Non-target screening in the Danube study

Nordic workshop
“Non-target screening of chemicals - to know the unknowns”
8 -9 April 2014,Ystad, Sweden

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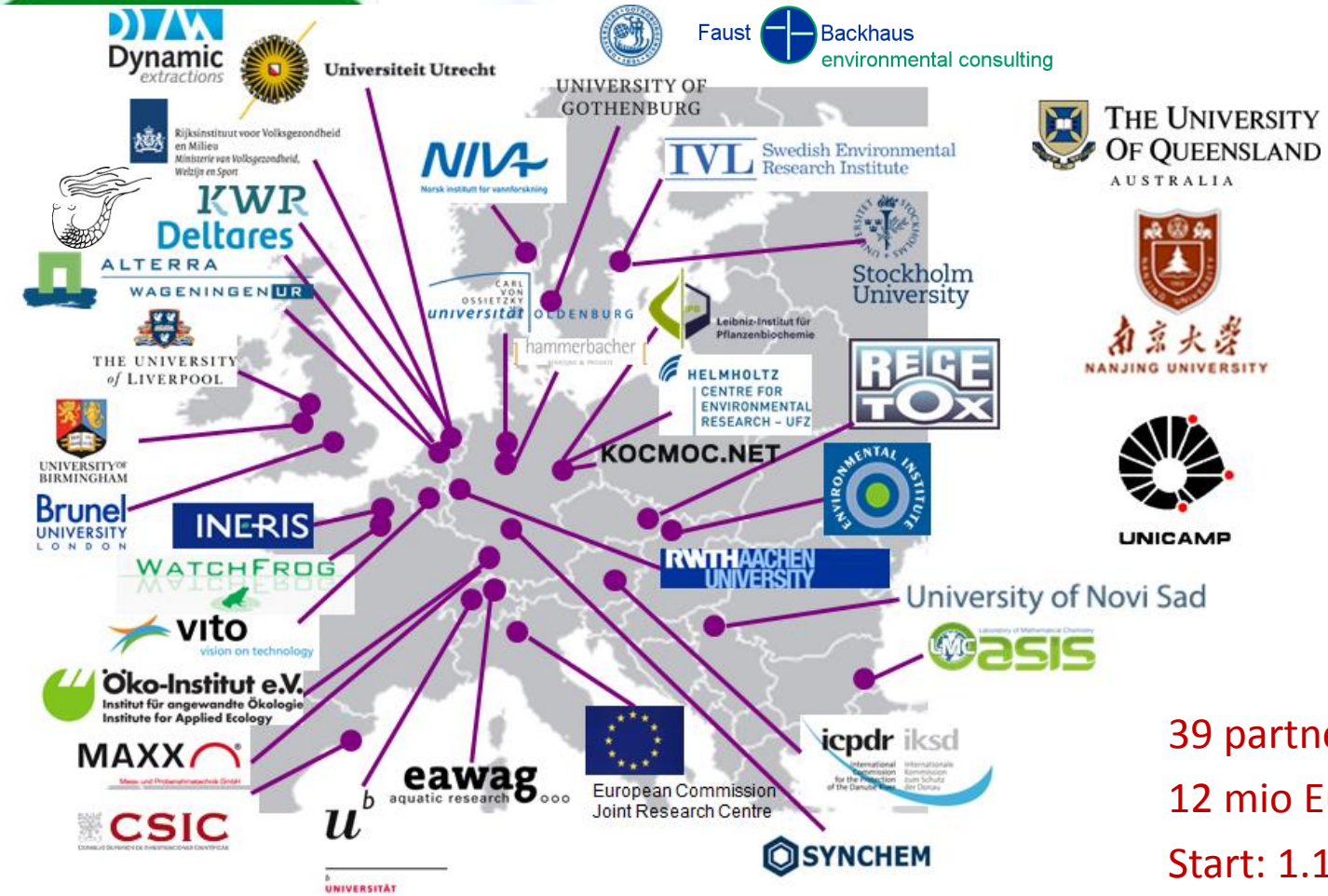
norman



39 partners

12 mio Euro

Start: 1.10.2013

Duration: 5 years

funded by the European Commission (Grant Nr. 603437)

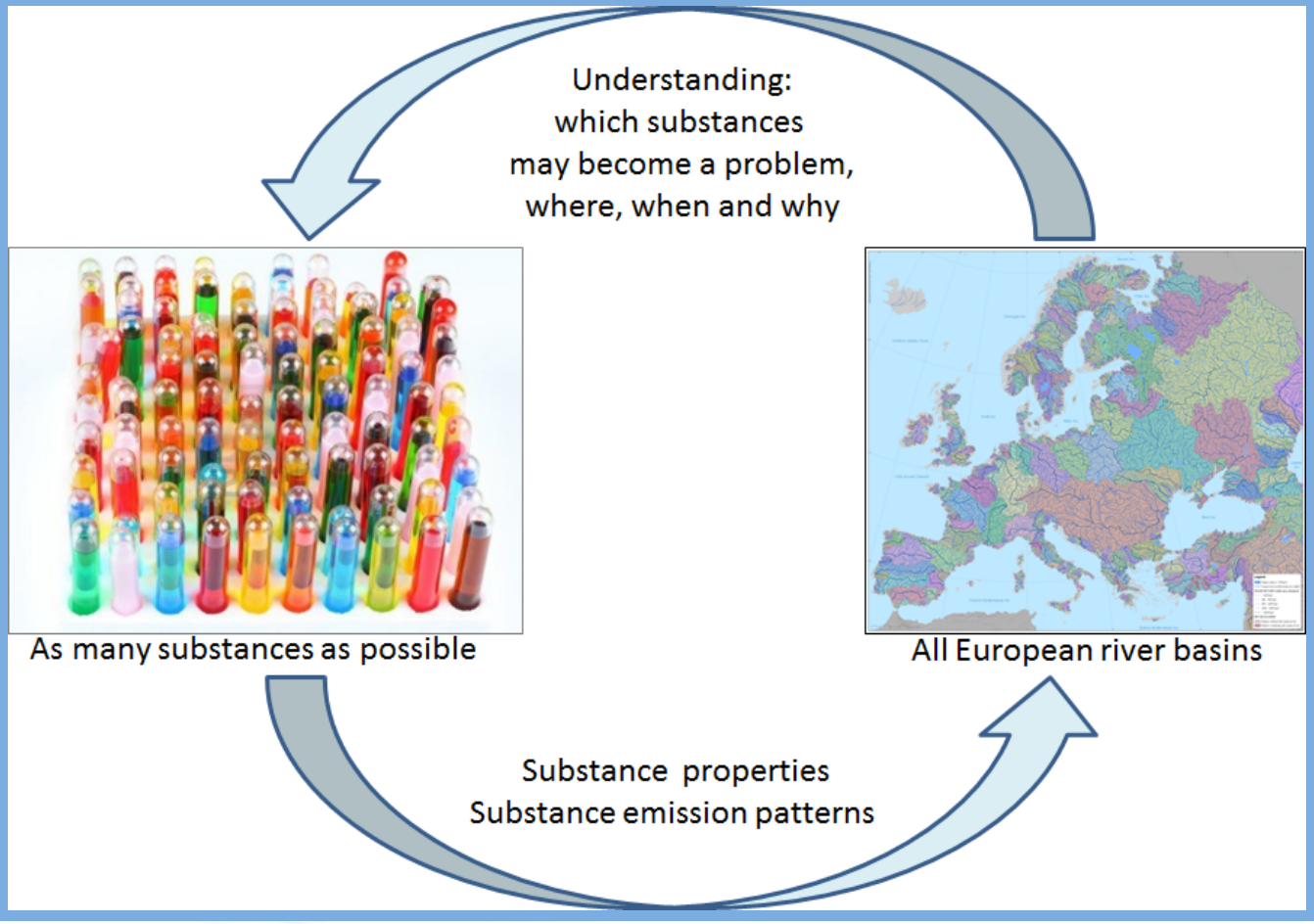
Objectives of SOLUTIONS:

- Conceptual framework for prioritisation, assessment and abatement of pollutants (eco and human health)
- **Efficient tools for identification of substances and mixtures posing a risk (e.g. River Basin Specific Pollutants, RBSPs):**
  - effect-based and analytical tools for early detection and identification
  - integrated models and databases for risk modelling exploiting data from chemical authorisation (e.g. REACH) and monitoring
- Demonstration in trans-European case studies (Danube, Rhine, Iberian Peninsula)

Modeling tools

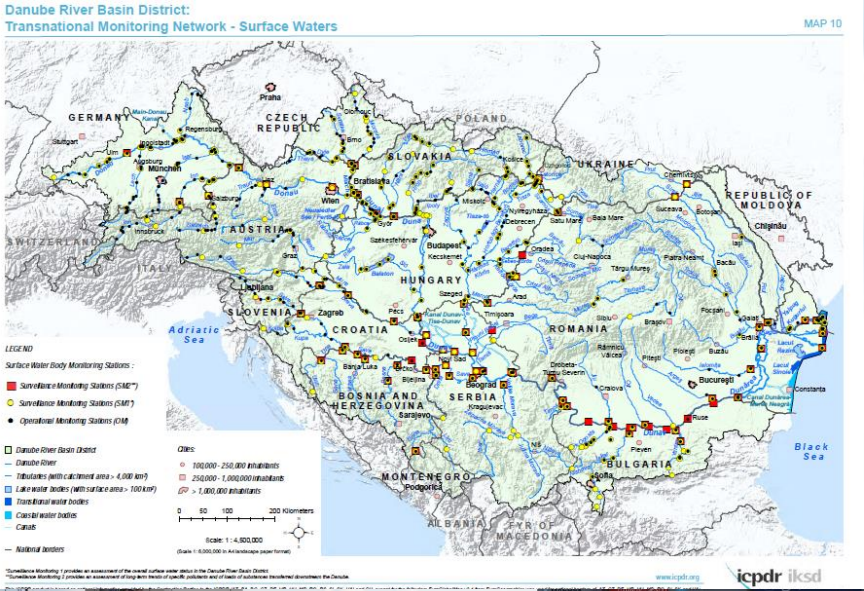
Europe-wide integrated risk modelling

⇒ extrapolation to future emerging compounds



Joint Danube Survey 3

ICPDR, the biggest river expedition in the world
11 August – 26 September 2013, 11 countries
http://danubesurvey.org/news



	Title	Author(s)
1	Introduction	Igor Liska
2	Survey preparation	Jaroslav Slobodnik
3	Hydromorphology	Katarina Holubova, Ulrich Schwarz, Peter Matok, Jan Busovsky, Radoslav Cuban
4	Ornithological survey & river bank assessment	Georg Frank
5	Macrozoobenthos	Wolfram Graf, Béla Csányi, Patrick Leitner, Momir Paunović, Thomas Huber, Claudia Nagy, Jozsef Szekeres
6	Phytobenthos	Jarmila Makovinska, Corina Carpentier
7	Macrophytes	Igor Stankovic, Karin Pall, Georg A. Janauer
8	Phytoplankton	Martin Dokulil, Ulrich Donabaum
9	Fish	Vinzenz Bammer, Michael Schabuss, Horst Zornig, Lachezar Zlatev Pehlivanov, Zoltan Szaloky, Andras Weiperth, Agnes Irma Gyorgy
10	Invasive species?	Momir Paunovic, Bela Csanyi, Wolfram Graf, macrophyta??
11	Zooplankton	Katalin Zsuga
12	MICROBIOLOGY – Bacterial Faecal Indicators	**Alexander Kirschner,** Stefan Jakwerth, Stoimir Kolarevic, Regina Sommer, Paul Blaschke, Gerhard Kavka, Georg Reischer & Andreas Farnleitner
13	MICROBIOLOGY - Microbial Faecal Source Tracking	Georg Reischer, Alexander Kirschner, Domenico Savio, Robert Mach, Arnold Bahlmann, Rudolf J. Schneider, Tobias Schulze, Werner Brack & **Andreas Farnleitner**
14	MICROBIOLOGY – Ecotoxicology and Antibiotic Resistance Patterns	Gernot Zarfel, Bettina Folli, Michaela Lipp, Bettina Pfeifer, Rita Baumert, Andreas Farnleitner, Alexander Kirschner & **Clemens Kittinger**
15	MICROBIOLOGY – Microbial Ecology	**Alexander Kirschner**, Stoimir Kolarevic, Stefan Jakwerth, Georg Reischer & Andreas Farnleitner
16	MICROBIOLOGY – Microbial Metagenomics	Teresa Lettieri, Valentina Ferrero, Alexander Kirschner, Andreas Farnleitner, Lourdes Duque & Armin Lahm

	Title	Author(s)
17	General physico-chemical determinands and nutrients	Carmen Hamchevici, Florentina Dumitrache, Fabrizio Sena, VUVH??
18	Quantity and Quality of Dissolved Organic Matter	**Thomas Hein**, Nina Welti, Tz-Ching Yeh, Alexander Kirschner & Elisabeth Bondar-Kunze, Damir Tomas, Marija Marjanovic-Rajcic
19	Petroleum hydrocarbons	Peter Literathy
20	Priority and other organic substances	Manferd Sengl, Karin Deutsch
21	Metals	Dietmar Krämer, Bernd Gawlik (Hg)
22	Target analysis of emerging polar substances	Robert Loos, Jerker Fick, Simona Tavazzi, Giulio Mariani, Gunther Umlauf
23	Target analysis of semivolatile organic compounds	Gunther Umlauf, Robert Loos, Simona Tavazzi, Giulio Mariani
24	Emerging substances in surface and groundwater	Florian Storck, Heinz Juergen Brauch
25	Chemical and immunochemical analysis of anthropogenic markers and organic contaminants	**Arnold Bahlmann**, Tina Lochen, Tobias Schulze, Alexander Kirschner, Werner Brack, Rudolf J. Schneider, Martin Krauss
26	Large volume sampling and Effect Directed Analysis (EDA)	**Tobias Schulze**, Martin Krauss, Werner Brack, Henner Hollert, Thomas-Benjamin Seiler, Zuzana Rabova, Klara Hilscherova, Selim Ait-Aissa, Nicolas Creusot, Knut Erik Tollefsen
27	Micronucleus and fish blood bioassays	**Bjoern Deutschmann**, Sandor Sipos, Ivana Teodorovic, Stoimir Kolarevic, Henner Hollert
28	Passive sampling: target and non-target screening and toxicological profiling	**Branislav Vrana**, Foppe Smedes, Ian Allan, Merete Grung, Zuzana Rabova, Klara Hilscherova, Tatsiana Rusina, Peter Tarabek, Jaroslav Slobodnik
29	Non-target screening of organic pollutants	**Drazenka Stipanicev,** Sinisa Repec, Peter Oswald, Wolfgang Schulz, Manfred Sengl, Jaroslav Slobodnik
30	Radioactive contamination	Franz Josef Maringer, Andreas Baumgartner, Michael Stietka, Claudia Seidel, Fabian Rechberger
31	Isotope analysis of fish and water samples	Andreas Zitek
32	Lessons learned	Editors
33	Conclusions	Editors

ICPDR Water Quality Database
http://www.icpdr.org/wq-db/

Databases | NORMAN | Water Quality | ICPDR - Int | Overview | – Danube River | Search | Ask.com

www.icpdr.org/wq-db/wq/overview

icpdr iksd
International Commission for the Protection of the Danube River
Internationale Kommission zum Schutz der Donau

Water Quality Database
- Introduction
- Overview
- Static data
- Results

JAROSLAV_SLOBODNIK
- Log out

Danube River Basin Water Quality Database

Overview

Database Overview

Data Source	Stations	Points	From	To	Samples	Determinands	Analytical Methods	Analytical Results
JDS1	98	273	13.08.2001	19.09.2001	967	169	288	27,030
JDS2	124	329	13.08.2007	26.09.2007	1,025	239	601	38,676
TNMN	131	185	01.01.1996	31.12.2011	281,117	273	4,172	1,226,344
Total	**353**	**787**	**01.01.1996**	**31.12.2011**	**283,109**	**455**	**5,061**	**1,292,050**

Please report errors and provide suggestions for improvements of this database to alex.hoebart@unvienna.org.

ENG 09:12 27/03/2014

Target, suspect and unknown screening of organic pollutants by UHPLC Coupled to iFunnel Q-TOF/MS

Draženka Stipaničev, Siniša Repec

Croatian waters, Central water management laboratory

Conditions

UHPLC Parameters:

1290 Infinity

IONIZATION MODIFIERS: both eluents, HPLC water NH_4AC 5mM / HAc (pH=4.7) and methanol (B), in the positive electrospray ionization (ESI+)

TOTAL RUN: 20 min

COLUMN: RP column - flow rate 0.4 mL /min was used (2,1x150 Acquity UPLC, HSS T3 1.8 µm)

INJECTION VOLUME : 100µL

MS only

QTOF Parameters – Mass Spectrometer

i-Funnel 6550
Instrument state: high res. mode (4GHz)

MS SCREENING METHOD:
MS 1 Mode: 2 spectra/sec,
Transients/spectrum : 4100
Mass Range: 50 – 1000 m/z
ESI+

METHOD : Gradient 0.4 mL/min
Nebulizer: 30 psig
Drying gas: 18 L N2/min
Sheath gas: 11 L N2/min
Nozzle: 0V
VCap: 3500 V
Fragmentor: 400V

All IONS

QTOF Parameters – Mass Spectrometer

i-Funnel 6550
Instrument state: high res. mode (4GHz)

All Ions SCREENING METHOD:
All ions Mode: 3 spectra/sec,
Transients/spectrum : 2705
Mass Range: 50 – 1000 m/z
ESI+
Three segments at CE: 0 eV, 20 eV, and 40 eV

METHOD : Gradient 0.4 mL/min
Nebulizer: 30 psig
Drying gas: 18 L N2/min
Sheath gas: 11 L N2/min
Nozzle: 0V
VCap: 3500 V
Fragmentor: 400V

The resolution power for ESI+ :
52296 at 922.009798 *m/z*
21801 at 118,086255 *m/z*

Screening workflow – Croatian Waters

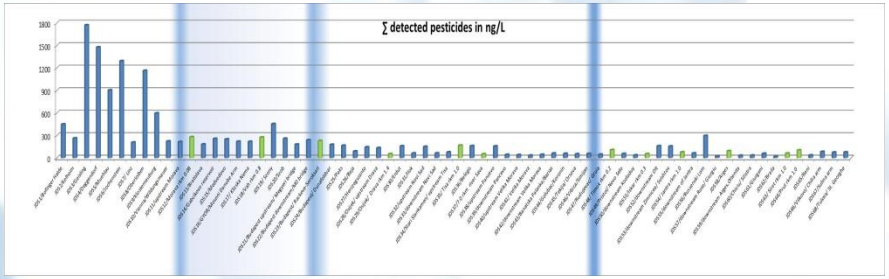
SCREENING	MS ONLY MFE-MP- FbF (Rt, exact mass, formula, isotopic pattern)	ALL IONS (CE 0,20,40 eV) FbF (Coelution score,Rt difference, fragment ions confirmation)	autoMSMS FbF	STANDARD IN LABORATORY	*PCD LIBRARY with MSMS SPECTRUM	*PCD LIBRARY without MSMS SPECTRUM	Web confirmation (PubChem, ChemID+, NIST...)
TARGET	+	+	+	+	+		
SEMI TARGET	+	+	+	-	+		
SUSPECT	+	+	+	-	-	+	
UNKNOWN	+/-	+/-	+	-	-	-	+
TOTAL UNKNOWN	+/-	+/-	+	-	-	-	-

•**MFE**- molecular feature extractor
•**FbF**- Find By Formula
•**MP**-MassProfiler

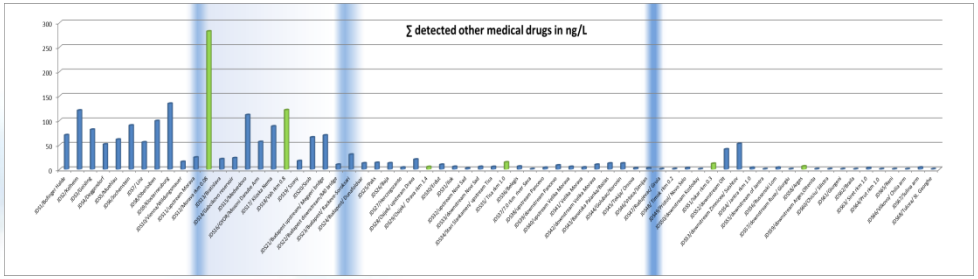
<u>*Agilent PCDL</u>:

- **MassHunter *METLIN metabolite* PCDL** ver 5 (database 64092 compounds, MS/MS library > 8040 compounds at three collision energies: 10, 20 and 40eV)
- **MassHunter *Forensic Toxicology* PCDL** ver. 4.1 (database 7509 compounds, MS/MS library > 2500+ compounds at three collision energies: 10, 20 and 40eV)
- **MassHunter *Pesticide* PCDL** ver. 4.1 (database 1664 compounds, MS/MS library > 600 compounds at three collision energies: 10, 20 and 40eV)

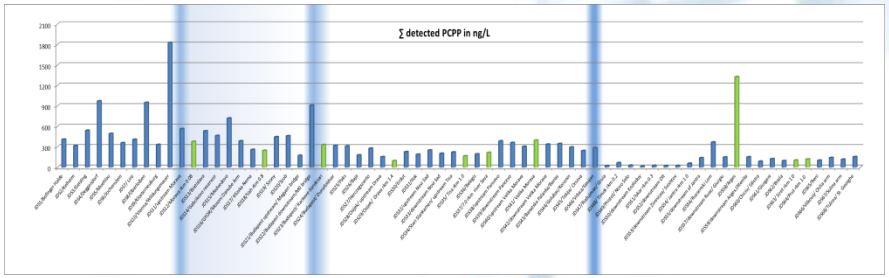
TARGET SCREENING (QUANT)



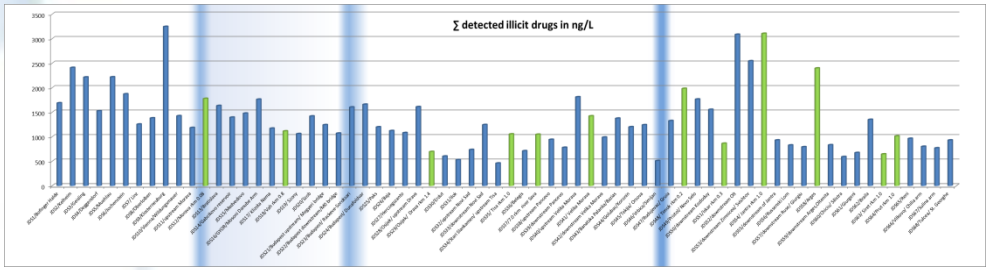
29 PESTICIDES



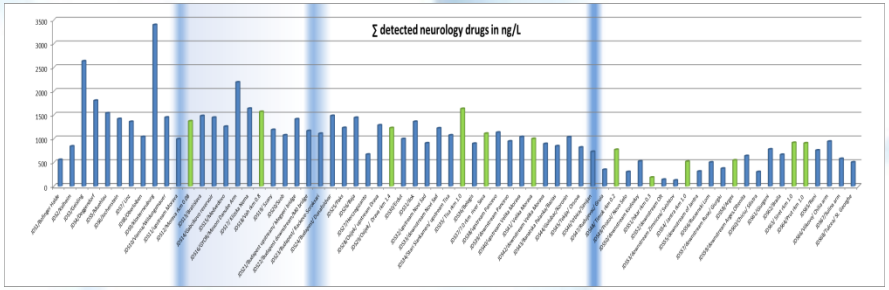
14 OTHER MEDICAL DRUGS



7 PCPP



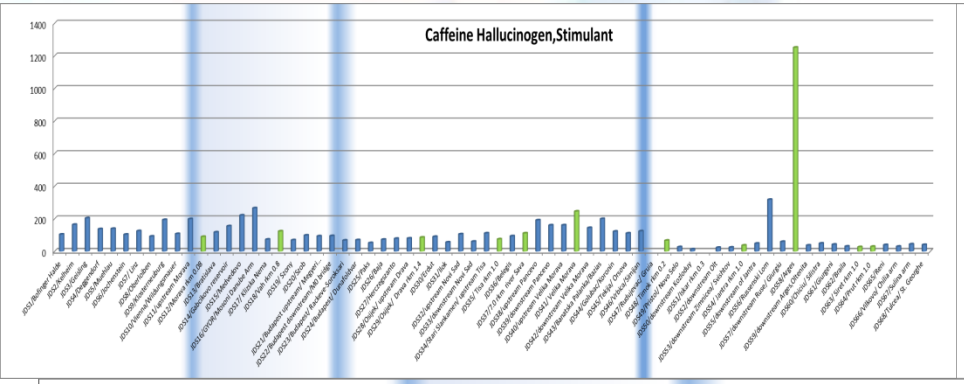
78 ILLICIT AND OTHER DRUGS

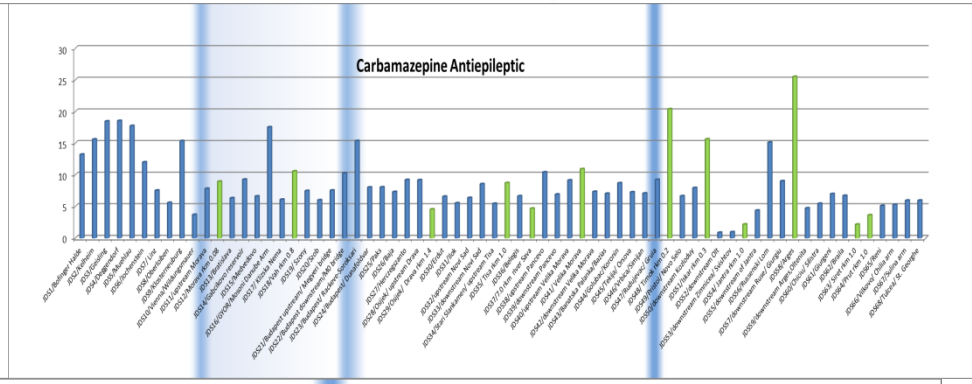


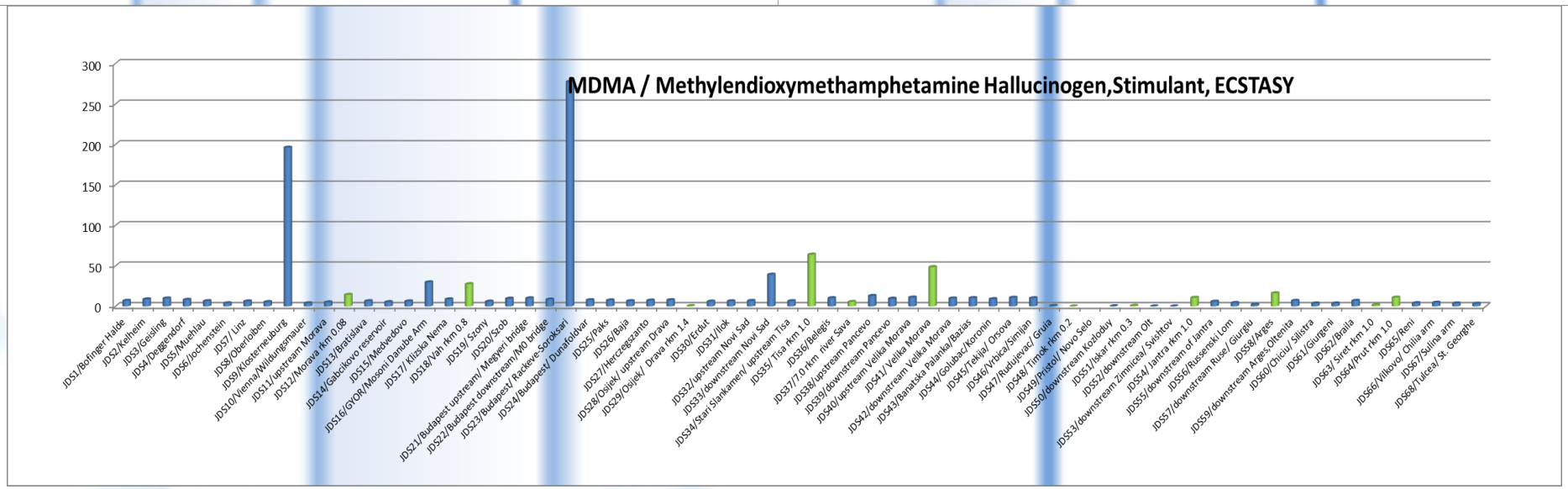
40 NEUROLOGY DRUGS

Rainy day 20.08.2014
26.08.2014.
12.09.2014.

TARGET SCREENING (QUANT)

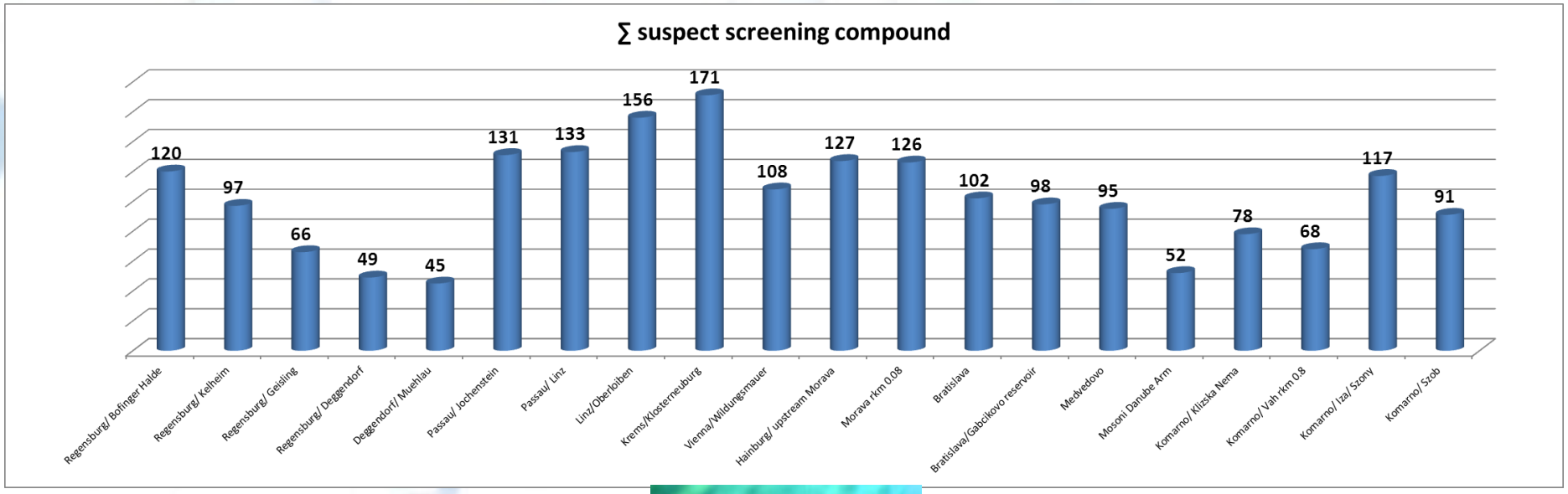






After weekend, 19.08. monday- JDS 9 Vienna, 26.08. monday – JDS 22 Budapest,
03.09. thursday – JDS 33 Novi Sad

NON-TARGET SCREENING (QUAL)



Suspect screening - JDS 58 phase I -MS only

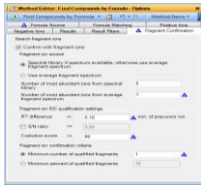
Data File	MS_JDS_58.d	**Sample Name**	MS_JDS_58
Sample Type	Sample	**Position**	P1-C4
Instrument Name	QTOF1	**User Name**	
Acq Method	MS_only_positive.m	**Acquired Time**	9/20/2013 7:43:47 PM
IRM Calibration Status	Success	**DA Method**	MS_PMU.m
Comment			

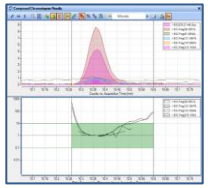
	Column2	Column3	Column4
Sample Group		**Info.**	
Funnel RF HP	150	**Funnel RF LP**	60
Acquisition SW	6200 series TOF/6500		
Version	series Q-TOF B.05.01 (B5125)		

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula	DB Diff (ppm)
Cpd 165: Fenchlorazole	1.065	372.87506	1016	Fenchlorazole	C10 H4 Cl5 N3 O2	372.87461	1.19	C10 H4 Cl5 N3 O2	1.45
Cpd 141: Fluorodifen	1.09	328.0309	1320	Fluorodifen	C13 H7 F3 N2 O5	328.03071	0.59	C13 H7 F3 N2 O5	2.44
Cpd 70: Cefazaflur	1.499	470.01153	3234	Cefazaflur	C13 H13 F3 N6 O4 S3	470.01125	0.59	C13 H13 F3 N6 O4 S3	-2.19
Cpd 95: 2,3-MDPEA / 2,3-Methylenedioxyphenethylamine	2.061	165.07883	3893	2,3-MDPEA / 2,3-Methylenedioxyphenethylamine	C9 H11 N O2	165.07898	-0.87	C9 H11 N O2	-0.21
Cpd 236: Merphos	2.078	298.10124	872	Merphos	C12 H27 P S3	298.10125	-0.04	C12 H27 P S3	1.89
Cpd 91: Vigabatrin	2.087	129.07889	7425	Vigabatrin	C6 H11 N O2	129.07898	-0.67	C6 H11 N O2	0.06
Cpd 78: o-Phenyleneurea (Benzimidazolone) (Droperidol-M)	3.381	134.048	8986	o-Phenyleneurea (Benzimidazolone) (Droperidol-M)	C7 H6 N2 O	134.04801	-0.07	C7 H6 N2 O	-0.02
Cpd 151: Anatoxin a	3.782	165.11506	1043	Anatoxin a	C10 H15 N O	165.11536	-1.87	C10 H15 N O	1.48
Cpd 255: (+)-cucurbic acid	3.799	212.14133	835	(+)-cucurbic acid	C12 H20 O3	212.14124	0.4	C12 H20 O3	-0.14
Cpd 212: L-Adrenaline	3.884	183.0896	1378	L-Adrenaline	C9 H13 N O3	183.08954	0.31	C9 H13 N O3	-0.55
Cpd 54: 6-Hydroxynicotine (S)	4.31	178.11061	9978	6-Hydroxynicotine (S)	C10 H14 N2 O	178.11061	-0.03	C10 H14 N2 O	-0.6
Cpd 51: Bufencarb	4.523	221.14166	15231	Bufencarb	C13 H19 N O2	221.14158	0.39	C13 H19 N O2	1.07
Cpd 265: Sotalol	4.676	272.11982	410	Sotalol	C12 H20 N2 O3 S	272.11946	1.33	C12 H20 N2 O3 S	-2.29
Cpd 129: Esculin (Polychrome)	4.702	340.07933	4476	Esculin (Polychrome)	C15 H16 O9	340.07943	-0.31	C15 H16 O9	0.8
Cpd 254: 4-[2-(Propylamino)ethyl]-1,3-dihydro-2H-indol-2-one	4.795	218.14194	691	4-[2-(Propylamino)ethyl]-1,3-dihydro-2H-indol-2-one	C13 H18 N2 O	218.14191	0.13	C13 H18 N2 O	-0.65
Cpd 1: Pramiracetam	4.864	269.21048	90695	Pramiracetam	C14 H27 N3 O2	269.21033	0.55	C14 H27 N3 O2	0.09
Cpd 248: Ciclacillin	4.957	341.14013	757	Ciclacillin	C15 H23 N3 O4 S	341.14093	-2.32	C15 H23 N3 O4 S	0.19
Cpd 121: Buformine	4.974	157.13254	3334	Buformine	C6 H15 N5	157.13275	-1.32	C6 H15 N5	-0.25
Cpd 79: N'-Hydroxymethylnorcotinine	4.983	192.08995	8963	N'-Hydroxymethylnorcotinine	C10 H12 N2 O2	192.08988	0.38	C10 H12 N2 O2	-0.26
Cpd 62: Pribecaine	5.059	277.16761	9104	Pribecaine	C16 H23 N O3	277.16779	-0.65	C16 H23 N O3	-0.05
Cpd 231: Tecomine	5.085	179.12958	674	Tecomine	C11 H17 N O	179.13101	-8	C11 H17 N O	6.81
Cpd 186: Diphacinone	5.179	340.11057	2460	Diphacinone	C23 H16 O3	340.10994	1.83	C23 H16 O3	0.09
Cpd 259: Aramite	5.196	334.10086	806	Aramite	C15 H23 Cl O4 S	334.10056	0.89	C15 H23 Cl O4 S	0.43
Cpd 193: Triazamate	5.204	314.14104	1211	Triazamate	C13 H22 N4 O3 S	314.14126	-0.71	C13 H22 N4 O3 S	-0.76
Cpd 170: 1-(3-Pyridinyl)-1,4-butanediol	5.264	167.09458	2380	1-(3-Pyridinyl)-1,4-butanediol	C9 H13 N O2	167.09463	-0.29	C9 H13 N O2	0.12
Cpd 125: Homatropine	5.383	275.15169	2890	Homatropine	C16 H21 N O3	275.15214	-1.63	C16 H21 N O3	0.75
Cpd 117: Gabapentin	5.4	171.12592	3718	Gabapentin	C9 H17 N O2	171.12593	-0.05	C9 H17 N O2	-0.71
Cpd 190: Arecoline	5.485	155.09469	665	Arecoline	C8 H13 N O2	155.09463	0.39	C8 H13 N O2	-1.22
Cpd 39: Ambenoxan	5.553	267.1468	30587	Ambenoxan	C14 H21 N O4	267.14706	-0.97	C14 H21 N O4	1.87
Cpd 63: Oxprenolol	5.57	265.16778	14740	Oxprenolol	C15 H23 N O3	265.16779	-0.06	C15 H23 N O3	0.46
Cpd 178: Gly Trp	5.681	261.11121	2204	Gly Trp	C13 H15 N3 O3	261.11134	-0.49	C13 H15 N3 O3	-0.05
Cpd 106: Theophylline	5.69	180.06446	4737	Theophylline	C7 H8 N4 O2	180.06473	-1.49	C7 H8 N4 O2	1.35
Cpd 86: Dacarbazine	5.724	182.09189	8310	Dacarbazine	C6 H10 N6 O	182.09161	1.56	C6 H10 N6 O	0.04
Cpd 42: Acetylhelenalin	5.826	304.13049	22757	Acetylhelenalin	C17 H20 O5	304.13107	-1.93	C17 H20 O5	0.37
Cpd 80: Ephedrine	5.869	165.11524	7912	Ephedrine	C10 H15 N O	165.11536	-0.76	C10 H15 N O	0.37
Cpd 75: O-Desmethyltramadol	5.979	249.17279	7869	O-Desmethyltramadol	C15 H23 N O2	249.17288	-0.34	C15 H23 N O2	0.42
Cpd 35: Arg Ser Ser	6.03	348.17585	37612	Arg Ser Ser	C12 H24 N6 O6	348.17573	0.33	C12 H24 N6 O6	-0.13
Cpd 163: 8-Hydroxychinolin (8-Hydroxyquinoline)	6.099	145.05283	2164	8-Hydroxychinolin (8-Hydroxyquinoline)	C9 H7 N O	145.05276	0.46	C9 H7 N O	-0.9
Cpd 243: Nifenazone	6.124	308.12719	537	Nifenazone	C17 H16 N4 O2	308.12733	-0.45	C17 H16 N4 O2	-0.93
Cpd 161: Acetylpheneturide	6.133	248.11601	2037	Acetylpheneturide	C13 H16 N2 O3	248.11609	-0.35	C13 H16 N2 O3	-0.83
Cpd 37: Cotinine	6.252	176.09515	33290	Cotinine	C10 H12 N2 O	176.09496	1.07	C10 H12 N2 O	-0.86
Cpd 245: Sudan 410	6.329	408.19579	789	Sudan 410	C26 H24 N4 O	408.19501	1.9	C26 H24 N4 O	1.5
Cpd 49: Thr Gln Gln	6.576	375.17526	24305	Thr Gln Gln	C14 H25 N5 O7	375.1754	-0.36	C14 H25 N5 O7	0.1
Cpd 131: Fencamine	6.601	384.22733	2617	Fencamine	C20 H28 N6 O2	384.22737	-0.13	C20 H28 N6 O2	0.19
Cpd 237: Propacetamol	6.644	264.14756	973	Propacetamol	C14 H20 N2 O3	264.14739	0.63	C14 H20 N2 O3	-0.98
Cpd 48: Etazolate	6.652	289.15384	22870	Etazolate	C14 H19 N5 O2	289.15387	-0.11	C14 H19 N5 O2	-0.14
Cpd 26: Octodrine	6.72	129.15172	21494	Octodrine	C8 H19 N	129.15175	-0.23	C8 H19 N	-0.15
Cpd 18: Thr Gln Asn	6.78	361.15968	50329	Thr Gln Asn	C13 H23 N5 O7	361.15975	-0.19	C13 H23 N5 O7	0.05
Cpd 195: Ampyrone	6.814	203.10596	12469	Ampyrone	C11 H13 N3 O	203.10586	0.47	C11 H13 N3 O	0.21
Cpd 73: Aminoantipyrine	6.814	203.10596	12469	Aminoantipyrine	C11 H13 N3 O	203.10586	0.47	C11 H13 N3 O	-0.28
Cpd 64: Dinaline	6.814	227.10595	13680	Dinaline	C13 H13 N3 O	227.10586	0.39	C13 H13 N3 O	-0.22
Cpd 33: Caffeine	6.916	194.08052	39014	Caffeine	C8 H10 N4 O2	194.08038	0.72	C8 H10 N4 O2	-0.08
Cpd 188: 8-Oxodeoxycoformycin	6.993	266.10146	3801	8-Oxodeoxycoformycin	C11 H14 N4 O4	266.1015	-0.19	C11 H14 N4 O4	0.17
Cpd 114: Sulfadimidine	7.061	278.08372	4822	Sulfadimidine	C12 H14 N4 O2 S	278.08375	-0.09	C12 H14 N4 O2 S	-0.44
Cpd 99: Isoproturon	7.197	206.14213	2214	Isoproturon	C12 H18 N2 O	206.14191	1.07	C12 H18 N2 O	-1.62
Cpd 46: Urapidil	7.248	387.22726	27154	Urapidil	C20 H29 N5 O3	387.22704	0.57	C20 H29 N5 O3	0.1
Cpd 269: Sulfamethoxazole	7.325	253.05192	632	Sulfamethoxazole	C10 H11 N3 O3 S	253.05211	-0.74	C10 H11 N3 O3 S	-0.89
Cpd 23: Clomiphene	7.359	405.18583	40060	Clomiphene	C26 H28 Cl N O	405.18594	-0.28	C26 H28 Cl N O	-0.31
Cpd 167: Arg Gln Arg	7.504	458.27174	2412	Arg Gln Arg	C17 H34 N10 O5	458.27136	0.82	C17 H34 N10 O5	0.35
Cpd 205: Aceclicline	7.657	169.11028	931	Aceclicline	C9 H15 N O2	169.11028	-0.01	C9 H15 N O2	0.13
Cpd 197: Gly Ser His	7.691	299.12272	984	Gly Ser His	C11 H17 N5 O5	299.12297	-0.83	C11 H17 N5 O5	-0.06
Cpd 105: Arg Gly Gly	7.87	288.15475	5239	Arg Gly Gly	C10 H20 N6 O4	288.1546	0.51	C10 H20 N6 O4	-0.18
Cpd 31: Vasicine	8.024	188.09511	43946	Vasicine	C11 H12 N2 O	188.09496	0.77	C11 H12 N2 O	-0.04
Cpd 230: Terbucarb	8.143	277.20392	1014	Terbucarb	C17 H27 N O2	277.20418	-0.94	C17 H27 N O2	0.65
Cpd 136: Homosalate	8.151	262.15673	2600	Homosalate	C16 H22 O3	262.15689	-0.61	C16 H22 O3	0.25
Cpd 68: N-Desmethylvenlafaxine	8.177	263.18848	10809	N-Desmethylvenlafaxine	C16 H25 N O2	263.18853	-0.19	C16 H25 N O2	0.08
Cpd 220: Congocidin (Netropsin)	8.296	430.2193	1313	Congocidin (Netropsin)	C18 H26 N10 O3	430.21893	0.86	C18 H26 N10 O3	-0.24
Cpd 41: 4-Nonynoic acid	8.509	154.09927	8665	4-Nonynoic acid	C9 H14 O2	154.09938	-0.73	C9 H14 O2	0.21
Cpd 16: Metoprolol	8.662	267.18347	1369	Metoprolol	C15 H25 N O3	267.18344	0.1	C15 H25 N O3	0.11
Cpd 43: Esaprazole	8.705	225.18421	15221	Esaprazole	C12 H23 N3 O	225.18411	0.44	C12 H23 N3 O	-0.05
Cpd 185: Anabaseine	8.773	160.10001	1443	Anabaseine	C10 H12 N2	160.10005	-0.24	C10 H12 N2	-0.68
Cpd 150: His Gly Thr	8.799	313.13853	3858	His Gly Thr	C12 H19 N5 O5	313.13862	-0.3	C12 H19 N5 O5	0.24
Cpd 101: Lamotrigine	8.85	255.0077	2929	Lamotrigine	C9 H7 Cl2 N5	255.00785	-0.6	C9 H7 Cl2 N5	0.4
Cpd 137: PYCC / 1-(Pyrrolidin-1-yl)cyclohexanecarbonitrile	8.867	178.147	1436	PYCC / 1-(Pyrrolidin-1-yl)cyclohexanecarbonitrile	C11 H18 N2	178.147	0.03	C11 H18 N2	-0.58
Cpd 210: Alprenolol	9.003	249.17255	1172	Alprenolol	C15 H23 N O2	249.17288	-1.33	C15 H23 N O2	1.01
Cpd 166: 2-Aminobenzimidazole	9.224	133.06415	3011	2-Aminobenzimidazole	C7 H7 N3	133.064	1.18	C7 H7 N3	-2.66
Cpd 158: Lomifylline	9.267	278.13863	2969	Lomifylline	C13 H18 N4 O3	278.13789	2.67	C13 H18 N4 O3	-1.56
Cpd 108: Leptacline	9.293	181.18291	3262	Leptacline	C12 H23 N	181.18305	-0.77	C12 H23 N	0.5
Cpd 249: Crenatine A	9.446	568.30585	1015	Crenatine A	C34 H40 N4 O4	568.30496	1.57	C34 H40 N4 O4	0.27
Cpd 57: Gly Ala Arg	9.531	302.17014	15617	Gly Ala Arg	C11 H22 N6 O4	302.17025	-0.37	C11 H22 N6 O4	-0.14
Cpd 160: Quinprenaline	9.625	246.13637	1504	Quinprenaline	C14 H18 N2 O2	246.13683	-1.84	C14 H18 N2 O2	1.73
Cpd 196: Arg Ser Ala	9.77	332.1804	1600	Arg Ser Ala	C12 H24 N6 O5	332.18082	-1.26	C12 H24 N6 O5	0.31
Cpd 222: Acetryptin	9.778	202.11051	1261	Acetryptin	C12 H14 N2 O	202.11061	-0.53	C12 H14 N2 O	-0.52
Cpd 184: Trepibutone	10.008	310.14128	1498	Trepibutone	C16 H22 O6	310.14164	-1.15	C16 H22 O6	2.64
Cpd 204: Dihydrocoumarin	10.255	148.05227	874	Dihydrocoumarin	C9 H8 O2	148.05243	-1.09	C9 H8 O2	0.21
Cpd 113: Ascaridole	10.315	168.11488	1730	Ascaridole	C10 H16 O2	168.11503	-0.86	C10 H16 O2	0.69
Cpd 25: 2-hydroxy-10-undecenoic acid	10.323	200.14137	21616	2-hydroxy-10-undecenoic acid	C11 H20 O3	200.14124	0.61	C11 H20 O3	0.17
Cpd 90: Cycluron	10.374	198.1732	3052	Cycluron	C11 H22 N2 O	198.17321	-0.05	C11 H22 N2 O	-0.02
Cpd 66: Methfuroxam	10.749	229.11018	11426	Methfuroxam	C14 H15 N O2	229.11028	-0.42	C14 H15 N O2	1.39
Cpd 93: Albuterol	10.826	239.15218	6144	Albuterol	C13 H21 N O3	239.15214	0.16	C13 H21 N O3	0.08
Cpd 14: 9-Riburonosyladenine	11.345	281.07622	58705	9-Riburonosyladenine	C10 H11 N5 O5	281.07602	0.72	C10 H11 N5 O5	-0.43
Cpd 98: 2-tert-Butyl-4-methoxyphenol	11.882	180.1148	4148	2-tert-Butyl-4-methoxyphenol	C11 H16 O2	180.11503	-1.26	C11 H16 O2	0.54
Cpd 110: Conhydrine	12.129	143.13091	4366	Conhydrine	C8 H17 N O	143.13101	-0.69	C8 H17 N O	-0.1
Cpd 45: 13,14-dihydro-16,16-difluoro Prostaglandin D2	12.367	390.22216	42117	13,14-dihydro-16,16-difluoro Prostaglandin D2	C20 H32 F2 O5	390.22178	0.97	C20 H32 F2 O5	0.11
Cpd 132: 2E,4E,6E-Nonatrienal	12.41	136.08868	1298	2E,4E,6E-Nonatrienal	C9 H12 O	136.08882	-0.97	C9 H12 O	-0.61
Cpd 97: Cyclobutoic acid	12.419	186.12577	4749	Cyclobutoic acid	C10 H18 O3	186.12559	0.93	C10 H18 O3	0.71
Cpd 118: 4-oxo-2E-Hexenal	12.47	112.05243	4669	4-oxo-2E-Hexenal	C6 H8 O2	112.05243	0.03	C6 H8 O2	-0.3
Cpd 198: p-Methoxycinnamaldehyde	12.58	162.06787	1321	p-Methoxycinnamaldehyde	C10 H10 O2	162.06808	-1.3	C10 H10 O2	-0.42
Cpd 252: Naphthalene	12.58	128.06252	1057	Naphthalene	C10 H8	128.0626	-0.63	C10 H8	-0.15
Cpd 47: DEET (Diethyltoluamide)	12.691	191.13106	27152	DEET (Diethyltoluamide)	C12 H17 N O	191.13101	0.25	C12 H17 N O	0.2
Cpd 52: Aspirin (Acetylsalicylic acid)	12.853	180.0423	19243	Aspirin (Acetylsalicylic acid)	C9 H8 O4	180.04226	0.22	C9 H8 O4	0.01
Cpd 20: Monoisobutyl phthalic acid	12.853	222.08938	20095	Monoisobutyl phthalic acid	C12 H14 O4	222.08921	0.79	C12 H14 O4	0.97
Cpd 138: Picaridin (Bayrepel)	13.228	229.16744	2962	Picaridin (Bayrepel)	C12 H23 N O3	229.16779	-1.55	C12 H23 N O3	1.58
Cpd 232: NECA / 5'-(N-Ethylcarboxamido)adenosine	13.415	308.12343	337	NECA / 5'-(N-Ethylcarboxamido)adenosine	C12 H16 N6 O4	308.1233	0.43	C12 H16 N6 O4	-2.06
Cpd 147: Triphenylphosphine oxide	13.466	278.08607	7248	Triphenylphosphine oxide	C18 H15 O P	278.08605	0.05	C18 H15 O P	-1.32
Cpd 214: Spectinomycin	13.594	332.15862	1023	Spectinomycin	C14 H24 N2 O7	332.15835	0.82	C14 H24 N2 O7	1.73
Cpd 175: Quercetin 3,3'-dimethyl ether 4'-(2-methylbutyrate)	13.671	414.13228	1348	Quercetin 3,3'-dimethyl ether 4'-(2-methylbutyrate)	C22 H22 O8	414.13147	1.95	C22 H22 O8	0.3
Cpd 85: Cyclandelate	13.679	276.17244	6069	Cyclandelate	C17 H24 O3	276.17254	-0.37	C17 H24 O3	-2.32
Cpd 262: Clebopride	13.688	373.15624	409	Clebopride	C20 H24 Cl N3 O2	373.1557	1.43	C20 H24 Cl N3 O2	-1.44
Cpd 38: Embelin	13.764	294.18254	9051	Embelin	C17 H26 O4	294.18311	-1.93	C17 H26 O4	0.54
Cpd 58: Torachrysone	13.798	246.08913	9027	Torachrysone	C14 H14 O4	246.08921	-0.31	C14 H14 O4	0.27
Cpd 267: Procinonide	13.977	508.22718	620	Procinonide	C27 H34 F2 O7	508.22726	-0.17	C27 H34 F2 O7	-0.34
Cpd 127: Precoxene II	14.667	220.11005	2686	Precoxene II	C13 H16 O3	220.10994	0.5	C13 H16 O3	-0.7
Cpd 28: His Lys Met	14.693	414.20389	19022	His Lys Met	C17 H30 N6 O4 S	414.20492	-2.5	C17 H30 N6 O4 S	0.03
Cpd 211: Benzylhydroxybenzoate	14.786	228.07865	1012	Benzylhydroxybenzoate	C14 H12 O3	228.07864	0.02	C14 H12 O3	0.22
Cpd 36: Xanthinol Nicotinate	14.795	434.19119	37453	Xanthinol Nicotinate	C19 H26 N6 O6	434.19138	-0.44	C19 H26 N6 O6	0.02
Cpd 227: Nifedipine	15.136	346.11705	1046	Nifedipine	C17 H18 N2 O6	346.11649	1.64	C17 H18 N2 O6	1.87
Cpd 111: Cyclandelate	15.221	276.1727	6588	Cyclandelate	C17 H24 O3	276.17254	0.56	C17 H24 O3	-2.53
Cpd 153: Triphenyl phosphate	15.408	326.07035	2806	Triphenyl phosphate	C18 H15 O4 P	326.0708	-1.36	C18 H15 O4 P	-0.47
Cpd 207: Dinocton	15.885	354.14327	1624	Dinocton	C16 H22 N2 O7	354.1427	1.61	C16 H22 N2 O7	-5
Cpd 77: Tributylphosphate	16.013	266.16479	43448	Tributylphosphate	C12 H27 O4 P	266.1647	0.37	C12 H27 O4 P	-0.36
Cpd 12: Caffeic acid	16.021	180.04231	56429	Caffeic acid	C9 H8 O4	180.04226	0.26	C9 H8 O4	-0.59
Cpd 72: Aspidodasycarpine	16.047	370.18858	11098	Aspidodasycarpine	C21 H26 N2 O4	370.18926	-1.84	C21 H26 N2 O4	0.33
Cpd 200: 6β-Hydroxyprednisolone	16.107	376.18804	1195	6β-Hydroxyprednisolone	C21 H28 O6	376.18859	-1.46	C21 H28 O6	1.49
Cpd 69: 3,5-Diprenyl-4-Hydroxyacetophenone	16.294	272.17721	7359	3,5-Diprenyl-4-Hydroxyacetophenone	C18 H24 O2	272.17763	-1.55	C18 H24 O2	0.34
Cpd 246: Simmondsin-2'-ferulate	16.328	551.19947	771	Simmondsin-2'-ferulate	C26 H33 N O12	551.20028	-1.47	C26 H33 N O12	0.24
Cpd 155: Butibufen	16.354	220.14626	16588	Butibufen	C14 H20 O2	220.14633	-0.33	C14 H20 O2	-0.26
Cpd 209: Met-Asn-OH	16.413	371.07672	1437	Met-Asn-OH	C14 H17 N3 O7 S	371.07872	-5.4	C14 H17 N3 O7 S	-1.04
Cpd 171: Amicetin	16.524	618.30149	1473	Amicetin	C29 H42 N6 O9	618.30133	0.27	C29 H42 N6 O9	0.01
Cpd 174: Dipyridamole	17.342	504.31802	884	Dipyridamole	C24 H40 N8 O4	504.31725	1.53	C24 H40 N8 O4	2.73
Cpd 225: Milbemectin A3	17.35	528.30815	653	Milbemectin A3	C31 H44 O7	528.3087	-1.05	C31 H44 O7	-0.1
Cpd 208: Thr Trp Met	17.469	436.17847	989	Thr Trp Met	C20 H28 N4 O5 S	436.17804	0.98	C20 H28 N4 O5 S	-0.84
Cpd 164: Xibornol	17.529	258.19813	2462	Xibornol	C18 H26 O	258.19837	-0.89	C18 H26 O	0.25
Cpd 82: Palmitic amide	17.555	255.25634	8212	Palmitic amide	C16 H33 N O	255.25621	0.5	C16 H33 N O	-0.56
Cpd 270: 2-Formyl-1-indanone	17.589	160.05223	717	2-Formyl-1-indanone	C10 H8 O2	160.05243	-1.24	C10 H8 O2	-0.82
Cpd 256: Aspoxicillin	17.963	493.16354	772	Aspoxicillin	C21 H27 N5 O7 S	493.16312	0.85	C21 H27 N5 O7 S	2.56
Cpd 100: Gentamicin A	18.279	468.24328	4372	Gentamicin A	C18 H36 N4 O10	468.24314	0.3	C18 H36 N4 O10	0.25
Cpd 172: 3'-Deoxystreptomycin 6,3''-bis-phosphate	18.474	725.2024	1633	3'-Deoxystreptomycin 6,3''-bis-phosphate	C21 H41 N7 O17 P2	725.20342	-1.41	C21 H41 N7 O17 P2	3.04

Suspect screening - JDS 58 phase II -All Ions



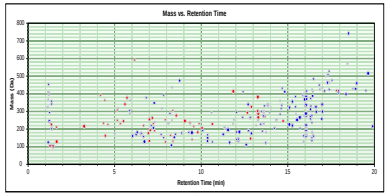


Data File	ALLMS_JDS_58_.d	**Sample Name**	ALLMS_JDS_58
Sample Type	Sample	**Position**	P1-C4
Instrument Name	QTOF1	**User Name**	
Acq Method	All_Ions_MS_positive.m	**Acquired Time**	9/30/2013 7:50:45 PM
IRM Calibration Status	Success	**DA Method**	MFE_FINAL.m
Comment			

Compound Table

Label	Tgt Name	Tgt Score	Mass Error (ppm)	MFG Formula	Tgt Formula	Obs. RT	Ref. Mass	Obs. Mass
Cpd 383: Methiuron	Methiuron	65.04	0.4	C10 H14 N2 S	C10 H14 N2 S	0.992	194.08777	194.08785
Cpd 1293: DCPA / Chlorthal-dimethyl (Dacthal)	DCPA / Chlorthal-dimethyl (Dacthal)	47.84	-2.97	C10 H6 Cl4 O4	C10 H6 Cl4 O4	1.057	329.90202	329.90104
Cpd 1054: Cloracetadol	Cloracetadol	51.17	-6.62	C10 H10 Cl3 N O3	C10 H10 Cl3 N O3	1.07	296.97263	296.97066
Cpd 794: Mesyl(methyl)carbamoylmethylaminomethylphosphonic acid	Mesyl(methyl)carbamoylmethylaminomethylphosphonic acid	60.57	1.72	C5 H13 N2 O6 P S	C5 H13 N2 O6 P S	1.083	260.02319	260.02364
Cpd 795: Chlorflurenol	Chlorflurenol	59.08	-2.76	C14 H9 Cl O3	C14 H9 Cl O3	1.096	260.02402	260.0233
Cpd 697: Chlorphenesincarbamate	Chlorphenesincarbamate	62.45	-3.86	C10 H12 Cl N O4	C10 H12 Cl N O4	1.109	245.04549	245.04454
Cpd 1254: Benodanil	Benodanil	59.19	1.45	C13 H10 I N O	C13 H10 I N O	1.448	322.98071	322.98118
Cpd 1576: Fluthiacet	Fluthiacet	60.95	-1.1	C14 H13 Cl F N3 O3 S2	C14 H13 Cl F N3 O3 S2	1.461	389.00709	389.00666
Cpd 1709: Cefazaflur	Cefazaflur	78.18	-0.26	C13 H13 F3 N6 O4 S3	C13 H13 F3 N6 O4 S3	1.474	470.01125	470.01113
Cpd 1119: 2-Iodohippuric acid	2-Iodohippuric acid	72.09	1.59	C9 H8 I N O3	C9 H8 I N O3	1.578	304.95489	304.95537
Cpd 1082: Erbitryl tetranitrate	Erbitryl tetranitrate	75.49	-1.01	C4 H6 N4 O12	C4 H6 N4 O12	1.591	301.99822	301.99792
Cpd 220: Linsidomine	Linsidomine	79.68	-6.3	C6 H11 N4 O2	C6 H11 N4 O2	1.604	171.0882	171.08712
Cpd 1123: Sulprofos oxon	Sulprofos oxon	83.85	2.32	C12 H19 O3 P S2	C12 H19 O3 P S2	1.617	306.05132	306.05203
Cpd 22: Taurine	Taurine	47.61	-0.26	C2 H7 N O3 S	C2 H7 N O3 S	1.63	125.01466	125.01463
Cpd 1285: Flurodifen	Flurodifen	81.38	2.81	C13 H7 F3 N2 O5	C13 H7 F3 N2 O5	1.643	328.03071	328.03163
Cpd 490: Nithiazid	Nithiazid	46.03	0.72	C6 H8 N4 O3 S	C6 H8 N4 O3 S	1.656	216.03171	216.03187
Cpd 204: Zoxazolamin	Zoxazolamin	47.45	1.07	C7 H5 Cl N2 O	C7 H5 Cl N2 O	1.669	168.00904	168.00922
Cpd 160: Acesulfame (Acesulfame-K)	Acesulfame (Acesulfame-K)	71.98	3.49	C4 H5 N O4 S	C4 H5 N O4 S	1.682	162.99393	162.9945
Cpd 30: Leucine	Leucine	47.62	-0.12	C6 H13 N O2	C6 H13 N O2	1.695	131.09463	131.09461
Cpd 211: Chlorzoxazone	Chlorzoxazone	54.92	5.65	C7 H4 Cl N O2	C7 H4 Cl N O2	1.695	168.99306	168.99401
Cpd 793: Penta erythrit-diphosphate	Penta erythrit-diphosphate	54.88	-2.79	C5 H10 O8 P2	C5 H10 O8 P2	1.708	259.98509	259.98437
Cpd 59: Methyl nicotinate	Methyl nicotinate	75.67	5.02	C7 H7 N O2	C7 H7 N O2	2.06	137.04768	137.04837
Cpd 194: Oxfenicine	Oxfenicine	79.91	-1.99	C8 H9 N O3	C8 H9 N O3	2.073	167.05824	167.05791
Cpd 1: Guanidine	Guanidine	47.31	3.18	C H5 N3	C H5 N3	2.281	59.04835	59.04854
Cpd 480: Ethylene, 1-(p-chlorophenyl)-1-phenyl- (Chlorphenoxamine artifact)	Ethylene, 1-(p-chlorophenyl)-1-phenyl- (Chlorphenoxamine artifact)	52.19	-5.81	C14 H11 Cl	C14 H11 Cl	3.505	214.05493	214.05368
Cpd 64: Deferiprone	Deferiprone	71.88	-2.84	C7 H9 N O2	C7 H9 N O2	3.74	139.06333	139.06293
Cpd 784: Sulnidazole	Sulnidazole	73.5	3.54	C9 H14 N4 O3 S	C9 H14 N4 O3 S	4.391	258.07866	258.07957
Cpd 555: Pirimicarb, desmethyl-	Pirimicarb, desmethyl-	86.9	-5.26	C10 H16 N4 O2	C10 H16 N4 O2	4.443	224.12733	224.12615
Cpd 519: Phenamazide	Phenamazide	85.58	-0.09	C13 H19 N O2	C13 H19 N O2	4.521	221.14158	221.14156
Cpd 1520: Lufuradom	Lufuradom	76.43	-5.12	C22 H20 F N3 O2	C22 H20 F N3 O2	4.691	377.15396	377.15202
Cpd 1439: Sulfaclorazole	Sulfaclorazole	56.17	3.01	C16 H15 Cl N4 O2 S	C16 H15 Cl N4 O2 S	4.704	362.06042	362.06151
Cpd 253: Glucosamine	Glucosamine	80.89	2.09	C6 H13 N O5	C6 H13 N O5	4.86	179.07937	179.07975
Cpd 872: Pramiracetam	Pramiracetam	87.33	0.29	C14 H27 N3 O2	C14 H27 N3 O2	4.873	269.21033	269.21041
Cpd 422: Tretamine	Tretamine	41.93	6.29	C9 H12 N6	C9 H12 N6	4.938	204.11234	204.11363
Cpd 141: Buformine	Buformine	47.59	-0.43	C6 H15 N5	C6 H15 N5	4.977	157.13275	157.13268
Cpd 1020: Enprazepine	Enprazepine	59.91	2.42	C20 H24 N2	C20 H24 N2	5.185	292.19395	292.19466
Cpd 1288: Tiapride	Tiapride	61.98	-1.34	C15 H24 N2 O4 S	C15 H24 N2 O4 S	5.589	328.14568	328.14524
Cpd 951: Cadralazine	Cadralazine	74.54	-3.71	C12 H21 N5 O3	C12 H21 N5 O3	5.667	283.16444	283.16339
Cpd 1657: Difenoxine	Difenoxine	71.71	-0.02	C28 H28 N2 O2	C28 H28 N2 O2	5.68	424.21508	424.21507
Cpd 1573: Cannogenin	Cannogenin	47.57	-0.34	C23 H32 O5	C23 H32 O5	5.693	388.22497	388.22484
Cpd 659: Tebatizole	Tebatizole	84.45	-2.48	C12 H21 N3 S	C12 H21 N3 S	5.824	239.14562	239.14503
Cpd 552: Cerulenin	Cerulenin	42.41	-5.11	C12 H17 N O3	C12 H17 N O3	5.837	223.12084	223.1197
Cpd 183: Ephedrine	Ephedrine	86.95	-0.42	C10 H15 N O	C10 H15 N O	5.863	165.11536	165.11529
Cpd 78: 1,2-Dimethyl-3-phenylaziridine	1,2-Dimethyl-3-phenylaziridine	80.4	-1.35	C10 H13 N	C10 H13 N	5.876	147.1048	147.1046
Cpd 1604: Morocromen	Morocromen	66.25	-0.58	C21 H27 N3 O5	C21 H27 N3 O5	6.032	401.19507	401.19484
Cpd 235: Cotinine	Cotinine	95.78	0.65	C10 H12 N2 O	C10 H12 N2 O	6.253	176.09496	176.09508
Cpd 622: Levofenfluramine	Levofenfluramine	82.11	1.55	C12 H16 F3 N	C12 H16 F3 N	6.566	231.12348	231.12384
Cpd 452: Ocrilate	Ocrilate	74.87	-0.6	C12 H19 N O2	C12 H19 N O2	6.579	209.14158	209.14145
Cpd 1240: Triciribine	Triciribine	83.69	0.83	C13 H16 N6 O4	C13 H16 N6 O4	6.592	320.1233	320.12357
Cpd 972: Lethane 60	Lethane 60	47.59	-5.29	C15 H27 N O2 S	C15 H27 N O2 S	6.605	285.17625	285.17474
Cpd 414: Ampyrone	Ampyrone	97.78	0.64	C11 H13 N3 O	C11 H13 N3 O	6.813	203.10586	203.10599
Cpd 1473: Amisulpride	Amisulpride	70.53	-1.84	C17 H27 N3 O4 S	C17 H27 N3 O4 S	6.84	369.17223	369.17155
Cpd 381: Caffeine	Caffeine	95.1	0.76	C8 H10 N4 O2	C8 H10 N4 O2	6.918	194.08038	194.08052
Cpd 1623: Alfaprostol	Alfaprostol	79.59	-1.42	C24 H38 O5	C24 H38 O5	7.23	406.27192	406.27135
Cpd 1474: Anidoxime	Anidoxime	46.82	-0.44	C21 H27 N3 O3	C21 H27 N3 O3	7.243	369.20524	369.20508
Cpd 1388: Dipipanone	Dipipanone	83.42	0.57	C24 H31 N O	C24 H31 N O	7.803	349.24056	349.24076
Cpd 329: Phenazone	Phenazone	92.63	1.5	C11 H12 N2 O	C11 H12 N2 O	8.025	188.09496	188.09524
Cpd 818: Tramadol	Tramadol	94.64	-0.48	C16 H25 N O2	C16 H25 N O2	8.168	263.18853	263.1884
Cpd 438: Somantadine	Somantadine	66.62	-7.72	C14 H25 N	C14 H25 N	8.507	207.1987	207.1971
Cpd 853: Metoprolol	Metoprolol	87.37	0.52	C15 H25 N O3	C15 H25 N O3	8.663	267.18344	267.18358
Cpd 640: Lidocaine	Lidocaine	76.87	-2.61	C14 H22 N2 O	C14 H22 N2 O	8.676	234.17321	234.1726
Cpd 1242: Cocaethylene-d3	Cocaethylene-d3	73.37	3.41	C18 H20 D3 N O4	C18 H20 D3 N O4	9.08	320.18154	320.18263
Cpd 396: Atrazine-2-hydroxy (Hydroxyatrazine)	Atrazine-2-hydroxy (Hydroxyatrazine)	77.48	-1.4	C8 H15 N5 O	C8 H15 N5 O	9.275	197.12766	197.12738
Cpd 1649: Pirepolol	Pirepolol	61.43	-1.58	C21 H32 N4 O5	C21 H32 N4 O5	9.431	420.23727	420.23661
Cpd 340: Dihydralazin	Dihydralazin	78.23	5.6	C8 H10 N6	C8 H10 N6	10.317	190.09669	190.09776
Cpd 404: Cycluron	Cycluron	78.49	-0.18	C11 H22 N2 O	C11 H22 N2 O	10.395	198.17321	198.17318
Cpd 660: Isoetarine	Isoetarine	98.64	-1.79	C13 H21 N O3	C13 H21 N O3	10.825	239.15214	239.15172
Cpd 762: Imuracetam	Imuracetam	87.73	4.47	C11 H18 N4 O3	C11 H18 N4 O3	11.346	254.13789	254.13903
Cpd 648: Carbamazepine	Carbamazepine	86.77	2.31	C15 H12 N2 O	C15 H12 N2 O	11.841	236.09496	236.09551
Cpd 1150: Betaxolol	Betaxolol	72.66	-5.67	C18 H29 N O3	C18 H29 N O3	12.427	307.21474	307.213
Cpd 349: DEET (Diethyltoluamide)	DEET (Diethyltoluamide)	92.67	0.38	C12 H17 N O	C12 H17 N O	12.687	191.13101	191.13109
Cpd 686: Cotoin	Cotoin	84.18	2.26	C14 H12 O4	C14 H12 O4	12.857	244.07356	244.07411
Cpd 606: Picaridin (Bayrepel)	Picaridin (Bayrepel)	86.83	0.94	C12 H23 N O3	C12 H23 N O3	13.234	229.16779	229.16801
Cpd 1487: Sulverapride	Sulverapride	71.49	2.29	C16 H25 N3 O5 S	C16 H25 N3 O5 S	13.586	371.15149	371.15234
Cpd 1365: Allethrin II	Allethrin II	72.45	1.99	C20 H26 O5	C20 H26 O5	13.599	346.17802	346.17871
Cpd 1611: Alpidem	Alpidem	57.79	2.08	C21 H23 Cl2 N3 O	C21 H23 Cl2 N3 O	13.677	403.12182	403.12266
Cpd 1431: Rosmarinic acid	Rosmarinic acid	75.66	-3.64	C18 H16 O8	C18 H16 O8	14.511	360.08452	360.08321
Cpd 1643: Estriol triacetate	Estriol triacetate	90.1	-1.13	C24 H30 O6	C24 H30 O6	14.693	414.20424	414.20377
Cpd 591: Oxybenzone	Oxybenzone	67.02	-1.2	C14 H12 O3	C14 H12 O3	14.784	228.07864	228.07837
Cpd 1036: Nonivamide	Nonivamide	62.12	3.28	C17 H27 N O3	C17 H27 N O3	15.214	293.19909	293.20006
Cpd 1378: Imidocarb	Imidocarb	57.18	-2.91	C19 H20 N6 O	C19 H20 N6 O	15.93	348.16986	348.16885
Cpd 765: Prohydrojasmon	Prohydrojasmon	90.4	-2.17	C15 H26 O3	C15 H26 O3	15.943	254.18819	254.18764
Cpd 695: Isoaminile	Isoaminile	70.49	-1.42	C16 H24 N2	C16 H24 N2	16.009	244.19395	244.1936
Cpd 302: Hydroxysimazine	Hydroxysimazine	83.15	-4.85	C7 H13 N5 O	C7 H13 N5 O	16.022	183.11201	183.11112
Cpd 1046: Nifurpipone	Nifurpipone	55.75	2.01	C12 H17 N5 O4	C12 H17 N5 O4	16.061	295.12805	295.12865
Cpd 274: Aspirin (Acetylsalicylic acid)	Aspirin (Acetylsalicylic acid)	90.32	6.07	C9 H8 O4	C9 H8 O4	16.074	180.04226	180.04335
Cpd 769: Dimethametryn	Dimethametryn	72.71	-4.53	C11 H21 N5 S	C11 H21 N5 S	16.282	255.15177	255.15061
Cpd 1744: Nonoxinol 9	Nonoxinol 9	82.16	-0.76	C33 H60 O10	C33 H60 O10	17.311	616.41865	616.41818
Cpd 1749: Midecamycin	Midecamycin	74.47	0.15	C41 H67 N O15	C41 H67 N O15	17.337	813.45107	813.45119
Cpd 943: Dodemorph	Dodemorph	98.56	-0.32	C18 H35 N O	C18 H35 N O	17.975	281.27186	281.27177

Unknown screening - JDS 58



Data File	MS_JDS_58_.d	**Sample Name**	MS_JDS_58
Sample Type	Sample	**Position**	P1-C4
Instrument Name	QTOF1	**User Name**	
Acq Method	MS_only_pozitive.m	**Acquired Time**	9/30/2013 7:43:17 PM
IRM Calibration Status	Success	**DA Method**	MFE_FINAL.m
Comment			

Sample Group		**Info.**	
Funnel RF HP	150	**Funnel RF LP**	60
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Digital Sample Banking

Web search

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula	DB Diff (ppm)
Cpd 165: C10 H3 N3 O3 S5	1.065	372.87421	1016	C10 H3 N3 O3 S5	372.87779	-9.61	C10 H3 N3 O3 S5	C10 H3 N3 O3 S5	3.73
Cpd 237: C10 H22 N2 O4 S2	2.078	298.10161	872	C10 H22 N2 O4 S2	298.1021	-1.65	C10 H22 N2 O4 S2	C10 H22 N2 O4 S2	0.32
Cpd 256: C12 H23 N O3	3.799	229.16788	835	C12 H23 N O3	229.16779	0.37	C12 H23 N O3	C12 H23 N O3	0.09
Cpd 260: C12 H18 N2 O9	5.196	334.10063	806	C12 H18 N2 O9	334.10123	-1.78	C12 H18 N2 O9	C12 H18 N2 O9	1.09
Cpd 119: C17 H18 N10 O	6.107	378.16677	3456	C17 H18 N10 O	378.16651	0.71	C17 H18 N10 O	C17 H18 N10 O	-0.2
Cpd 166: C7 H7 N3	9.224	133.06415	3011	C7 H7 N3	133.064	1.18	C7 H7 N3	C7 H7 N3	-2.66
Cpd 25: C9 H14 N6 O	10.323	222.12222	20444	C9 H14 N6 O	222.12291	-3.1	C9 H14 N6 O	C9 H14 N6 O	4.86
Cpd 14: C10 H14 N6 O5	11.345	298.10278	58705	C10 H14 N6 O5	298.10257	0.7	C10 H14 N6 O5	C10 H14 N6 O5	-0.26
Cpd 230: C10 H10 N2 O4	13.143	222.06337	866	C10 H10 N2 O4	222.06406	-3.09	C10 H10 N2 O4	C10 H10 N2 O4	-0.77
Cpd 138: C12 H23 N O3	13.228	229.16744	2962	C12 H23 N O3	229.16779	-1.55	C12 H23 N O3	C12 H23 N O3	1.58
Cpd 252: C8 H14 N6 O5 S2	13.577	338.04614	794	C8 H14 N6 O5 S2	338.04671	-1.68	C8 H14 N6 O5 S2	C8 H14 N6 O5 S2	1.36
Cpd 168: 17.342	17.342	874.50391	1137		874.5038	0.13			-0.13
Cpd 268: C20 H16 N4 O7	17.35	424.10065	558	C20 H16 N4 O7	424.1019	-2.95	C20 H16 N4 O7	C20 H16 N4 O7	0.6
Cpd 226: C16 H40 N12 O8	17.35	528.30818	788	C16 H40 N12 O8	528.30921	-1.94	C16 H40 N12 O8	C16 H40 N12 O8	-0.16

Screening with GC-MS

Peter Oswald
Environmental Institute, Kos, Slovakia

GC-MS screening of water samples

Sample preparation:

1000 ml raw water sample

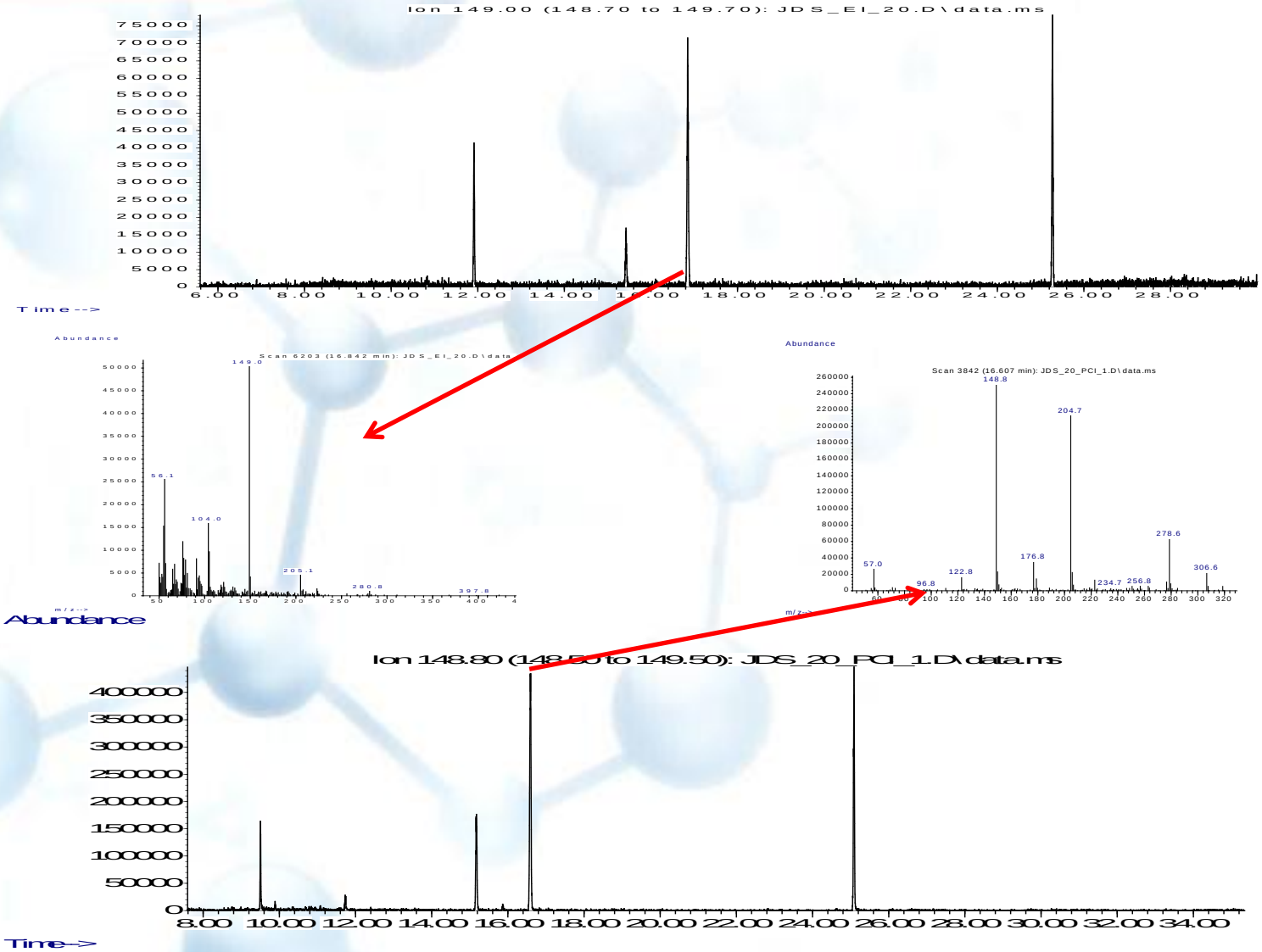
Spiked with deuterated standards D-10 Phenanthrene and D8-DDT at 0,01 ug/L

- LLE
  - Sample volume: 1000 ml
  - Extration solvent: 2 x 30ml hexane
  - Final extract volume: 1 ml

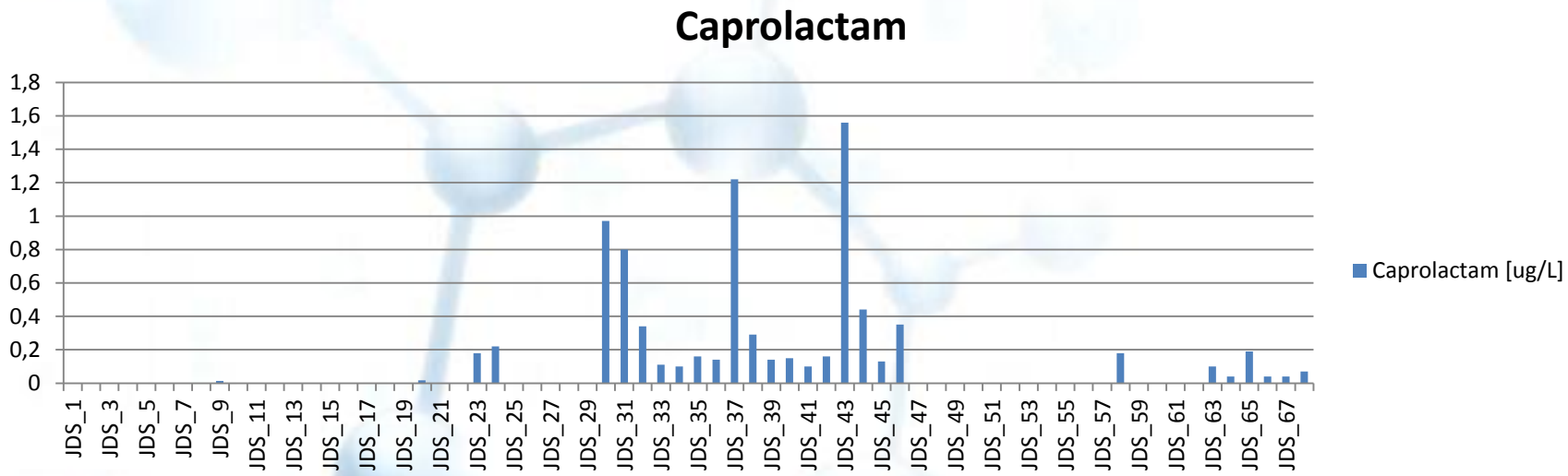
- GC-MS
  - Injection volume 10 ul
  - EI, PCI and NCI
  - SCAN mode (m/z50-800)
  - Wiley, NIST, manual interpretation

- Outputs
  - DCT- storage of analytical information and results

EI vs. PCI - Dibutyl phthalate , MW 278



Caprolactam distribution over JDS sampling sites



European demonstration Programme

FP7 EDA EMERGE Project -

Preliminary results

Sampling campaign - August 2013

Large volume solid phase extraction (50L) with in-situ fractionation

Danube RB - 3 sites

Results of bioassays

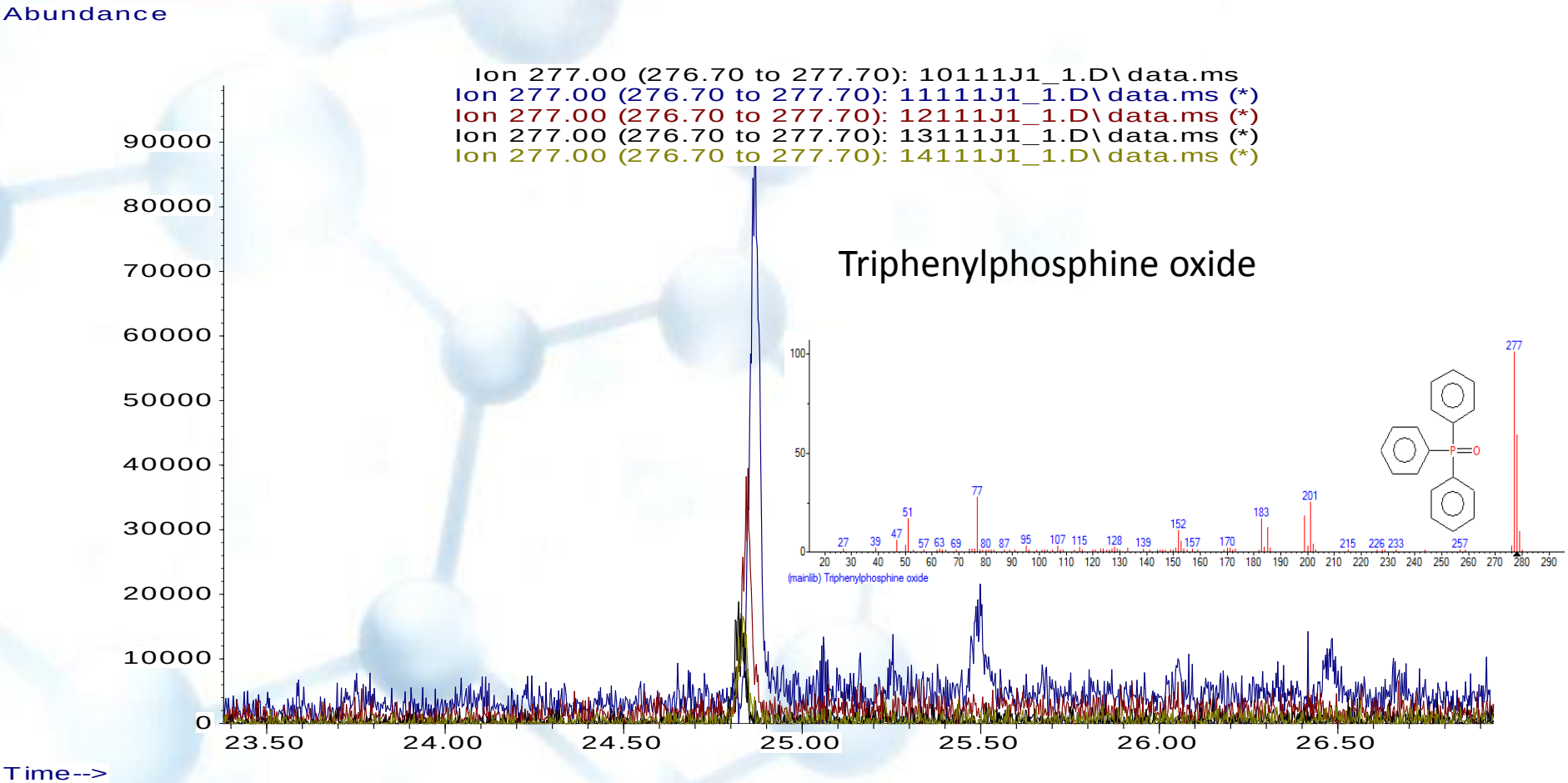
Code	Fraction	Sampling location	**Algae**		**Ames**	ER	antiER	AR	antiAR	GR	AchE inh.
			inhibition	stimulation							
10111C1	HR-X	laboratory blank	MODERATE	NO	250 (LOEC)	n.d.	u.i	n.d.	Positive	n.d.	n.d.
10112C1	HR-XAW	laboratory blank	MEDIUM	NO	n.d.	n.d.	u.i	n.d.	n.d.	n.d.	n.d.
10113C1	HR-XCW	laboratory blank	NO	MODERATE	n.d.	n.d.	u.i	n.d.	n.d.	n.d.	n.d.
12111C1	HR-X	Morava-confluence to Danube	MEDIUM	MODERATE	n.d.	n.d.	u.i	n.d.	Positive	n.d.	8.38
12112C1	HR-XAW	Morava-confluence to Danube	MODERATE	NO	n.d.	n.d.	u.i	n.d.	n.d.	n.d.	n.d.
13111C1	HR-X	Danube-Bratislava innercity	MODERATE	NO	n.d.	< LOQ	u.i	n.d.	n.d.	n.d.	n.d.
13112C1	HR-XAW	Danube-Bratislava innercity	MODERATE	NO	n.d.	< LOQ	u.i	n.d.	n.d.	n.d.	n.d.
13113C1	HR-XCW	Danube-Bratislava innercity	MODERATE	MEDIUM	n.d.	n.d.	u.i	n.d.	n.d.	n.d.	62.82
14111C1	HR-X	Danube-Szob	MODERATE	MODERATE	n.d.	72	u.i	n.d.	Positive	n.d.	n.d.
14112C1	HR-XAW	Danube-Szob	MODERATE	SEVERE	n.d.	38	u.i	n.d.	n.d.	n.d.	n.d.
14113C1	HR-XCW	Danube-Szob	SEVERE	MODERATE	n.d.	n.d.	u.i	n.d.	n.d.	n.d.	-11.38

- Sample from Morava confluence most toxic to algae, antiandrogenicity, AchE inhibition
- Sample from Szob – estrogenicity and antiandrogenicity, stimulation of algal growth

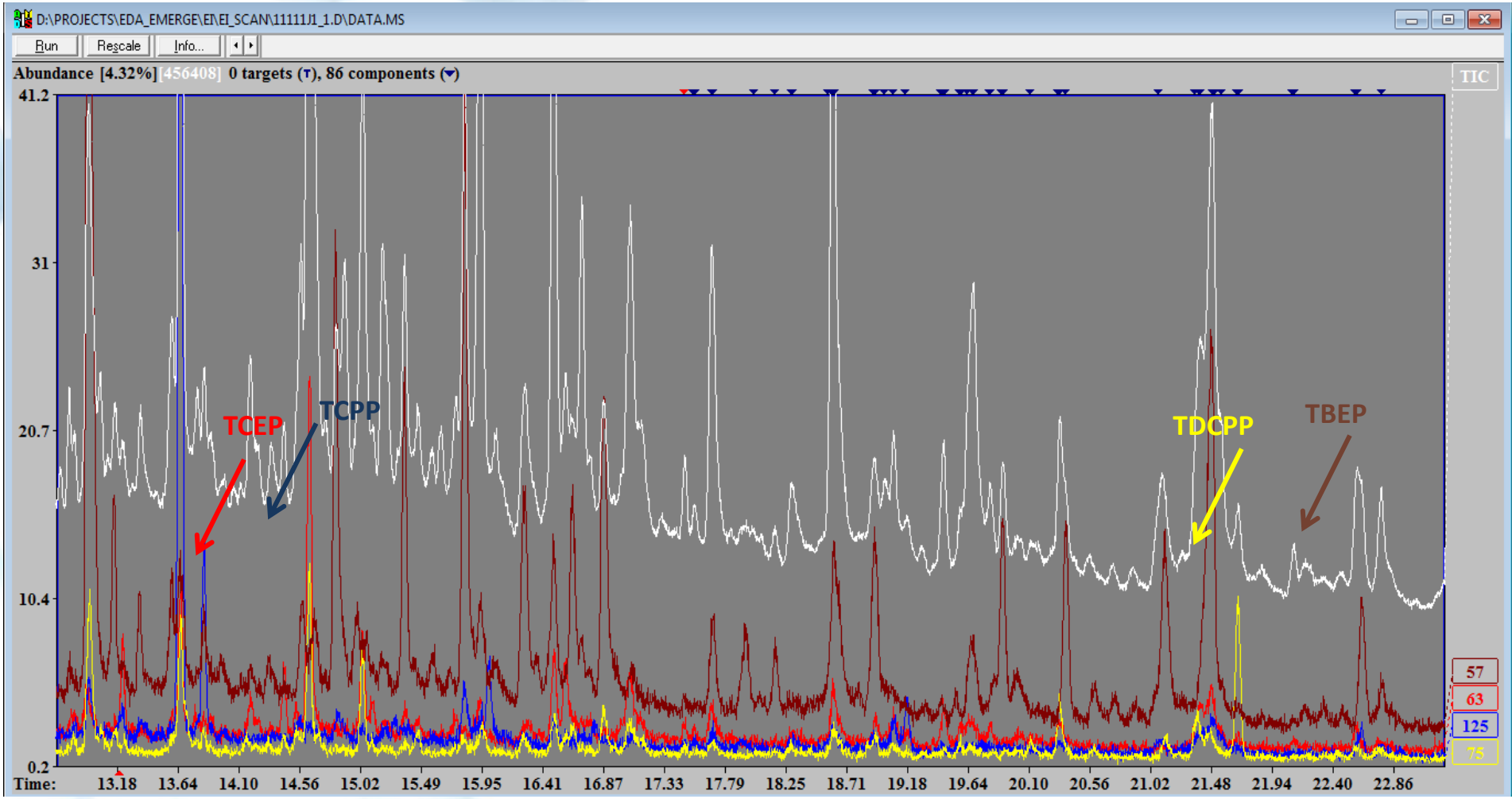
European Demonstration Programme

Sampling site	Screening	Target	Bioassay
Svratka-WWTP effluent	44 (3)	6 (1)	Severe
Morava-confluence to Danube	40 (3)	3	Medium
Danube - Bratislava	17 (1)	1	Moderate
Danube - Szob	15 (1)	2	Moderate

Retrospective analysis of full scan GC-MS chromatograms HR-X: Triphenylphosphine oxide (m/z 277) found in samples 10111 (5 ng/l), 11111 (24.8 ng/l), 12111 (9.4 ng/l), 13111 (3.4 ng/l), 14111 (4.3 ng/l)



Retrospective analysis of full scan GC-MS chromatogram HR-X Organophosphorus flame retardants(TCEP – 50.8 ng/l, TCPP – 1081.8 ng/l, TDCPP – 120.2 ng/l, TBEP – 152.9 ng/l) found in sample 11111



Full scan GC-MS chromatograms of HR-X 11111: **comparison of target and non-target analyses** (occurrence/concentrations in ng/l)

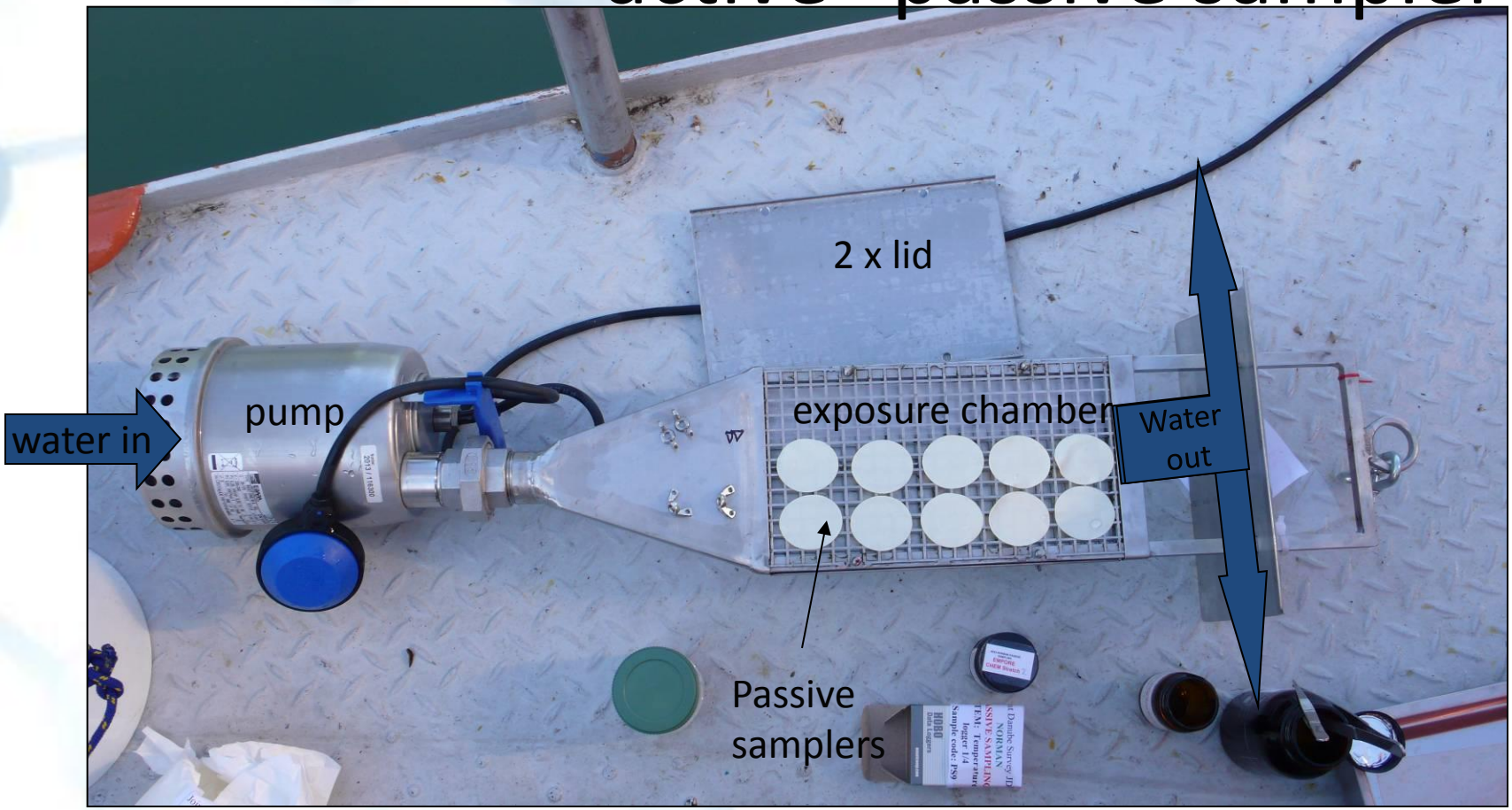
EI_11111J1	Non-target	Target UFZ	Target eawag	Target IRB
5-Methyl-Benzotriazol	1612.5		>200	
Diethyltoluamide (DEET)	324.1	397.6	>200	
Atrazine	27.6	23.7		
Tris(2-chloroethyl) phosphate - TCEP	50.8			
Caffeine	93.7	n.d	n.d.	
Triclosan	133.0	5		
Bisphenol A	74.0			64
Diclofenac	410.6		410	
Carbamazepine	707.0	629.9	>200	
Tri(butoxyethyl)_phosphate	152.9	147.1		
Triphenylphosphine oxide	24.8	49.4		
Verapamil	9.4		31	

Passive sampling in the JDS3

- **"Active" passive sampler system** on board of the expedition ship + collection of **sediment/SPM** at highly relevant sites
- temporally- and **spatially- integrative sampling approach** - defined stretches
- passive samplers for **hydrophobic and for polar compounds**

- **Target analysis** for priority substances + relevant river basin specific compounds
- **Non-target screening** analysis (GC-TOF-MS; LC-HR-MS(MS))
- **Toxicity profiling** – set of bioassays harmonised with EDA EMERGE project

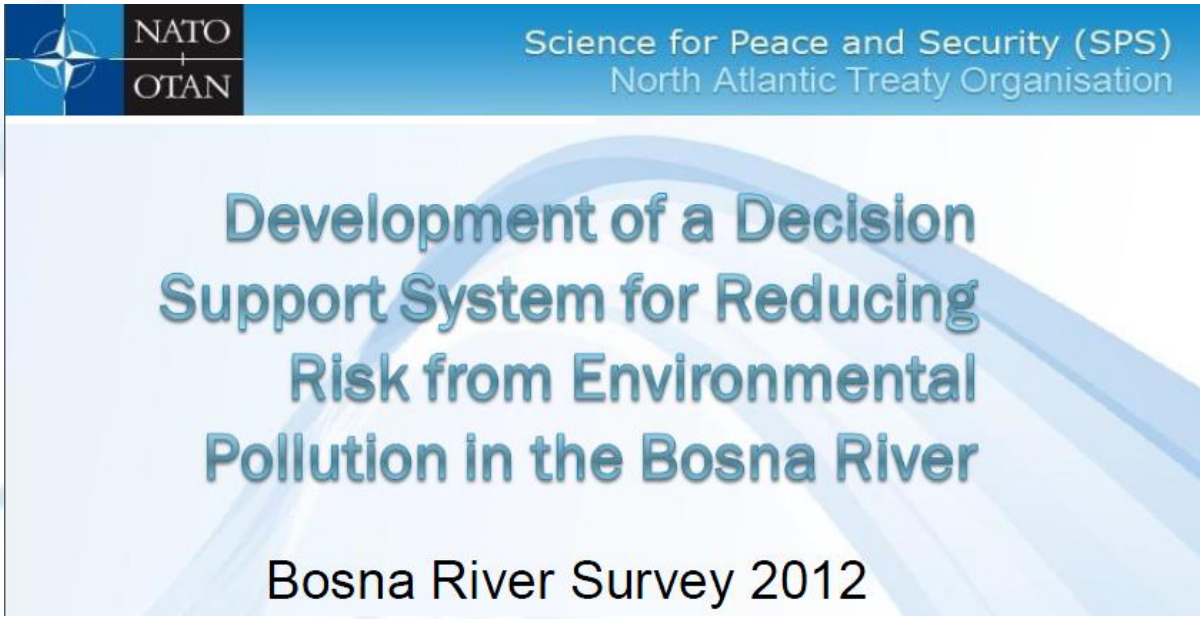
Design of a prototype "active" passive sampler



Passive sampling on board

Analysis in water column

		Type of analysis			
Compound class	**Passive sampler**	**Target analysis**	**Non-target screening**	**Toxicity profiling**	**Digital sample banking**
Nonpolar compounds log Kow >3	Silicone rubber sheet (SR) Low density polyethylene sheet (LDPE)	RECETOX/ GC/MS NIVA / GC/MS	NIVA / GC/TOF	RECETOX/EI EDA EMERGE bioassay battery	Later use in FP7 SOLUTIONS
Polar compounds log Kow < 3	SDB-RPS Empore discs	RECETOX/ Waters XEVO TQ-S RECETOX/ AB SCIEX 5500 Q Trap	RECETOX/ Agilent 6550 QTOF Croatian Waters/JDS3 methodology	RECETOX/EI bioassay battery	Later use in FP7 SOLUTIONS
Result		Absolute or relative concentrations of target compounds in the dissolved phase	List of compounds subject to prioritisation for identification of RBSP	measure of the response and effect pattern of bioavailable and bioaccumulative pollutants in water	



Aims:

- To provide screening of urban and industrial waste water streams for identification of major polluters and discharged substances

➔ **Prioritisation of most relevant "Bosna RBSPs" and establishment of emission limit values for major polluters**

Adaptation to the project needs

Prioritisation of polluters and relevant substances

- Use of wastewater samples from the 11 polluters
  ➔ **Ranking of sites based on the ranking of substances**

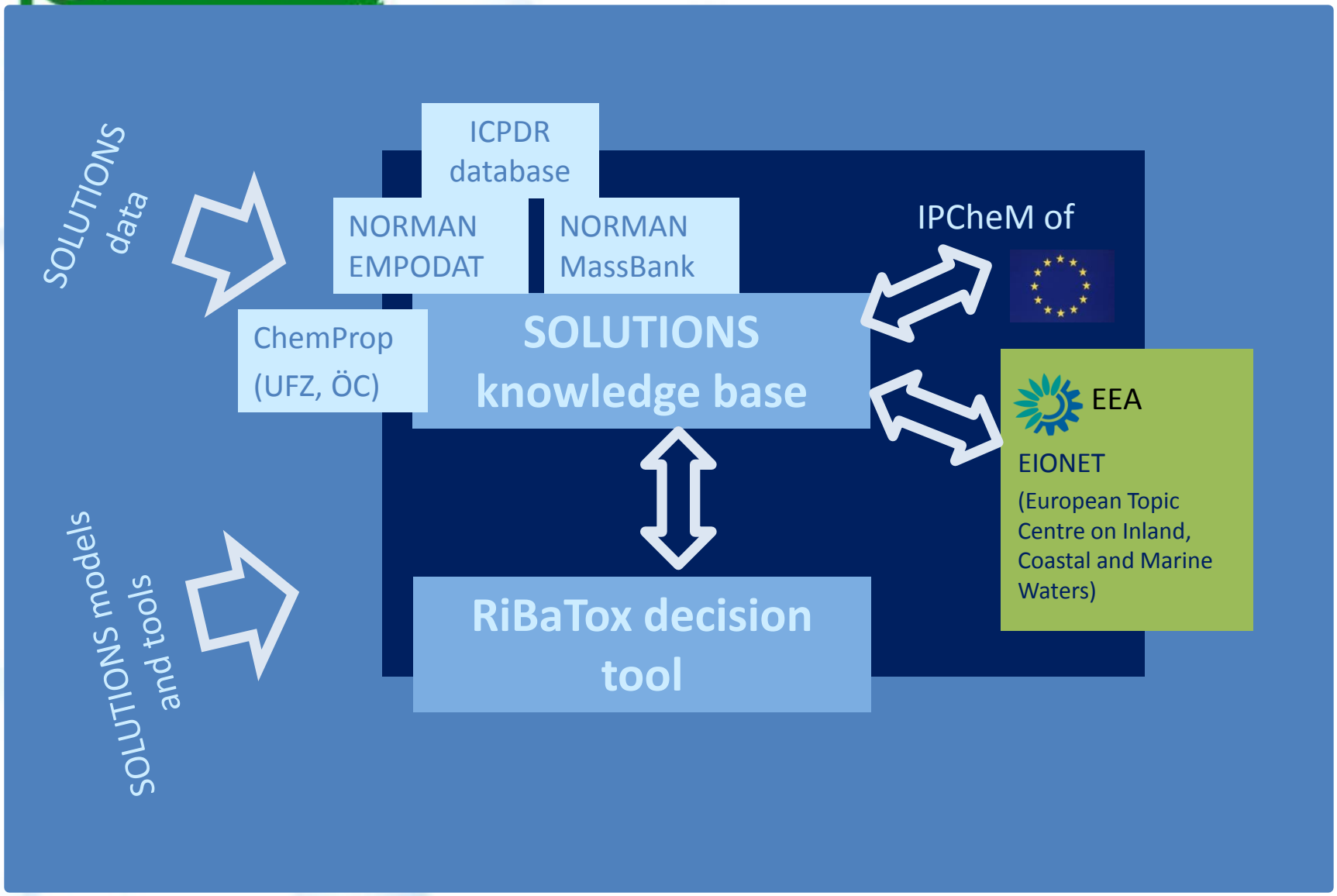
- Target + non-target analysis data for each pollution site
  ➔ **Long list of substances for each polluter**

- Use of local "*Extent of Exceedance*" (EoE) indicator for ranking
- ➔ **Priority based on MEC_{site}/PNEC ratio**

Priority Rank of the 11 polluters based on non-target analysis

SITE	CAS	Compound Name	Conc	Lowest PNEC	Exce.
I-1 Coal thermo power plant	107304-12-1	Stigmastan-6,22-dien, 3,5-dedihydro-	0.0016	0.00036	4
I-1 Coal thermo power plant	57-88-5	cholesterol	0.0014	0.00053	3
I-2 Coal thermo power plant	207-08-9	benzo[k]fluoranthene	0.0021	0.00017	12
I-2 Coal thermo power plant	205-99-2	Benzo(b)fluoranten	0.0018	0.00017	11
I-2 Coal thermo power plant	57-88-5	cholesterol	0.0045	0.00053	8
I-3 Prevent Leather	59-48-3	2H-Indol-2-one, 1,3-dihydro-	50	0.50	100
I-3 Prevent Leather	620-92-8	Phenol, 4,4'-methylenebis-	33	1.69	20
I-3 Prevent Leather	106-44-5	p-Cresol	28	1.4	20
I-3 Prevent Leather	57-11-4	octadecanoic acid	0.03	0.0013	20
I-3 Prevent Leather	141-02-6	2-Butenedioic acid (E)-, bis(2-ethylhexyl) ester	0.14	0.0094	14
I-3 Prevent Leather	3735-92-0	Carbamodithioic acid, dimethyl-, methyl ester	0.26	0.049	5
I-3 Prevent Leather	80-05-7	Bisphenol A	0.27	0.2	1
I-3 Prevent Leather	78-51-3	Tri(butoxyethyl)phosphate	8.9	6.8	1

SOLUTIONS data and knowledge



Conclusions

- Cooperation of SOLUTIONS, NORMAN and ICPDR at
  - **Draft list of Danube RBSPs** – draft Danube RBMP in autumn 2014
  - Prioritisation of target substances (NORMAN Prioritisation framework)
  - **Prioritisation of non-target screening substances** – EG established – open for interested parties
  - **Toxicity profiling** – outcomes of EDA EMERGE and NORMAN ILS
  - Modelling – derivation of P-PNECs via QSAR
  - Assessment of the role of **passive sampling** in the WFD-compliant investigative monitoring and the identification of RBSPs
  - Non-target screening at setting **emission limit values** for individual polluters