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Perfluorinated Chemicals, especially Perfluorinated Alkyl Sulfonates and Carboxylates: European Distribution and Legislation



Robert LOOS

Robert.Loos@jrc.ec.europa.eu

Directorate H - Institute for Environment and Sustainability
Water Resources Unit (H.01)

Ispira, Italy

Introduction

Acronyms, Use of PFASs, Sources / Precursors / Emissions, Contamination

Legislation

Voluntary phase-out, Restriction of PFOS in Europe, Stockholm Convention, WFD, Directive 2013/39/EU

Occurrence

General, Surface waters, European Hotspots, Rhine River, The Sauerland Case, Groundwater, Tap / Drinking Water

JRC EU-wide Monitoring Surveys

Surface waters, Po River, Danube River, Groundwater, PFOS Modelling, Adriatic Sea

Conclusions

Acronyms

- Term “**PFCs**” was used for substances such as **PFOS or PFOA**.
- Term “**perfluorinated chemicals**” includes a wide range of chemicals with varied uses, characteristics and environmental profiles.
- “**PFCs**”: first used to describe **perfluorocarbons**, a family of **greenhouse gases** (CF₄), in official Kyoto Protocol documents.
- Acronym “**PFASs**” (singular **PFAS**) should be used for “**perfluoroalkyl and polyfluoroalkyl substances**” such as perfluorooctane sulfonate (**PFOS**) and perfluorooctanoic acid (**PFOA**).

Buck, R.C., Franklin, J., Berger, U., Conder, J.M., Cousins, I.T., de Voogt, P., Astrup Jensen, A., Kannan, K., Mabury, S.A., van Leeuwen, S.P.J. *Perfluoroalkyl and polyfluoroalkyl substances in the environment: Terminology, classification, and origins*. *Integrated Environmental Assessment and Management* 7(4) (2011) 513–541.

- Since 1950, **PFASs, surfactants and polymers** made with the aid of PFASs have been widely used in numerous industrial and commercial applications.
- C–F bond is extremely strong and stable.
- **Chemical and thermal stability** of a perfluoroalkyl moiety, in addition to its **hydrophobic and lipophobic nature**, lead to highly useful and enduring properties in **surfactants and polymers**.
- **Polymer applications** include **textile repellents, stain-resistant coatings for carpets and grease-proof food-contact paper**.
- **Surfactant applications** that take advantage of the aqueous surface tension–lowering properties include **processing aids for fluoropolymer manufacture**, and **aqueous film–forming foams (AFFFs)** used to extinguish fires.

Use of PFASs



PFOS uses and potential sources

Firefighting foams



Water and oil repellents (impregnation) for carpets, leather, paper, textiles, food packaging



Inks, varnishes, waxes, metal plating and cleaning, coating formulations, lubricants, photography, photolithography, and semiconductor industries, hydraulic fluids

PFOS deliberately manufactured, present as an impurity and potentially as a degradation by-product

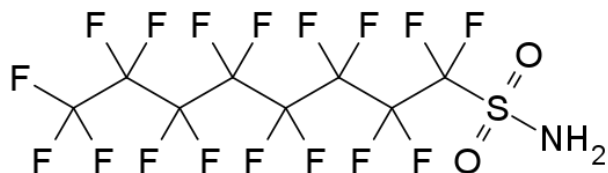
Sources / Precursors

- Widespread use of **industrial raw materials and consumer products and articles.**
- Contain **PFASs** (also as impurities)
- Perfluoroalkyl moiety in polymers or precursors degrades in the environment to form **PFOS, PFOA**, and similar substances.
- **Precursor substances:**
 - Perfluorooctane sulfonamides (PFOSA)
 - N-ethyl perfluorooctane sulfonamidoethanol (NEt-FOSE)
 - Fluorotelomer alcohols (FTOHs)
 - Polyfluoroalkyl phosphate esters (PAPs) (phosphoric acids)

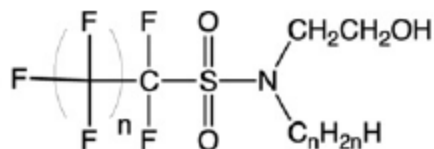
PFOA



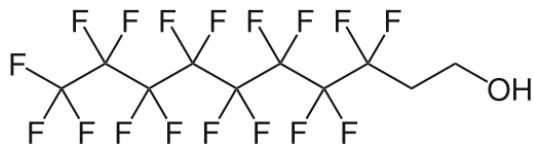
PFOS



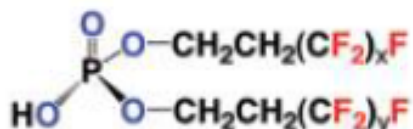
Perfluorooctanesulfonamide (PFOSA) was an ingredient in 3M's former Scotchgard stain protection formulation.



N-ethyl Perfluorooctane sulfonamidoethanol (NEtFOSE)



8:2 Fluorotelomer alcohol: volatile precursor of PFACs



Polyfluoroalkyl phosphate esters (PAPs) (phosphoric acids)

Sources and Emissions

- **Main sources of PFASs to the aquatic environment are **municipal and industrial wastewater treatment plants.****
- **Direct discharges**
- **Landfill leachates**
- **PFOA:** released directly from fluoropolymer production plants.
- **Indirect emissions:** caused by atmospheric degradation of the **precursor compounds.**

Worldwide Contamination

- **Widespread use of PFASs / Emissions.**
- **Broad range of these substances have been detected in the environment, wildlife, and humans.**
- **The global extent of this contamination was first demonstrated for PFOS in wildlife (fish, birds, and marine mammals) by Giesy and Kannan (2001).**
- **Human blood by Hansen et al. (2001).**
- **PFASs: ubiquitously found in the (aqueous) environment.**

Giesy JP, Kannan K. *Global distribution of perfluorooctane sulfonate in wildlife.* *Environ. Sci. Technol.* 35 (2001) 1339–1342.
Hansen KJ, et al. *Compound-specific, quantitative characterization of organic fluorochemicals in biological matrices* *Environ. Sci. Technol.* 35 (2001) 766-770.

Voluntary phase-out of PFOS / PFOA production

Concerns about the potential **environmental and toxicological impact** of long-chain PFASs:

- 1) **The voluntary phase-out of production of PFOS** and related compounds by their major global manufacturer in 2000 (3M Company).
- 2) **Stewardship agreement** launched in 2006 between the USEPA and 8 leading global companies to **reduce emissions and product content of PFOA** and related chemicals by 95% by 2010 and to work toward their elimination by 2015.

Legislation in Europe

- **2006: European Union Marketing and Use Directive** restricting the use of “perfluorooctane sulfonates” in the European Union.
- **2009: Inclusion of PFOS in the Stockholm Convention** on Persistent Organic Pollutants as an Annex B substance, i.e., restricted in its use.
- **2013: PFOS becomes priority substance** under the WFD.



DIRECTIVE 2006/122/ECOF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL

of 12 December 2006

amending for the 30th time Council Directive 76/769/EEC on the approximation of the laws, regulations and administrative provisions of the Member States relating to restrictions on the marketing and use of certain dangerous substances and preparations (perfluorooctane sulfonates)

An OECD (Organisation for Economic Cooperation and Development) hazard assessment has been done on the basis of information that was available by July 2002. This assessment concluded that perfluorooctane sulfonates (hereinafter 'PFOS') are persistent, bioaccumulative and toxic to mammalian species and, therefore, indicate cause for concern.

The risks posed to health and environment by PFOS have been assessed in accordance with the principles of Council Regulation (EEC) No 793/93 of 23 March 1993 on the evaluation and control of the risks of existing substances⁽³⁾. The risk assessment identified a need to reduce the risks to health and the environment.

In order to protect health and the environment, it therefore appears necessary that the placing on the market and the use of PFOS should be restricted. This Directive is intended

- **Industries which cannot operate without PFOS are bound to use the **best available techniques (BAT)** to reduce emissions to the environment.**
- **Consumer products and articles may not contain more than 0.1% of PFOS.**

COMMISSION RECOMMENDATION
of 17 March 2010
on the monitoring of perfluoroalkylated substances in food
(Text with EEA relevance)
(2010/161/EU)

- Scientific opinion of **EFSA** considered it unlikely that **adverse effects of PFOS and PFOA** are occurring in the general population.
- Uncertainties with regard to **developmental effects** in living organisms.
- EFSA recommended that further data on **PFASs levels in food** and in humans would be desirable.

Directive 2013/39/EU – Priority Substances

Perfluorooctansulfonic acid (PFOS)



EQS: 0.65 ng/L
(0.13 ng/L)

Biota: 9.1 µg/kg

- **Expert laboratories can achieve LOQs of 0.1 ng/L**
- **Biota EQS (9.1 µg/kg) is more easy to achieve**



- Central and south European rivers have higher concentrations and mass discharges of **PFASs** than Northern European countries.
- High **PFASs** levels in **groundwater** are often restricted to some contaminated areas, e.g., due to illegal waste deposition on agricultural land or in the vicinity of a fluoropolymer producing factory.
- Sites with former **fire-fighting activities** are also potential “**hot spot**” areas.
- Concentrations in **drinking water** remain fairly **low** on average.
- Typical concentrations are in the low **ng/L range** with the exception of highly contaminated areas, like in the **Moehne and Ruhr area in Germany**.

Eschauzier, C., de Voogt, P., Brauch, H.-J., Lange, F.T. *Polyfluorinated chemicals in European surface waters, ground- and drinking waters*. In: *Polyfluorinated chemicals and transformation products*, *Hdb Env. Chem.* 17 (2012) 73–102; Springer-Verlag, Berlin, Heidelberg.



- **Rhone, Rhine, Danube, and Po rivers** have the highest discharges of European rivers (between 810 and 2,200 m³/s) and in part also **high PFASs concentrations**, thus generating a considerable mass flux even at low water contamination levels.
- **Rhine: PFOS and PFOA** < 10 ng/L.
- **PFBA and PFBS** were found at high concentrations (335 and 181 ng/L).
- At the Dutch–German border at **Lobith** high concentrations of **PFBA and PFBS** were observed in 2008 with average concentrations of **70 and 47 ng/L**.

Eschauzier, C., de Voogt, P., Brauch, H.-J., Lange, F.T. *Polyfluorinated chemicals in European surface waters, ground- and drinking waters. In: Polyfluorinated chemicals and transformation products. Hdb Env. Chem.* 17 (2012) 73–102; Springer-Verlag, Berlin, Heidelberg.

Möller, A., et al. Distribution and sources of polyfluoroalkyl substances (PFAS) in the River Rhine watershed. *Environmental Pollution* 158 (2010) 3243–3250.

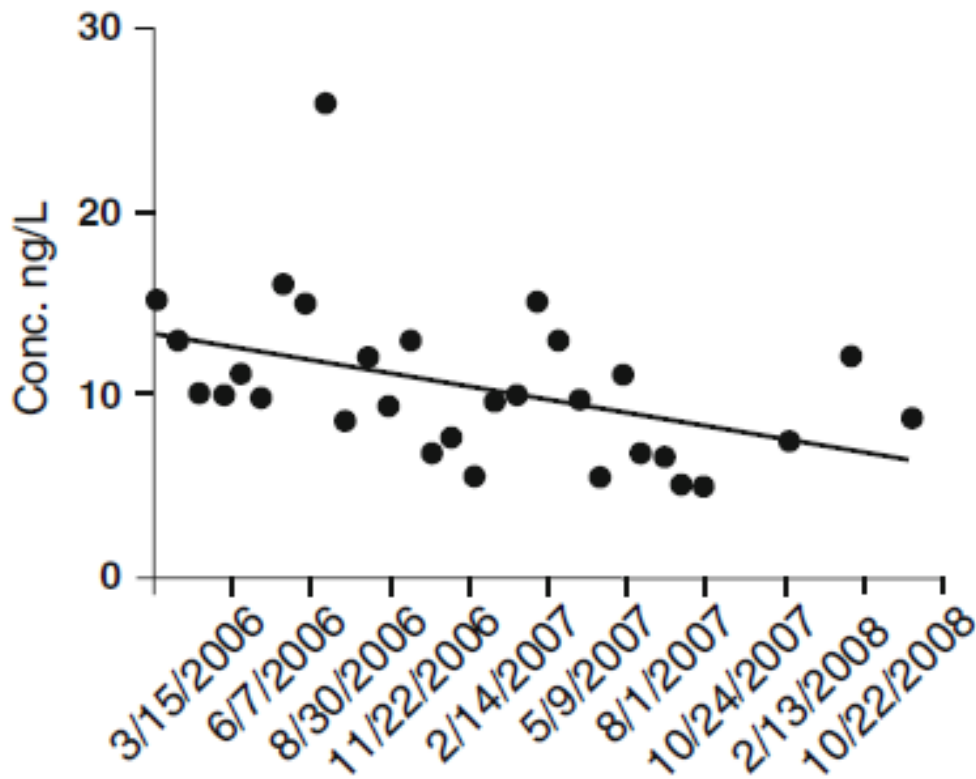


Fig. 5 Concentration of PFOS (in ng/L) in the Rhine River at Lekkanaal (Nieuwegein, the Netherlands) sampled in the period from 2006 to 2008

Eschauzier, C., de Voogt, P., Brauch, H.-J., Lange, F.T. Polyfluorinated chemicals in European surface waters, ground- and drinking waters. In: Polyfluorinated chemicals and transformation products, *Hdb Env. Chem.* 17 (2012) 73–102; Springer-Verlag, Berlin, Heidelberg.

Surface waters

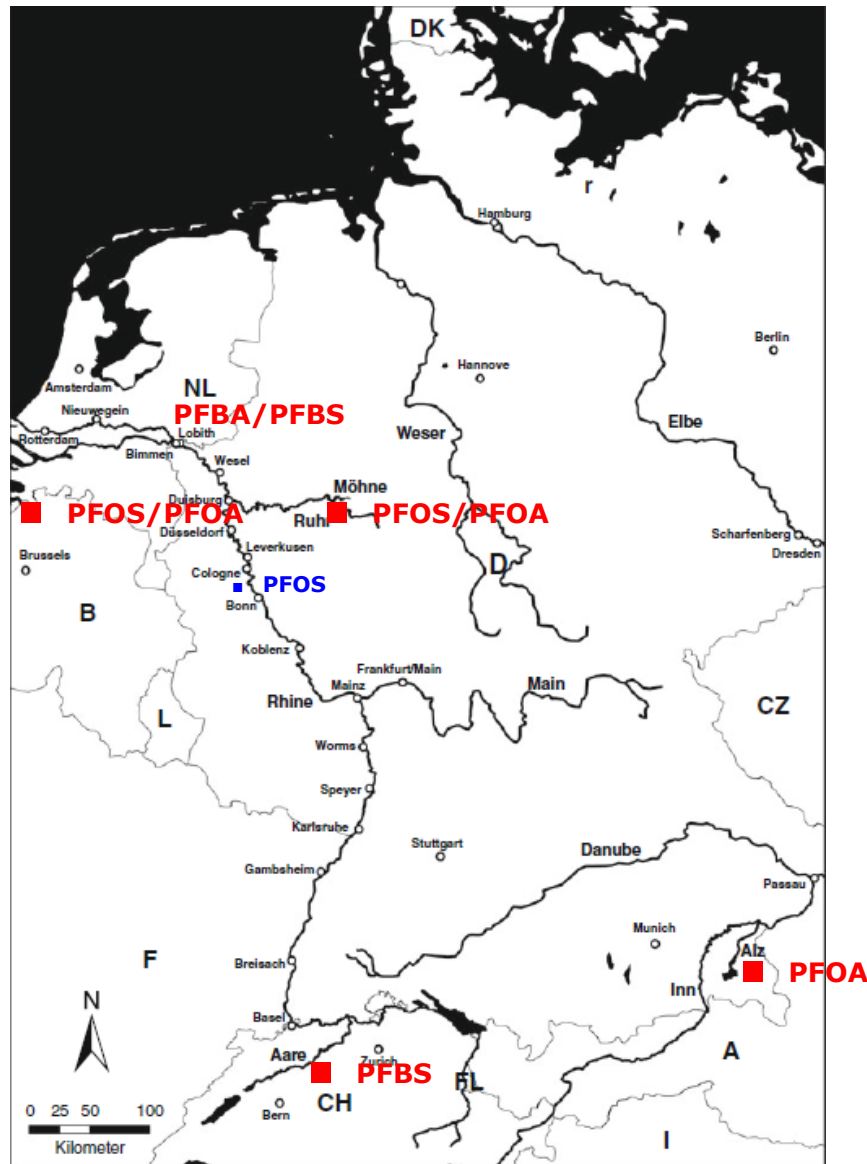
- **Elbe River: PFOS 6.4 ng/L** (in 2008)
- Predominating diffuse PFASs inputs, **PFOS to PFOA ratio of 3:1** can be deduced.
- **Alz River** (south Germany)
- **PFOA: up to 7.5 µg/L** in the vicinity of a fluoropolymer manufacturing facility in Gendorf (in 2007).
- **Inn and Danube**: concentrations of **100 and 50 ng/L PFOA**.

Eschauzier, C., de Voogt, P., Brauch, H.-J., Lange, F.T. Polyfluorinated chemicals in European surface waters, ground- and drinking waters. In: Polyfluorinated chemicals and transformation products, *Hdb Env. Chem.* 17 (**2012**) 73–102; Springer-Verlag, Berlin, Heidelberg.

PFASs Hotspots



European
Commission



Rhine: PFBA and PFBS

Aare: PFBS

Sauerland (Möhne/Ruhr): PFOS/A

Alz/Inn (Gendorf): PFOA

River Scheldt (BE/NL)

Rhone / Seine (Fr)

Barcelona; Llobregat River

Cologne: Groundwater (PFOS)

(Fire brigade training area)

Eschauzier, C., de Voogt, P., Brauch, H.-J., Lange, F.T. Polyfluorinated chemicals in European surface waters, ground- and drinking waters. In: Polyfluorinated chemicals and transformation products, *Hdb Env. Chem.* 17 (2012) 73–102; Springer-Verlag, Berlin, Heidelberg.

4,8-Dioxa-3H-Perfluorononanoate



Regulatory Toxicology and Pharmacology 59 (2011) 64–80



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Contents lists available at ScienceDirect

Regulatory Toxicology and Pharmacology

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



Toxicological evaluation of ammonium 4,8-dioxa-3H-perfluorononanoate, a new emulsifier to replace ammonium perfluorooctanoate in fluoropolymer manufacturing

Steven C. Gordon*

3M Company, Toxicology Assessment and Compliance Assurance, St. Paul, MN 55144, USA

ARTICLE INFO

Article history:
Received 8 June 2010
Available online 25 September 2010

Keywords:
Ammonium 4,8-dioxa-3H-perfluorononanoate
ADONA
Genotoxicity
NOAEL
Developmental toxicity
Subchronic toxicity
Ammonium perfluorooctanoate
APFO
PPAR α

ABSTRACT

Ammonium 4,8-dioxa-3H-perfluorononanoate (ADONA) was developed to replace ammonium perfluorooctanoate (APFO) as an emulsifier in the manufacture of fluoropolymers. The toxicity of ADONA was evaluated in acute and repeat-dose studies of up to 90-days duration, and in eye and skin irritation, dermal sensitization, genotoxicity, and developmental toxicity studies. ADONA was also evaluated as a peroxisome proliferator-activated receptor alpha (PPAR α) agonist in rats. ADONA was moderately toxic orally and practically non-toxic dermally in acute studies in rats. It was a mild skin irritant and a moderate to severe eye irritant in rabbits. It was a weak dermal sensitizer in local lymph node assays in mice. ADONA was not genotoxic based on the weight of evidence from five assays. It was not developmentally toxic in rats except at maternally toxic doses. ADONA was a possible PPAR α agonist in male rats. The liver was the primary target organ in male rats and the kidney was the primary target organ in female rats. NOAELs in 28- and 90-day oral studies in rats were 10 mg/kg/day for males and 100 mg/kg/day for females. These findings demonstrate that the toxicity profile for ADONA is acceptable for its intended use and is superior to that of APFO.

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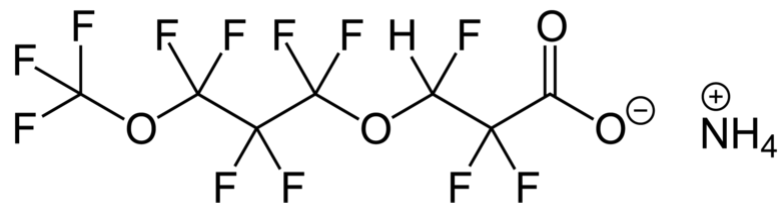
1. Introduction

Ammonium 4,8-dioxa-3H-perfluorononanoate (CF₃OCF₂CF₂CF₂-OCH₂CF₂COO⁻ NH₄⁺; ADONA) was developed to replace ammonium perfluorooctanoate (APFO) as an emulsifier used in the aqueous emulsion polymerization of fluoropolymers made from tetrafluoroethylene (TFE). Use of a fluorinated emulsifier in this process is essential, both to stabilize the final fluoropolymer dispersion and as a process safety measure to control the exothermic polymerization kinetics of TFE. APFO has been used since the 1950s for this purpose and as an intentionally added component in a variety of industrial and consumer products. However, evidence of widespread distribution and persistence of perfluorooctanoic acid (the aqueous dissociation product of APFO) in the environment, its biopersistence in humans, and its toxicity in laboratory animal studies [reviewed by Kennedy et al. (2004) and Lau et al. (2004, 2007)] have raised serious concerns about its continued use. In 2006, the US Environmental Protection Agency announced a Stewardship Program challenging fluoropolymer manufacturers to eliminate perfluorooctanoic acid and related chemicals from emissions and products by the year 2015 (USEPA, 2006). As a re-

sult, considerable efforts have been made to reduce the use of these compounds and to develop safer alternatives.

ADONA is a white to off-white crystalline solid at room temperature but it is generally made and used as a 30% aqueous solution. ADONA readily dissolves in aqueous media and dissociates to the corresponding carboxylic acid in equilibrium with its anion. The pK_a of the carboxylic acid is less than 3; therefore, the anion is expected to predominate at physiological pH. ADONA is chemically highly stable and non-reactive. Additional properties of ADONA are listed in Table 1.

ADONA is not intentionally added to products. Most of the ADONA used in fluoropolymer manufacturing is either recaptured from waste streams and products for reuse or is thermally destroyed during processing. Certain fluoropolymers manufactured using ADONA as the emulsifier, such as polytetrafluoroethylene (PTFE), are used as non-stick coatings on cookware and other food contact surfaces. These fluoropolymers are sintered onto the substrate at temperatures exceeding 280°C. ADONA decomposes at approximately 125–175°C. Fluoropolymers and fluoroelastomers made using ADONA as the emulsifier are functionally indistinguishable from those made using APFO.



- **ADONA, Ammonium 4,8-dioxa-3H-perfluorononanoate**, is 3M's PFOA replacement in the emulsion polymerization of fluoropolymers.
- Most of the ADONA used in fluoropolymer manufacturing is either recaptured from waste streams and products for reuse or is **thermally destroyed during processing**.
- These fluoropolymers are sintered onto the substrate at temperatures exceeding 280° C. ADONA decomposes at approximately 125–175° C.

The Sauerland Case, Germany



Perfluorinated Compounds in Fish and Blood of Anglers at Lake Möhne, Sauerland Area, Germany

Jürgen Hölzer,^{*,†} Thomas Göen,[‡] Paul Just,[§] Rolf Reupert,^{||} Knut Rauchfuß,^{||} Martin Kraft,^{||} Johannes Müller,[‡] and Michael Wilhelm[†]

[†]Department of Hygiene, Social and Environmental Medicine, MA 1/33, Ruhr-University Bochum, Universitätsstrasse 150, 44801 Bochum, Germany

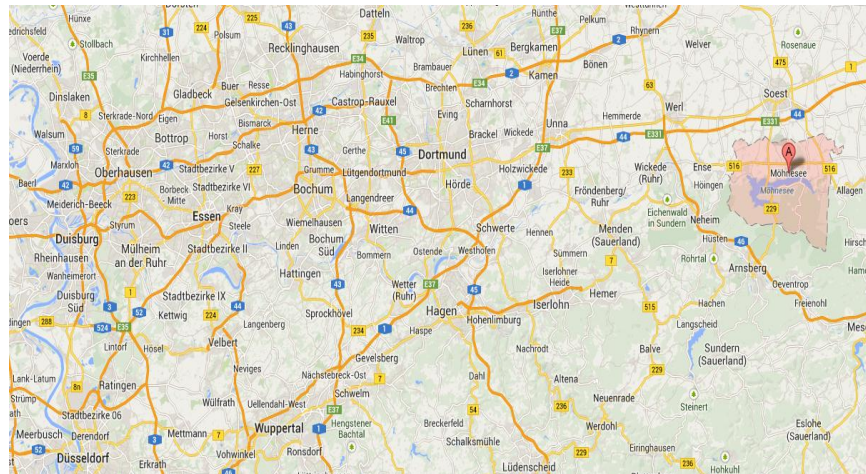
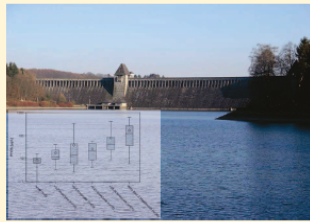
[‡]Institute and Outpatient Clinic of Occupational, Social and Environmental Medicine, University Erlangen-Nuremberg, Germany

[§]Staatliches Veterinäruntersuchungsamt, Arnsberg, Germany

^{||}North Rhine-Westphalia State Agency for Nature, Environment and Consumer Protection, Recklinghausen, Germany

Supporting Information

ABSTRACT: Perfluorinated compounds (PFCs) were measured in fish samples and blood plasma of anglers in a cross-sectional study at Lake Möhne, Sauerland area, Germany. Human plasma and drinking water samples were analyzed by solid phase extraction, high-performance liquid chromatography (HPLC), and tandem mass spectrometry (MS/MS). PFCs in fish fillet were measured by ion pair extraction followed by HPLC and MS/MS. PFOS concentrations in 44 fish samples of Lake Möhne ranged between 4.5 and 150 ng/g. The highest median PFOS concentrations have been observed in perch (median: 96 ng/g) and eels (77 ng/g), followed by pikes (37 ng/g), whitefish (34 ng/g), and roaches (6.1 ng/g). In contrast, in a food surveillance program only 11% of fishes at retail sale contained PFOS at detectable concentrations. One hundred five anglers (99 men, 6 women; 14–88 years old; median 50.6 years) participated in the human biomonitoring study. PFOS concentrations in blood plasma ranged from 1.1 to 650 µg/L (PFOA: 2.1–170 µg/L; PFHxS: 0.4–17 µg/L; LOD: 0.1 µg/L). A distinct dose-dependent relationship between fish consumption and internal exposure to PFOS was observed. PFOS concentrations in blood plasma of anglers consuming fish 2–3 times per month were 7 times higher compared to those without any fish consumption from Lake Möhne. The study results strongly suggest that human internal exposure to PFC is distinctly increased by consumption of fish from PFC-contaminated sites.



[Environ. Sci. Technol. 45 \(2011\) 8046–8052.](#)



- **Surface waters** along the **River Möhne** became contaminated with PFCs, mainly by application of **polluted soil conditioner on agricultural lands**.
- **Soil conditioner** (53,000 tons) has been distributed on more than 1300 farm lands between 2000 and 2006.
- Maximum **PFOA** and **PFOS** concentrations were **2.4 and 33 mg/kg**.
- Contamination of **drinking water**.
- Whereas soil samples contained more PFOS than PFOA, **PFOA was the predominant compound observed in drinking water**
(2006: **500-640 ng PFOA/L**).
- A **human biomonitoring study** was implemented, which revealed **4-8-fold increased PFOA blood concentrations in residents exposed to contaminated drinking water** compared to a reference population.



- **Fish samples of Lake Möhne**

PFOS was the predominant PFAS found

Highest median PFOS concentrations:

Perches (median: 96 ng/g), Eels (77 ng/g), Pikes (37 ng/g)

Whitefish (34 ng/g), Roaches (6.1 ng/g)

- **PFOS concentrations in blood plasma of anglers**

Ranged from 1.1 to **650 µg/L**

(**PFOA: 2.1-170 µg/L**; PFHxS: 0.4-17 µg/L).

- A distinct dose-dependent relationship between fish consumption and internal exposure to PFOS was observed.
- **PFOS concentrations in blood plasma of anglers** consuming fish 2-3 times per month were **7 times higher** compared to those without any fish consumption from Lake Möhne.

Hölzer et al., *Environ. Sci. Technol.* 45 (2011) 8046–8052.
Hölzer et al., *Environ. Health Perspect.* 116 (5) (2008) 651-657.

- Bavaria, industrial area **Gendorf**, Alztal aquifer, Germany:
Groundwater contamination with **PFOA: up to 7 µg/L**.
- near **Cologne**, Germany. The source of the contamination was identified to be a **fire brigade training area** (AFFFs). ΣPFASs reached levels up to **4 µg/L**, with **PFOS** and **PFHxS** prevailing.

Eschauzier, C., de Voogt, P., Brauch, H.-J., Lange, F.T. *Polyfluorinated chemicals in European surface waters, ground- and drinking waters*. In: *Polyfluorinated chemicals and transformation products*, *Hdb Env. Chem.* 17 (2012) 73–102; Springer-Verlag, Berlin, Heidelberg.

Tap / Drinking Water



Concentrations of PFAAs in seven tap water samples collected from six European cities. For PFOA and PFOS the percentage of the linear isomer and sum of branched isomers (% lin/br) is additionally given.

Sampling location	Concentrations of PFCAs [ng/L]							
	PFPeA	PFHxA	PFHpA	PFOA (% lin/br)	PFNA	PFDA	PFUnDA	PFDoDA
SU ^a	nd ^h	2.86	1.09	6.18 (92/8)	0.433	0.506	nd	<MLQ
IES ^b	<MLQ ⁱ	2.10	1.19	4.92 (90/10)	0.522	0.612	nd	<MLQ
UA ^c	1.39	3.00	0.996	2.70 (100/0)	0.339	0.182	nd	<MLQ
UvA ^d	0.734	3.06	1.47	8.56 (80/20)	<MLQ	<MLQ	nd	<MLQ
VU ^e	2.69	5.15	1.91	5.66 (65/35)	<MLQ	<MLQ	nd	<MLQ
NILU ^f	<MLQ	0.806	0.434	2.20 (100/0)	<MLQ	0.094	<MLQ	<MLQ
FI ^g	<MLQ	<MLQ	<MLQ	0.302 (100/0)	<MLQ	<MLQ	nd	<MLQ

Sampling location	Concentrations of PFASs and PFPAs [ng/L]						
	PFBS	PFHxS	PFOS (% lin/br)	PFDS	PFHxPA	PFOPA	PFDPa
SU ^a	0.955	2.50	8.81 (68/32)	<MLQ	nd	nd	nd
IES ^b	0.502	1.15	6.92 (74/26)	<MLQ	nd	nd	nd
UA ^c	2.94	0.909	2.71 (62/38)	0.074	nd	nd	nd
UvA ^d	7.61	0.556	0.861 (62/38)	0.045	nd	<MLQ	nd
VU ^e	18.8	1.34	0.397 (58/42)	<MLQ	nd	<MLQ	nd
NILU ^f	<MLQ	<MLQ	0.573 (70/30)	0.195	nd	nd	nd
FI ^g	0.092	<MLQ	0.847 (71/29)	<MLQ	nd	nd	nd

^a SU: Stockholm University, Sweden.

^b IES: Institute for Environment and Sustainability, Ispra, Italy.

^c UA: University of Antwerp, Belgium.

^d UvA: University of Amsterdam, The Netherlands.

^e VU: VU University, Amsterdam, The Netherlands.

^f NILU: Norwegian Institute for Air Research, Tromsø, Norway.

^g FI: Fraunhofer Institute, Schmallenberg, Germany.

^h nd: not detected (for method detection limits see Table 2).

ⁱ <MLQ: detected but below the method limit of quantification (Table 2).

Barcelona: PFOA 9 ng/L; PFOS 50 ng/L
Amsterdam: PFBA <9.5 to 52 ng/L,
PFBS 11 to 42 ng/L

Ericson, I., et al. *Levels of perfluorinated chemicals in municipal drinking water from Catalonia, Spain: public health implications.* Arch. Environ. Contam. Toxicol. 57 (2009) 631–638.

Ullah, S., Alsberg, T., Berger, U. *Simultaneous determination of perfluoroalkyl phosphonates, carboxylates, and sulfonates in drinking water.* Journal of Chromatography A 1218 (2011) 6388– 6395.

Eschauer, C., et al. *Impact of treatment processes on the removal of perfluoroalkyl acids from the drinking water production chain.* Environ. Sci. Technol. 46 (2012) 1708–1715.

21 October 2013



- **2007: Surface water survey**

“EU-wide survey of polar organic persistent pollutants in European river waters”

[Environmental Pollution 157 \(2009\) 561–568](#)

- **Joint Danube Survey (2007)**

[Water Research 44 \(2010\) 2325–2335](#)

- **2008: Groundwater survey**

“Pan-European survey on the occurrence of selected polar organic persistent pollutants in ground water”

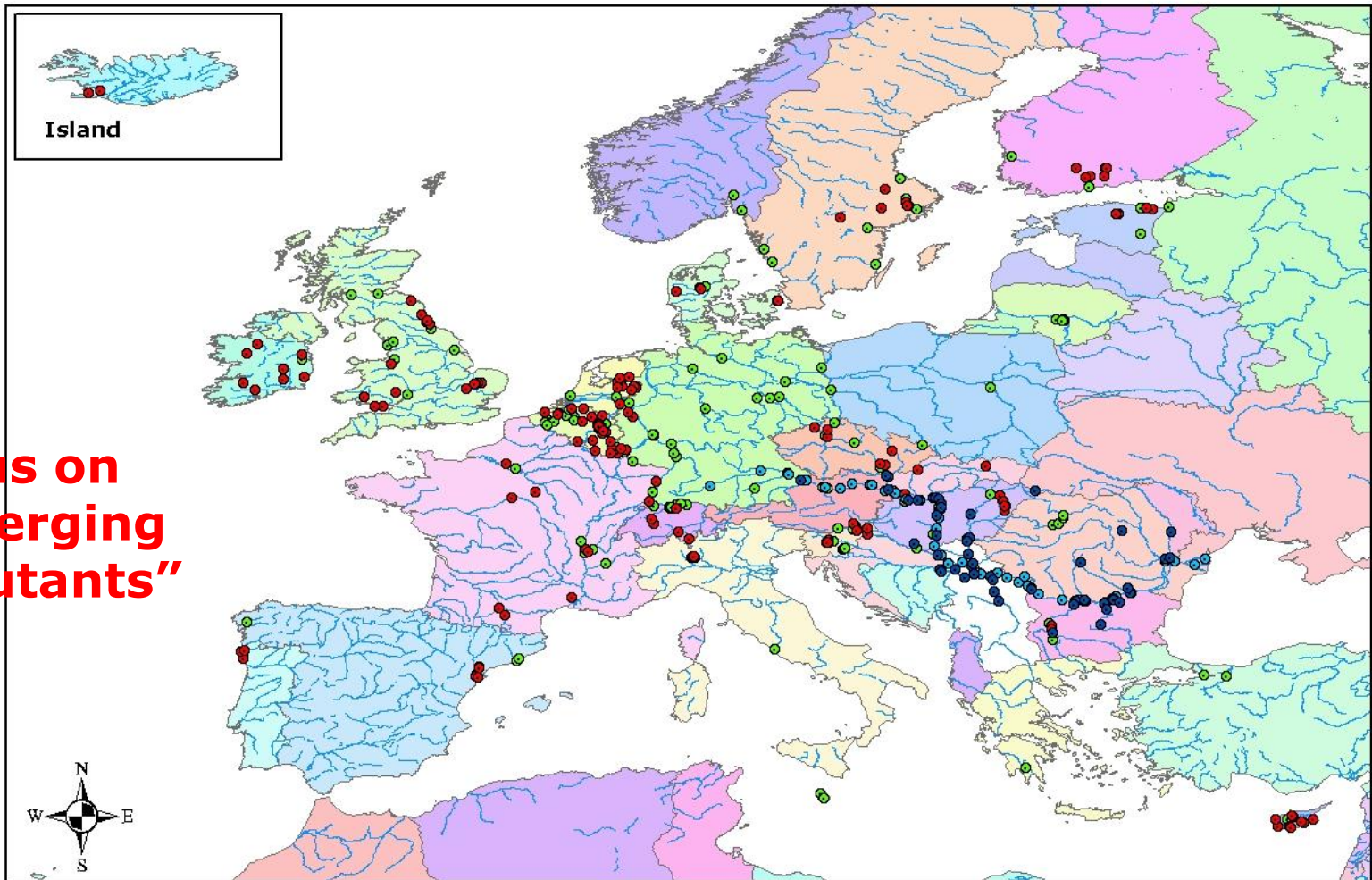
[Water Research 44 \(2010\) 4115–4126](#)

- **2010: Waste water treatment plant (WWTP) effluents**

[Water Research, in press](#)

- **2012: Pilot exercise for WFD “Watch List” (surface waters)**

Sampling Stations: Rivers and Groundwater



**Focus on
“emerging
pollutants”**

Legend

- Groundwater sampling point
- Surface water sampling point
- Main rivers
- JDS2-Tributaries sampling point
- JDS2-Danube sampling point

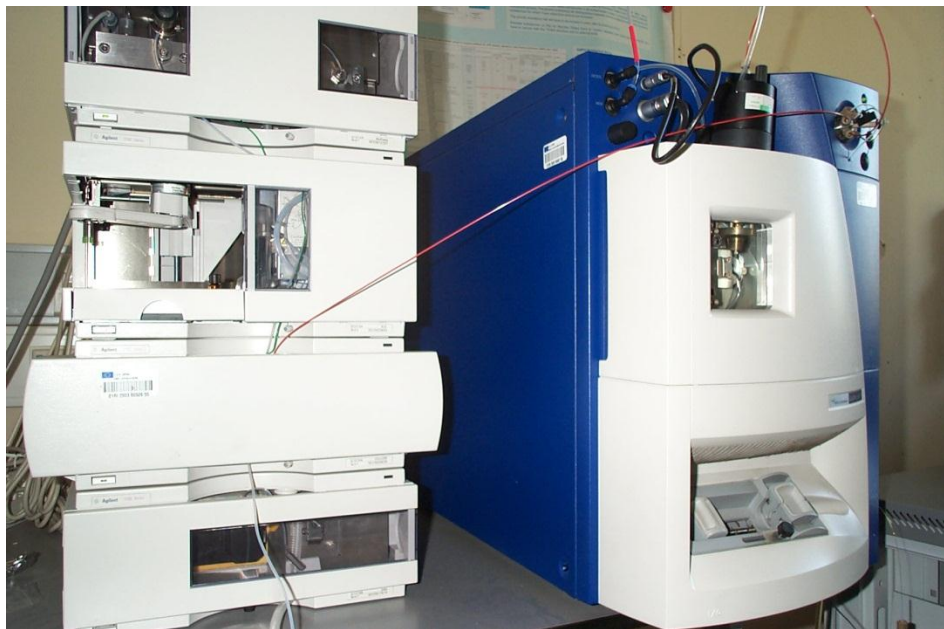
Surface water: 122 samples

Ground water: 164 samples

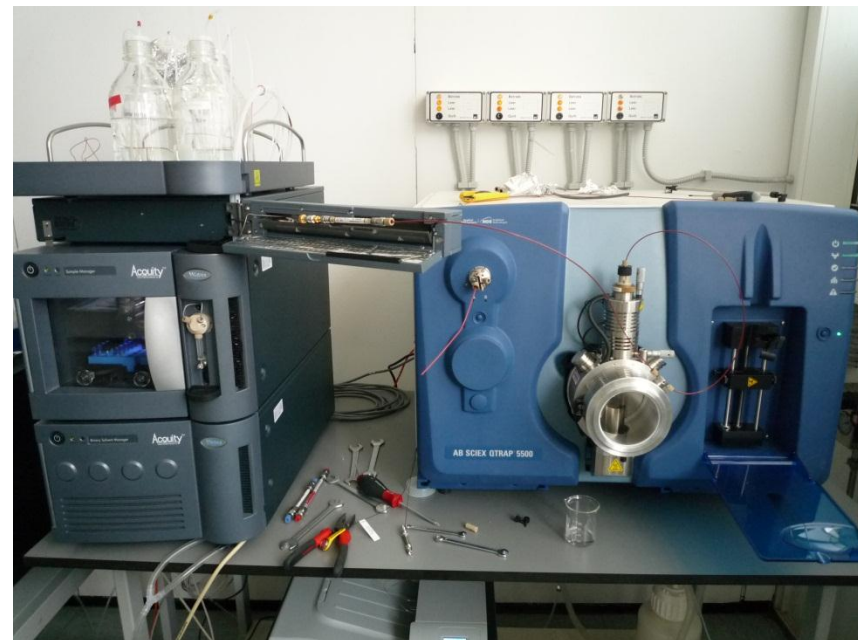
Danube and tributaries: 103 samples

Autotrace 280 system





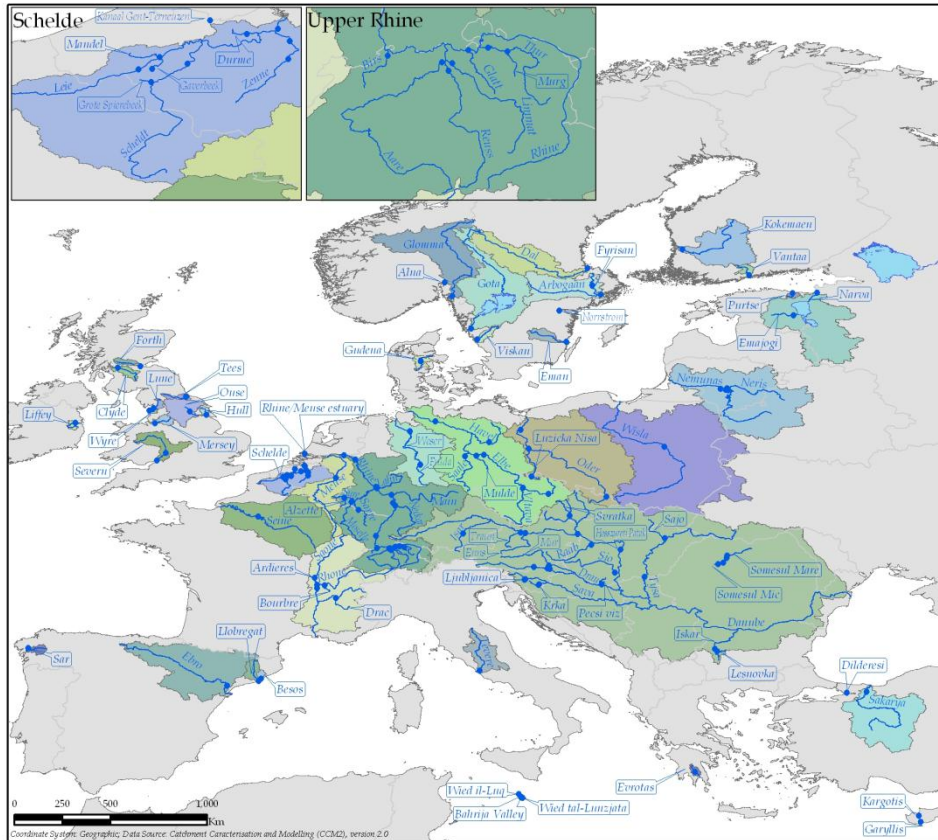
LC-MS-MS



UHPLC-MS-MS

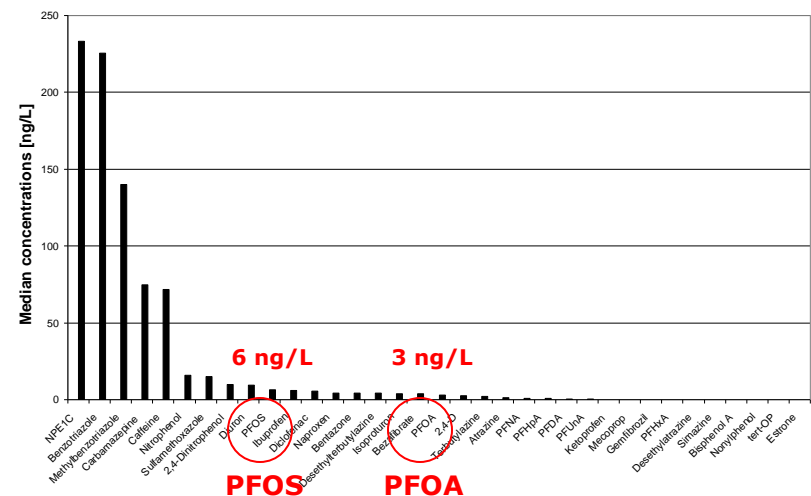
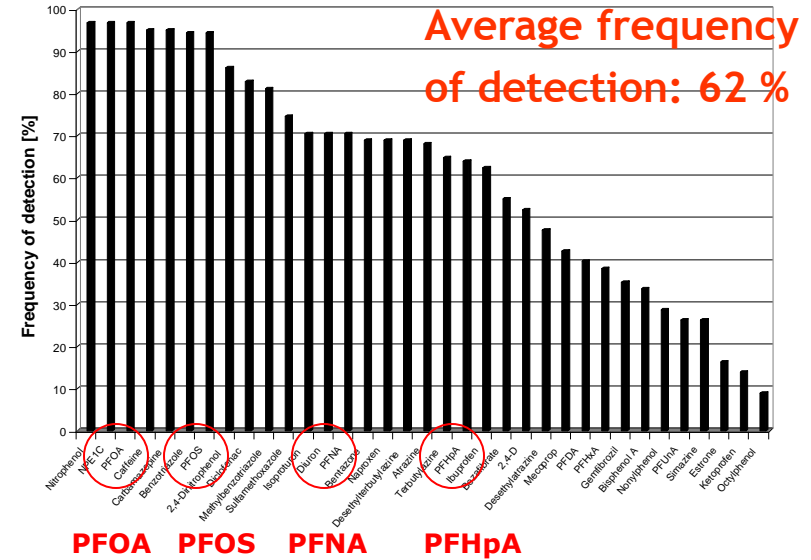
Ultra-high pressure liquid chromatography
Particles < 2 μm
Higher pressure; faster; more efficient

EU-wide Survey of Polar Pollutants in Rivers



The **most frequently detected compounds** were 1-nitrophenol (freq 97%), NPE₁C (97%), **PFOA (97%)**, caffeine (95%), carbamazepine (95%), **PFOS (94%)**, benzotriazole (94%), 2,4-dinitrophenol (86%), diclofenac (83%), and tolyltriazole (81%).

The **highest median concentrations** were measured for NPE₁C (233 ng/L), benzotriazole (226 ng/L), tolyltriazole (140 ng/L), carbamazepine (75 ng/L), and caffeine (72 ng/L).



Loos et al., *Environ. Poll.* 157 (2009) 561-568.

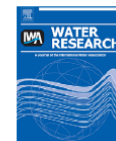


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Occurrence of polar organic contaminants in the dissolved water phase of the Danube River and its major tributaries using SPE-LC-MS² analysis

Robert Loos^a, Giovanni Locoro, Serafino Contini

European Commission, Joint Research Centre, Institute for Environment and Sustainability, Via Enrico Fermi, 21020 Ispra, Italy

ARTICLE INFO

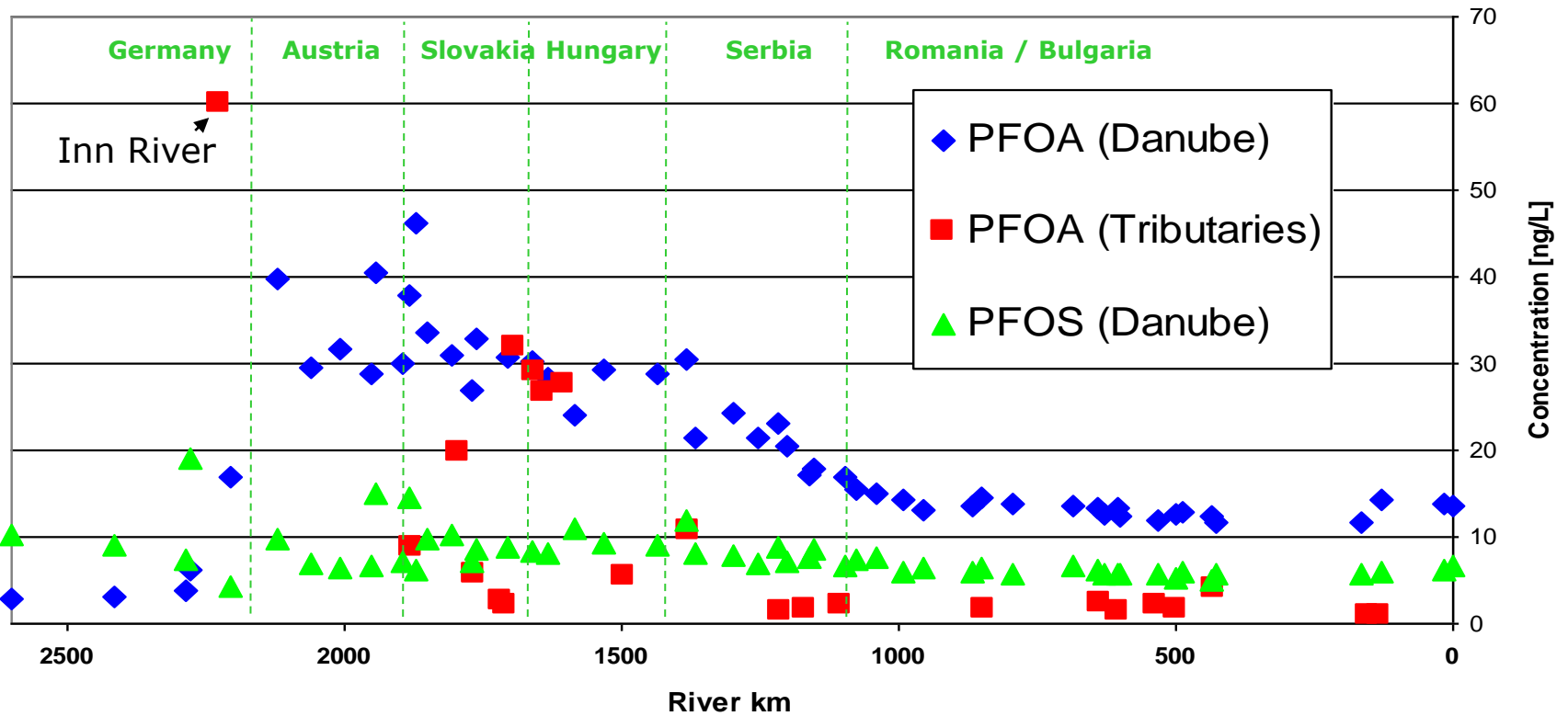
Article history:
Received 1 July 2009
Received in revised form
17 December 2009
Accepted 21 December 2009
Available online 4 January 2010

Keywords:
Danube River
Water monitoring
SPE-LC-MS²
Water framework directive
Emerging contaminants

ABSTRACT

Polar water-soluble organic contaminants were analysed in the dissolved liquid water phase of river water samples from the Danube River and its major tributaries (within the Joint Danube Survey 2). Analyses were performed by solid-phase extraction (SPE) followed by triple-quadrupole liquid chromatography mass spectrometry (LC-MS²). In total, 34 different polar organic compounds were screened. Focus was given on pharmaceutical compounds (such as ibuprofen, diclofenac, sulfamethoxazole, carbamazepine), pesticides and their degradation products (e.g. bentazone, 2,4-D, mecoprop, atrazine, terbutylazine, desethylterbutylazine), perfluorinated acids (PFOS; PFOA), and endocrine disrupting compounds (nonylphenol, NPE, C, bisphenol A, estrone). The most relevant polar compounds identified in the Danube River basin in terms of frequency of detection, persistency, and concentration levels were 1H-benzotriazole (median concentration 185 ng/L), caffeine (87 ng/L), tolyltriazole (73 ng/L), nonylphenoxy acetic acid (49 ng/L), carbamazepine (33 ng/L), 4-nitrophenol (29 ng/L), 2,4-dinitrophenol (19 ng/L), PFOA (17 ng/L), sulfamethoxazole (16 ng/L), desethylterbutylazine (11 ng/L), and 2,4-D (10 ng/L). The highest contamination levels were found in the area around Budapest and in the tributary rivers Arges (Romania), Timok (Bulgaria), Rusenski Lom (Bulgaria), and Velika Morava (Serbia).

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PFOA

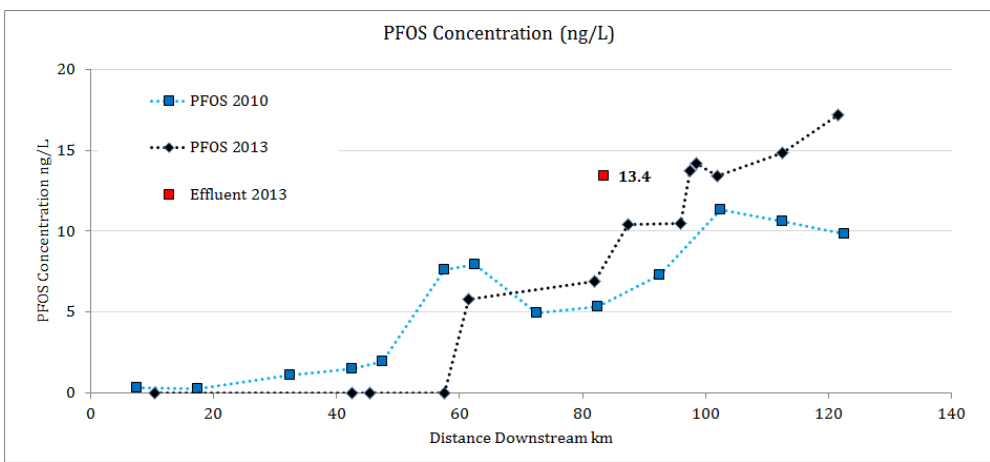
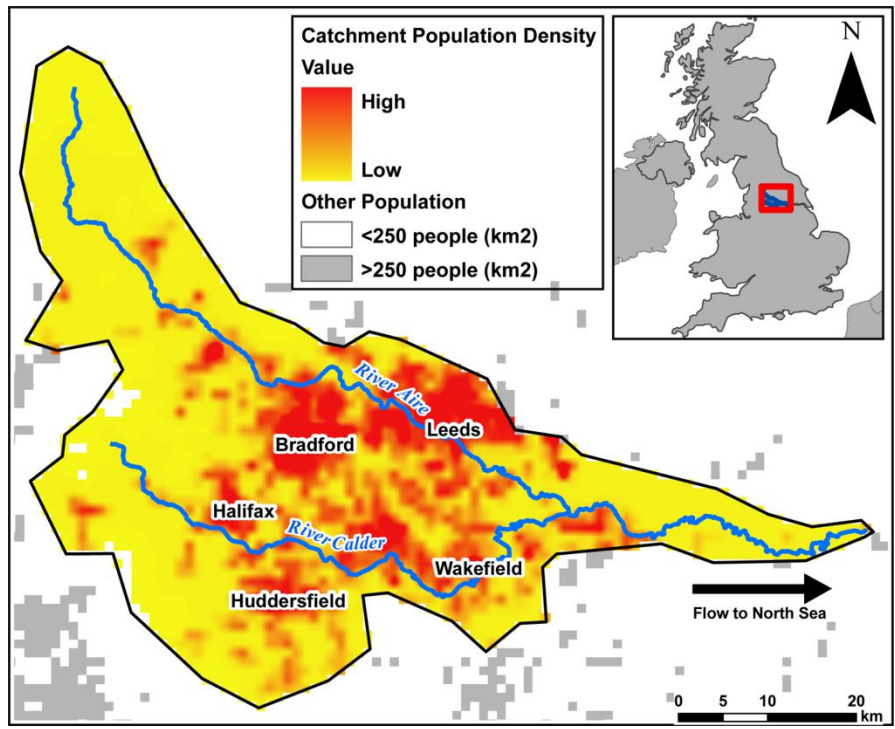


PFOS



River Aire (UK)

PFOS: 10-15 ng/L



JRC cooperation with Lancaster University (Mark Earnshaw, Alex Paul, Andy Sweetman, Kevin Jones)



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Pan-European survey on the occurrence of selected polar organic persistent pollutants in ground water

Robert Loos^{a,*}, Giovanni Locoro^a, Sara Comero^a, Serafino Contini^{a,1}, David Schwesig^b, Friedrich Werres^b, Peter Balsaa^b, Oliver Gans^c, Stefan Weiss^c, Ludek Blaha^d, Monica Bolchi^e, Bernd Manfred Gawlik^a

^aEuropean Commission, Joint Research Centre, Institute for Environment and Sustainability, Via Enrico Fermi, 21020 Ispra, Italy

^bIWU Water Centre, Moritzstr. 26, 45476 Muelheim an der Ruhr, Germany

^cUmweltbundesamt GmbH, Spittelauer Lände 5, 1090 Vienna, Austria

^dMasaryk University, RECETOX, Kamenice 3, CZ 62500 Brno, Czech Republic

^ePerkin Elmer Italia S.p.A., Via Tiepolo, 24, I 20052 Monza (MI), Italy

ARTICLE INFO

Article history:
Received 9 February 2010
Received in revised form
17 May 2010
Accepted 22 May 2010
Available online 1 June 2010

Keywords:

Ground water
Pan-European monitoring
Non-probabilistic sampling
Polar organic contaminants
SPE-LC-MS²

ABSTRACT

This study provides the first pan-European reconnaissance of the occurrence of polar organic persistent pollutants in European ground water. In total, 164 individual ground-water samples from 23 European Countries were collected and analysed (among others) for 59 selected organic compounds, comprising pharmaceuticals, antibiotics, pesticides (and their transformation products), perfluorinated acids (PFAs), benzotriazoles, hormones, alkylphenolics (endocrine disruptors), Caffeine, Diethyltoluamide (DEET), and Triclosan. The most relevant compounds in terms of frequency of detection and maximum concentrations detected were DEET (84%; 454 ng/L), Caffeine (83%; 189 ng/L), PFOA (66%; 39 ng/L), Atrazine (56%; 253 ng/L), Desethylatrazine (55%; 487 ng/L), 1H-Benzotriazole (53%; 1032 ng/L), Methylbenzotriazole (52%; 516 ng/L), Desethylterbutylazine (49%; 266 ng/L), PFOS (48%; 135 ng/L), Simazine (43%; 127 ng/L), Carbamazepine (42%; 390 ng/L), nonylphenoxy acetic acid (NPE;C) (42%; 11 µg/L), Bisphenol A (40%; 2.3 µg/L), PFHxS (35%; 19 ng/L), Terbutylazine (34%; 716 ng/L), Benzotriazole (32%; 11 µg/L), Propazine (32%; 25 ng/L), PFHpA (30%; 21 ng/L), 2,4-Dinitrophenol (29%; 122 ng/L), Diuron (29%; 279 ng/L), and Sulfamethoxazole (24%; 38 ng/L). The chemicals which were detected most frequently above the European ground water quality standard for pesticides of 0.1 µg/L were Chloridazon-desphenyl (26 samples), NPE;C (20), Bisphenol A (12), Benzotriazole (8), N,N'-Dimethylsulfamid (DMS) (8), Desethylatrazine (6), Nonylphenol (6), Chloridazon-methyl-desphenyl (6), Methylbenzotriazole (5), Carbamazepine (4), and Benzotriazole (4). However, only 1.7% of all single analytical measurements (in total 8000) were above this threshold value of 0.1 µg/L; 7.3% were > than 10 ng/L.

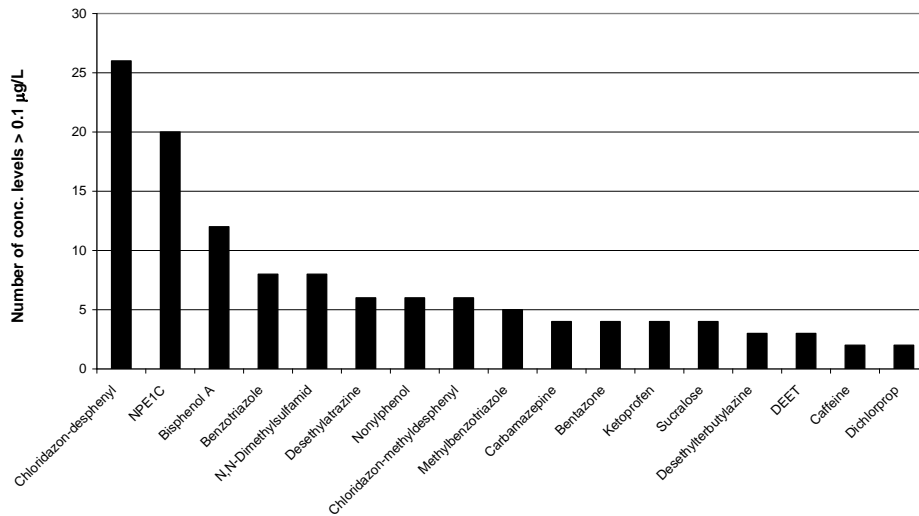
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Table 1 – Summary of analytical results for polar organic pollutants in EU ground waters.

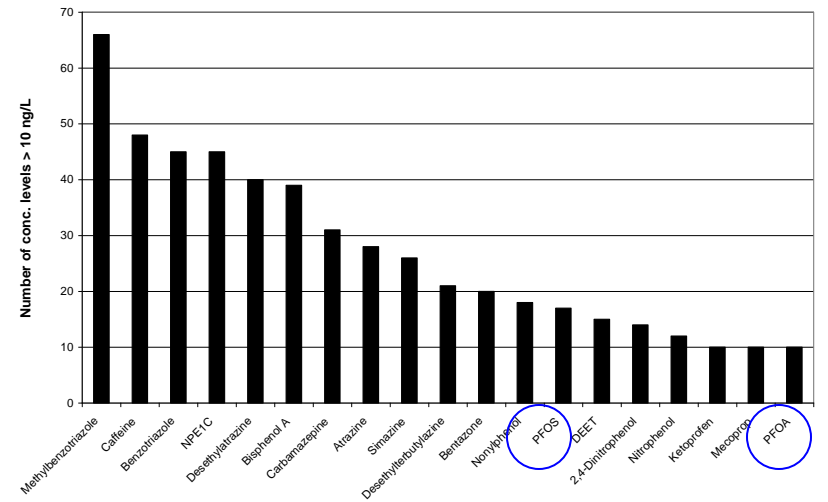
Chemical	LOD [ng/L]	Freq [%]	max [ng/L]	Average [ng/L]	med [ng/L]	Per90 [ng/L]
DEET	0.4	83.5	454	9	1	9
Caffeine	1.0	82.9	189	13	4	32
PFOA	0.4	65.9	39	3	1	6
Atrazine	0.4	56.1	253	8	1	24
Desethylatrazine (DEA)	0.4	54.9	487	17	1	50
1H-Benzotriazole	1.0	53.0	1032	24	1	40
Methylbenzotriazole	1.0	51.8	516	20	4	42
Desethylterbutylazine (DET)	0.4	49.4	266	7	0	12
PFOS	0.4	48.2	135	4	0	11
Simazine	0.5	43.3	127	7	0	17
Carbamazepine	0.5	42.1	390	12	0	20
NPE;C	0.5	41.5	11316	263	0	127
Bisphenol A	1.0	39.6	2299	79	0	73
PFHxS	0.4	34.8	19	1	0	5
Terbutylazine	0.3	33.5	716	6	0	2
Benzotriazole	0.4	31.7	10550	116	0	15
Propazine	0.3	31.7	25	1	0	2
PFHpA	0.4	29.9	21	1	0	1
2,4-Dinitrophenol	1.0	29.3	122	4	0	6
Diuron	0.3	28.7	279	3	0	3
Sulfamethoxazole	0.5	24.4	38	2	0	4
PFDA	0.4	23.8	11	0	0	1
tert-Octylphenol (OP)	0.4	23.2	41	1	0	2
Metolachlor	0.3	20.7	209	2	0	2
Nitrophenol	4.0	20.1	152	4	0	8
Isoproturon	0.2	20.1	22	0	0	0
Hexazinone	0.3	17.7	589	4	0	1
Chloridazon-desphenyl	50	16.5	13000	176.9	0	217
PFBS	0.3	15.2	25	0	0	1
PFNA	0.4	15.2	10	0	0	0
Mecoprop	0.2	13.4	785	7	0	1
N,N'-Dimethylsulfamid (DMS)	50	11.6	52000	332	0	50
Nonylphenol (NP)	30.0	11.0	3850	83	0	39
Ketoprofen	1.0	10.4	2886	26	0	2
Diazinon	0.3	9.1	1	0	0	0
MCPA	0.1	7.9	36	0	0	0
Chlortoluron	0.3	7.9	91	1	0	0
Ibuprofen	0.2	6.7	395	3	0	0
Chloridazon-methyl-desphenyl	50	6.1	1200	19.1	0	0
Methabenzthiazuron	0.3	5.5	104	1	0	0
Dichlorprop	0.1	4.9	3199	36	0	0
Diclofenac	0.2	4.9	24	0	0	0
Alachlor	0.3	4.9	27	0	0	0
2,4-D	0.1	3.7	12	0	0	0
2,4,5-T	0.2	3.7	3	0	0	0
Linuron	0.3	2.4	293	2	0	0
Triclosan	2.0	1.8	9	0	0	0
Estrone	1.0	0.6	4	0	0	0

Number of samples, 164; LOD = limit of detection; freq = frequency of detection [%]; max = maximum concentration; med = median concentration; Per90 = 90th percentile [%]; priority compounds of the WFD in blue. In green: Pesticide metabolites analysed by IWW Water centre (Germany). (For interpretation of the references to colour in this Table legend, the reader is referred to the web version of this article). Not included are Naproxen, Propanil, Fenarimol, Bezaflubate, Gemiflurozil, PFHxS, PFUnA, Metaxuron, Carbaryl, and Molinate which were not detected.

Number of detections > 0.1 µg/L



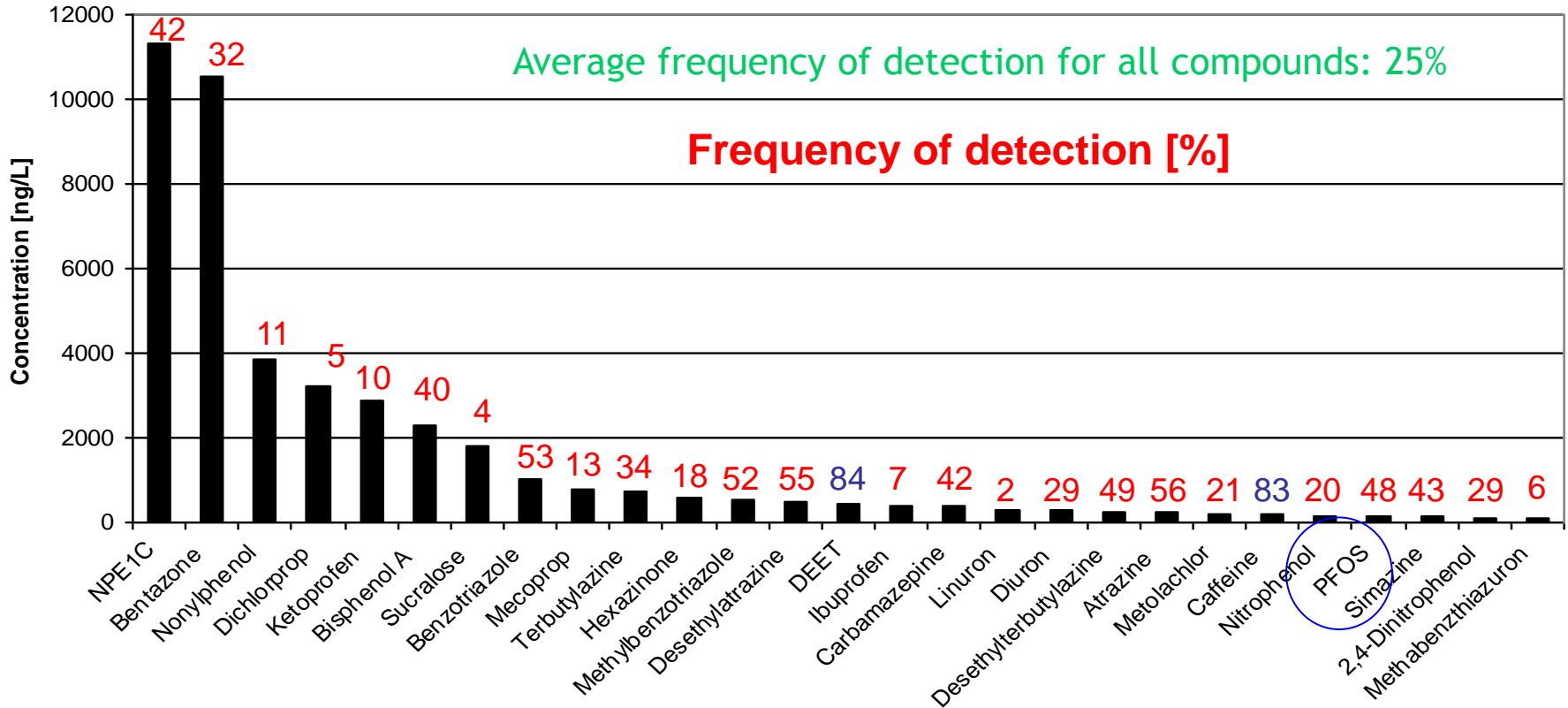
Number of detections > 10 ng/L



PFOS

PFOA

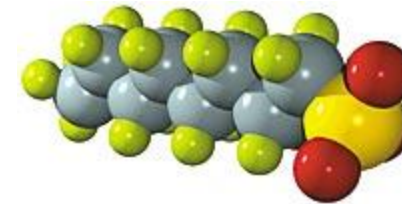
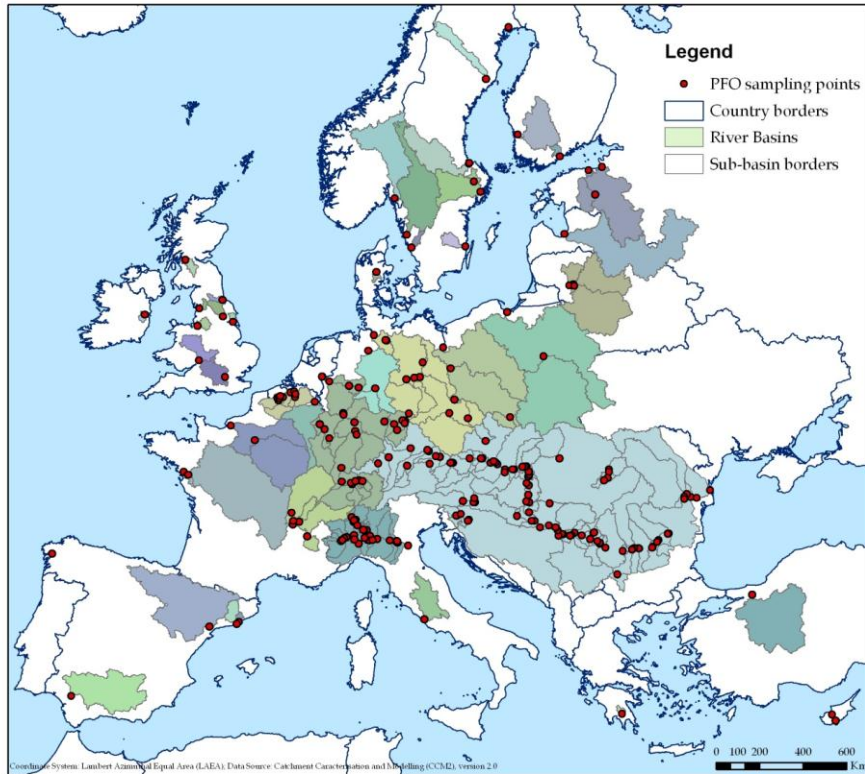
Max. Concentrations in Groundwater



Not included:

Dimethylsulfamid (max. 52 µg/L in one sample; freq. 12%), Chloridazon-desphenyl (max. 13 µg/L; freq. 17%), Chloridazon-methyl-desphenyl (max. 1.2 µg/L; freq. 6%), PFOA (max. 39 ng/L; freq. 66 %).

Loos et al. Pan-European survey on the occurrence of selected polar organic persistent pollutants in ground water. [Water Res.](#) 44 (2010) 4115-4126.



Environ. Sci. Technol. 2009 43, 9237–9244

A Map of European Emissions and Concentrations of PFOS and PFOA

ALBERTO PISTOCCHI AND
ROBERT LOOS*

*European Commission, Joint Research Centre, Institute for
Environment and Sustainability, Via Enrico Fermi,
21020 Ispra, Italy*

*Received April 28, 2009. Revised manuscript received
October 20, 2009. Accepted October 26, 2009.*

A spatially distributed data set of measured concentrations of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) was used, together with climatological averages of river flow, to estimate their overall aqueous emissions from the European continent. This estimate is based on the assumption that

listed as chemical for regulation within the Stockholm Convention (5) and was banned in the European Union in 2007 for most applications (6). Contrary to other classical POPs, PFOS and PFOA are primarily emitted to water and they accumulate in seawater, which represent their major reservoir in the environment, as well as the most important medium for their global transport (7–9).

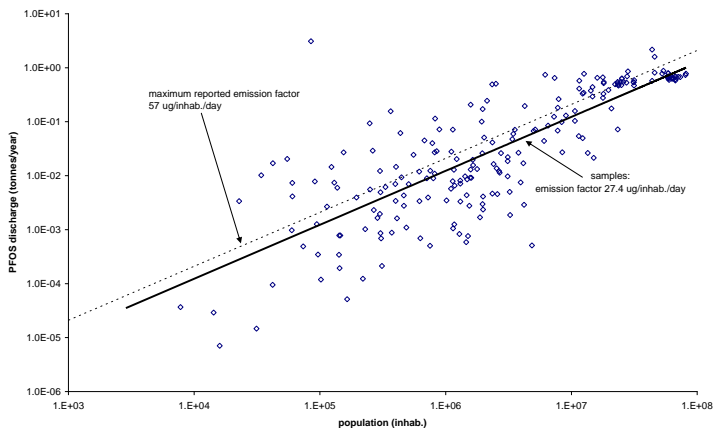
The main direct PFOA contamination source is the manufacturing of ammonium perfluorooctanoate (APFO) and its subsequent use in fluoropolymer production. The total estimated emissions of APFO over the time period 1950–2010 are thought to be in the range of ~2600–5050 t, from an estimated total global production volume of between 4400 and 8000 tons. In 1999, global annual APFO production (which is still not regulated) was approximately 260 tons. Indirect PFOA sources from fluorotelomer alcohols (FTOHs) and perfluorooctane sulfonyl fluoride (POSF)-based chemicals (e.g., perfluorooctyl sulfonamidoethanols), which can degrade in the environment to yield PFOA only account for ~10% of all PFOA emissions (7–9).

- 46 river monitoring points for rivers which “discharge” into the seas
- Around the same number of relevant rivers / streams (~50) is missing

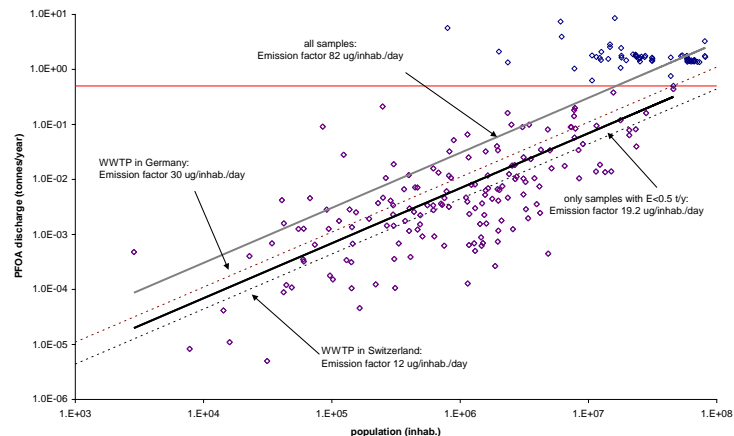
Correlation of PFOA/S with Population



PFOS



PFOA



Estimation for the year 2007:

PFOS and PFOA discharges along the whole European river network to coastal areas in Europe: ~ **20 t/a PFOS** and **30 t/a PFOA**.

Emission factor: **27** $\mu\text{g}/\text{day}/\text{person}$ **PFOS**, and **82** $\mu\text{g}/\text{day}/\text{person}$ **PFOA**.

A. Pistocchi, R. Loos, *Environ. Sci. Technol.* 43 (2009) 9237-9244.



Analysis of polar organic contaminants in surface water of the northern Adriatic Sea by solid-phase extraction followed by ultrahigh-pressure liquid chromatography–QTRAP[®] MS using a hybrid triple-quadrupole linear ion trap instrument

Robert Loos · Simona Tavazzi · Bruno Paracchini · Elisabetta Canuti · Christof Weissteiner

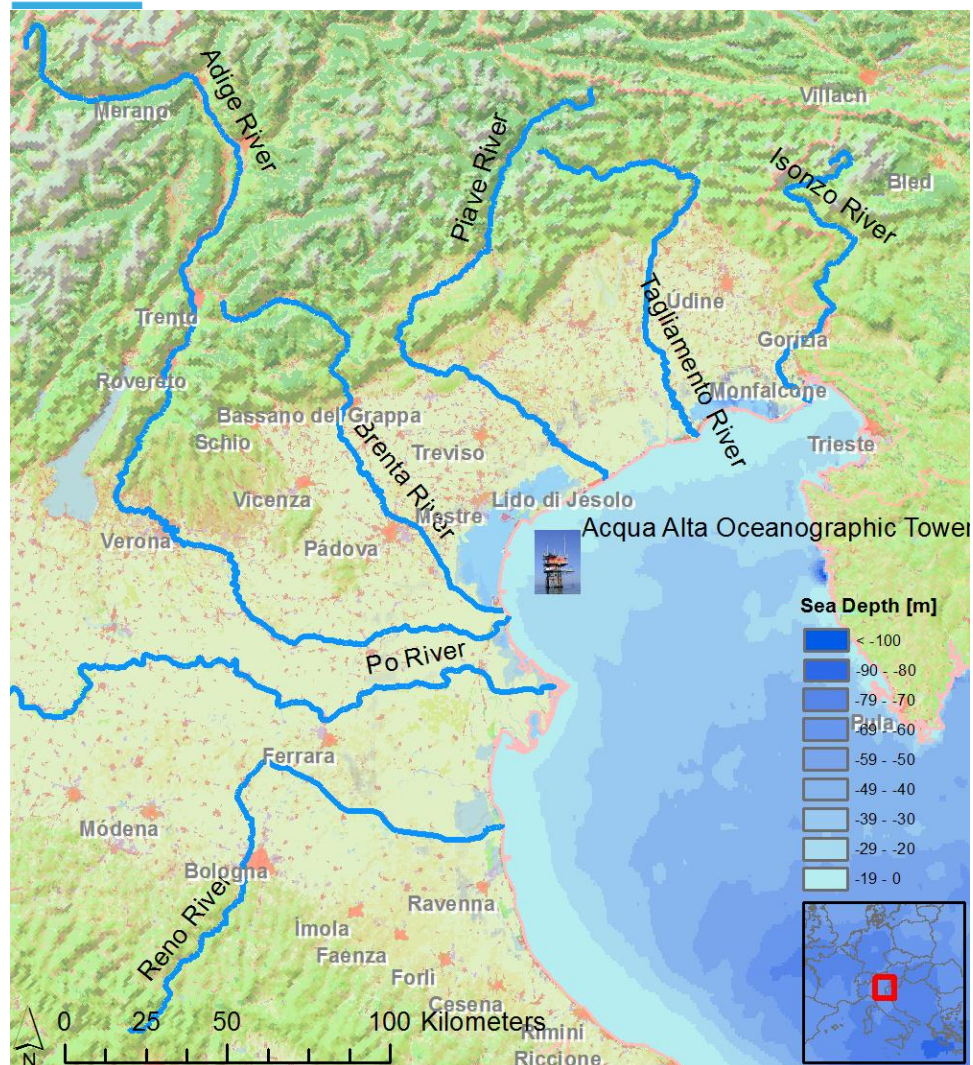
Received: 22 January 2013 / Revised: 20 March 2013 / Accepted: 25 March 2013 / Published online: 9 May 2013
© Springer-Verlag Berlin Heidelberg 2013

Abstract Water-soluble polar organic contaminants are discharged by rivers, cities, and ships into the oceans. Little is known on the fate, pollution effects, and thresholds of toxic chemical mixtures in the marine environment. A new trace analytical method was developed for the multi-compound analysis of polar organic chemical contaminants in marine waters. The method is based on automated solid-phase extraction (SPE) of one-liter water samples followed by ultrahigh-pressure liquid chromatography triple-quadrupole linear ion-trap mass spectrometry (UHPLC–QTRAP[®] MS). Marine water samples from the open Adriatic Sea taken 16 km offshore from Venice (Italy) were analyzed. Method limits of quantification (LOQs) in the low picogram per liter (pg/l) concentration range were achieved. Among the 67 target chemicals analyzed, 45 substances could be detected above the LOQ. The chemicals detected at the highest concentrations were caffeine (up to 367 ng/l), nitrophenol (36 ng/l), 2,4-dinitrophenol (34 ng/l), 5-methyl-

1*H*-benzotriazole (18.5 ng/l), sucralose (11 ng/l), 1*H*-benzotriazole (9.2 ng/l), terbutylazine (9 ng/l), alachlor (7.7 ng/l), atrazine-desisopropyl (6.6 ng/l), diethyltoluamide (DEET) (5.0 ng/l), terbutylazine-desethyl (4.3 ng/l), metolachlor (2.8 ng/l), perfluorooctanoic acid (PFOA) (2.5 ng/l), perfluoropentanoic acid (PFPeA) (2.3 ng/l), linuron (2.3 ng/l), perfluorohexanoic acid (PFHxA) (2.2 ng/l), diuron (2.0 ng/l), perfluorohexane sulfonate (PFHxS) (1.6 ng/l), simazine (1.6 ng/l), atrazine (1.5 ng/l), and perfluorooctane sulfonate (PFOS) (1.3 ng/l). Higher concentrations were detected during summer due to increased levels of tourist activity during this period.

Keywords Adriatic Sea · UHPLC-MS/MS · Multicomponent chemical analysis · Pesticides · Pharmaceuticals · Perfluoroalkyl substances

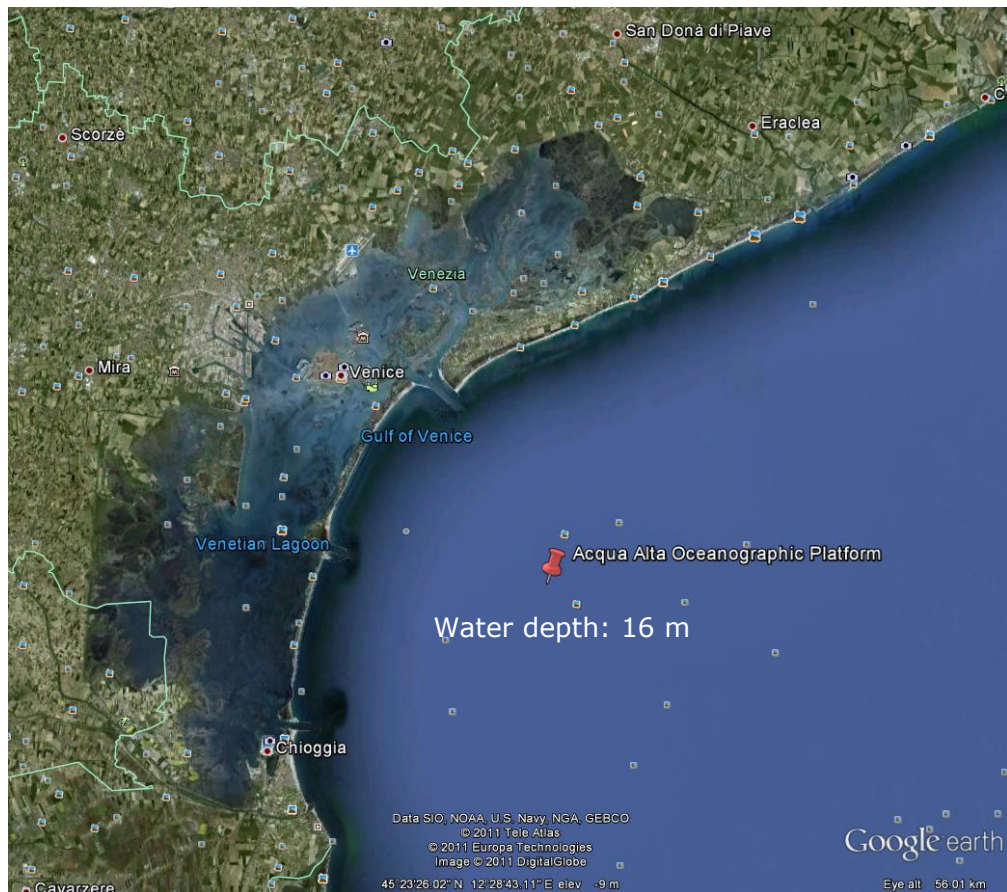
Water samples taken in 2011/12.



Adriatic Sea



Venice, 16 km off-shore



Acqua Alta Oceanographic Platform (CNR)

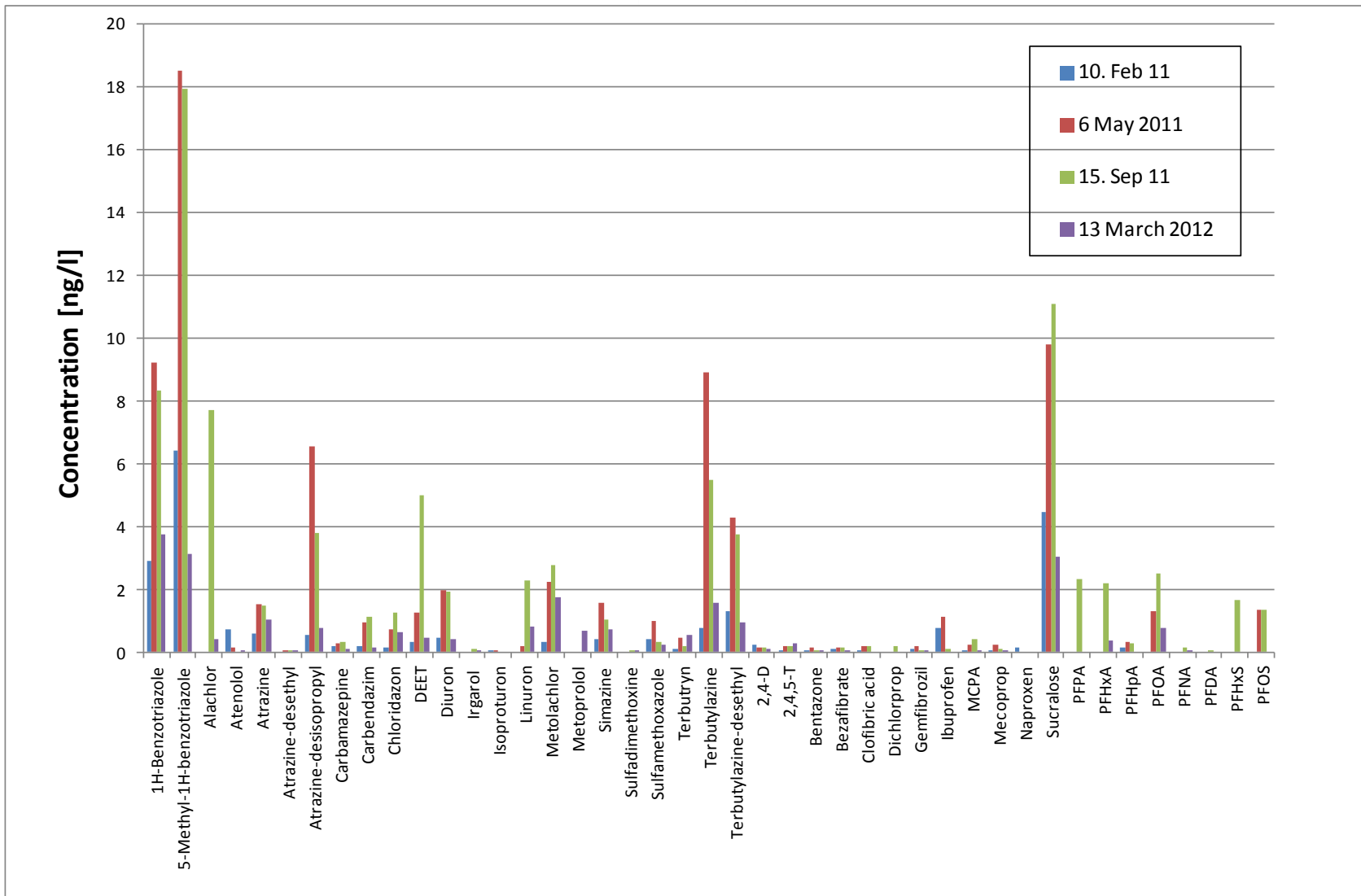


21 October 2013

Joint
Research
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Adriatic Sea



Caffeine	367 ng/l	Metolachlor	2.8 ng/l
Nitrophenol	36 ng/l	PFOA	2.5 ng/l
2,4-Dinitrophenol	34 ng/l	Linuron	2.3 ng/l
		PFPeA	2.3 ng/l
		PFHxA	2.2 ng/l
5-Methyl-1H-benzotriazole	18.5 ng/l	Diuron	2.0 ng/l
Sucralose	11.1 ng/l	Simazine	1.6 ng/l
1H-Benzotriazole	9.2 ng/l	PFHxS	1.6 ng/l
Terbutylazine	9 ng/l	Atrazine	1.5 ng/l
Alachlor (one sample)	7.7 ng/l	PFOS	1.3 ng/l
Atrazine-desisopropyl	6.6 ng/l	Chloridazon	1.3 ng/l
DEET	5.0 ng/l	Ibuprofen	1.1 ng/l
Terbutylazine-desethyl	4.3 ng/l	Carbendazim	1.1 ng/l
		Sulfamethoxazole	1.0 ng/l

Chemical

Adriatic Sea

PFOA

0.79 – 2.51 ng/l

PFOS

1.34 ng/l

Literature

North Sea: **0.11–7.8 ng/l**

(Ahrens et al. 2009. Chemosphere 76, 179-184;
Möller et al. 2010. Environ. Pollut. 158, 3243-3250;
Willie et al. 2010. J. Chromatogr. A 1217, 6616–6622)

China (Pacific; coastal): **38-1542 pg/l**

(Cai et al. 2012. Environ. Pollut. 161, 162-169)

Catalonian Coast: **<0.08-1.86 ng/l**

(Sánchez-Avila et al. 2010. Mar. Pollut. Bull. 60 (1), 103–112)

Atlantic Ocean: **229-439 pg/l (max.)**

(Ahrens et al. 2009. ES&T 43, 3122–3127;
Yamashita et al. 2005. Mar. Pollut. Bull. 51, 658–668)

North Sea: **0.25-4.2 ng/L**

China (Pacific; coastal): **<20.7-70.3 pg/l**

Catalonian Coast: **<0.03-3.93 ng/L**

Atlantic Ocean: **73-291 pg/l (max.)**

PFASs in Aquatic Biota



European Commission

Monitoring of Perfluorinated Compounds in Aquatic Biota: An Updated Review

PFCS in Aquatic Biota

Magali Houde,^{*,†} Amila O. De Silva,[‡] Derek C. G. Muir,[‡] and Robert J. Letcher[§]

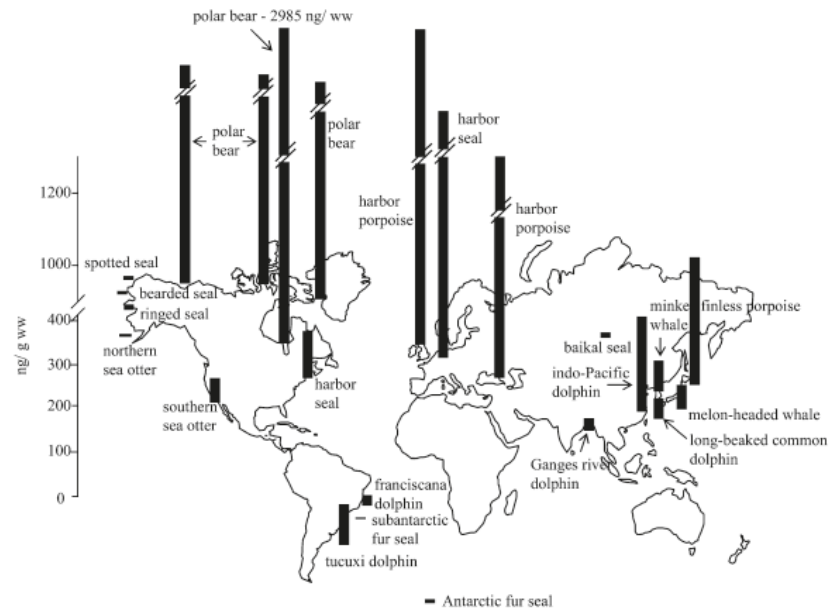
[†]Environment Canada, Centre Saint-Laurent, 105 McGill Street, Montréal, Québec, Canada, H2Y 2E7

[‡]Environment Canada, National Water Research Institute, 867 Lakeshore Road, Burlington, Ontario, Canada, L7R 4A6

[§]Environment Canada, Wildlife and Landscape Science Directorate, National Wildlife Research Centre, Carleton University, 1125 Raven Road, Ottawa, Ontario, Canada, K1A 0H3

Supporting Information

ABSTRACT: The goal of this article is to summarize new biological monitoring information on perfluorinated compounds (PFCs) in aquatic ecosystems (post-2005) as a followup to our critical review published in 2006. A wider range of geographical locations (e.g., South America, Russia, Antarctica) and habitats (e.g., high-mountain lakes, deep-ocean, and offshore waters) have been investigated in recent years enabling a better understanding of the global distribution of PFCs in aquatic organisms. High concentrations of PFCs continue to be detected in invertebrates, fish, reptiles, and marine mammals worldwide. Perfluorooctane sulfonate (PFOS) is still the predominant PFC detected (mean concentrations up to 1900 ng/g ww) in addition to important concentrations of long-chain perfluoroalkyl carboxylates (PFCA; sum PFCA up to 400 ng/g ww). More studies have evaluated the bioaccumulation and biomagnification of these compounds in both freshwater and marine food webs. Several reports have indicated a decrease in PFOS levels over time in contrast to PFCA concentrations that have tended to increase in tissues of aquatic organisms at many locations. The detection of precursor metabolites and isomers has become more frequently reported in environmental assessments yielding important information on the sources and distribution of these contaminants. The integration of environmental/ecological characteristics (e.g., latitude/longitude, salinity, and/or trophic status at sampling locations) and biological variables (e.g., age, gender, life cycle, migration, diet composition, growth rate, food chain length, metabolism, and elimination) are essential elements in order to adequately study the environmental fate and distribution of PFCs and should be more frequently considered in study design.



Recently reported (2006–2010) PFOS concentrations in liver of marine mammals worldwide.^{6–8,10,12,13,15,19,21,30,36,65–67,102–104}

Environ. Sci. Technol. 45, 2011, 7962–7973.

Harbor seals in the German Bight
PFOS: predominant compound with a maximum concentration of **3676 ng/g**.

Ahrens, L.; Siebert, U.; Ebinghaus, R. Total body burden and tissue distribution of polyfluorinated compounds in harbor seals (*Phoca vitulina*) from the German Bight. *Mar. Pollut. Bull.* 2009, 58, 520–525.

PFASs in wild fish from Northern Germany

Fish filet samples (eels, bream, herring, mackerel, carp, trout)

PFOS was found in the filet samples caught from densely populated regions at levels between **8.2 and 225 µg/kg**.

Marine or remote locations: **PFOS: up to 50.8 µg/kg**.

Schuetze, A.; Heberer, T.; Effkemann, S.; Juergensen, S. Occurrence and assessment of perfluorinated chemicals in wild fish from Northern Germany. *Chemosphere* **2010**, 78, 647–652.

PFASs in muscle tissue from edible fish species from the second largest freshwater lake in Sweden, Lake Vättern (LV), and in brackish water of the Baltic Sea

PFOS concentrations were higher in LV (medians **2.9–12 ng/g**) than in Baltic Sea water fish (medians **1.0–2.5 ng/g**).

Berger, U.; Glynn, A.; Holmstrom, K.E.; Berglund, M.; Ankarberg, E. H.; Tornkvist, A. fish consumption as a source of human exposure to perfluorinated alkyl substances in Sweden: Analysis of edible fish from Lake Vattern and the Baltic Sea.

Chemosphere **2009**, 76, 799–804.

PFASs in high mountain lakes in France

PFOS: 3.61–4.24 ng/g.

Ahrens, L.; Maruszczak, N.; Rubarth, J.; Dommergue, A.; Nedjai, R.; Ferrari, C.; Ebinghaus, R. Distribution of perfluoroalkyl compounds and mercury in fish liver from high-mountain lakes in France originating from atmospheric deposition. *Environ. Chem.* **2010**, 7, 422–428.

Environ. Chem. **2010**, 7, 422–428.

Conclusions

- **Lack of monitoring data** (spatial and temporal variability)
- **The JRC focuses its monitoring on EU-wide studies**
- **Important for the prioritization of chemicals**
- **Next year the JRC will take over the lead on the prioritization under the WFD**
- **Monitoring data, effect data, and modelling**
- **PFOS compliance monitoring (EQS: 0.65 ng/L)**
- **Biota EQS: 9.1 µg/kg**

Conclusions

- **Dietary intake** is supposed to be an important path of exposure for the general population to **PFOS and PFOA**.
- Contamination of **drinking water** led to significantly increased PFOA concentrations, in **blood samples** of the affected populations in Little Hocking, Ohio, **USA**, Arnsberg, **Germany**, and Minnesota, **USA**.
- **Fish** is an important part of the diet and recently the significance of fish consumption on the internal exposure to **PFOS** was emphasized.
- **Continued use of PFOA and PFOS precursors**

“Polar Bear Brains have High Levels of Environment Toxins”

Nature World News; 24 July 2013



Greaves, A.K., et al.
BRAIN REGION DISTRIBUTION AND PATTERNS OF BIOACCUMULATIVE PERFLUOROALKYL CARBOXYLATES AND SULFONATES IN EAST GREENLAND POLAR BEARS (*URSUS MARITIMUS*)
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21 October 2013

Thank you for your attention !



Monte Rosa
(4.634 m)

Lago Maggiore

Ispra

JRC