance with the relevant regulations applicable to the activity of an accredited Conformity Assessment Body.

This Certificate of Accreditation replaces, to the full extent, Certificate No.: 586/2019 of 8. 11. 2019, or any administrative acts building upon it.

The Certificate of Accreditation is valid until: 17. 3. 2027

Prague: 17. 3. 2022

Český institut pro akreditaci, o.p.s.

-3-

Lukáš Burda
Director of the Department
of Testing and Calibration Laboratories
Czech Accreditation Institute
Public Service Company

Accredited matrices:

- Outdoor / indoor air
- Surface water
- Sediments, soils
- Food, foodstuffs
- Cell tissues
- Human samples

Analytes:

- PCDDs/Fs, dl-PCBs, ind. PCBs
- PBDEs
- PAHs, OCPs
- PFCs
- metals

GC/MS, LC/MS, ICP-MS

Compounds analysed

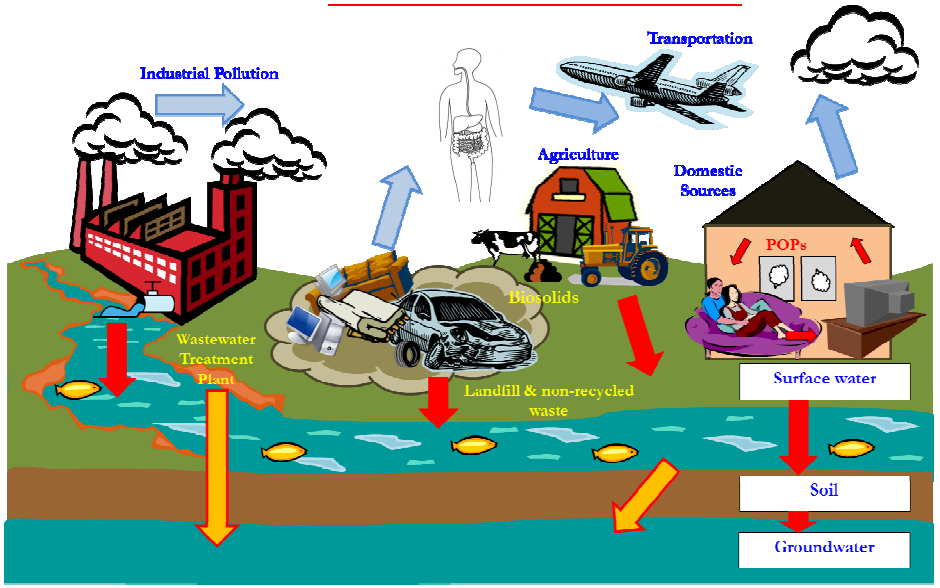
Organic pollutants:

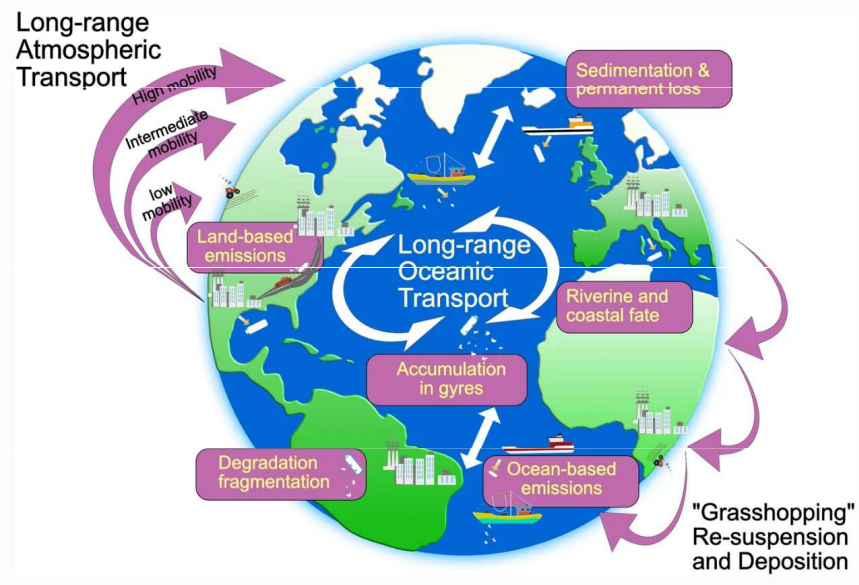
- Polychlorinated dibenzo-*p*-dioxins/furans (PCDDs/Fs)
- Polychlorinated biphenyls (PCBs) – indicator and dioxin-like
- Brominated and organophosphorus flame retardants
- Organochlorine, cyclodiene, and polar pesticides
- Polycyclic aromatic hydrocarbons (PAHs), NOx-, and oxy-PAHs
- Perfluorinated compounds (PFAS)
- Bisphenols
- Thyroid hormones
- UV filters – benzophenone
- Mycotoxins

Metabolites:

- OH-PAHs
- Phthalates metabolites + DINCH
- Pesticides metabolites

Trace elements, heavy metals, and species

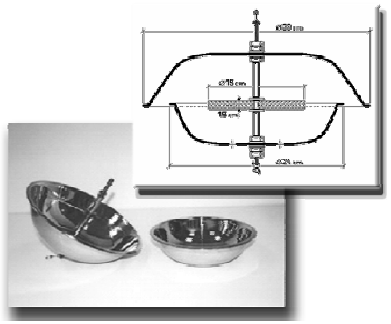


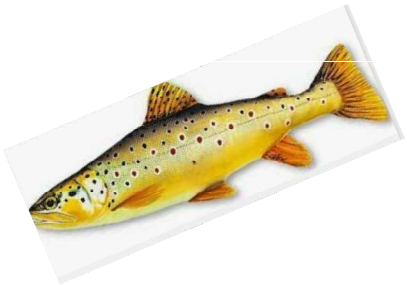


Gouin T., Microplastics and Nanoplastics, 2021 (14)

Matrices

- **Environmental matrices** – water, sediments, soils, air (indoor/outdoor, passive/active) – on XAD resin
- **Biotic matrices** - pine needles, moss, small animals
- **Materials** – micro/macro plastic, firefighter foam
- **Human matrices** – urine, serum, breast milk, hair





CETOX

Sample preparation

- Extraction of the samples (Automated hot solvent extractors – Büchi B-811, Soxhlet extraction, ultrasonic extraction, SPE, ASE, QuEChers)

- Analytical clean-up (adsorption column chromatography on the different sorbents, GPC)

Analysis

- GC-MS/MS, GC-HRMS techniques
- LC-MS/MS techniques
- (LC)-ICP-MS/MS techniques
- AAS

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Zatížení populací chemickými látkami

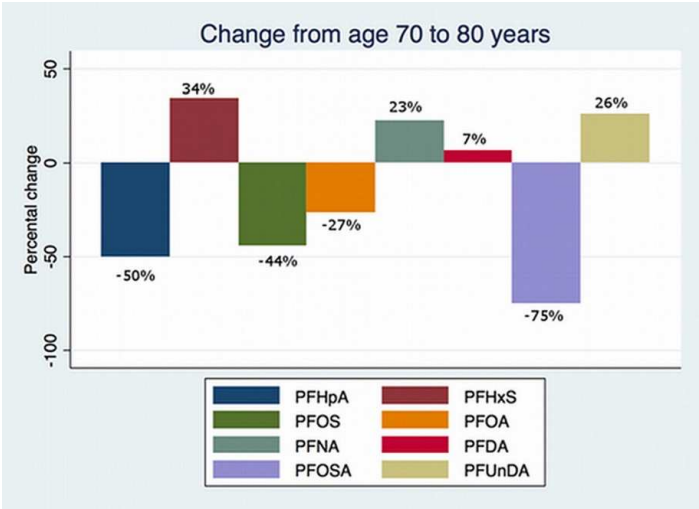
Petr Kukučka – laboratoř stopové analýzy

Chemické látky v lidských matricích

- Často residuální koncentrace různých organických, anorganických sloučenin (ať už z historické zátěže nebo pokračující expozice), lipofilní látky jsou vázány na tuk.
- Hlavní expoziční cesta je často příjem potravním řetězcem (jídlo, nápoje).
- Koncentrace látek v lidské krvi, při metabolitech v moči.

Persistentní organické polutanty

- (Poly)halogenované sloučeniny (aromáty, alifáty)
- Perzistence
- „Grasshopper effect“
- Lipofilita
- Schopnost bioakumulace



Stubleski, S. Salihovic, L. Lind, P.M. Lind, B. van Bavel, A. Kärrman
Changes in serum levels of perfluoroalkyl substances during a 10-year follow-up period in a large population-base cohort
Environ. Int., 95 (2016), pp. 86-92, 10.1016/j.envint.2016.08.002

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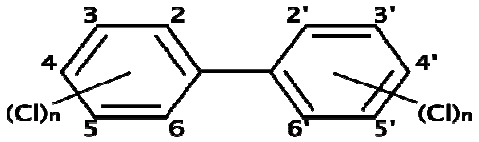
Persistentní organické polutanty

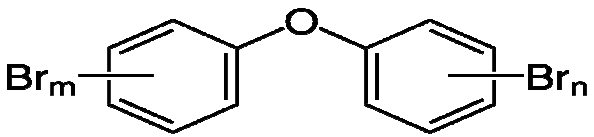
- Expoziční cesty – **GI trakt**, respirační systém, dermální kontakt
- Endokrinní disruptory
- Karcinogeny
- Kongenery – obtížné omezení použití
- Stockholm convention 2001 omezení POPs (celkem 12)

Persistentní organické polutanty

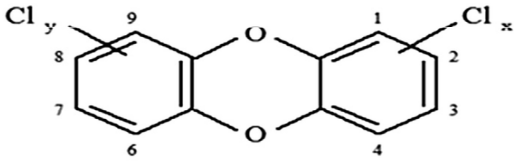
PCB



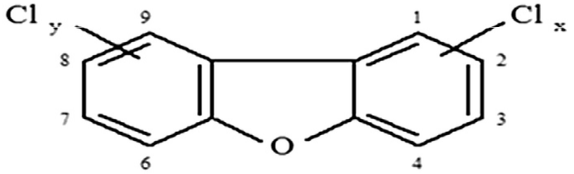
PBDE



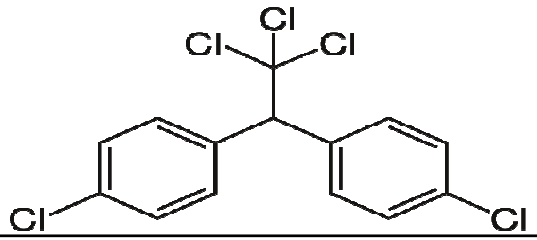
PCDD



PCDF



DDT



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PCBs

- 1929 - 1979 (byly zakázány)
- SOUČÁSTÍ elektrických či topných zařízení – kapacitátorů a transformátorů
- Hydraulických tekutin
- Změkčovadel = aditiva do barev či laků
- Plastů
- Bezuhlíkového kopírovacího papíru
- Vypínačů, izolace kabelů

PCBs

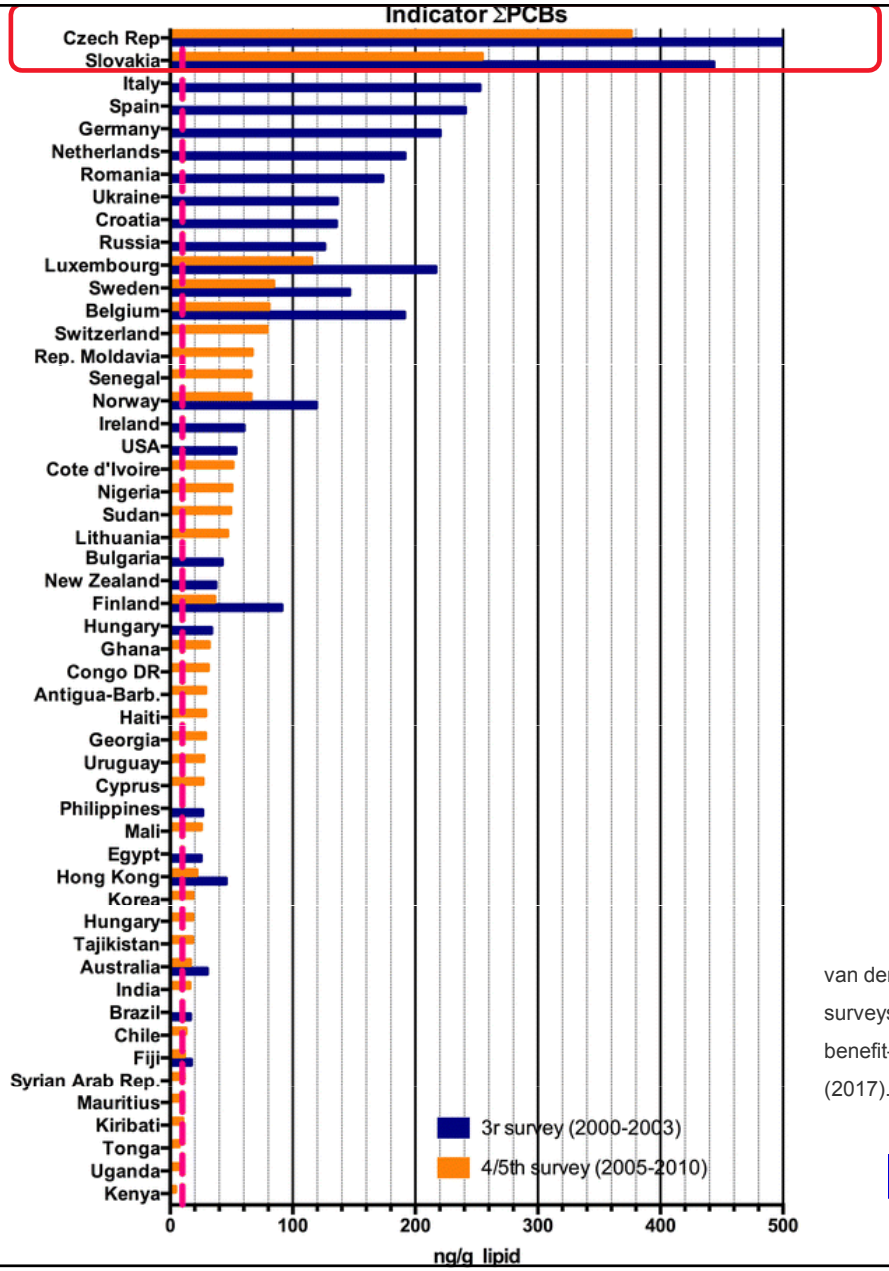
- V 2. polovině 20. stol. – detekovány PCBs u zvířat
- Únik do atmosféry:
- Špatné zacházení s odpadem obsahujícím PCBs
- Z transformátorů
- Při pálení materiálů obsahujících PCBs

PCBs

- Detekovány v tkáních – vrcholných predátorů, v rybách, také v mléce, mase, másle či vejcích
- Studie na zvířatech prokázaly vliv na imunitní, hormonální či rozmnožovací sst.
- Prokázaná karcinogenita u zvířat
- Přenos mateřským mlékem – zpomalený mentální vývoj dítěte

PCBs

- WHO šetření
- mateřské mléko



van den Berg, M., Kypke, K., Kotz, A. *et al.* WHO/UNEP global surveys of PCDDs, PCDFs, PCBs and DDTs in human milk and benefit–risk evaluation of breastfeeding. *Arch Toxicol* **91**, 83–96 (2017). https://doi.org/10.1007/s00204-016-1802-z

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BFRs

- Polybromované difenylethery(PDBEs)
- Perzistentní, lipofilní a bioakumulativní látky
- Zabraňují hoření materiálů a zpomalují progresi požáru
- Vznikají přímou bromací difenyl etheru – 209 možných kongenerů – komerčně technické směsi
- Ve mnoha zemích již byly zakázány

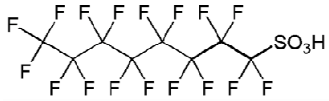
PBDEs

- U zvířat je prokázáno, že tyto látky mají vliv na:
- Vývoj nervové soustavy – neurotoxikanty – u myší došlo ke zhoršenému vývoji motorických schopností (McDonald, 2001)
- Hormonální systém - endokrinní disruptory – struktura PDBEs je podobná thyroidním hormonům
- Rozvoj rakoviny – u myší a krys, které byly exponovány směsi PDBEs, došlo ke zvýšení oxidativního stresu a změnám v hormonální homeostáze

DDT a metabolity

- 1,1,1-trichlor-2,2-bis(4-chlorfenyl)ethan
- Poprvé vyrobeno na konci 19. stol.
- Ochrana před malárií a tyfem – používáno během 2. sv. války
- V Africe proti malárii používáno dodnes
- Patří mezi endokrinní disruptory, má několik metabolitů – DDE a DDD
- Toxické efekty: ovlivněna plodnost: malformace pohl. orgánů., tenké skořápky u ptáků atd.

PFAS



- široká skupina poly- a perfluorovaných látek
- vazba C-F velmi silná, těžko rozložitelná, velmi perzistetní
- hydrofobní a oleofobní vlastnosti
- dobrovolné ukončení použití po roce 2000, v roce 2009 zákaz různými úmluvami, náhradní produkty
- imunotoxicita

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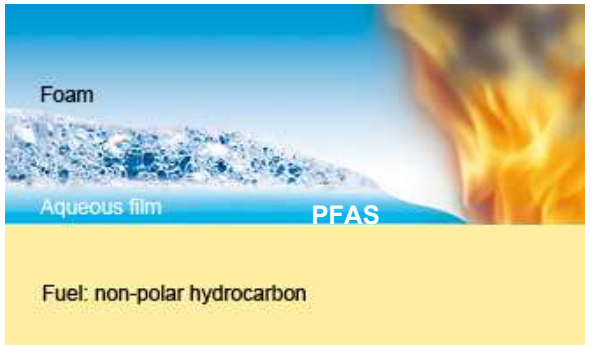
PFAS v hasících pěnách

- Aqueous film forming foams (AFFFs), PFAS snižují povrchové napětí vody (lepší rozptyl pěny na povrchu hořící kapaliny), tepelně odolný – stabilní film
- většinou pro hašení kapalin (hořlavin, např. paliv)
- značná expozice ŽP, kontaminované lokality – cvičiště hasičů, letiště, vojenské letecké základny
- pracovní expozice hasičů AFFFs (vdechnutí aerosolu, dermalní kontakt)
- v některých zemích již zakázané (PFOS), nicméně stále existují fluorované alternativy



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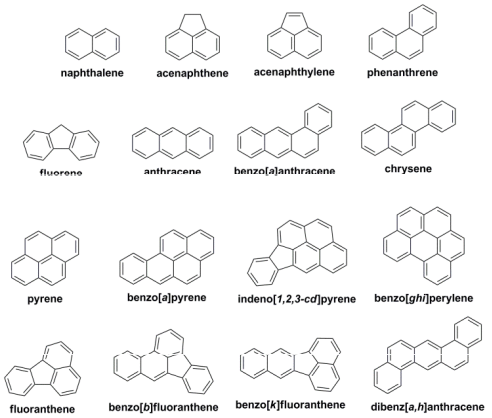
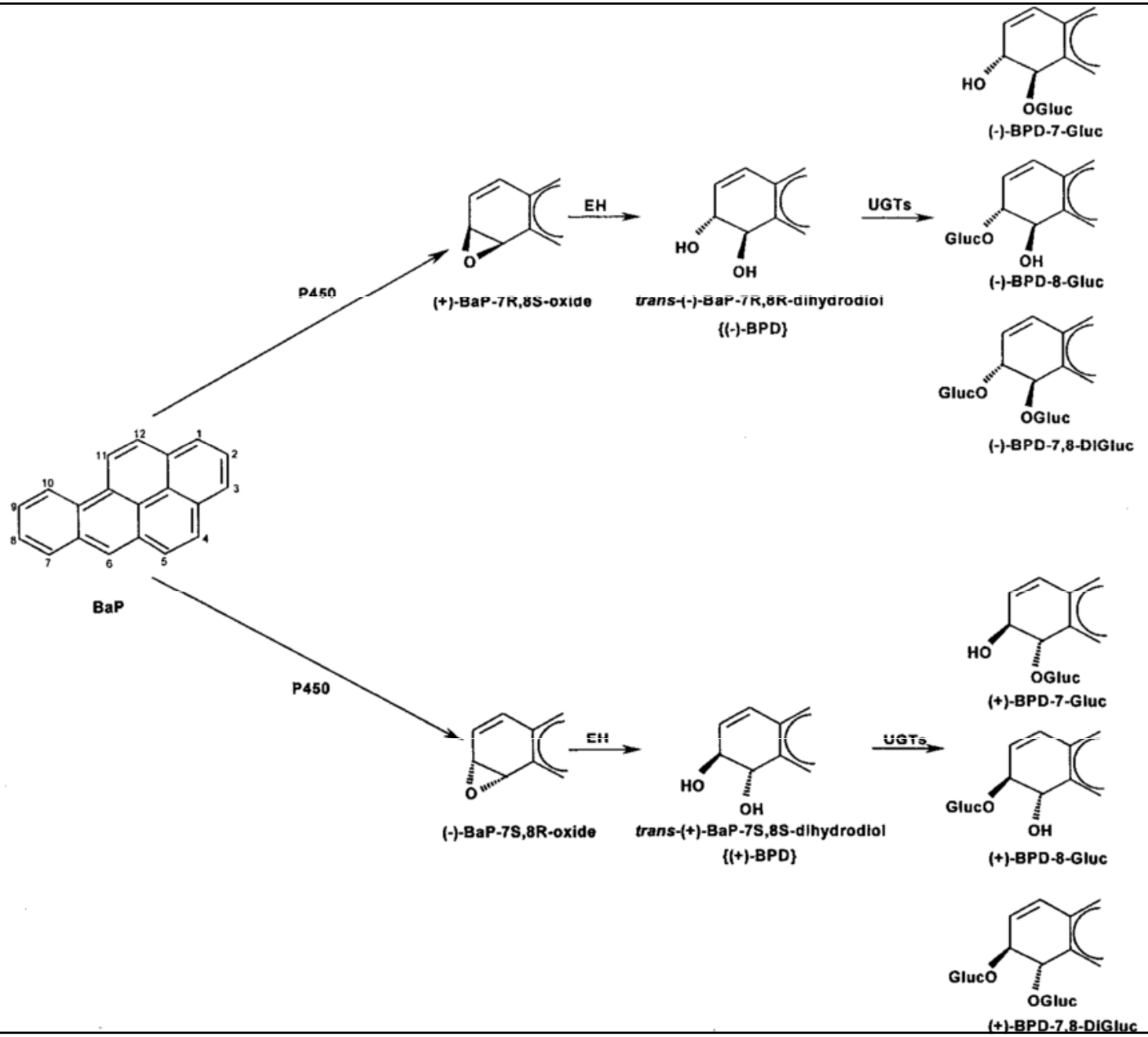
PAHs



- polykondezované aromáty
- všudypřítomné, v prostředí relativně stabilní
- produkty nedokonalého hoření, nebo petrogenní původ
- uzené maso, kouřem sušené ovoce, pracovní expozice (asfaltování, stavební práce)
- podezřelé nebo prokázané karcinogeny – po metabolické aktivaci

Metabolizace BaP



Analýza POPs v krvi

- obvykle nativní sloučeniny (nemetabolizované)
- Náročnost matrice (naše analyty jsou malé molekuly): proteiny, lipidy, sacharidy atd.
- Extrakční metody musí pokrývat široký rozsah analytů
- Předčišťovací postupy pro odstranění hlavních interferentů při instrumentální analýze: proteiny, lipidy, sacharidy atd.
- Často omezené množství dostupného vzorku vs. stanovení širokého rozsahu analytů, často v nízkých koncentracích

Analýza POPs v krvi

LLE: jednoduchá, levná, pracná pro širší sady vzorků, nutnost dodatečných přečištění, snadná modifikace

Contents lists available at ScienceDirect

Journal of Chromatography A

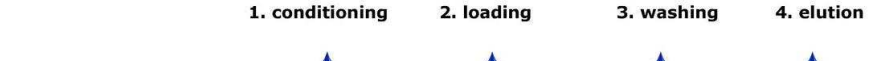
journal homepage: www.elsevier.com/locate/chroma

Determination of selected perfluorinated alkyl acids and persistent organic pollutants from a small volume human serum sample relevant for epidemiological studies

Jani Koponen*, Panu Rantakokko, Riikka Airaksinen, Hannu Kiviranta

National Institute for Health and Welfare, Department of Environmental Health, P.O. Box 95, FI-70701 Kuopio, Finland

SPE: potřeba speciálního zařízení, vyšší náklady, pracná pro širší sady vzorků, nutnost dodatečných přečištění, omezený výběr sorbentů pro SPE



Contents lists available at SciVerse ScienceDirect

Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

A rapid method for screening of the Stockholm Convention POPs in small amounts of human plasma using SPE and HRGC/HRMS

Samira Salihovic[a,*], Lisa Mattioli[a], Gunilla Lindström[a], Lars Lind[b], P. Monica Lind[c], Bert van Bavel[a]

[a] MTM Research Centre, School of Science and Technology, Örebro University, SE-701 82 Örebro, Sweden
[b] Department of Medical Sciences, Acute and Internal Medicine, Uppsala University Hospital, SE-751 85 Uppsala, Sweden
[c] Department of Medical Sciences, Occupational and Environmental Medicine, Uppsala University Hospital, SE-751 85 Uppsala, Sweden

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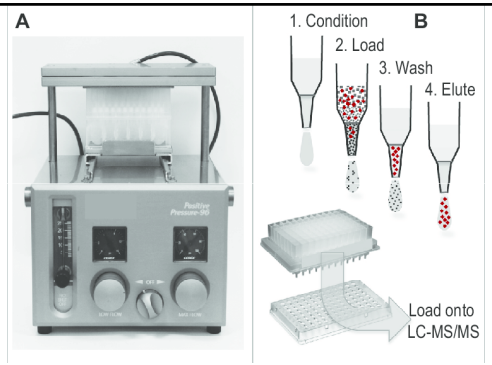
Deskové SPE



– 96 jamkové SPE desky: rychlé a jednoduché, vyšší náklady, nutnost zvláštního vybavení, nízké objemy vzorků (<250 µL)

– lze zpracovat cca 88 vzorků najednou (+ QCs)

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

A method for analysis of marker persistent organic pollutants in low-volume plasma and serum samples using 96-well plate solid phase extraction

Jordan Stubleski[a,*], Petr Kukucka[b], Samira Salihovic[a,c], P. Monica Lind[d], Lars Lind[e], Anna Kärrman[a]

[a] MTM, School of Science and Technology, Örebro University, 701 82 Örebro, Sweden
[b] RECETOX, Masaryk University, Kamenice 5, 62500 Brno, Czech Republic
[c] Department of Medical Sciences, Molecular Epidemiology and Science for Life Laboratory, Uppsala University, 75141 Uppsala, Sweden
[d] Department of Medical Sciences, Occupational and Environmental Medicine, Uppsala University, Uppsala, Sweden
[e] Department of Medical Sciences, Cardiovascular Epidemiology, Uppsala University, Uppsala, Sweden

Contents lists available at SciVerse ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

A rapid method for the determination of perfluoroalkyl substances including structural isomers of perfluorooctane sulfonic acid in human serum using 96-well plates and column-switching ultra-high performance liquid chromatography tandem mass spectrometry

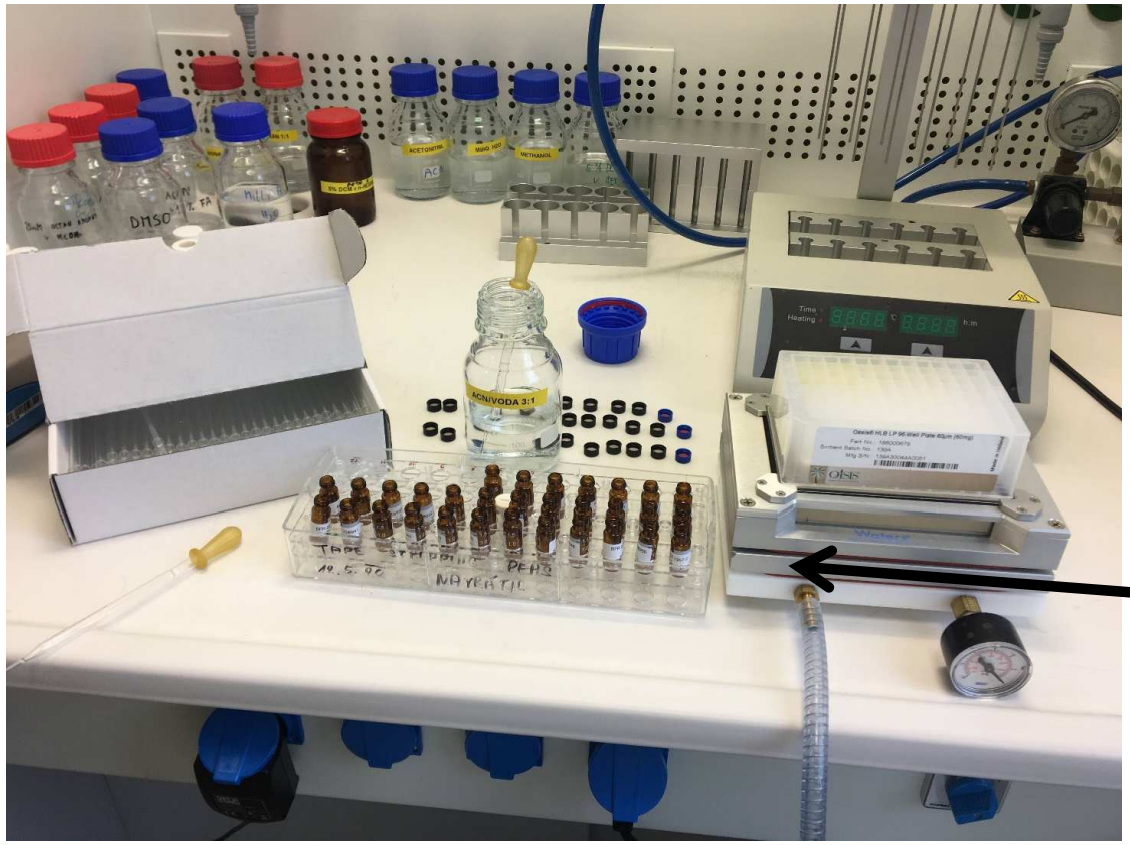
Samira Salihovic[a,*], Anna Kärrman[a], Gunilla Lindström[a], P. Monica Lind[b], Lars Lind[c], Bert van Bavel[a]

[a] MTM Research Centre, School of Science and Technology, Örebro University, Örebro, Sweden
[b] Occupational and Environmental Medicine, Uppsala University, Uppsala, Sweden
[c] Acute and Internal Medicine, Uppsala University, Uppsala, Sweden

Příprava vzorků

- kondicionace SPE desk (Waters Oasis HLB 60 mg/well, 60 µm)
- předčišťovací deska (Phenomenex Phree phospholipid removal plate, 30 mg/well, *genericky označena jako* C18)
- analyty: PCBs, OCPs, BFRs, PAHs, PFRs
- krevní sérum, NIST SRM 1957, 1958(nativní a obohacené krevní sérum)

Přenos vzorků na HLB desku



Vysrážení proteinů krevní plasmy
Pomocí acetonitrilu

Vialky
se vzorky krevní plasmy/séra

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Pracovní postup

150 až 250 µL objem vzorku, obohacení směsí vnitřních standardů (roztoky v IPA)

vysrážení krevních proteinů: 3x objem vzorku ACN

HLB deska kondicionovaná

SPE podtlakový manifold

Příprava vzorků

Klíčové je pro analýzu PCBs a pod odstranění vlhkosti z deskyHLB

…15 min centrifuga (4300 rpm), profuk 30 min N2

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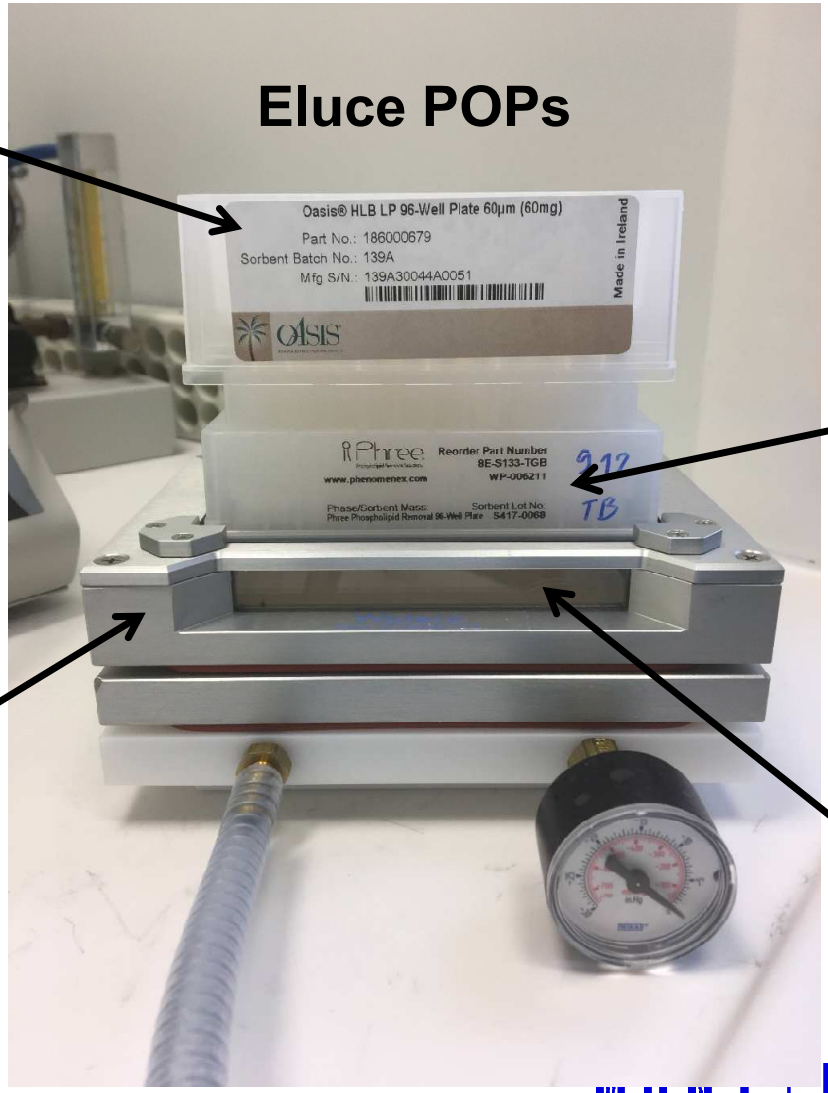
Eluce POPs

Vysušená HLB deska se vzorky



Phree deska

Vakuový manifold

Eluce do skleněných inzertů

MONI | RECETOX

Příprava vzorku

Eluce a přečištění, Na2SO4 do Phree desky, HLB deska polona na Phree, 1.2 mL *n*-hexane/DCM, převod doGC vialk, přídavek recovery standardů, N2 odpaření, GC-MS anlaýza

Stanovení PFAS

- vysrážení proteinů (Phenomenex Phree), matricová kalibrace, ^{13}C vnitřní standardy(Wellington labs)

- UPLC-ESI-MS/MS analýza

- ANlayty:
  - PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, PFTDA
  - PFBuS, PFHxS, PFOS (incl. branched isomers), PFDS, PFOSA

Průtočnost laboratoří, obecně

příprava vzorků (SPE) 1-2 dny pro 88 vzorků

instrumentální analýza 88 vzorků na 2 skupiny analytů 5 dnů

Stanovení prvků a specií

Stanovení toxických a esenciálních prvků v plasmě nebo moči metodou ICP-MS

Toxické a esenciální prvky – vztah k plodnosti

V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Cd, Sn, Sb, Hg a Pb

Příprava: pouze 10x ředění do Triton X-100 (0,04%), amoniak (1%), butanol (2%),

EDTA (0,04%) + vnitřní standardy (Sc, Ge, In, Lu, Bi)

Detekce: Agilent 8900 ICP-MS/MS, pouze He SQ mód

QA/QC CRM Seronorm Serum Level 1 + Level 2

Výhody oproti mineralizaci:

Malá spotřeba vzorku (0,2 ml)

Nízké riziko kontaminace

Produktivita – 100 vzorků/8 h

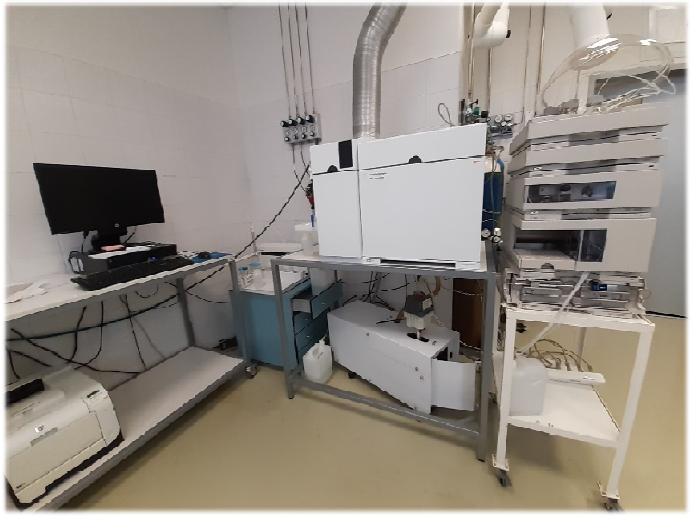
Stanovení prvků a specií

ICP-MS

- 7500ce ICP-MS (2004 – 2020)
- 7700x ICP-MS (2012 – dosud)
- 8900 ICP-MS/MS (2020 – dosud)

Separační techniky

- Bio-inert 1260 Infinity II HPLC (2020 – dosud)
- 1100 HPLC (2004 – dosud)
- 6890N GC (2004 – dosud)

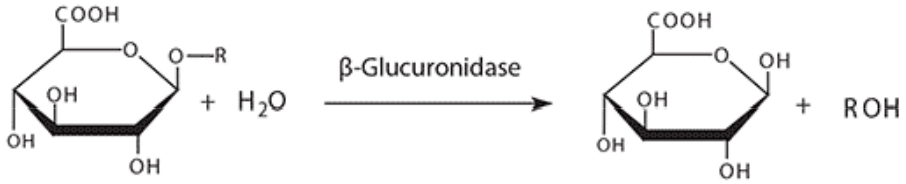


Analýza polutantů v moči

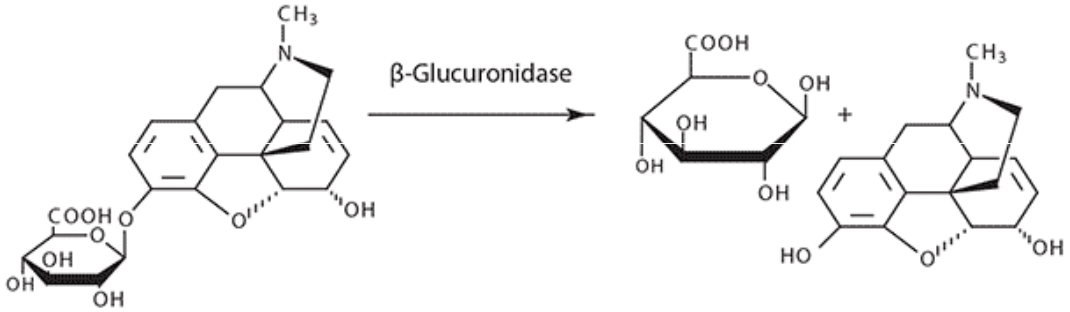
– dekojugace glukuronidů (promíchání vzorků s roztokem enzymu, inkubace za vhodné teploty, třepání po vhodnou dobu)
– úprava pH, použití vhodného SPE sorbentu (ionexy), dle povahy analytu (kationty, anionty, neutrální)
– obohacení značenými vnitřními standardy a extrakce na SPE, poté eluce a analýza UPLC-MS/MS

Dekonjugace

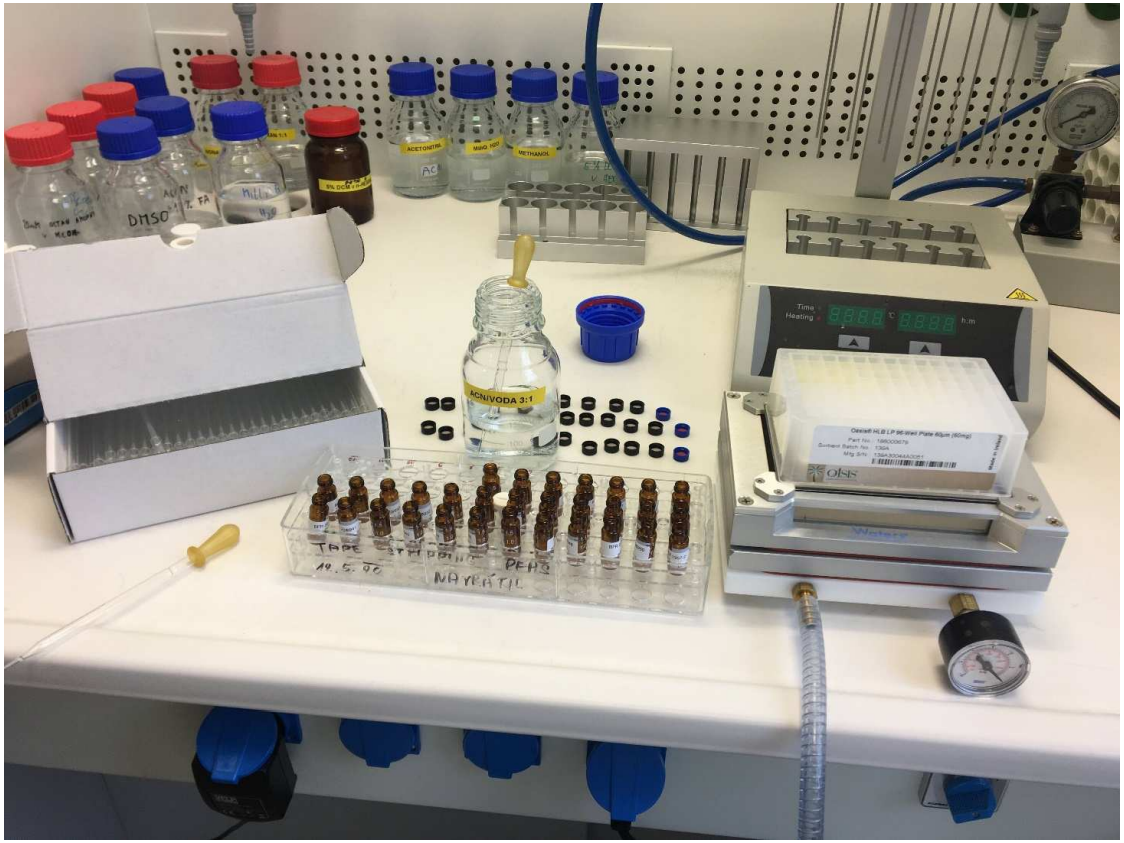
General β-Glucuronidase Hydrolysis Reaction



β-Glucuronidase Hydrolysis Reaction for Morphine 3-β-glucuronide



Příprava vzorků



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Shrnutí

- pracovní postupy s velkou průtočností vzorků (časová náročnost především instrumentální analýza)
- malé použité objemy vzorků, QAQC
- metody pro dlouhodobý biomonitoring – časové sledování expozice populací

Díky za pozornost!

Publications

Top publications from last years

- **Sobotka, J., Smedes, F., & Vrana, B.** (2022). Performance comparison of silicone and low-density polyethylene as passive samplers in a global monitoring network for aquatic organic contaminants. Environmental Pollution, 302, 119050. https://doi.org/10.1016/j.envpol.2022.119050
- **White, K.B.; Sanka, O.; Melymuk, L.; Pribylova, P.; Klanova, J.** Application of land use regression modelling to describe atmospheric levels of semivolatile organic compounds on a national scale. Science of The Total Environment (2021), 793, Article Number: 148520.
- **White, K. B.; Kalina, J.; Scheringer, M.; Pribylova, P.; Kukucka, P.; Kohoutek, J.; Prokes, R.; Klanova, J.** Temporal Trends of Persistent Organic Pollutants across Africa after a Decade of MONET Passive Air Sampling. Environmental Science & Technology (2021), 55(14), 9413-9424, SI.
- **Smedes, F.; Sobotka, J.; Rusina, T. P.; Fialova, P.; Carlsson, P.;** Kopp, R.; **Vrana, B.** Unraveling the Relationship between the Concentrations of Hydrophobic Organic Contaminants in Freshwater Fish of Different Trophic Levels and Water Using Passive Sampling. Environmental Science & Technology (2020), 54 (13), 7942-7951.
- **van der Schyff, V.; du Preez, M.; Blom, K.; Kwet Yive, N. S. Ch.; Klanova, J.; Pribylova, P.; Audy, O.; Martinik, J.; Bouwman, H.** Chlorinated and brominated persistent compounds in hard coral, soft coral, and parrotfish from remote Mascarene islands. Chemosphere (2021), 267, Article Number: 129316.

Top publications in cooperation with the South America

- **Pozo, K., Moreira, L. B., Karaskova, P., Přibylová, P., Klánová, J., de Carvalho, M. U., Maranho, L. A., & de Souza Abessa, D. M. (2022).** Using large amounts of firefighting foams releases per- and polyfluoroalkyl substances (PFAS) into estuarine environments: A baseline study in Latin America. Marine Pollution Bulletin, 182, 113938. https://doi.org/10.1016/j.marpolbul.2022.113938
- **Corsolini, S.; Metzdorff, A.; Baroni, D.; Roscales, J. L.; Jimenez, B.; Cerro-Galvez, E.; Dachs, J.; Galban-Malagon, C.; Audy, O.; Kohoutek, J.; Pribylova, P.; Poblete-Morales, M.; Avendano-Herrera, R.; Bergami, E.; Pozo; K.** Legacy and novel flame retardants from indoor dust in Antarctica: Sources and human exposure. Environmental Research (2020), online. DOI: 10.1016/j.envres.2020.110344.
- **Corsolini, S.; Metzdorff, A.; Baroni, D.; Roscales, J. L.; Jimenez, B.; Cerro-Galvez, E.; Dachs, J.; Galban-Malagon, C.; Audy, O.; Kohoutek, J.; Pribylova, P.; Poblete-Morales, M.; Avendano-Herrera, R.; Bergami, E.; Pozo; K.** Legacy and novel flame retardants from indoor dust in Antarctica: Sources and human exposure. Environmental Research (2021), 196, Article Number: 110344.
- **Pozo, K., Gómez, V., Přibylová, P., Lammel, G., Klánová, J., Rudolph, A., & Ahumada, R. (2022).** Multicompartmental analysis of POPs and PAHs in Concepción Bay, central Chile: Part I – Levels and patterns after the 2010 tsunami. Marine Pollution Bulletin, 174, 113144. https://doi.org/10.1016/j.marpolbul.2021.113144
- **Pozo, K., Gómez, V., Tucca, F., Galbán-Malagón, C., Ahumada, R., Rudolph, A., Klánová, J., & Lammel, G. (2022).** Multicompartmental analysis of POPs and PAHs in Concepción Bay, central Chile: Part II – Air-sea exchange during Austral summer. Marine Pollution Bulletin, 177, 113518. https://doi.org/10.1016/j.marpolbul.2022.113518

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List of publications (ACTRIS RI, RECETOX RI)

- The most impacted publications in last 5 years

Kalina, J.; Scheringer, M.; Boruvkova, J.; Kukucka, P.; Pribylova, P.; Sanka, O.; Melymuk, L.; Vana, M.; Klanova, J. Characterizing spatial diversity of passive sampling sites for measuring levels and trends of semivolatile organic chemicals. Environmental Science & Technology (2018), 52 (18), 10599-10608.	6,653
Kalina, J.; Scheringer, M.; Boruvkova, J.; Kukucka, P.; Pribylova, P.; Bohlin-Nizzetto, P.; Klanova, J. Passive Air Samplers As a Tool for Assessing Long-Term Trends in Atmospheric Concentrations of Semivolatile Organic Compounds. Environmental Science & Technology (2017), 51 (12), 7047-7054.	6,198
Nezikova, B.; Degrendele, C.; Bandowe, B. A. M.; Holubova Smejkalova, A.; Kukucka, P.; Martinik, J.; Mayer, L.; Prokes, R.; Pribylova, P.; Klanova, J.; Lammel, G. Three years of atmospheric concentrations of nitrated and oxygenated polycyclic aromatic hydrocarbons and oxygen heterocycles at a Central European background site. Chemosphere (2021), 269, Article Number: 128738.	5,778
Degrendele, C.; Wilson, J.; Kukucka, P.; Klanova, J.; Lammel, G. Are atmospheric PBDE levels declining in central Europe? Examination of the seasonal and semi-long-term variations, gas-particle partitioning and implications for long-range atmospheric transport. Atmospheric Chemistry and Physics (2018), 18 (17), 12877-12890.	5,509
Degrendele, C.; Fiedler, H.; Kocan, A.; Kukucka, P.; Pribylova, P.; Prokes, R.; Klanova, J.; Lammel, G. Multiyear levels of PCDD/Fs, dl-PCBs and PAHs in background air in central Europe and implications for deposition. Chemosphere (2020), 240, Article Number: UNSP 124852.	5,108
Kalina, J., White, K. B., Scheringer, M., Přibylová, P., Kukučka, P., Audy, O., Martiník, J., & Klánová, J. (2022). Comparability of semivolatile organic compound concentrations from co-located active and passive air monitoring networks in Europe. Environmental Science: Processes & Impacts, 24(6), 898–909. https://doi.org/10.1039/D2EM00007E	4,238

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