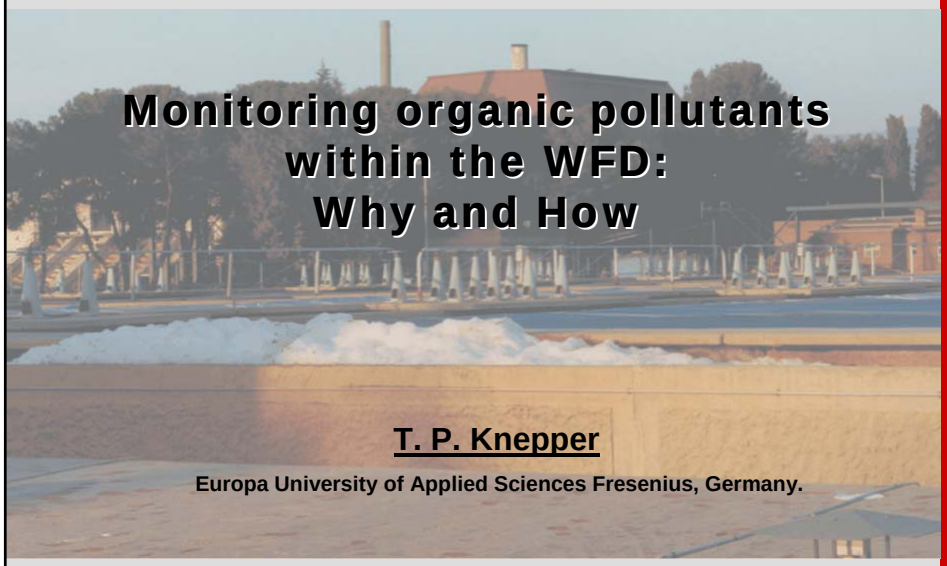

Monitoring organic pollutants within the WFD: Why and How

T. P. Knepper

Europa University of Applied Sciences Fresenius, Germany.

Outline:

- Why do we need to monitor within the WFD ?
  - Strategy of monitoring
  - Results of monitoring
  - (WWTP-influent & -effluent; surface & ground water; soil)
- How to monitor
  - Sampling campaigns
  - Analysis
  - Quality Assurance

Strategy of monitoring (WFD):

Each ***compound***
- and also ***metabolite*** –
which could possibly appear has to be investigated.

....if an analytical method is available and the analysis not too costly

Time shedule WFD:

Dec. 2000	In force
Dec. 2003	National law implementation
Dec. 2004	Characterisation and inventory
Dec. 2006	Programs for monitoring are ready for application
Dec. 2009	Program for measures and plans for management of river basin are finished
Dec. 2012	Program for measure is implemented
Dec. 2015	„good condition“ (ecological + chemical); new plans for management of river basin

Priority Substances and Other Pollutants

The Commission proposal (COM(2006)397 final) setting environmental quality standards for surface waters of 41 dangerous chemical substances includes the 33 priority substances and 8 other pollutants.

1) Priority Substances

33 substances or group of substances are on the list of priority substances including selected existing chemicals, plant protection products, biocides, metals and other groups like Polyaromatic Hydrocarbons (PAH) that are mainly incineration by-products and Polybrominated Biphenylethers (PBDE) that are used as flame retardants..

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Chemical status - 33 priority compounds

Organic compounds (n = 16)

(2) Anthracene
(4) Benzene
(5) Brominated diphenylethers
(7) Chloroalkanes (C_{10}-C_{13})
(10) 1,2-Dichloroethane
(11) Dichloromethane
(12) DEHP
(15) Fluoranthene
(17) Hexachlorobutadiene
(22) Naphthalene
(24) Nonylphenols (4-para-N)
(25) Octylphenols (para-tert-O)
(26) Pentachlorobenzene
(28) PAK (Benzo-a-pyrene, Benzo-b-fluoranthene, Benzo-g,h,i-perylene, Benzo-k-fluoranthene, Indeno-1,2,3-cd-pyrene)
(31) Trichlorobenzenes (1,2,4-TB)
(32) Trichlormethane

Metals (n = 4)

(6) Cadmium
(20) Lead
(21) Mercury
(23) Nickel

Pesticides (n = 13)

(1) Alachlor
(3) Atrazine
(8) Chlorfenvinphos
(9) Chlorpyrifos
(13) Diuron
(14) Endosulfan
(16) Hexachlorobenzene
(18) HCH (Lindan)
(19) Isoproturon
(27) Pentachlorophenol
(29) Simazine
(30) TBT-cation
(33) Trifluralin

Identified as priority dangerous compounds (n = 13)

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European Water Framework Directive (WFD)

Starting 2007 the EU-member states have to conduct monitoring programs upon organic pollutants and others.

Hesse, Germany carried out a prelimary monitoring to find appropriate sampling points.

Pesticides: Monitoring of surface waters 2004/2005 (preliminary monitoring)

- 119 sampling locations
- 95 substances
- 6 measurements 2004 or 2005

  therefrom

  4 in April - June

  2 in October -November

  → **Increased mean values during application time**
- Presetting of WFD

  For I → *12 samples/a*

  For II, III and IV → *4 samples/a*

European Water Framework Directive (WFD) – Regulation for Pesticides in surface waters

I priority pesticides
quality specifications by EU (in preparation)
e.g. IPU, Diuron, Atrazine

II river basin targeted pesticides
quality specifications by Hesse/Working Group on water issues (LAWA)

III further basin targeted pesticides
so far no regulation

IV river basin targeted pesticides
quality standard suggestion by LAWA, e.g. Terbutryn

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Pesticide monitoring in 2004/2005

pesticide	quality standard [µg/L]	90-perc. [µg/L]	maximum value [µg/L]
Isoproturon	0.3 / 1.0 *	0.47	15
Mecoprop (MCPP)	0.1	0.12	11
Dichlorprop (2,4-DP)	0.1	0.11	10
n-Chloridazon	0.1	0.1	9.7
Bentazone	0.1	0.14	9
MCPA	0.1	0.16	7.7
Metazachlor	0.4	< l.d.	4.6
Diuron	0.2 / 1.8 *	0.21	4.5
Metobromuron		< l.d.	4.4
Metamitron		0.2	4.3
Ethofumesate		0.12	3.9
Terbuthylazine	0.5	0.04	2.5
Metolachlor	0.2	< l.d.	1.6
Atrazine	0.6 / 2.9 *	< l.d.	1.4
Terbutryn	0.03	0.09	1.3
Epoxiconazole		0.04	1
2,4-D	0.1	< l.d.	0.91
Propiconazole		0.07	0.8
Metribuzin		< l.d.	0.75
Fluoxypyr		0.04	0.55
Tebuconazole		0.05	0.51
Fenpropimorph		< l.d.	0.49
Terbutylazine-desethyl		< l.d.	0.47
Dichlobenil		< l.d.	0.46
Haloxyfop		< l.d.	0.46

approx. 700 samples

74 pesticides found, therefrom

25 with max. values

l.d. = limit of detection

•annual average value / maximum value

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sampling point	pesticide	detection limit [µg/L]	quality standard [µg/L]	mean [µg/L]	maximum value [µg/L]
Gersprenz Babenhs.-Harreshs.	Dichlorprop (2,4-DP)	0.03	0.1	0.23	0,70
	MCPA	0.04	0.1	0.16	0.62
	Bentazon	0.03	0.1	0.19	0.63
	Dimethoat	0.05	0.1	0.04	0.14
	n-Chloridazon	0.05	0.1	0.32	1.50
	Metolachlor	0.03	0.2	0.25	1.40
	Terbuthylazine	0.03	0.5	0.46	2.50
	Diuron	0.04	0.2 / 1.8	0.12	0.20
	Isoproturon	0.03	0.3 / 1.0	0.24	0.56
	Terbutryn	0.03	0.03	0.07	0.13
	Dicamba	0.05		0.05	0.17
	Ethofumesat	0.03		0.81	3.50
	Haloxyfop	0.03		0.05	0.21
	Iprodion	0.03		0.04	0.14
	Metamitron	0.10		0.80	4.20
	Metobromuron	0.10		0.11	0.40
	Metribuzin	0.03		0.05	0.12
	Tebuconazole	0.04		0.05	0.13
	Terbutylazine-desethyl	0.03		0.10	0.47
Fanggraben Biebesheim	Mecoprop (MCPP)	0.03	0.1	0.08	0.20
	Diuron	0.04	0.2 / 1.8	0.60	1.70
	Terbutryn	0.03	0.03	0.29	0.74

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Exceedings of quality standards of pesticides at sampling locations 2004/2005:

Pesticide with quality standard

compound	No. of QS-exceedings	% positive results
Terbutryn	44	33
Isoproturon	29	53
MCPA	24	31
Bentazone	23	35
Mecoprop (MCPP)	19	38
Dichlorprop (2,4-DP)	14	27
Diuron	12	36
n-Chloridazon	10	15
Metolachlor	3	4
2,4-D	2	5
Metazachlor	2	8
Terbuthylazine	1	15
Atrazine	1	9

Pesticide without quality standard:

compound	No. of sampling locations with mean > 0.1µg/L	% positive results
Metamitron	38	24
Ethofumesate	18	23
Propiconazole	6	30
Metobromuron	4	1
Metribuzin	4	6
Epoxiconazole	2	12
Fluroxypyr	2	11
Tebuconazole	2	24
Fenpropimorph	1	2
Haloxyfop	1	2

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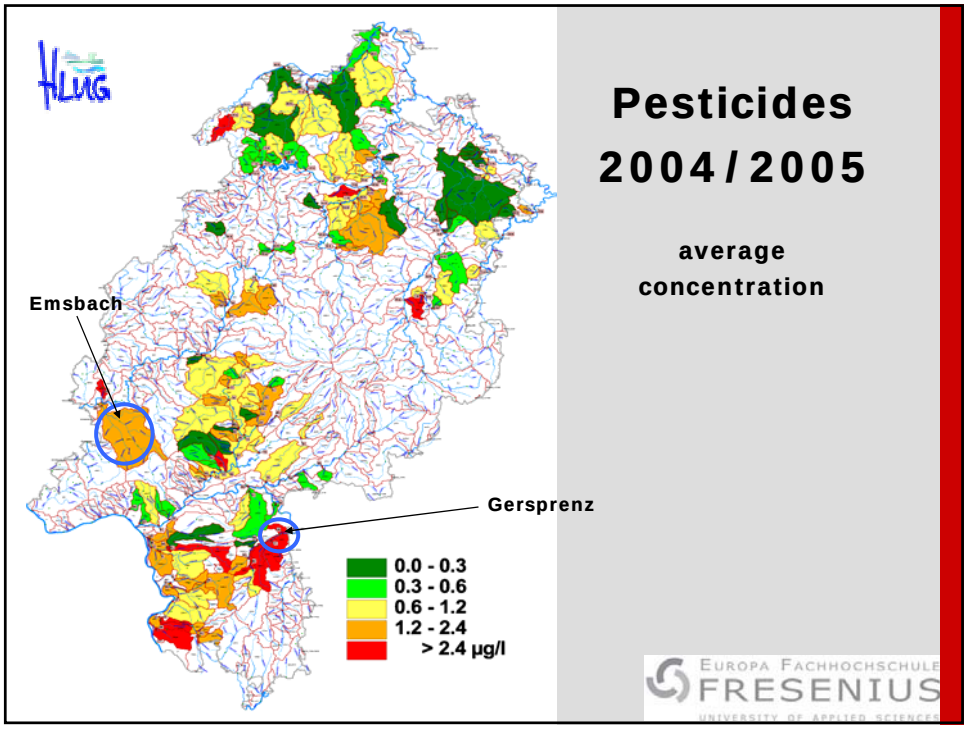
Pesticides
2004/2005
average
concentration
Emsbach
Gersprenz
0.0 - 0.3
0.3 - 0.6
0.6 - 1.2
1.2 - 2.4
> 2.4 µg/l
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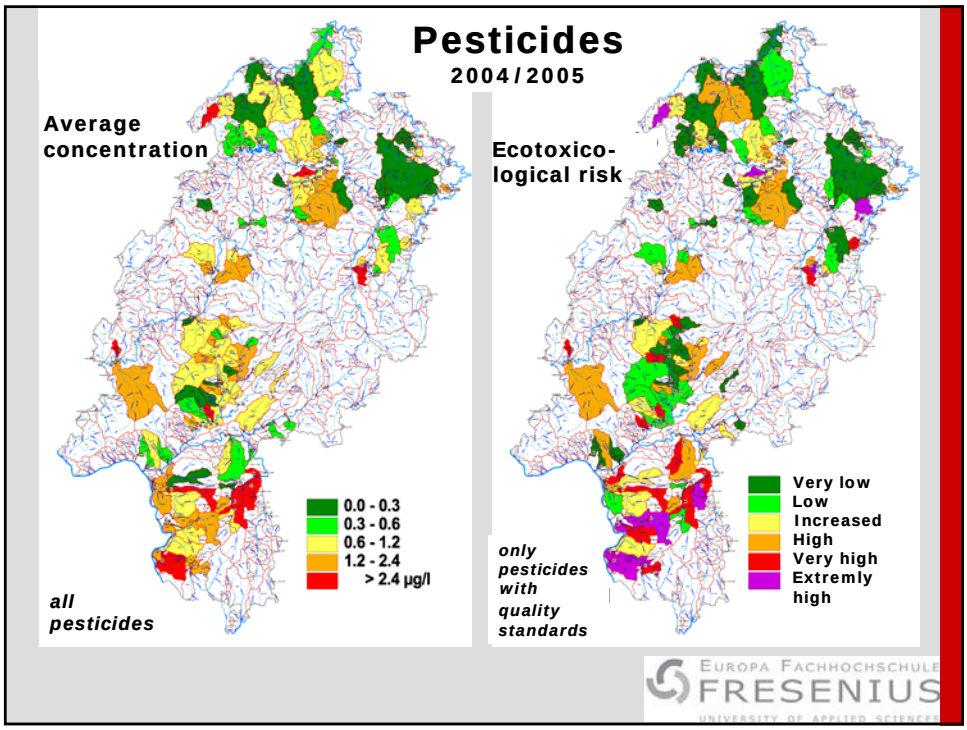
Pesticides
2004/2005
Average
concentration
0.0 - 0.3
0.3 - 0.6
0.6 - 1.2
1.2 - 2.4
> 2.4 µg/l
all
pesticides
Ecotoxico-
logical risk
Very low
Low
Increased
High
Very high
Extremly
high
only
pesticides
with
quality
standards
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	preliminary monitoring WFD 2005-2006		WFD – Monitoring since 2007	
	Number of sampling locations	Number of measurements/ sampling location	Number of sampling locations	Number of measurements/ sampling location
Basic physical parameter / Phosphorous	204	>12	182	>12
Pesticides 2004-2005	**119**	**6**	**96**	**8-17**
Heavy metal, PAH, PCB, organotin compounds in suspended matter	29	8	28	further 4

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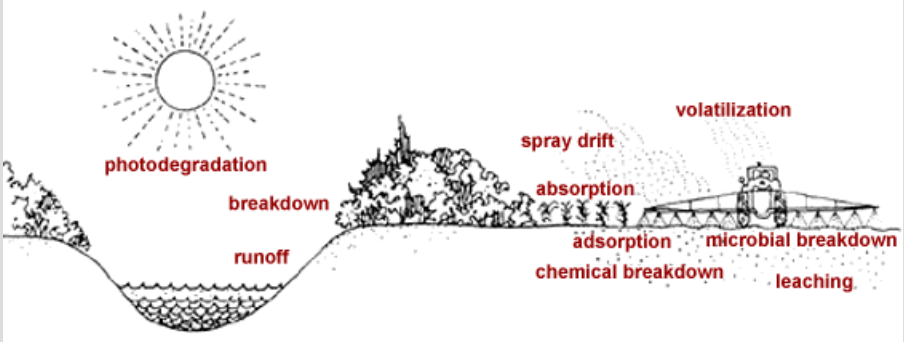
How do pesticides enter surface waters?



http://www.agf.gov.bc.ca/pesticides/images/c/c_2_1.gif

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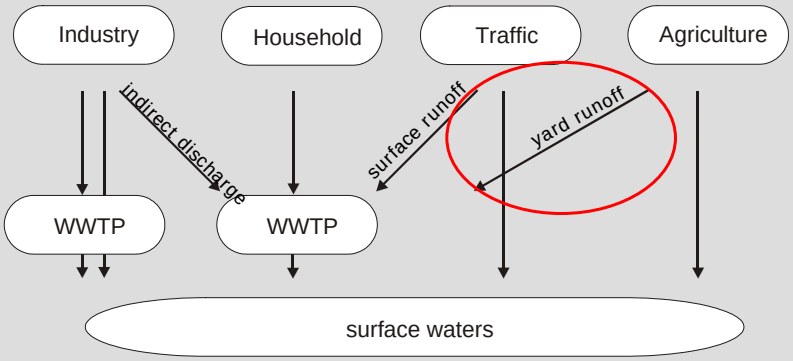
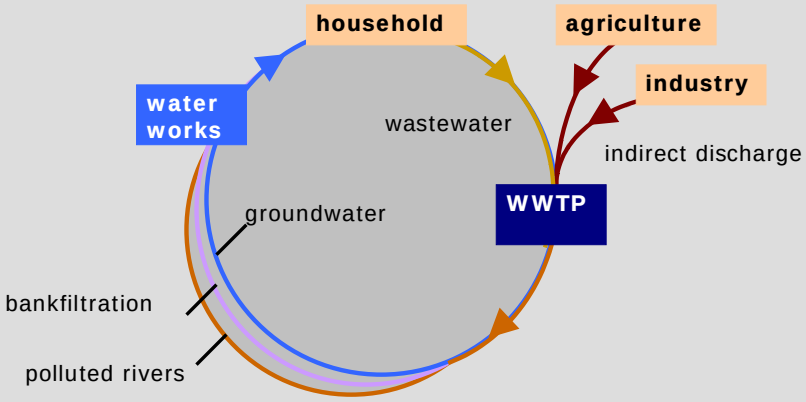
Sources of organic pollutants in municipal WWTP



Pollutants: Entry into the watercycle



Waste water treatment in agglomerations affected by sensitive areas and organic loads (data January 2002)

Member State	Agglomeration concerned	Complying treatment Level	Non complying treatment level
	Load [p.e.]	%	%
Belgium	8 952 516	29	71
Denmark	6 698 384	96	4
Germany	124 876 488	P-Reduction 90% N-Reduction 74%	-
Greece	609 400	40	60
Spain	5 740 260	25	75
France	16 728 379	36	64
Ireland	3 362 856	8	92
Italy	3 024 094	72	22
Luxembourg	804 500	14	86
Netherlands	15 906 991	P-Reduction 79% N-Reduction 66%	-
Austria	1 851 885	100	0
Portugal	1 372 700	11	90
Finland	6 377 300	7	93
Sweden	7 672 670	73	27
United Kingdom	6 221 177	29	71
Total	**210 199 600**	**-**	**-**
MS not applying Article 5	**69 416 121**	**42**	**58**

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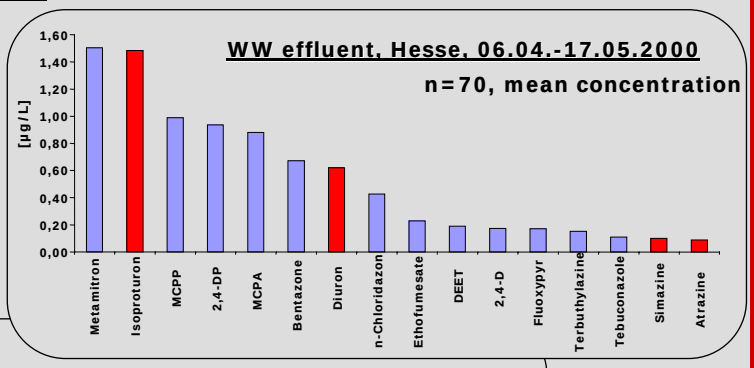
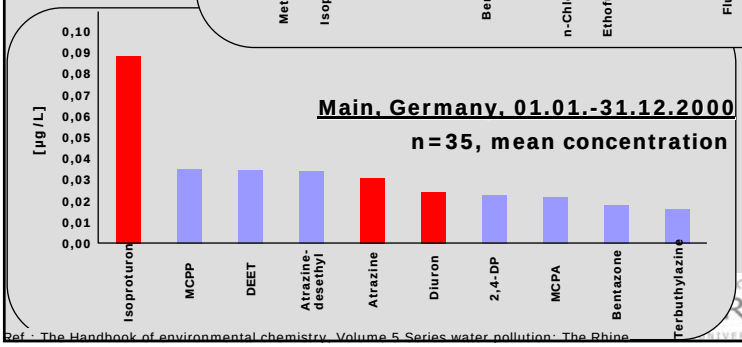
Comparison of pesticide concentrations in waste and surface waters





Ref.: The Handbook of environmental chemistry, Volume 5 Series water pollution: The Rhine

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Balance of the stream Nidda to bordering WWTPs

Compound	Waste water treatment plant pestcide load in kg	Nidda pesticide load in kg	Share of load of pesticides from WWTP to total load in Nidda in %
Atrazine	1,9	3,4	57
MCPP	5,2	7,7	67
2,4-DP	4,4	6,9	63
Isoproturon	8,5	14,0	61
Diuron	6,6	10,4	64

Data gained 23/04/1994 to 24/05/1994

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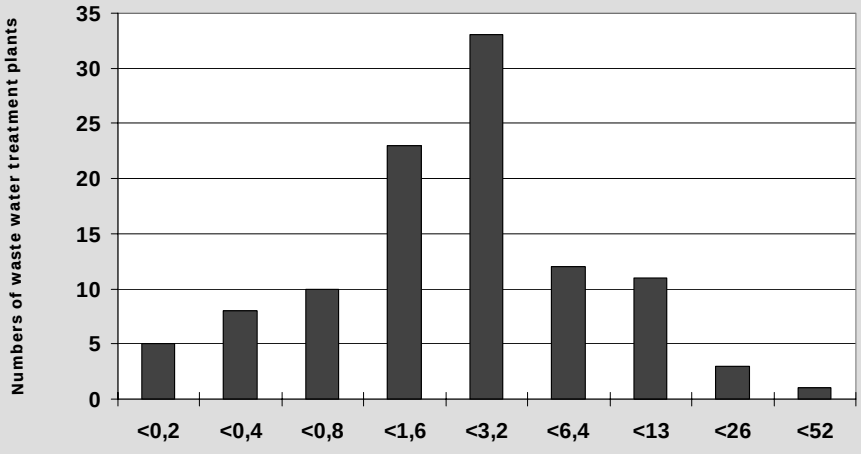
Average pesticide entry from 106 Hessian waste water treatment plants (April/May 1999)



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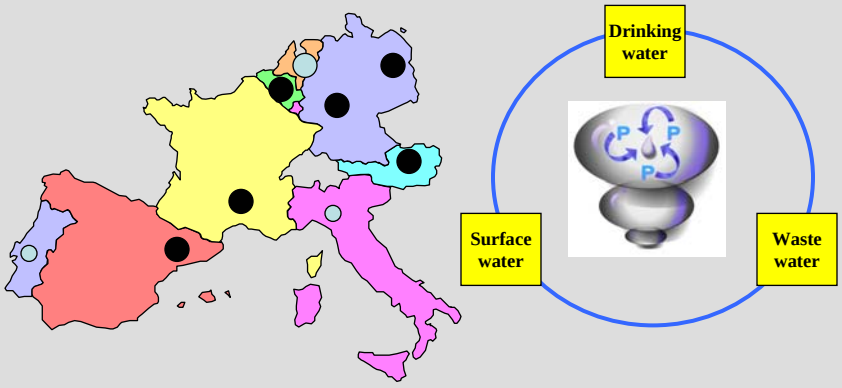
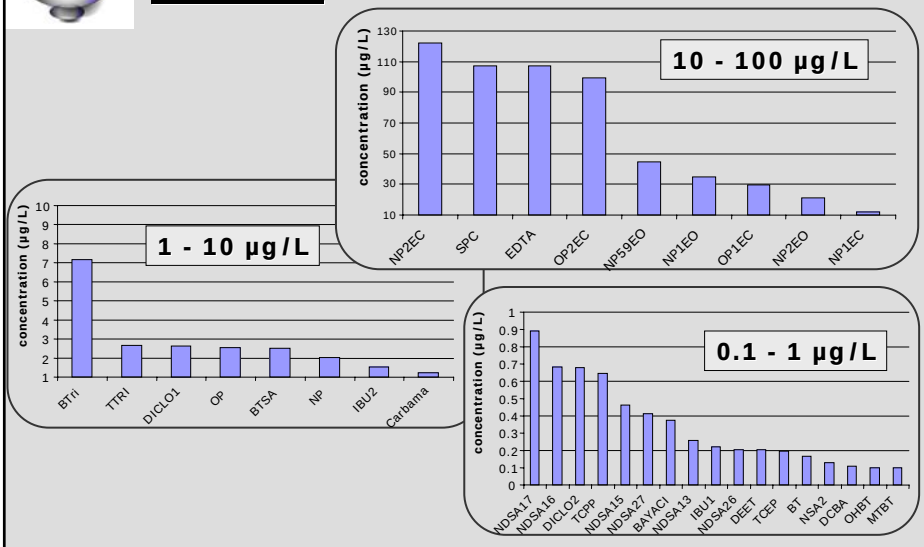
How to monitor ?

Sampling campaign!

Monitoring



P^3-compounds in European wastewater effluents:



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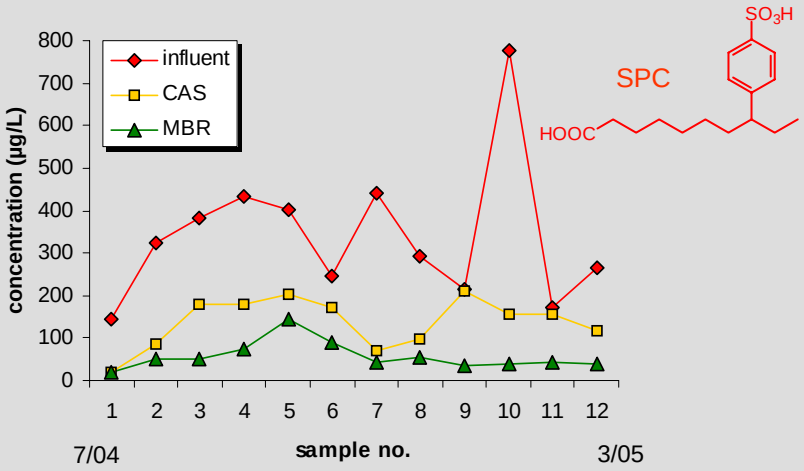
Sulfophenyl carboxylates



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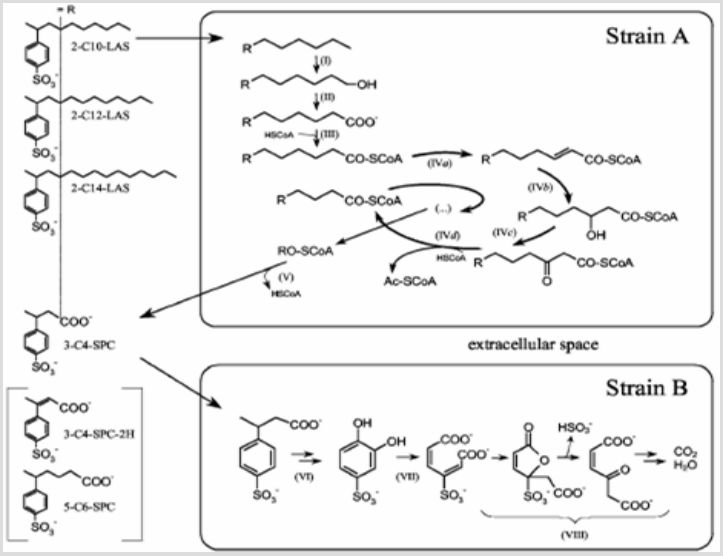
Aerobic degradation of LAS by bacteria



Schleheck, D., Knepper, T.P., Fischer, K., Cook, A.M.; *Appl. Environ. Microbiol.*, 2004.

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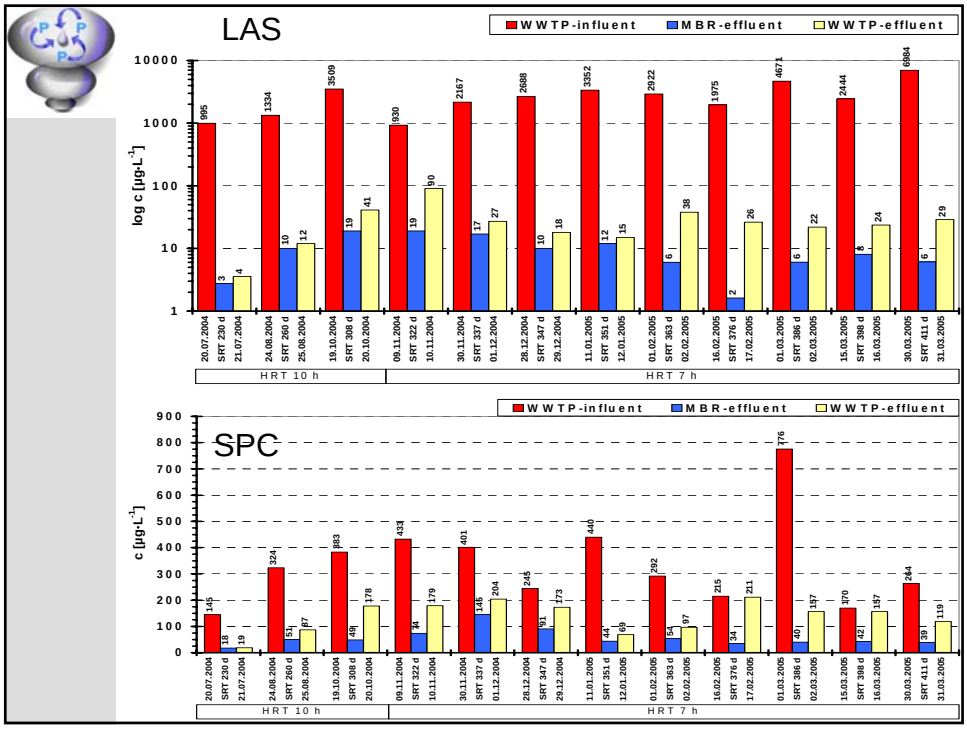
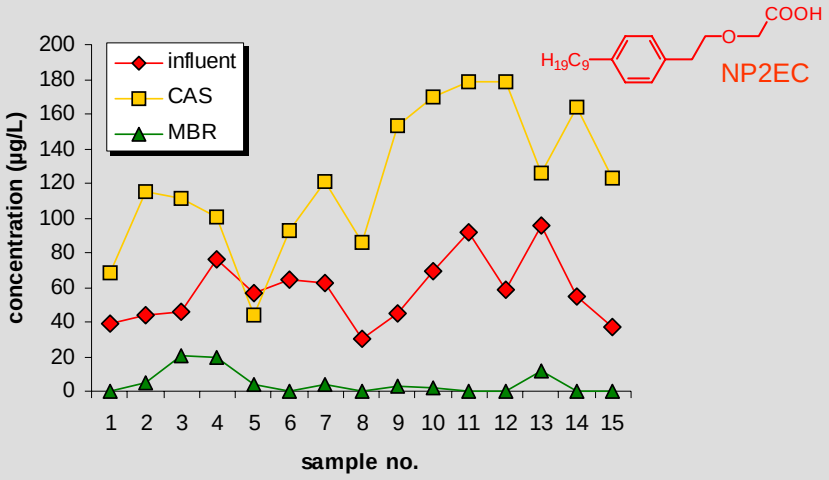


Nonylphenol-diethoxycarboxylate



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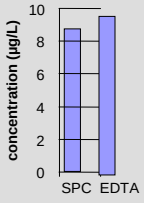
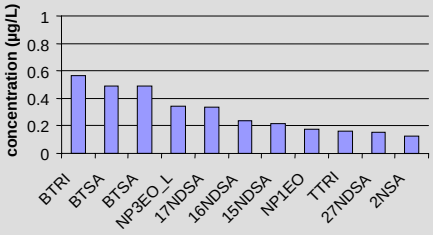
P³-compounds in European surface waters

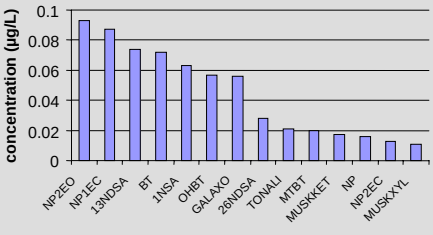


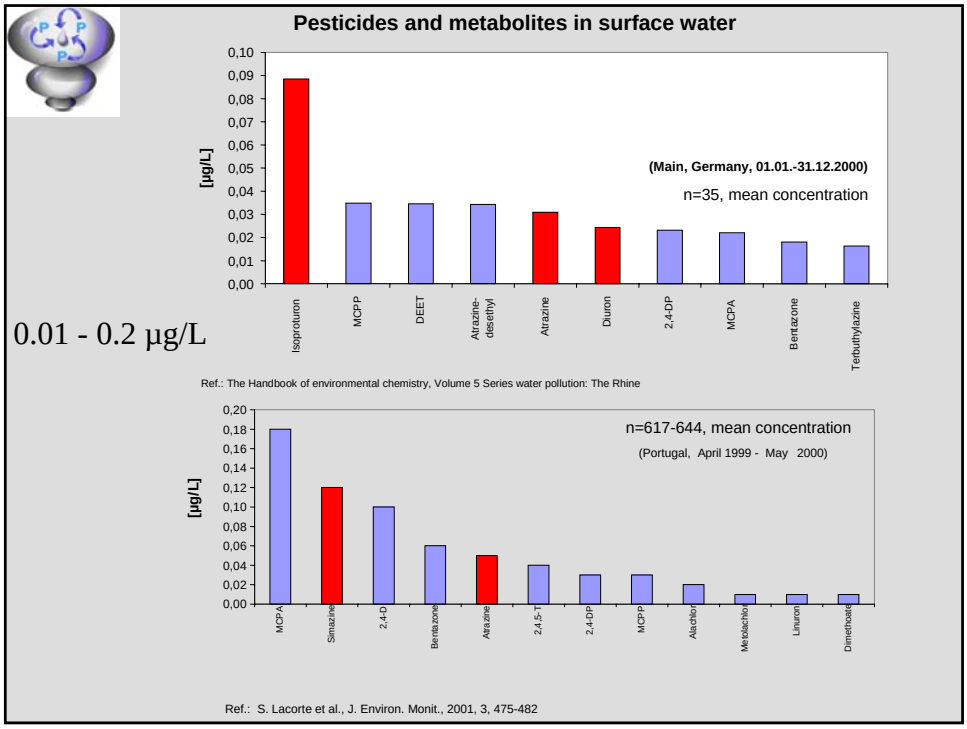
> 1 µg/L

0.1 - 1 µg/L



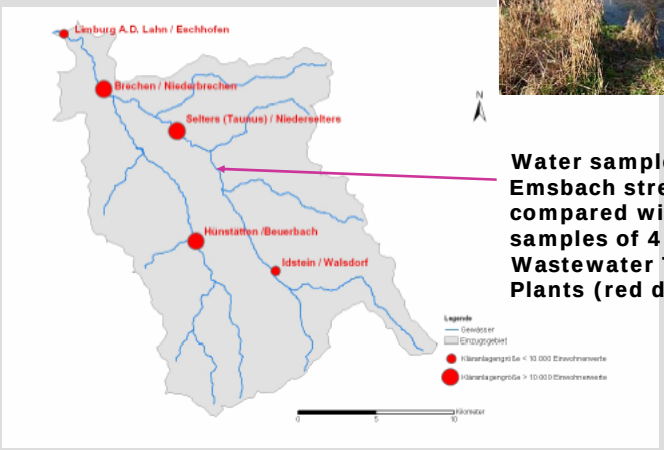
0.01 - 0.1 µg/L





How is the concentration in the water cycle correlated with the elimination during wastewater treatment??

Balance of data gained for Hessian project* to fulfill requirements of WFD



Water samples of Emsbach stream compared with water samples of 4 Wastewater Treatment Plants (red dots)

*„Auswahl der kosteneffizientesten Maßnahmenkombinationen unter Berücksichtigung der Umweltziele und Ausnahmen nach Art. 4 WRRL anhand ausgewählter Wasserkörper im hessischen Teil des Bearbeitungsgebiets Mittelrhein“

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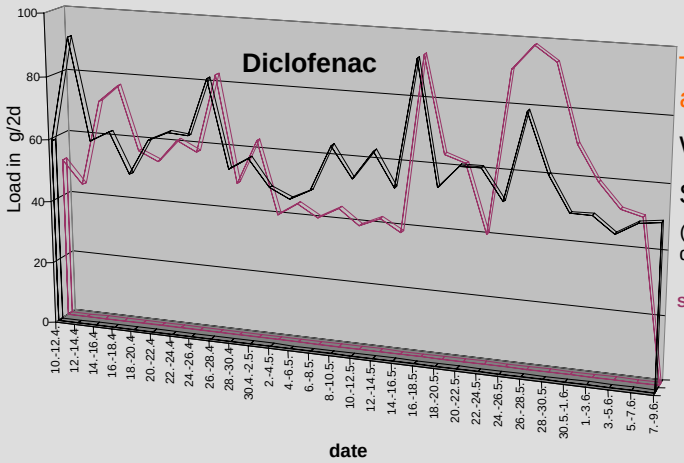
Balance of entry of emerging contaminants into the aquatic environment via WWTP:



Total for 2 month analysis (2006):

WWTP: 1.83 kg

Stream: 1.77 kg

(result different for degradable compounds)

Stream

Total of 4 WWTP effluents

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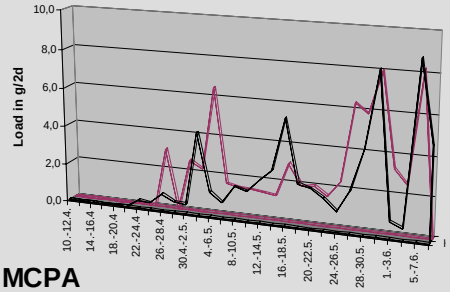
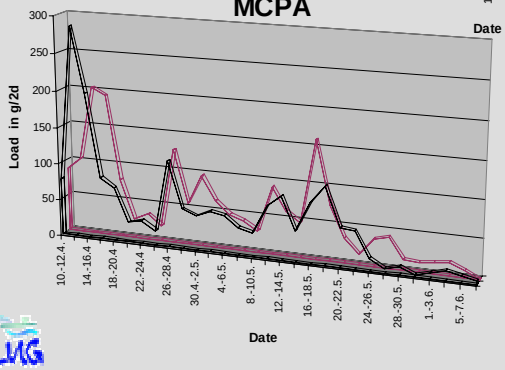
Ethofumesat



MCPA



WWTP Effluent

(Total of 4 WWTP effluents)

Stream: Emsbach

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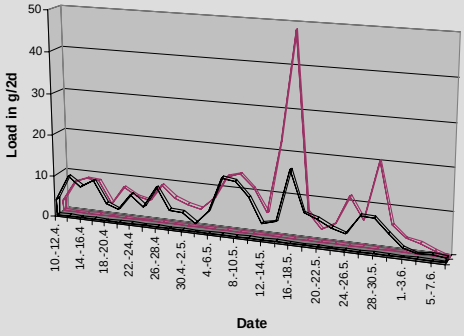
Isoproturon



Stream Emsbach

Total of 4 WWTP effluents

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Can we estimate the concentration of P³ in the water cycle??

What is the Water Cycle Spreading Index (WCSI)?

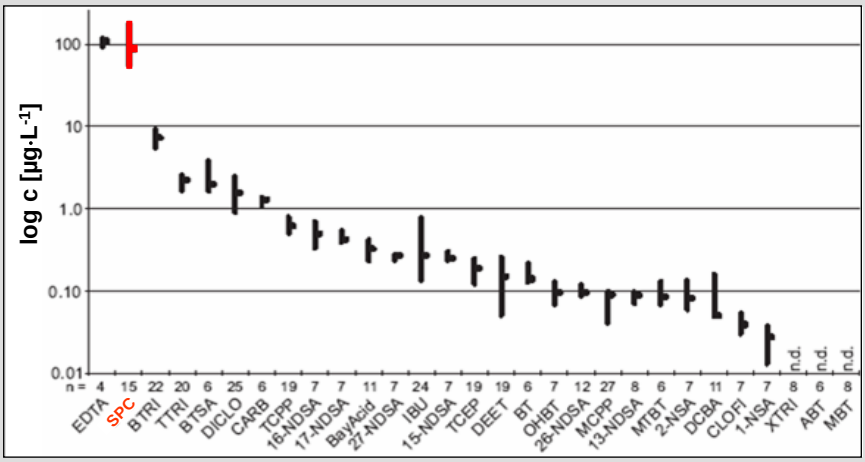
Implemented by Reemtsma et. al.:

$$WCSI = \frac{\text{effluent concentration}}{\text{normalized removal in WWTP}} = \frac{c_{in} \cdot c_{out}}{c_{in} - c_{out}}$$

Reemtsma, T., Weiss, S., Müller, J., Petrovic, M., Gonzalez, S., Barcelo, D., Ventura, F., Knepper, T.P.; *Environ. Sci. Technol.*, 2006.

WCSI:

Concentration in the effluents of 3 WWTPs in a 10 month study period:



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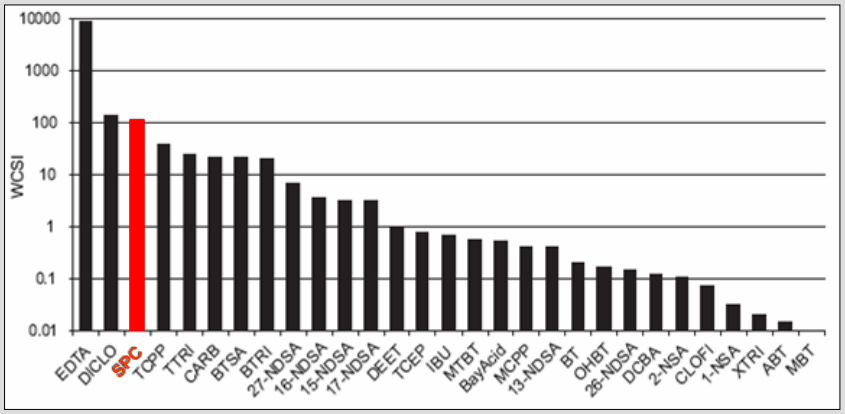
WCSI: SPCs in contrast to other compounds:



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Where do persistent polar priority pesticides and metabolites remain in the environment?

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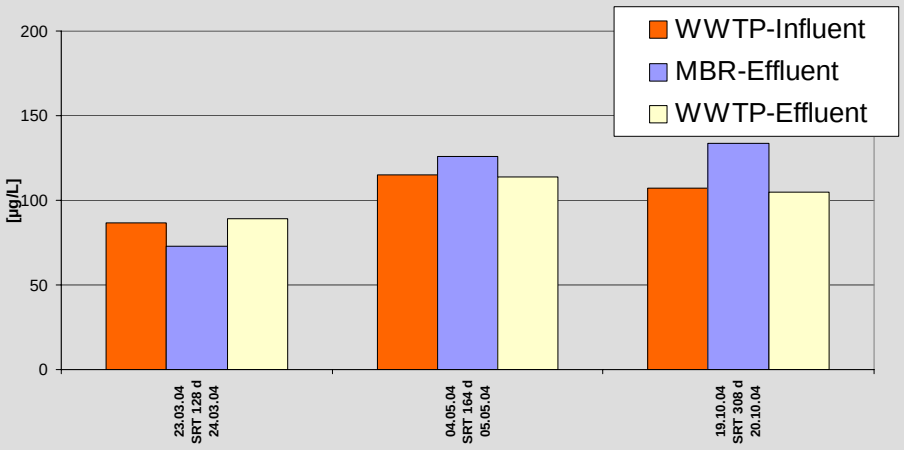
EDTA



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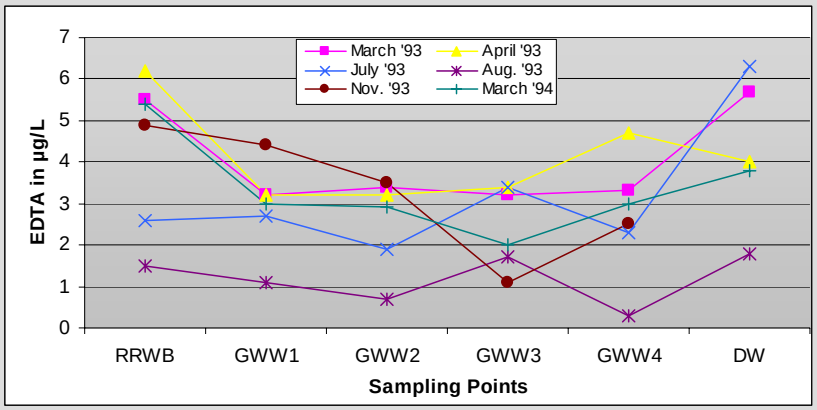
Values of EDTA in ground water wells



RRWB = River Rhine Water Basin; GWW1 = Groundwater Well (1 m); GWWW2 (80 m); GWW3 (145 m); GWW4 (160 m); DW = Drinking Water

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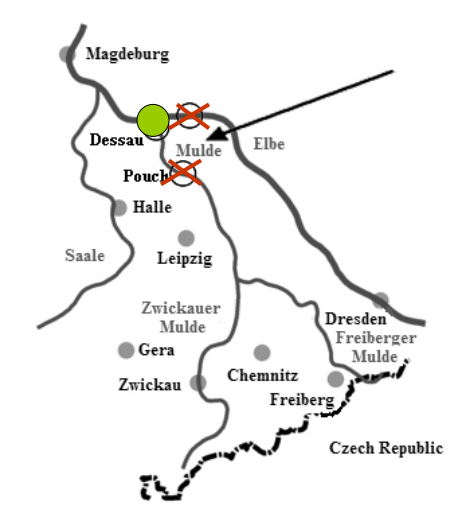
Occurrence of barbiturates in surface water



Mulde at Pouch and Elbe: not detected !

....but permanently in Dessau !!!

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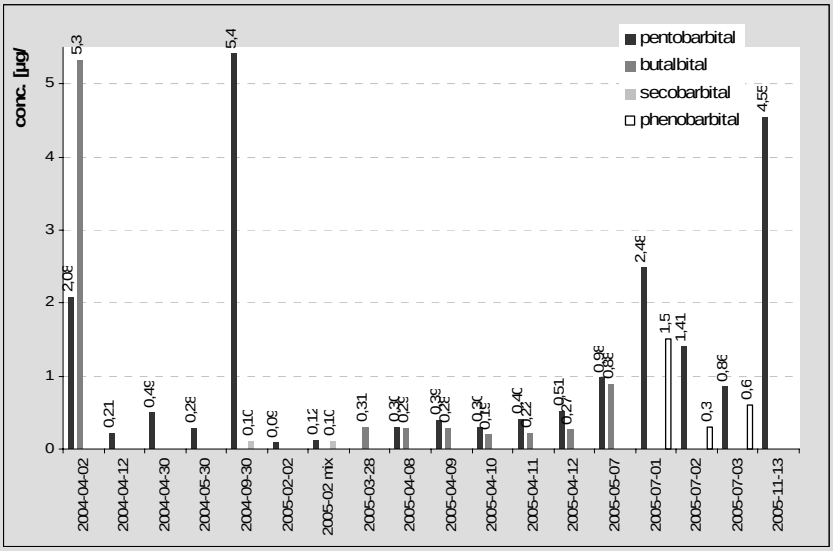
Occurrence of barbiturates in the river mulde



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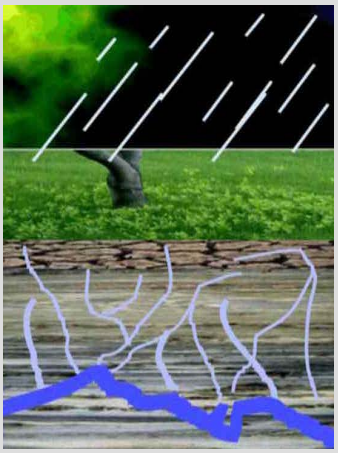
Occurrence of barbiturates in irrigated ground water (Berlin)



Groundwater, Berlin

WW infiltration 40 y ago:

Phenobarbital:

up to 1.3 µg/L

Others:

between 0.05 and 0.08 µg/L

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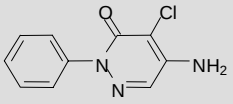
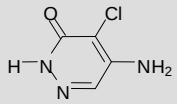
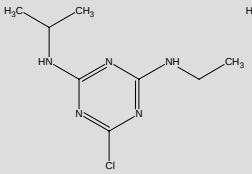
Selected pesticides and metabolites



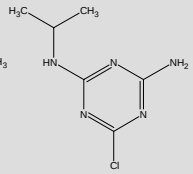
Chloridazon



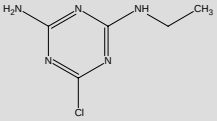
Desphenyl - chloridazon



atrazine



desethyl-atrazine



desisopropyl-atrazine

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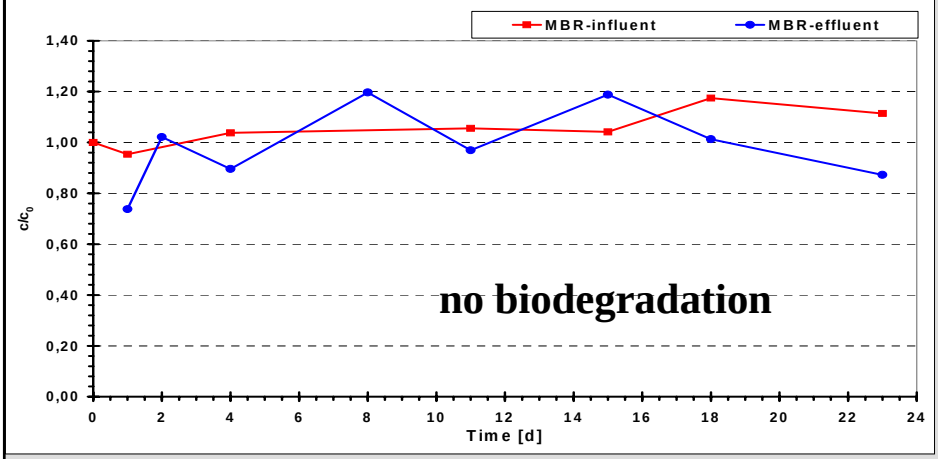
Fate of atrazine degradation after spiking into bioreactor-influent (c ~25 µg·L^{-1}):



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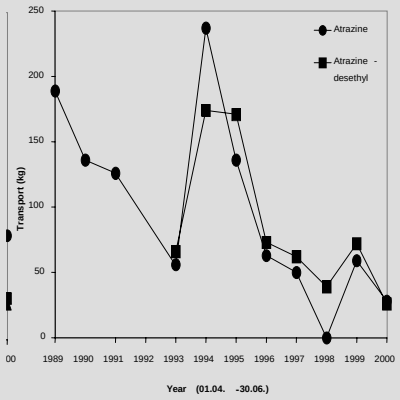
Atrazine in surface water

In Germany baned since 1991, but still detectable

Formation of metabolites in soil



Transport rates of atrazine, atrazine-desethyl in the Main river during the period of 01.04.-30.06. over the years 1989 to 2000.

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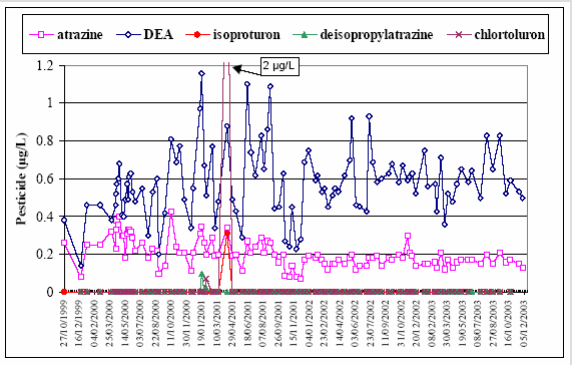
The problem:

- **atrazine application in the watershed was stopped in 2000 (replaced by acetochlor)**
- **groundwater of the Brévilles spring still exhibits contamination by atrazine and desethylatrazine with concentrations of 0.19 ± 0.7 µg/L and 0.59 ± 0.18 µg/L, respectively**



Do soil / rocks act as „storage tank“ ?

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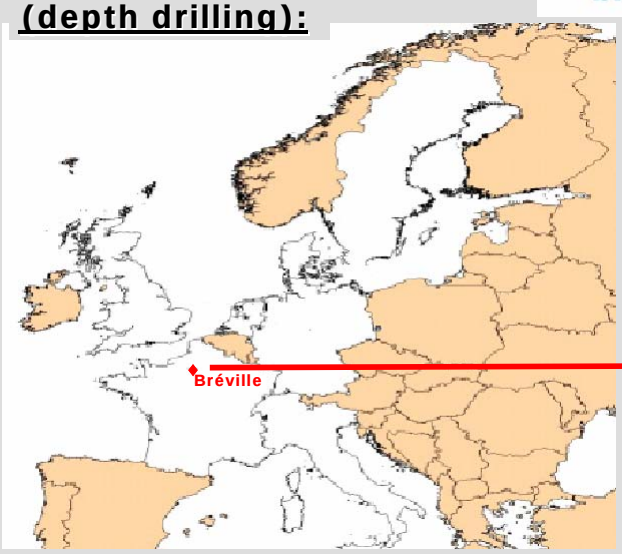
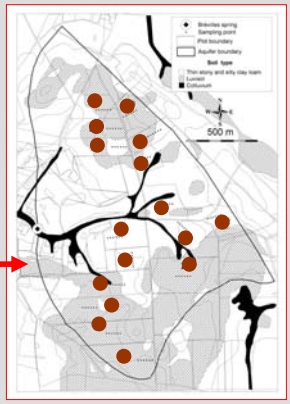
Sampling points (depth drilling):





● sampling points

Results:

Analyte (ng/g)	P10			P11			P12		P13			P15		
	0-20cm	40-60cm	60-75cm	0-20cm	20-40cm	40-60cm	0-20cm	20-40cm	0-20cm	20-40cm	40-60cm	0-20cm	20-40cm	40-60cm
atrazine	2	n.d.	n.d.	2	1	n.d.	3	1	4	<1	n.d.	2	<1	n.d.
desethylatrazine	n.d.	n.d.	n.d.	<LOD	<LOD	n.d.	n.d.	n.d.	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.
desisopropylatrazine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
acetochlor	6	n.d.	n.d.	16	4	<LOD	23	5	22	3	4	<LOD	<LOD	<LOD

Analyte (ng/g)	P18		P19			P20			P22			P23		
	0-20cm	20-40cm	0-20cm	20-40cm	40-60cm	0-20cm	20-40cm	40-60cm	60-80cm	0-20cm	20-40cm	40-60cm	0-20cm	20-40cm	40-60cm
atrazine	2	<1	1	1.3	n.d	<1	n.d.	n.d.	n.d.	5	n.d.	n.d.	2	<1	n.d.

No atrazine nor metabolites could be detected in samples from below the root zone.

Analyte (ng/g)	P24				P25		P26		P27				P28		P29		
	0-20cm	20-40cm	40-60cm	60-80cm	0-20cm	20-40cm	0-20cm	20-40cm	0-20cm	20-40cm	40-60cm	60-80cm	0-20cm	20-40cm	0-20cm	20-40cm	40-60cm
atrazine	3	n.d.	n.d.	n.d.	4	1	<1	<1	<1	<1	n.d.	n.d.	4	1	2	1	n.d.
desethylatrazine	n.d.	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.
desisopropylatrazine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
acetochlor	n.d.	n.d.	n.d.	n.d.	38	3	3	n.d.	2	n.d.	n.d.	n.d.	10	1	<LOD	<LOD	n.d.

How to monitor ?

Analysis & QA

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Analytical determination of micropollutants and their metabolites

Complex matrices Low detection limits	Selective/sensitive analytical methods	Time and labour consuming procedures
Extraction clean-up →	**Separation** →	**Detection**
Filtration, solid phase extraction with various materials, derivatisation	High pressure liquid chromatograpy gas chromatography ion chromatography	electrospray ionisation mass spectrometry (ESI-MS/MS) Electron impact quadrupole and ion trap MS/MS ESI-Q-TOF-MS....QQQQQQQ

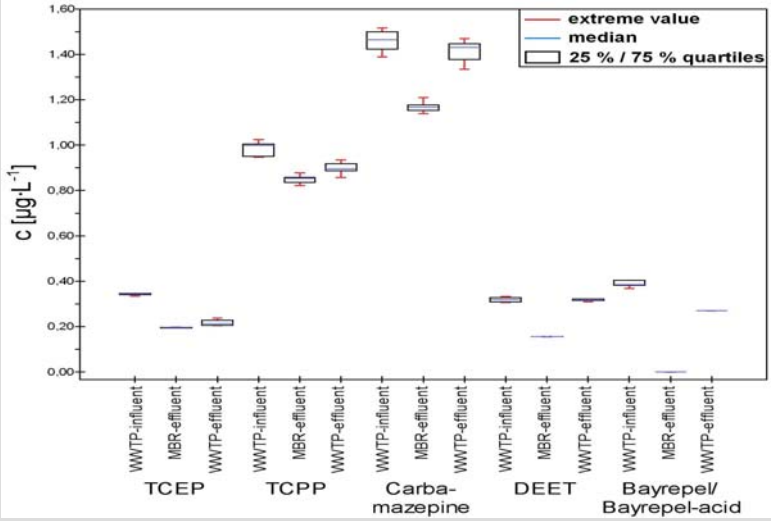
Identification of P^3 and their metabolites (after MBR treatment)

and degradation products (after AOP treatment) via

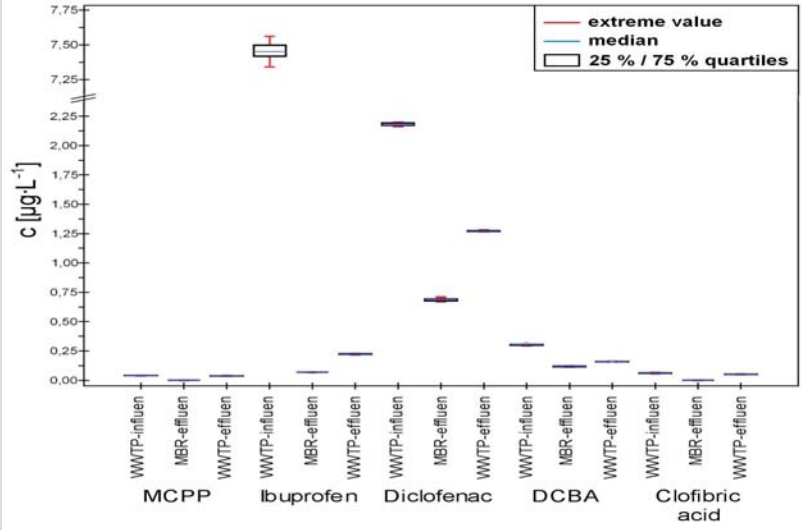
mass spectra and library searches (GC/MS)

ESI very suitable for polar and ionic compounds

SPE enrichment statistics for neutral analytes in wastewater , n=5



SPE enrichment statistics for acidic analytes in wastewater , n=5



Interlaboratory Experiments

Table 1: participants, analysed compounds and used analytical method

participant	laboratory	atrazine	DEA	DIA	IPU	acetochlor	OXA	ESA	analytical method
AGBAR (Barcelona, Spain)	1	X	X	X	X	X			IPU: SPE and HPLC-FL other pesticides: SPE and GC/MS
BRGM (Orleans, France)	2	X	X	X	X	X	X	X	atrazine, DEA, DIA, IPU, ESA: SPE and LC/MS acetochlor: SPE and GC/ECD; OXA: SPE and LC/MS/MS
CEMAGREF (Lyon, France)	3	X	X	X	X	X			SPE and LC/MS/MS
Centre d'Analyses et de Recherches (Illkirch, France)	4	X	X	X	X	X			atrazine, DEA, DIA, IPU: SPE and HPLC/UV acetochlor: LL, GC/MS
CSIC (Barcelona, Spain)	5	X	X	X	X	X	X	X	SPE and LC/MS/MS
Europa University of Applied Science (Idstein, Germany)	6	X	X	X		X			GC/MS
Institut Fresenius (Taunusstein, Germany)	7	X	X	X	X	X			IPU: LL and LC/MS/MS other pesticides: SPE and GC/MS
Institut Pasteur de Lille (France)	8	X	X	X	X	X			SPE and LC/MS/MS
Laboratoire Departemental d'Analyses de la Drôme (Valence, France)	9	X	X	X	X	X			SPE and LC/MS/MS
Laboratoire National de Métrologie et d'Essais (Paris, France)	10	X	X	X	X	X			SPE and LC/MS/MS
Monsanto Company (Saint Luis, USA)	11						X	X	direct injection LC/MS/MS

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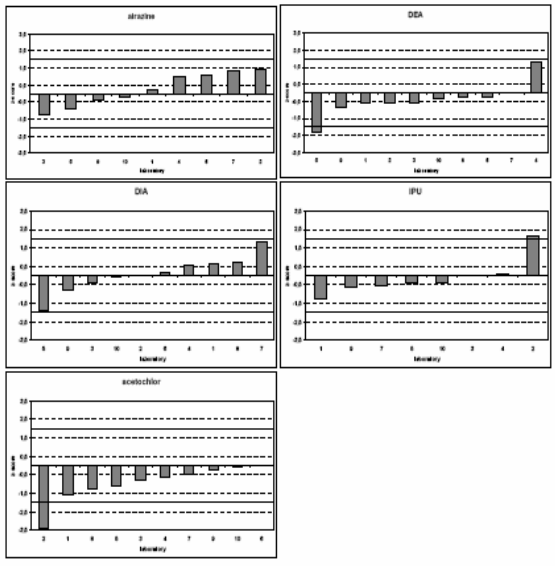
Interlaboratory Experiments

z-scores - LEVEL 1



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Conclusions

- The more pollutants are analysed the more can be detected – main source for entry into the aquatic environment are WWTP.
- Monitoring campaigns need to be well thought of and organized – metabolites need to be included
- Organic pollutants are present in surface waters all over Europe at comparable concentrations (for pesticides during application time)
- The **WCSI** may be used as an indicator for the potential of polar pollutants to be spread in the aquatic environment.
- Analytical methods need to be harmonized and thoroughly checked in the matrix analyzed.

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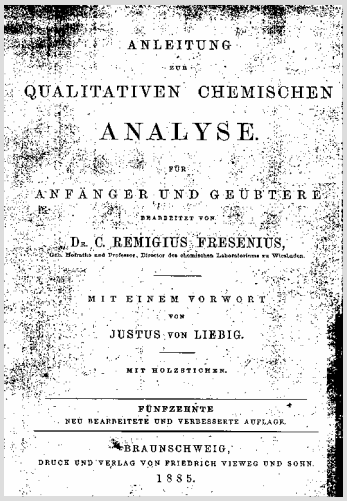
Peter Seel


ANLEITUNG
ZUR
QUALITATIVEN CHEMISCHEN
ANALYSE.
FÜR
ANFÄNGER UND GEÜBTERE.
BEARBEITET VON
Dr. C. REMIGIUS FRESENIUS,
MIT EINEM VORWORT
VON
JUSTUS VON LIEBIG.
MIT HOLZSTICHEN.
FÜNFZEHNTE
NEU BEARBEITETE UND VERBESSERTE AUFLAGE.
BRAUNSCHWEIG,
DRUCK UND VERLAG VON FRIEDRICH VIEWEG UND SOHN.
1885.

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