

WELCOME TO ISSUE N° 1

OF THE NORMAN NETWORK NEWSLETTER

The aim of the activities of the NORMAN network is to enhance the exchange of information on emerging environmental substances, and to encourage the validation and harmonisation of common measurement methods and monitoring tools so that the requirements of risk assessors and risk managers can be better met. The NORMAN newsletter is for everyone interested in emerging substances in the environment. This newsletter keeps you up to date on scientific advances in this area and highlights the activities and events of the EU NORMAN Network.

Editorial

« Emerging Pollutants »

Questions, Challenges, and the Future

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Before venturing further down the "emerging pollutants" path, let's pause and consider our destination. How will we know when we're there? Is it when we've made significant progress in cataloging that portion of the chemical universe ("chemical space") important to the environment and have the capability to monitor for these substances in real time? Or is it rather when we know the significance and ramifications of these substances, and have the ability to monitor their impacts on ecological systems? The first is an endeavor for the physical sciences (and one that may not be achievable given the enormity of chemical space), and the second requires major advances in the natural sciences (particularly human and environmental toxicology). Should we not marry the two?

"Emerging pollutants" as the focus of the NORMAN Project marks the first time an international program will tackle this rather ill-defined aspect of environmental science. The project will foster more attention on unregulated pollutants, a topic that has blossomed over the last 10 years — primarily as a result of analytical chemistry's penchant for ever-lower levels of detection

and improved abilities in identifying "mystery unknowns", not necessarily because of demonstrated negative impacts of these substances on the environment or human health.

Are we clear in what we strive to communicate with the term "emerging" pollutants? In the U.S., this term was first used in the late 1990s, most prominently by the National Research Council ("Identifying Future Drinking Water Contaminants: Based on the 1998 Workshop on Emerging Drinking Water Contaminants", National Academy Press, Washington, DC, 1999; <http://www.nap.edu/catalog/9595.html>) and by the USGS in their Toxic Substances Hydrology "Emerging Contaminants" Project (<http://toxics.usgs.gov/regional/emc/index.html>), where the acronym "EPOCs" was coined ("Emerging Pollutants of Concern"; <http://sofia.usgs.gov/projects/epocs/>). In response to the Safe Drinking Water Act Amendments of 1996, the U.S. EPA implemented the Contaminant Candidate List (CCL) in 1998 to identify drinking water contaminants that could be candidates for future regulation. The CCL, updated every 5 years, is a process that draws partly on emerging knowledge of new pollutants.

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Usage of the term "emerging pollutants" has proliferated across the technical/trade literature as well as the popular press. Unfortunately, expansion in the term's usage has muddied its meaning — growing so broad that at times it seems to encompass all chemical pollutants not regulated, and, paradoxically, even some whose environmental presence is sufficiently understood (e.g., certain highly fluorinated chemicals) that some actions are already underway regarding their regulation or control.

Has popularization of the term "emerging pollutants" diluted its meaning, especially when used to categorize substances long-known to persist in the environment? If we simply mean "unregulated pollutants," what is the value added by "emerging"? Three of many possible examples of pollutants sometimes still categorized as "emerging" but which were identified as problematic in the last century include PBDEs (e.g., *Chemosphere* 1987, 16[10-12]:2343-2349), DBPs (e.g., *Water Res.* 1988, 22[7]:803-813), and perchlorate (e.g., *J. Environ. Manage.* 1999, 56[2]:79-95). Perhaps what is intended by use of the term is not "emerging pollutants of concern" but rather "pollutants of emerging concern." Discussions of semantics might seem rather academic but they can prove important in understanding the scope and objective of the topic.

The pie delineated by "chemical space" can be sliced in any number of dimensions to yield portions of the chemical universe defined and named in various ways, producing a babel of terms (e.g., EDCs, PPCPs, PBTs, POPs, EPOCs, etc.), all of which overlap to various degrees, sharing subsets of substances with one another. Concerted discussion of the context of the term "emerging pollutants" didn't occur until 2001 (*J. Am. Soc. Mass. Spectrom.* 2001, 12[10], 1067-1076), where it is noted that "emerging" had been used to describe substances that span several different categories, most of which are not truly emerging; the term was further elaborated upon in 2004 (*Environ. Impact Assess. Rev.* 2004, 24[7-8]:711-732) and 2005 (*Renew. Resourc. J.* 2005, 23[4]:6-23).

Why does the notion of emerging pollutants exist, and why is it still ill-defined? Initially, environmental chemists wanted to distinguish certain "novel" unregulated pollutants from those that are regulated. But the universe of non-regulated chemicals is extraordinarily large (*Renew. Resourc. J.* 2005, 23[4]:6-23). In its most restricted sense, the term should perhaps be applied only to those substances that have been newly introduced to the environment (three examples being new drug entities, new pesticide active ingredients, and new nanomaterials). In its broad usage, however, this facet of the term seems to be the minor one. The major ones seem to be driven more from our "emerging recognition" of problematic substances, even those that have long been present.

To emphasize the strict meaning of "emerging" pollutants as "mystery" substances, we could refer to them as "stealth" pollutants. These substances have eluded attention because they are neglected, overlooked, omitted, or ignored as a result of the design of environmental monitoring programs (e.g., pre-defined, target-based monitoring) or because of the limitations of analytical chemistry.

Establishing a stealth environmental presence results from any number of reasons — because these substances:

(i) are concealed, hidden, cloaked, or masked (e.g., by properties of the environment or matrix that deter or foil their detection; examples being low levels obscured by interferences and irreversible matrix sequestration);

(ii) are indiscernible (e.g., concentrations are below analytical limits of detection);

(iii) result from clandestine or surreptitious operations (introduced to the environment by unknown or unidentifiable activities or processes, or have simply been overlooked [e.g., illicit drugs]; or they reside in compartments that previously escaped suspicion [e.g., acrylamide in food]);

(iv) have analytical signatures that are undecipherable or cryptic (e.g., convoluted mass spectra); or

(v) were previously unsuspected (e.g., unanticipated degradates/transformation products; so-called inert ingredients in pesticide and consumer/food product formulations). These distinctions, while seeming to merely play with semantics, are important for catalyzing discussion as to the what, where, why, and how "emerging" pollutants originate.

Returning to the question of our destination, perhaps our ultimate objective should be to advance our knowledge and environmental monitoring abilities to the point where the need for the term "emerging" disappears altogether. This could be done by:

(i) developing the ability to predict the probability of any new substance to enter and distribute in the environment as well as the presence of preexisting stealth pollutants (accompanied by the design and implementation of early-warning environmental monitoring networks for detecting any substance that newly appears — not by target analysis, but rather by "change detection");

(ii) shifting from a chemical-centric focus to that of actual biological impacts (biological effects-directed vigilance as opposed to chemical-directed monitoring), and

(iii) minimizing the possibility of stealth pollutants via concerted application of environmental monitoring, life-cycle analysis, and environmental stewardship.

NOTICE:

The United States Environmental Protection Agency through its Office of Research and Development funded and managed the research described here. It has been subjected to Agency's administrative review and approved for publication as an EPA document.

Monitoring and Bio-monitoring

Occurrence and removal of pharmaceuticals in sewage treatment plants in Italy and effects on human health

Human pharmaceuticals are an important class of emerging environmental pollutants. The potential toxicological risk associated with the presence of low levels of pharmaceuticals in aquatic environments is currently under debate.

Pharmaceutical compounds such as non-steroid anti-inflammatory drugs (ibuprofen or diclofenac) and carbamazepine can be found in the environment. Researchers in Europe began in the early 1990s to identify trace amounts of certain therapeutic drugs in surface waters

and groundwater. Since then, survey campaigns in Europe and the U.S. have found about 100 such compounds in surface waters, groundwater, sewage, effluents from wastewater treatment plants, and even tap water. Pharmaceutical compounds enter the environment via several

different pathways, including improper disposal and the excretion of non-metabolized drugs. Conventional biological wastewater treatments are quite inefficient in removing pharmaceutical compounds, since they are found in significant amounts in STP effluents and receiving waters.

Scientists from the University of Insubria (Varese, Italy) and the Mario Negri Institute (Milan, Italy) have in recent years studied the occurrence and the environmental fate of pharmaceuticals in sewage treatment plants (STPs) and the corresponding receiving rivers in Italy. Their results have recently been published in ES&T [1].

The Italian scientists first performed a listing of "priority pharmaceuticals" for human use in Italy, which resulted in the selection of 26 pharmaceuticals, belonging to 11 therapeutic classes. These pharmaceuticals were analyzed by liquid chromatography-tandem mass spectrometry (LC-MS-MS). Their occurrence was assessed in six sewage treatment plants (STPs) in towns spread over different Italian locations, and the loads and the removal rates (RR) were studied. Influent and effluent samples of all the STPs were sampled for mass balance calculations taking 24-hour composite pool samples. Surface water composite samples were also taken at the receiving rivers.

Total pharmaceutical loads ranged from 1.5 to 4.5 g/day/1000 inhabitants in influents and 1.0 and 3.0 g/day/1000 inhabitants in effluents. Total RR in STPs were mostly lower than 40%. Pharmaceuticals could be divided into three groups according to their behaviour in STPs: one group with RR higher in summer than in winter, one group with RR similar in summer and winter, and a last group not removed.

Occurrence, distribution and fate of pharmaceutical compounds were also studied in the surface waters receiving the effluents of the STPs. Degradation and sorption were identified as the major factors affecting attenuation of the pharmaceuticals in the investigated rivers. In these Italian studies ciprofloxacin, ofloxacin, sulfamethoxazole (antibiotics), atenolol (cardiovascular drug), ibuprofen (anti-inflammatory), furosemide, hydro-chlorothiazide (diuretics), ranitidine (gastrointestinal drug), and benzafibrate (lipid regulator) were the most abundant residual drugs, and therefore of environmental concern.

Behaviour and removal of pharmaceutical compounds in STPs has also been studied in other countries. For example, results for carbamazepine

show that only 29% of this compound was removed from the aqueous phase during treatment in a Canadian STP, while the metabolites of carbamazepine were not effectively removed at all [3]. Similar RR were found for carbamazepine in Austria [4], Germany and Switzerland.

For ibuprofen however, a RR of > 90 % was reported [4,5]. In a Spanish study the overall removal efficiency in a STP was between 45% and 70% for acidic pharmaceuticals (e.g. ibuprofen), around 67% for estradiol and 57% for the antibiotic sulfamethoxazole [6].



HUMAN HEALTH RISKS FROM PHARMACEUTICALS?

Researchers agree that aquatic species face the greatest risk from exposure to low levels of pharmaceuticals, such as synthetic hormones, which can act as endocrine disrupters at environmental levels. However, little is known about the potential human health effects arising from complex drug mixtures.

The same Italian scientists investigated whether drugs found in water can be harmful to humans. The researchers designed a cocktail of 13 drugs — including several antibiotics, the popular pain reliever ibuprofen, and a highly toxic cancer medicine — to mimic the mixtures found in several Northern Italian rivers and in wastewater. In the first study to examine the effects of a drug mixture at environmental levels on human cells, they report that a combination of pharmaceutical compounds inhibits the growth of embryonic kidney cells in laboratory tests. These results suggest that water-borne pharmaceuticals can be potential effectors on aquatic life [2].

Pharmaceuticals and personal care products have also been found in drinking water (in the USA) produced from rivers affected by sewage treatment plant effluents [7-8]. This leads to human health concerns because little is known about health risks from such compounds.

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Sources, fate and transport of perfluorocarboxylates

Perfluoroalkylcarboxylates [$F(CF_2)_nCO_2$, $n \geq 7$; PFCAs] and their potential precursors are of increasing and regulatory interest because they have been found globally in wildlife and in humans. However, their physical-chemical properties, fate and transport are not well known. This article reviews the direct and indirect sources of PFCAs released in the environment and provides indications about their fate and transport, focussing on perfluorooctanoate (PFO) as representative of PFCAs in general.

The authors estimate that the global historical emissions of total PFCAs from direct sources (manufacture and use of PFCAs) and indirect sources (PFCAs present as chemical reaction impurities or degradation of precursors to form PFCAs) is 3200-7300 tonnes.

The principal direct source of PFCA emissions to the environment is associated with the use of PFCAs in the manufacture of fluoropolymers. As a matter of fact, ammonium salts of perfluorooctanoic acid (PFOA) and perfluorononanoic acid (PFNA) have been used for over fifty years as processing aids to facilitate aqueous polymerisation of fluoropolymers such as, polytetrafluoroethylene (PTFE) and polyvinylidene fluoride (PVDF). The emissions of PFCAs from this process are reported to be approximately 60% of the total PFCAs used, with 23%, 65% and 12% distributed to air, water and land, respectively. Fluoropolymer manufacturers have recently installed additional capability to capture and recycle APFO, thereby leading to dramatic (→90%) reductions in overall environmental releases over the past few years (from hundreds of t in 1999 to less than 50 t projected in 2006). Fluoropolymer producers who use APFN have forecast a 67% reduction in emissions by 2006.

Additional direct sources of PFCA emissions include:

- The manufacture of PFO/APFO and PFN/APFN
- The processing of fluoropolymer dispersions
- The use of aqueous fire-fighting foams (AFFF) containing PFCAs
- The use of PFCA and derivatives as additives in industrial and consumer products.

The main indirect sources of PFCA emissions to the environment include:

- The impurities contained in perfluorooctyl sulphonyl-based (POSF) products made by the ECF process: the production of these products began in the 1960s and was phased out in 2003. They were used world-wide in industrial and consumer products (e.g. food-contact paper, spray cans for do-it-yourself application). POSF-based products contained between 200 and 600 ppm of PFO. The PFCA impurities present in POSF-based products were emitted to the environment (mainly air and water) from point sources as well as dispersive use and disposal of industrial and consumer products. POSF-based products were also used from the 1970s forward as surfactants in AFFF in replacement of PFCA-based foams.
- The impurities contained in fluorotelomer-based products and the degradation of precursors: fluorotelomer alcohol (FTOH) and fluorotelomer olefin (FTO), are typically present in the fluorotelomer-based polymers as residual raw material and reaction by-products, respectively. They may react during chemical processing and form PFCAs. Moreover, if not removed, they remain in the ultimate sales. Releases to the environment (air or water) were reported from manufacturing facilities and from industrial application of fluorotelomer-based products to textiles. Finally, environmental degradation of FTOH and FTO is also a potential indirect source of PFCAs. FTOH present in air in the gas phase are reported to be transformed to PFCA by reaction with HOx in the absence of NOx. FTO are likely to degrade by reaction with HOx by the same transformation pathway. FTO may also form PFCAs by reaction with ozone.

However, due to substantial data gaps it is not possible at present to draw clear conclusions on the atmospheric partitioning and fate of these substances.

The physical-chemical properties of PFO (negligible vapour pressure, high solubility in water, and moderate sorption to solids) suggest that PFO would accumulate in surface waters.

Environmental monitoring data have been reviewed and used to estimate mass inventories in different environmental compartments. In spite of limitations associated with lack of monitoring data and wide variability in reported environmental concentrations, calculations confirm that oceans contain the majority of PFO historically released, followed by freshwater and sediment.

As regards air, PFCAs have not been reported in the vapour or particle phase of background air samples. However, the presence of PFO in rainfall and snow indicated the atmospheric presence of PFCAs.

This could be the result of point sources in the immediate vicinity of the sampling stations, or the atmospheric transport of PFCAs themselves or volatile precursors which undergo transformation to PFCAs. More research on this topic is needed. In any case it is expected that the total mass in the atmosphere would be low compared to the masses in surface waters and sediments.

It was not possible to calculate a soil inventory. Soils receive PFCAs via atmospheric wet deposition and possibly dry deposition, but retention by soil is expected to be low compared to other hydrophobic organic compounds. Monitoring of PFCAs in background soils is needed to test this hypothesis.

The only environmental sinks for PFO are considered to be sediment burial and transport to the deep oceans. This implies a very long residence time. It is therefore believed that only a small percentage of the PFO historically released has been removed from the environment.

Various transport pathways have been investigated to explain the presence of PFCAs in remote areas such as Arctic and their widespread global distribution. The degradation and transport of volatile precursors such as FTOHs have been hypothesised as the main source of long-chain PFCAs in remote regions such as the Arctic. Yet to be investigated are potential precursors such as fluorotelomer olefins (FTOs) and perfluorosulphonyl chemicals.

There is also evidence that APFO/PFOA is directly released to the atmosphere at fluoropolymer manufacturing facilities. However, further studies are required to determine whether direct atmospheric transport of APFO/PFOA is a significant pathway for long-range transport of PFCA.

Another possible contributor to long-range transport is the marine aerosol. Due to their high solubility in water and their accumulation in the upper sea layers, PFCAs can be supplied to the atmosphere through marine aerosols. Studies are needed to determine whether and to what extent marine aerosols contain PFCAs and may contribute to their global transport.

In addition to atmospheric transport, ocean water transport could significantly contribute to long-range transport of PFCAs. PFCAs are water soluble and persistent chemicals which therefore have a high potential for long-range aquatic transport to the Arctic. By taking the total flow of water entering the Arctic surface ocean and the lowest reported open ocean PFO water concentrations of 0.015-0.062 ng/l as representative of background level in the northern hemisphere, the

authors estimated the amount of PFO that reaches the Arctic at 2 and 12 t per year. Even though ocean water transport is relatively slow compared to atmospheric transport, considering that the PFCA have been emitted for about 50 years and have been accumulating in the oceans, the amount of PFCA delivered to the Arctic through ocean water

transport is likely to exceed the amount delivered from northern atmosphere from FTOH degradation and subsequent atmosphere deposition, which is between 0.1 and 1 t per year. In any case, the relative importance of these two pathways should be further examined.

SOURCE:

Konstantinos Prevedouros, Ian T. Cousins, Robert C. Buck, Stephen H. Korzenioski. (2006). "Sources, fate and transport of perfluorocarboxylates." *Environ. Sci. Technol.* 40(1): 32-44.

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Environmental and human health risk assessment

Ecotoxicogenomics: linkages between exposure and effects in assessing risks of aquatic contaminants to fish

Genomics technologies are a quickly emerging sub-field within ecotoxicology, rapidly displaying their potential implications for human and ecological risk assessment issues. By means of the similarly booming field of bioinformatics, it becomes possible to link data from 'omics' technologies such as gen-, prote-, transcript-, and metabonomics with data from population genetics, histopathology and ecology to produce a new generation of cause-effect relations of events occurring within an organism, from sources of stressors through exposure and, ultimately, to outcomes. The current generation of risk assessment models is incapable of examining dose-effect relations without extrapolation. Ecotoxicogenomics build upon establishing large exposure-to-outcome databases and could ultimately allow for a substantial reduction of uncertainties in risk assessment. In the review by Miracle and Ankley, the aquatic ecotoxicology of fish is used as an example to illustrate the need to link classic and emerging toxicological disciplines.

A historical perspective of the evolution of fish testing over the last 40 years is given, starting from short-term lethality studies and ending with full life-cycle assays, extrapolation to effects at the community level, and the recognition of mechanisms of action that can result in very sensitive responses (such as endocrine disrupting activity). The merits and disadvantages of currently common fish tests are discussed and it is made clear that a dynamic picture of adverse effects in biological systems is currently lacking.

Dynamic diagnostics by means of the 'omics' technologies indicated above (and discussed in more detail in Miracle and Ankley) and increased understanding of modes of action and disease are clearly highlighted. It is on the other hand recognised that bioinformatics is the limiting step when it comes to the interpretation of the wealth of data generated. Thereupon, potential application of 'omics' technologies in risk assessment of mixtures of chemicals, identification of chemicals with similar modes of action and species extrapolation are clearly highlighted. Nevertheless, it is concluded that, given the multitude of the potential advantages to diagnostic and predictive risk assessment, bioinformatics are not broadly sought or used. This is probably due to the fact that identification of modes of action can be achieved only by collecting data at lower biological levels of organisation (usually molecular and cellular level). The significance of alterations at these lower levels is in general uncertain in terms of effects at individual and population levels. Research on the delineation of the cascade of events that occur from a molecular initiating event (receptor interactions, enzyme induction, etc.) through responses at the cellular and tissue levels, to adverse outcome in the animal, is clearly needed before ecotoxicogenomics are to be considered a mature tool for more accurate ecological effect and risk assessment. As such, the paper provides a nice brief overview of current developments within this relatively new research area.

SOURCE:

A.L. Miracle, G.T. Ankley, "Ecotoxicogenomics: linkages between exposure and effects in assessing risks of aquatic contaminants to fish." *Reproductive Toxicology*, 19 (2005), 321-326.

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Lessons from endocrine disruption and their application to other issues concerning trace organics in the aquatic environment

This article reviews the past 10 years of research on endocrine disruptors, focusing on the exotoxicological effects. The authors do an admirable job summarising the literature, summarising it into 10 “lessons learned”:

1. One must observe biological systems carefully, as in all cases to date endocrine disrupting effects were identified by accidental observation of wildlife.
2. It is important to define what is “normal”, since many effects related to endocrine disruption (e.g. imposex) can occur in unexposed populations.
3. The response to endocrine disrupting chemicals may vary widely between groups of organisms. While most vertebrates respond in a similar way, invertebrates can respond very differently than vertebrates, and there can be large differences between vertebrates.
4. The potency of endocrine disruptors varies widely. Almost all of the evidence to date suggests that the very potent natural and synthetic steroidal estrogens are the primary causes of feminisation of fish in most cases. A few situations have been identified where the concentrations of less potent alkylphenolic chemicals contribute to feminization.
5. Chemical degradation and transformation can be important, such as when alkylphenol polyethoxylates break down to nonylphenol and related compounds, or when inactive conjugates of steroidal estrogens excreted by humans are cleaved in sewage treatment plants to give the free estrogen.

6. Endocrine disrupting effects are often only observed after continual exposure to low doses, which means that low volume chemicals cannot be excluded a priori. In addition, organisms are generally exposed to mixtures of chemicals, and mixture effects must be taken into consideration.
7. One must be aware of unusual pollutants from unexpected sources. Natural steroidal estrogens are one good example.
8. Acute toxicity tests may not be very helpful, as illustrated by the fact that the EC₅₀ of EE₂ for vitellogenin is more than 6 orders of magnitude lower than the LC₅₀ in the fathead minnow.
9. Sewage treatment plays a key role, both as the source of endocrine disruptors to aquatic ecosystems and in attenuating their release.
10. Hydrology, and in particular the extent to which sewage treatment plant effluents are diluted by the recipient waters, is a key factor in determining where and when endocrine disrupting effects may occur.

Based on these lessons, the authors make several suggestions. They emphasise that research should focus on key issues, e.g. on chemicals that have been demonstrated to have effects in wildlife, rather than looking for yet another chemical which is weakly estrogenic. It is convincingly argued that multidisciplinary teams could be more effective at bringing the science forward. The review closes with a plea for a more structured approach to risk assessment of emerging complex chemical issues such as pharmaceuticals, one that uses available knowledge on chemical use, release, properties, fate, dilution, exposure, bioaccumulation, and mechanisms to prioritise chemicals, locations, and organisms that are most likely to be affected. In summary, a broad, insightful, and accessible analysis with a lesson for everyone.

SOURCE:

Sumpter, John P. ; Johnson, Andrew C. (2005). "Lessons from endocrine disruption and their application to other issues concerning trace organics in the aquatic environment." *Environ. Sci. Technol.* 39(12): 4321-4332.

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Environmental exposure pathways for decaBDE

Remarkable findings have recently been published on the occurrence of decabromo diphenyl ether (BDE₂₀₉) in terrestrial top-predators (Lindberg et al., 2004; Vorkamp et al., 2005; Christensen et al., 2005; Voorspoels et al., 2006). In grizzly bears from British Columbia and red foxes from Belgium the dominant polybrominated diphenyl ether (PBDE) congener was unexpectedly BDE₂₀₉. BDE₂₀₉ was frequently detected in fox liver (40% of the analysed samples) in levels ranging from < 4 to 760 ng/g lipid weight (Voorspoels et al., 2006). The relatively high levels of BDE₂₀₉ in mammals were surprising, as this conflicts with the expected short half-lives of BDE₂₀₉ in mammals; 7 days (Hagmar et al., 2000), 15 days (Thuresson et al. 2006) in humans and 2.5 days in rats (Mörck et al., 2003). In general, BDE₂₀₉ is metabolised much faster than the lower brominated PBDEs, and the assimilation efficiency in rats is low (about 10%) (Mörck et al., 2003) due to the large

molecule size and extreme hydrophobicity. Interestingly, BDE₂₀₉ could not be found in the main prey species, mice and voles, of the red fox (Voorspoels et al., 2005). The grizzly bear study showed that different PBDE profiles were found between maritime bears – mainly feeding on marine and terrestrial foods – and inland bears – exclusively feeding on terrestrial food. The profiles in maritime bears were dominated by BDE₄₇ > BDE₂₀₉ > BDE₉₉ > BDE₁₀₀ > BDE₁₅₃, while the inland bears' profiles were dominated by the higher brominated congeners: BDE₂₀₉ > BDE₂₀₆ > BDE₄₇ > BDE₂₀₇ > BDE₂₀₈ (Christensen et al., 2005). These results contrast with the PBDE profiles found in terrestrial birds (peregrine falcons) from Sweden (Lindberg et al., 2004) and Norway (Vorkamp et al., 2005), where BDE₁₅₃, BDE₉₉, BDE₄₇, and BDE₁₀₀ are the dominant congeners, and not the higher brominated congeners (e.g. BDE₂₀₉).

These findings suggest that i) terrestrial mammals are exposed to relatively high levels of BDE209, and ii) exposure pathways other than the main food constituents are important for terrestrial mammals. Possible exposure pathways for these animals could be of food with soil, vegetation, air particles, human garbage and sewage sludge. An overview of the exposure pathways for BDE209 is given in Figure 1. Christensen and co-workers (2005) suggest that the air-to-plant partitioning of BDE209 may play an important role in the exposure pathway. Airborne PBDEs are mainly bound to particles and dominated by BDE209 (e.g. ter Schure et al., 2004), and the total deposition of PBDEs in e.g. Sweden is dominated by BDE209 (ter Schure and Larson, 2002). Spray irrigation with treated municipal wastewater has recently been found as a potential source of PBDEs in the atmosphere (Goel et al. 2006). The use of sewage sludge on agricultural and other land might be another source of PBDE exposure for the terrestrial environment. High levels of PBDEs, dominated by BDE209, have been found in sewage sludge (e.g. de Boer et al., 2003). The role of human garbage as a source of PBDE exposure for terrestrial animals has been indicated (e.g. Christensen et al., 2005; Voorspoels et al., 2006), but never studied in detail. For birds, e.g. herring gulls, this might also be a source of exposure, but this exposure pathway needs further attention.

In conclusion, the PBDE exposure pathways in terrestrial wildlife are poorly understood and attention should be given to describing the flows and sources of exposure.

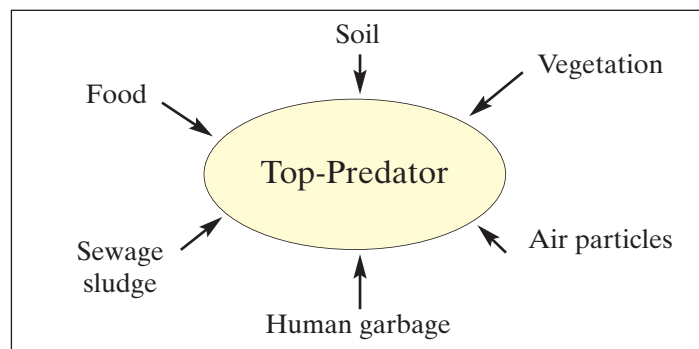


Figure 1: Suggested exposure pathways of BDE209 for terrestrial top-predators. The significance and flows of the pathways has never been studied in detail.

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Sub-nanogram per litre detection of the emerging contaminant progesterone with a fully automated immunosensor based on evanescent field techniques

The paper by Tschmelak et al. describes the analysis of progesterone found in various surface waters and anticipated to be a human carcinogen. The technique is the enzyme-linked immunosorbent assay (ELISA) using a polyclonal antibody anti-progesterone IgG from sheep and an analyte derivative for the surface modification.

The originality of the technique is that it is fully automatised for routine analyses.

The technique is very promising, as tests with spiked Milli-Q water showed a limit of detection (LOD), calculated using three times the standard deviation of the twelve independent blank measurements, of 0.37 ng/l. The sample volume necessary for the measurement is only 900 µl; sample water does not need any preconcentration. Common analytical methods like GC/MS and HPLC/MS require pre-concentration of the water sample and derivatisation for some compounds to reach

LOD from 1 to 10 ng/l. In addition, this is a low cost system.

Few other studies showed similar LOD for endocrine-disruptor compounds; Schneider et al. observed a LOD of 0.2 ng/l for ethynylestradiol in water, but the system is not yet automatised.

However, this paper presents the preliminary tests and a further work is needed to complete the study and to establish that it is a very powerful tool in aquatic analysis. As mentioned by the authors, cross-reactivity tests and multi-analyte measurements with other endocrine-disrupting compounds have to be conducted with surface waters and effluents of sewage treatment plants. Real samples from various locations have to be tested. The LOD and LOQ were obtained in compliance with the IUPAC rules but it is necessary to further verify them using real samples spiked to the estimated LOD.

SOURCE:

J. Tschmelak, G. Proll, G. Gauglitz 2004, *Sub-nanogram per litre detection of the emerging contaminant progesterone with a fully automated immunosensor based on evanescent field techniques*, Analytica Chimica Acta 519, 143-146.

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FURTHER READING:

Schneider C., Schöler H.F. and Schneider R.J. 2005, *Direct sub-ppt detection of the endocrine disruptor ethynylestradiol in water with a chemiluminescence enzyme-linked immunosorbent assay*, Analytica Chimica Acta 551, 92-97.

Use of Identification Point (IP) approach for confirmation of emerging contaminants in environmental samples

Increasing concern about confirmation of positive data favoured the development of different criteria to assure data quality and to avoid the reporting of false positives. One of the relatively new concepts in environmental analysis is confirmation of contaminants based on the use of identification points (IP) proposed by the European Commission Guidelines (EU Commission Decision 2002/657/EC) for identification and quantification of organic residues and contaminants. Originally defined for the determination of organic contaminants in food samples, it has been expanded to other matrices, including environmental samples. The Decision describes a set of minimum performance characteristics which have to be fulfilled by method to be used for two groups of contaminants: Group A – (banned compounds) stilbenes, stilbene derivatives, and their salts and esters; antithyroid agents; steroids; resorcylic acid lactones including zeranol, and beta-agonists;

and Group B – (compounds with established residue level) antibacterial substances, including sulphonamides, quinolones; other veterinary drugs; anthelmintics; anticoccidials, including nitroimidazoles; carbamates and pyrethroids; sedatives; non-steroidal anti-inflammatory drugs (NSAIDs); other pharmacologically active substances; other substances and environmental contaminants, such as organochlorine compounds including PCBs, organophosphorus compounds, chemical elements, mycotoxins and dyes. The Decision proposes a system of IPs, where at least three IPs are required (four in the case of banned compounds) to confirm a positive finding. In addition, the deviation of the relative intensity of the recorded ions must not exceed a certain percentage of the reference standard, and the retention time must not deviate more than 2.5%. This means that it is necessary to acquire three ions in single-mass spectrometry instruments (3 IPs), or the selection

of one precursor ion and the recording of two product ions at low resolution in MS2 instruments, resulting in 4 IPs.

F. Hernandez et al. adopted the IP approach for confirmation of pesticides in environmental waters and described it in several papers. The paper published in Trends in Analytical Chemistry discusses different strategies that could be used in order to reach the number of IPs required to confirm positive findings, depending basically on the instrument available. Two approaches were considered:

- (i) acquisition of two (or more transitions) in QqQ instruments; and,
- (ii) acquisition of the total product-ion spectra with Q-TOF instruments.

The usefulness of the methods developed was tested by analyzing around 100 groundwater and surface-water samples. The results of the study pointed out that although the confirmation criterion based on collection of sufficient number of IPs is useful, the quality of the transitions in tandem MS-based methods has to be carefully considered, as non-selective transitions (e.g., loss of water, carbon dioxide, and

hydrochloric acid) are more prone to be hampered and might lead to the reporting of false positives or negatives in the samples.

Another paper, published in Analytical Chemistry, by the same group, compared the capabilities of triple quadrupole mass spectrometry (QqQ), time-of-flight mass spectrometry (TOF), and hybrid quadrupole time-of-flight mass spectrometry (QTOF) to achieve the required IPs. The number of IPs collected, the sensitivity, and the practical advantages and disadvantages of these techniques were discussed. The authors conclude that the QqQ instrument allows the confirmation of detected pesticides even at very low concentrations (ng/l) achieving between four and five IPs when adding confirmatory transitions. The direct confirmation with a TOF instrument is only feasible for those compounds showing sufficient sensitivity, isotopic pattern, or easy in-source fragmentation. In other cases, the required IPs could be reached by adding IPs earned with this technique to those obtained from the MS/MS screening method. Finally, the use of a QTOF instrument allows up to 20 IPs in a single run at relatively high concentrations (submicrograms per litre) to be obtained, as no "ion shopping" was required.

SOURCES:

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Strategies for quantification and confirmation of multi-class polar pesticides and transformation products in water by LC-MS2 using triple quadrupole and hybrid quadrupole time-of-flight analysers

This article describes different approaches for chemical analysis of polar pesticides and their transformation products (TPs) in water. The instrumental technique chosen for qualitative and quantitative determination is mass spectrometry (MS) combined with high-performance liquid chromatography (HPLC or LC). The authors discuss the difficulties associated with the determination of such a large group of substances with varying chemical properties (cationic, anionic and neutral). One major issue is to find mobile phase compositions that

yield good chromatographic behaviour, while at the same time do not hamper detection. The optimal solution, according to the authors, is to use two methods. In one, formic acid is added to the mobile phase, giving satisfactory chromatography of anionic and neutral analytes. In the other method, used for cationic and neutral analytes, heptafluorobutyric acid (HFBA) is added to the sample vial and into the cartridge-conditioning solvent (the cartridge being an in-line SPE-cartridge), which is being pumped by a different pump than that employed for analyte

elution. By doing this, the ion-pairing agent HFBA is taken advantage of for achieving enrichment and retention without interfering with the detection. Detection was performed with a triple quadrupole (QqQ) MS.

The authors differentiate between screening, quantitative, and confirmatory methods, respectively. Screening methods are further divided into pre-target, post-target, and non-target screening. Both of the above described methods were characterised as pre-target screening or quantitative methods, but also as confirmatory, since two ion transitions were monitored for each analyte in selected reaction monitoring mode (SRM).

Two different MS analysers, one QqQ and one Quadrupole Time-of Flight (QToF) were evaluated for pesticide residue analysis (PRA). The strengths/weaknesses of the two systems were compared in terms of

sensitivity, mass resolution, and specificity. It was concluded that the QqQ was the best for quantitation of pre-selected analytes present at concentrations below the compliant level of 0.1 µg/L, whereas the QToF was superior for screening of unknowns and for confirmation of positive findings.

Optimised analytical conditions for 27 compounds, e.g. polarity, precursor ion and product ion masses (exact mass for precursor ions), as well as cone voltage and collision energy, are presented. The article lacks information of instrumental details e.g. make, model and model year. The ongoing instrumental development leading to more sensitive QToFs with larger linear range and QqQ with higher scan speeds require that this information is included in order to enable the reader to relate the results to existing equipment. However, this information may be available among the 50 referenced articles.

SOURCE:

Hernandez, F., O. J. Pozo, et al. (2005). "Strategies for quantification and confirmation of multi-class polar pesticides and transformation products in water by LC-MS2 using triple quadrupole and hybrid quadrupole time-of-flight analyzers." *Trac-Trends in Analytical Chemistry* 24(7): 596-612.

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Research projects/findings

Engineered Nanoparticles – emerging pollutants? Launch of the Environmental Nanosciences Initiative in the UK

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<http://www.nerc.ac.uk/funding/thematics/eni/>

A new programme of research has just been launched in the UK to investigate the fate, behaviour and environmental effects of engineered nanoparticles. The Environmental Nanosciences Initiative (ENI), launched in May by the UK Natural Environmental Research Council, UK Government (Defra) and Environment Agency will act as a portal for environmental nanosciences research, funding exploratory grants and bringing scientists and policy makers together to investigate the environmental risks and benefits of nanotechnologies.

Nanotechnology has been described as the new Industrial Revolution and is projected to have a global market value of \$U.S.1 trillion within the next 5 – 10 years. It involves the production and application of substances and structures at the nanoscale, typically with several dimensions below 100nm (0.1microns). Substances manufactured in this very small size range can have very different properties when compared to their larger counterparts, reflecting surface properties and quantum effects that become important at the nanoscale. Such enhanced properties of nanomaterials offer huge potential socio-economic, health and environmental benefits, from drug delivery to environmental remediation, which has in turn fuelled a rapid increase in nanomaterials manufacture.

Engineered nanoparticles are one important sector of nanotechnologies, representing a diverse range of substances. These include metal and metal oxide based nanoparticles and carbon – based nanotubes and fullerenes, many of which are already on the market in many products, from cosmetics to fuel additives (if you want to know more

visit www.nanotechproject.org/inventories).

While engineered nanoparticles offer significant potential benefits, there are also considerable uncertainties with regards to potential risks to environment and human health. If nanoparticles have enhanced properties, are they also more toxic or more persistent? Are they more mobile within organisms, do they have novel effects? Do they affect the behaviour and toxicity of other substances they interact with in the environment? Are risk assessments for substances in their bulk form sufficient for the same materials as nanoparticles? Will engineered nanoparticles become a new class of emerging pollutants?

Answering these questions will require basic research into fate and behaviour, toxicity and environmental effects, underpinned by methods and instrumentation for detection. It will also require scientists from different disciplines to work together, transferring knowledge between for example human toxicology and ecotoxicology. The Environmental Nanosciences Initiative will begin to address these questions. A two day conference (Environmental Effects of Nanoparticles and Nanomaterials) will be held in London, September 18-19th (see www.emerging-pollutants.net/workshops)

If you want to learn more about the Environmental Nanosciences Initiative contact Richard Owen (richard.owen@environment-agency.gov.uk). The ENI website (www.nerc.ac.uk/funding/thematics/eni/) will be launched in late June 2006.

MODELKEY: Towards better impact and cause analysis

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Water is an inherited good that has to be protected and used in a sustainable way. Based on that understanding, the EU Water Framework Directive demands a good ecological status of European waters by 2015. This is a reasonable but challenging task for European water managers. In order to address their needs to identify, rank and mitigate the forces which contribute to low ecological quality, a new Integrated Project called MODELKEY (Models for Assessing and Forecasting the Impact of Environmental Key Pollutants on Marine and Freshwater Ecosystems and Biodiversity (<http://www.modelkey.org/>)) was funded by the EU and started its work in 2005. 26 prestigious European institutes from 14 different countries under the co-ordination of

Because priority pollutants are often poor predictors of toxic risks in complexly contaminated environments, the MODELKEY approach focuses on the identification of effect-based key toxicants in sediments, water and biota. Basin scale exposure models are designed to predict mobilisation, fate and transport of these key toxicants to downstream ecosystems.

There are enormous amounts of monitoring data on contamination, ecology, effects and hydrology that are frequently collected by water agencies and scientific projects and stored locally. MODELKEY is compiling them in one central database and making them available for European scale effect and risk modelling. A kit of modelling tools will



SPMD for passive sampling of lipophilic organic compounds after three weeks of exposure in the River Elbe close to Pardubice (Czech Republic)



Kick-off meeting in Palma



Sediment sampling at the River Elbe in P_{elou} (Czech Republic)

the UFZ Centre for Environmental Research in Leipzig (Germany) have come together for 5 years to develop and integrate advanced analytical and bioanalytical tools as well as interlinked and verified diagnostic and predictive models to unravel causal relationships between contamination and ecological quality and to assess risks to aquatic ecosystems. This goal is being addressed by looking at the three example river basins of the Llobregat (Spain), the Scheldt (France, Belgium and The Netherlands), and the Elbe (Czech Republic and Germany) and is to be achieved by two closely interlinked approaches:

- a deterministic approach that focuses on an understanding and modelling of functions, processes and mechanisms on a site scale
- a stochastic approach, exploiting existing monitoring data for the identification of relationships by statistical means on a river basin scale.

Major elements of the deterministic approach include advanced effect analysis, integrating early-warning tools on the basis of in vitro assays and biomarkers, toxicity testing on all trophic levels and community effect assessment in laboratory and field. Strict exposure assessment and control together with state-of-the-art effect and accumulation modelling in food webs will help to bridge the missing links between measurable contamination and observable ecosystem deterioration.

developed that allows the simulation of a broad range of hydrological, chemical, toxicological and biological conditions with a single water quality evaluation system. Geographical Information Systems (GIS) and modern statistical approaches, including Artificial Neural Networks and Monte Carlo simulation techniques, are used to calculate exposure distribution in defined habitat patches and to predict effects on communities. Site-specific risk maps and a decision support system will help MODELKEY end-users to assess and prioritise risks and contamination sources. An end user communication board (<http://www.modelkey.org>) provides the platform for a close interaction between MODELKEY scientists and water managers from the beginning of the project allowing stakeholders to address their needs to the project, to benefit directly from project results and to critically evaluate the project's progress.

For more information, please visit the project website at: <http://www.modelkey.org> or download the project newsletters from <http://www.modelkey.org/index.php?en=5636>. If you would like to stay informed about MODELKEY and receive its newsletters in future, please just send an e-mail to Michaela Hein at michaela.hein@ufz.de.

NoMiracle: The integrated project "Novel methods for integrated risk assessment of cumulative stressors in Europe"

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To support current and future European strategies, in particular for environment and health, the NoMiracle integrated project will support the development and improvement of a coherent series of methodologies that will be underpinned by mechanistic understanding, while inte-

grating the risk analysis approaches of environmental and human health. The project has a special focus on mixtures of chemicals with a specific mode of action, such as pesticides, biocides and pharmaceuticals. The cumulative stress caused by natural stressors and their combined action

with chemicals is a main issue of NoMiracle. By developing and using improved assessment tools and novel models, the project will quantify, and aim to reduce, uncertainty in current risk assessment and screening methodologies, e.g. by improving the scientific basis for setting safety factors. The new methods will take into account geographical, ecological, social and cultural differences across Europe. The NoMiracle Consortium consists of 38 partners from 17 European countries.

The aims of NoMiracle are condensed into seven major science & technology objectives:

1. to develop new methods for assessing the cumulative risks from combined exposures to several stressors including mixtures of chemical and physical/biological agents;
2. to achieve more effective integration of the risk analysis of environmental and human health effects;
3. to improve our understanding of complex exposure situations and develop adequate tools for exposure assessment;
4. to develop a research framework for the description and interpretation of cumulative exposure and effect;
5. to quantify, characterise and reduce uncertainty in current risk assessment methodologies, e.g. by improvement of the scientific basis for setting safety factors;
6. to develop assessment methods which take into account geographical, ecological, social and cultural differences in risk concepts and risk perceptions across Europe;
7. to improve the provisions for the application of the precautionary principle and to promote its operational integration with evidence-based assessment methodologies.

The endpoint of NoMiracle is to provide tools to quantify the exposure and effects of multiple stressors to which humans and ecosystems are exposed under real-life conditions. Tools will be developed to integrate geo-referenced pollution and exposure data with climatic GIS maps. The main tool from NoMiracle will make it possible to identify deviations of chemical mixtures from conventional concepts such as Concentration Addition (CA) and Independent Action (IA) in order to identify synergistic effects and high risk situations. The model can be used to predict the likelihood and magnitude of deviations for risk assessment purposes (e.g. derivation of safety factors in risk assessment for mixtures). Furthermore, NoMiracle will explore potential mechanistic explanations for the deviations of the CA and IA models.

The toolbox also contains:

1. techniques to identify and prioritise realistic cumulative exposure scenarios;
2. novel algorithms to predict exposure levels under real-life conditions based on the analysis of matrix-compound interactions;
3. novel algorithms to predict the effects of exposure to multiple stressors based on the analysis of interactions between multiple stressors (chemical, biological and physical);
4. scientific integration of human and ecological effect assessment by incorporation of mechanistic information on biochemical and molecular processes;
5. a paradigm change from substance-oriented to receptor-oriented risk assessment, in which the receptor (e.g. an individual organism) is considered as an integrator of stressors over space and time;
6. a comprehensive evaluation of the uncertainties involved in the novel assessment methods and an indication of the implications for decision making, especially in relation to the precautionary principle.

During the first 18 months the project has promoted strong interactions among interdisciplinary research lines. The data background for scenario selection, exposure assessments, uncertainty assessments and risk mapping has been established, and problems with data gaps and uncertainty related to data values and estimation have been dealt with. Procedures are under development which can be included in a systemic way based on a paradigm for criteria setting. In the area of computer science and database management, the concept of ontology-based knowledge is investigated for adoption into a hierarchical criteria development principle, as published in Sørensen, PB, Lerche, D, Brüggemann, R (2005) Resources and uncertainties in evaluation of chemicals, *Water Sci. Techn.* 52 (6) 235-242). The focus is on a methodological implementation of precaution into the screening phase. A new paradigm is developed for multi-criteria ranking to support risk assessment.

Preliminary results from a state-of-the-art overview of cumulative risk mapping shows that a lot has been done on mapping of pollution, pollution sources and environmental risks of single pollutants, but that mapping cumulative or integrated risks seems to be a new area with very little done to date. An ongoing study on aggregation of spatial data demonstrates the potential impact of different aggregation methods on risk maps.

PERFORCE:

Perfluorinated organic compounds in the European Environment

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<http://www.science.uva.nl/perforce/index.htm>

Perfluorinated compounds are chemicals that have attracted increasing scientific and socio-economic interest, following their detection in organisms from remote regions – including the polar regions – and in human blood.

Perfluorinated alkylated substances (PFAS) have numerous useful applications in consumer and other products. They can be found in textile, carpet and leather treatments (for water and dirt proofing), surfactants, polymerisation aids, fire-fighting foams and paper additives. These chemicals are highly stable, do not degrade in the environment and can accumulate in living organisms. To make a full risk assessment of the environmental impact of PFAS, accurate data on their physico-chemical properties and new tools to assess the movement and distribution of PFAS are needed.

PERFORCE is a project aimed at establishing Europe as an international scientific leader in environmental research and exposure assessment of perfluorinated organic compounds. The lack of pertinent data and information on deviating properties of these newly emerging persistent

hazardous substances presents urgent needs for elucidation on both the socio-economical and the scientific level. In addition, PFAS present interesting and challenging possibilities for the acquisition of new knowledge on environmental chemical mechanisms, of e.g., surfactants and ionic compounds. In particular the sources and routes of PFAS detected in remote areas will be elucidated. The major objective of the project is to introduce and evaluate new chemical and biological techniques and tools to assess the occurrence and distribution of PFAS in European ecosystems. This exposure assessment will, together with ongoing hazard assessment and toxicity testing elsewhere enable a proper environmental risk assessment of PFAS to be made in the near future.

The PERFORCE project is financially supported by the EU-New and Emerging Science and Technology (NEST) programme. The project brings together expert teams from academia, government and industry to significantly boost our understanding of PFAS. The project, led by the University of Amsterdam, brings together researchers from the Netherlands, Norway, Sweden and Belgium (including DuPont, a leading PFAS manufacturer) and aims to establish Europe as an international



Figure 1: Overview of sampling location and sample types collected in the PERFORCE monitoring programme.

scientific leader in environmental research and exposure assessment for PFAS. Work is split between developing new chemical analytical

methods, bioanalytical tools, physicochemical property and fate modelling, environmental modelling and validation of techniques. An important component of the project will be the transfer of know-how from industry to the academic teams. The final exposure assessment will be based on both field data obtained from a European monitoring campaign (see Fig. 1) and on modelling results.

The project will also link with other international initiatives, especially in North America and OECD projects, to provide calibration and comparison/standardisation of techniques to give a global dimension to this global problem. PERFORCE has co-organized the first international interlaboratory study on PFAS. Results showed that, while blood analysis showed acceptable variabilities, large in-between laboratory variabilities exist for most environmental matrices (e.g. water, fish tissue, sediment).

Understanding the behaviour and distribution of PFAS in the environment will enable Europe to properly assess their societal risk and, if necessary, to develop an ecologically sound chemical replacement policy which can provide competitive advantages for European chemical industries.

Life of the network, NORMAN Activities

The activities of the NORMAN project started officially on 1st September 2005, with a kick-off meeting in Paris on 7-8 September 2005. The project is in its starting phase: we are laying the foundations on which to build the services that will be provided by the network. The ultimate aim is to meet users' needs in the exchange and production of good-quality and comparable data in a field where data are typically scarce and insufficient for sound decision-making. Below is a summary of the activities carried out so far and forthcoming results. More information on each of these activities is provided on the project website (www.norman-network.net).

IMPLEMENTATION OF A NETWORK OF CONTACT POINTS

In order to help with the gathering of data and information about on-going initiatives on emerging substances, NORMAN is establishing a network of Contact Points (CP) in Europe.

The NORMAN Contact Points are experts from leading European institutes, organisations and research centres in the Member States, who are expected to carry out the following tasks within their own country:

- support the NORMAN network in creating links to the key research centres, reference laboratories, institutions, etc. and in providing first-contact information about the on-going research activities, projects, monitoring programmes and other initiatives relating to emerging substances
- help to disseminate information about NORMAN reports, workshops and activities to relevant institutions / organisations.

The current list of CPs already appointed is available on the project website.

DEFINITION OF A SET OF CRITERIA TO IDENTIFY AND SELECT EU REFERENCE LABORATORIES, RESEARCH CENTRES AND RELATED ORGANISATIONS DEALING WITH EMERGING POLLUTANTS

In light of the implementation of a permanent European network to deal with all analytical issues relating to emerging environmental substances, it is crucial to define a set of criteria for the identification and selection of European reference laboratories, research centres dealing with emerging pollutants and stakeholders identified as potential users of NORMAN outcomes.

For this it was deemed necessary to start by reaching a consensus within the NORMAN consortium about the terms: reference laboratory, expert

laboratory, research institute, etc.

For the term "National reference laboratory", an overview search was made of all possible available sources, including in sectors other than the environmental.

The definition proposed in the current glossary of terms (soon available on the project website) consists of a list of the technical competencies as well as the tasks that a national reference laboratory, or a research centre dealing with emerging pollutants etc. should perform in the light of the ultimate objectives of the NORMAN network.

In order to allow a common understanding and approach, the current glossary includes some key terms of common use within the NORMAN project. This glossary should be seen as a living document and suggestions, comments and improvements can be sent by email to a.stru-geon@brgm.fr

EXCHANGE OF INFORMATION – WORKSHOP ON "EMERGING ENVIRONMENTAL POLLUTANTS: KEY ISSUES AND CHALLENGES" (STRESA, ITALY - 19-20 JUNE 2006)

The first important opportunity for an exchange of information and discussion about emerging environmental substances will be the workshop "Emerging environmental pollutants: key issues and challenges" which took place in Stresa, Italy on 19-20 June 2006 (followed by the plenary meeting – 21 June at JRC-IES, Ispra).

The workshop programme brought together experts and stakeholders from different scientific communities and environmental compartments (air, water and soil) to discuss the latest research findings and evaluate the main gaps and needs for risk assessment and management of emerging pollutants (further details on the project website).

A written report about the outcomes of the workshop will be available on the project website in September.

The event represented also an important input for NORMAN network for the future strategy of the network and development of its services.

NORMAN DATABASES

One of the services that will be provided by the NORMAN network will consist of a regular and up to date overview of available information / data about experts, organisations and projects dealing with emerging substances, geo-referenced monitoring and bio-monitoring data on emerging substances, mass spectra of unknown and provisionally identified substances.

This service will be offered through three different databases: EMPOMAP, EMPODAT and EMPOMASS, which are still under construction.

EMPOMAP is expected to be published on the internet early this summer (June / July 2006). At that time the database will be open for registration of experts, organisations and projects from all over Europe dealing with emerging substances.

For the design of EMPODAT a special effort is being made to make this database compatible (in terms of metadata) with the existing databases and, in particular, with the WISE information system that is under development as part of the work of EEA and DG ENV. The publication of the EMPODAT database on the web is planned for the end of 2006. Contributions from all partners and Contact Points will be fundamental from the start, in order to collect the data that are at present available but highly dispersed in different databases, written reports, academic research studies and publications.

QA/QC ISSUES ACTIVITIES

Methods validation protocols

The exchange and production of reliable and comparable data constitute one of the most critical and fundamental objectives of NORMAN; they are, after all, essential prerequisites of any successful assessment and management of environmental risks. In the case of emerging pollutants, poor data comparability at European level is a common problem. The measurement methods have often been developed in-house for a specific task and not necessarily validated at international level. The methods are often not well-established in the scientific community and are therefore far from being harmonised or standardised. Furthermore, methods developed by different institutes may target different environmental sub-compartments, adding further obstacles to data comparability.

To address this problem, NORMAN is developing a framework of protocols especially designed for the validation of methods (both, biological and chemical) for measuring emerging pollutants in the different environmental matrices. These protocols will guide the user through the following steps:

- evaluation and classification of existing methods with respect to their "level of maturity" in terms of validation
- method selection (in cases where more than one candidate method exists for a specific purpose)
- optimisation of a method in terms of extending its applicability
- validation steps to be undertaken in order to demonstrate effectively the validation status of a selected method.

The first draft of these protocols will be published on the NORMAN website by September 2006 and will be tested in the three case studies organised within the project. Each case study will address a different class of emerging substances of particular environmental interest today and a different level of method maturity.

Case studies

Three different inter-laboratory studies will be undertaken during the course of the project:

- 1) Validation and harmonisation of an analytical methodology for research laboratories: Oestrogens in sewage treatment effluents (Case Study 1 – Leader: UK Environment Agency, UK);
- 2) Validation and harmonisation of an analytical methodology for reference laboratories: Non-steroidal anti-inflammatory drugs (NSAIDs) (Case Study 2 – Leader CSIC, Spain);
- 3) Validation and harmonisation of an analytical methodology for routine laboratories: brominated flame retardants (DecaBDE) (Case Study 3 – Leader UBA, Germany).

Based on a review of existing literature and using the criteria defined in the validation protocols the most promising methods will be selected for the inter-laboratory studies.

The results of these will be used to test and improve the validation protocols and to test the organisation of validation activities as part of the services of a permanent network.

Case 1- Oestrogens in sewage treatment effluents

The endpoint for this case study is validation at the intra-laboratory level. Three levels of method are being reviewed: in vitro assays for total oestrogenic activity, in vivo biomarker assays for oestrogenic exposure and in vitro assays for direct measurement of oestrogens. A list is being produced of laboratories to be invited to undertake the intra-laboratory validation. It is envisaged that three laboratories will be selected for each of the method levels identified. A first meeting with the laboratories that will confirm their participation in the trial is planned for early October in London.

Effluent samples will be sent out to laboratories in early November 2006. Two well-characterised domestic sewage treatment final effluents will be used, one with a high oestrogenic loading, one with a low oestrogenic loading.

The deadline for laboratories submitting their data reporting templates will be the end of February 2007.

A meeting with the laboratories to review the draft report will be held in June 2007, with a final report on the study (peer reviewed publication) which will be published in July 2007.

Case 2 - Non-steroidal anti-inflammatory drugs

A review of existing analytical methods (LC and GC methods published in the last 5 years) for ibuprofen, naproxen, ketoprofen and diclofenac, including the degree of validation, domain of application and performances, has been completed. Based on the review, the list of expert laboratories that developed the methods, as well as laboratories using the methods for analysis of NSAIDs in research or monitoring programmes, was compiled and used to prepare a final list of laboratories to be invited to participate in the 1st inter-laboratory exercise.

A preparatory meeting was organised in Barcelona on 19th May 2006 to discuss the methods used by the participants and to agree on QA/QC issues.

Two rounds of inter-lab studies are planned. The first round of inter-lab studies will take place between October and December 2006, whereas the second inter-lab study is planned for October – December 2007, with meetings with the laboratories and publication of the results on the project website in May 2007 and March 2008.

