Proposals for the Estonian State Monitoring Programme

Completed within the project LIFE07 ENV/EE/000122
"Baltic Actions for Reduction of Pollution of the Baltic Sea from Priority Hazardous Substances"

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1.Introduction

One of the key issues in the project "Baltic Actions for the Reduction of Pollution of the Baltic Sea from Priority Hazardous Substances" (BaltActHaz) was to investigate the **occurrence** of selected WFD, HELCOM priority substances and nationally important pollutants in the environment, as well as track them down to the sources in order to work further on the **reduction of discharges, or exercise the "phasing out" of hazardous substances from the sources**.

The results of screening of hazardous substances in the environment, WWTPs as well as investigation of potential sources of hazardous substances in Estonia, Latvia and Lithuania, are described in the separate reports which are available for downloading on the project website www.baltacthaz.bef.ee.

So far many of those substances were not really monitored in the Baltic countries due to different reasons, i.e. lack of resources, lack of laboratory capacities, etc. Therefore one of the project activities is focused on the **elaboration of proposal for improvements with regard to detection and monitoring of hazardous substances, including the analysis of state-of-the-art of current environmental monitoring system**. The proposals are mainly based on the results of the screening activities performed within the project. However, also other available data and experience of other countries are considered.

This report focuses on the following issues:

- Legal framework for the monitoring of hazardous substances and the purpose of monitoring in general,
- Analytical requirements for the chemical analysis and monitoring of water status and related challenges,
- Analysis of current practices of monitoring of hazardous substances in Estonia,
- Proposals for improvement of the current monitoring system,
- Management of emerging substances.

The report is mainly targeted to the authorities who are responsible for the implementation and enforcement of policies for the control of hazardous substances (WFD and HELCOM BSAP), especially those developing environmental monitoring programmes.

2. Background on the monitoring of hazardous substances in water

2.1. Legal framework for the monitoring of hazardous substances

2.1.1. EU requirements for the chemical monitoring

A strategy for dealing with **pollution of water from chemicals is laid down in Article 16 of the Water Framework Directive 2000/60/EC (WFD)**. As a first step of this strategy, a list of priority substances was adopted identifying 33 substances of priority concern at the Community level. It has the objective to ensure a high level of protection against risks to, or via the aquatic environment arising from these 33 priority substances, by setting European quality of the environment standards. In addition, the WFD requires Member States to identify specific pollutants in the River Basins and to include them in the monitoring programmes. **Monitoring of both WFD priority substances and other pollutants for the purpose of determination of chemical and ecological status shall be performed according to Article 8 and Annex V to the WFD.**

Article 8 of WFD lays down the main requirements to establish the monitoring of surface water status, groundwater status and protected areas. Member States shall ensure the implementation of programmes for the monitoring of water status in order to establish a coherent and comprehensive overview of water status within each river basin district. The programmes have to be operational at the latest by 22 December 2006, and must be in accordance with the requirements of Annex V, which provides details of how the programme should be designed, what should be monitored and how the results should be presented.

The **main reasons** for undertaking monitoring for the WFD are to:

- Develop an overview of the water status of each river basin district,
- Classify individual water bodies as to their water status.

Three types of **monitoring techniques** are required for surface waters under WFD:

- **Surveillance monitoring**: to validate the characterisation pressure and impact assessments, to identify long term changes and trends- priority list substances discharged into the river basin or sub-basins must be monitored; other pollutants also need to be monitored if they are discharged in significant quantities in the river basin or sub-basin;
- **Operational monitoring**: to help classify water bodies which are at risk of failing to meet 'good status' objectives;
- **Investigative monitoring**: to assess why a water body is failing to achieve its objectives and decide what action is needed (it starts when data from surveillance and operational monitoring are available).

The **chemical monitoring according to WFD covers**:

- All surface waters (rivers, lakes and artificial waters; transitional waters; coastal waters up to one nautical mile and territorial waters, extending to 12 nautical miles from territorial baseline of the Member State),
- Groundwater.

Parameters to be monitored:

- **Priority substances**: compliance with European Quality of the environment Standards (AA- EQS and MAC-EQS),
- Other pollutants (river basin specific substances): compliance with national EQS,
- Physico-chemical parameters supporting the interpretation of biological data,
- Parameters required for interpretation of the results of chemical measurements (e.g. DOC, Ca, SPM).

Monitoring in biota is compulsory only for mercury, HCB, and hexachlorobutadiene. Instead of checking compliance with biota EQS Member States may set up a more stringent EQS for water (replacing the one suggested by the Commission) to provide the same level of protection as the biota standard.

The monitoring frequencies given in WFD, Annex V 1.3.4 **of once a month for priority substances and once in three months for other pollutants** will result in certain confidence and precision. Reduced monitoring frequencies, and under certain circumstances even no monitoring may be justified when monitoring reveals / has revealed that concentrations of substances are far below the EQS, declining or stable and there is no obvious risk of increase.

Directive 2008/105/EC sets the quality of the environment standards for 41 substances in the water matrix, but also gives an option to the Member States to derive **EQS for sediment and/or biota**. The frequency of monitoring priority substances in the water column (whole water or dissolved) differs from those in sediment and biota and it is clear that the choice of the matrix to be monitored is strategic in terms of costs and resources for compliance checking. The minimum frequency required for water monitoring of priority substances is once per

month (once every 3 months for river basin-specific pollutants), but for sediment and biota the monitoring frequency can be once per year unless technical knowledge and expert judgment justify another interval.

An overall methodological approach to monitoring for the implementation of the WFD is provided in **guidance documents**; however they are not legally binding documents:

- Guidance Document No. 7 - Monitoring under the Water Framework Directive
- Guidance Document No. 19 - Guidance on surface water chemical monitoring
- Guidance Document No: 25 - Guidance on chemical monitoring of sediment and biota

They can be found on CIRCA site:

http://circa.europa.eu/Public/irc/env/wfd/library?l=/framework_directive/guidance_documents

2.1.2. International requirements for the chemical monitoring

Monitoring is also necessitated by several international environmental agreements where the most relevant for the Baltic countries are the HELCOM Convention which sets the requirements to monitor the Baltic Sea status. The aims of the monitoring as decided by HELCOM are:

- to identify and quantify the effects of anthropogenic discharges / activities in the Baltic Sea in the context of the natural variations in the system, and
- to identify and quantify the changes in the environment as a result of regulatory actions.

It also provides the guidance on it, so-called COMBINE (Cooperative Monitoring in the Baltic Marine Environment) manual: http://www.helcom.fi/groups/monas/CombineManual/en_GB/Contents/

2.2. Purpose of the monitoring of hazardous substances

The overall aim of environmental monitoring is to describe the state of the environment, to show how well our environmental objectives are being met, and to warn about new environmental issues.

Despite the regulatory requirement to perform the monitoring of hazardous substances and in such a way to assess the compliance with standards and objectives there are a number of benefits what monitoring data can provide and why it is necessary:

- it describes the state of the environment and reflects whether the "good status of the environment" (pollutants' concentrations not exceeding EQS, what means that it is safe for the environment and people) is achieved,
- it helps to assess the threats to the environment (as well as human health through the environment) in the early stage, i.e. to identify the substances of concern and what the level of concern is,
- it provides data that can serve as a basis for concrete remedial actions and helps to monitor the progress towards changes and the efficacy of action decided upon, and/or the measures applied (by juxtaposing current and past states we can detect changes in the environment what enables to see whether the past measures have had the desired effect, or if further study is necessary to determine whether or not an observed change is a sign of a problem),
- allows to estimate the pollution loads' transfers across international boundaries or into the sea and provide a basis for analysing the national and international environmental impacts of different emission sources,
- it helps to ascertain formerly unidentified reasons for failure to achieve environmental objectives,
- it supports the assessment of impact of accidental pollution,
- the measured environmental concentrations of harmful substances are needed in the consumer and industrial chemicals risk assessment carried out in the EU; the information gained can also be utilized in national risk assessment work and emission source identification,
- it strengthens the capacity of environmental authorities for the decision making in relation to integrated planning and control of emissions of hazardous substances.

Environmental monitoring is a long-term activity. Measurements must often be taken over long periods in order to show whether a change is due to human activity or natural variation.

3. Analytical requirements for chemical analysis and monitoring of water status and related challenges

3.1. Technical specifications for chemical analysis according to 2009/90/EC

The quality and comparability of analytical results generated by laboratories to perform water chemical monitoring pursuant to Article 8 of Directive 2000/60/EC should be ensured. Therefore the Commission directive **2009/90/EC laying down technical specifications for chemical analysis and monitoring of water status was adopted**. It establishes minimum performance criteria for analytical methods to be applied by Member States when monitoring water status, sediment and biota, as well as rules for demonstrating the quality of analytical results.

The main requirements as described in the directive 2009/90/EC:

- all analytical methods used for the purposes of chemical monitoring programmes carried out under Directive 2000/60/EC are validated and documented in accordance with EN ISO/IEC-17025 standard or other equivalent standards accepted at international level,
- minimum performance criteria for all analytical methods applied are based on an uncertainty of measurement of 50 % or below (k = 2) estimated at the level of relevant quality of the environment standards and a limit of quantification equal or below a value of 30 % of the minimum quality of the environment standards (LOQ ≤ 0.3*AA-EQS),
- in the absence of relevant EQS for a given parameter, or in the absence of method of analysis meeting the minimum performance criteria, monitoring is carried out using best available techniques not entailing excessive costs,
- laboratories apply quality management system practices in accordance with EN ISO/IEC-17025 or other equivalent standards accepted at international level (EN ISO/IEC-17025 standard on general requirements for the competence of testing and calibration laboratories provides appropriate international standards for the validation of the analytical methods used).

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this Directive at the latest two years after its entry into force, it means in 2011.

3.2. Challenges related to technical specifications of 2009/90/EC

3.2.1. Availability of standard methods

Some priority substances are very difficult to analyse as there is a problem also faced on EU level with regard to availability of standardized methods meeting technical specifications of the Directive 2009/90/EC:

- PBDEs: no standard for water available, problems with sensitivity of the methods (LOQ hard to meet as the sum concentration of 6 congeners has to be below 0.5 ng/l and 0.2 ng/l respectively),
- SCCP: no standard for water available, analysis not under control also in the research laboratories, most frequently applied method is GC-ECNI-MS (but with this method there are some unsolved problems: isomers with less than five chlorine cannot be detected; problems with calibration due to dependency of response on degree of chlorination),
- TBT: extremely low LOQ required due to very low EQS – 0.2 ng/l, existing standard methods not sensitive enough,
- PAH (5- and 6-ring PAH): sensitivity for some parameters (in particular for the 6-ring isomers) not sufficient with respect to the low EQS; method is not suitable to cope with samples with SPM content (requirement for whole water samples),
- Organochlorine pesticides: sensitivity of existing standard methods insufficient for cyclodiene pesticides, endosulfane and pentachlorbenzene; difficulties of meeting required LOQ for DDT, hexachlorcyclohexane and hexachlorbenzene.

Table 3.2.1.1. Overview on the standard methods available (Source: Circa website).

Priority Substance	Standard	Principle	LLOA Standard [µg/l]	Target LOQ (AA-QS *0,3) for inland surface waters [µg/l]	Target LOQ (AA-QS *0,3) for other surface waters [µg/l]	Status: inland surface waters	Status: other surface waters
Alachlor[1]	EN ISO 6468:1996	GC/ECD		0,1	0,1	A	A
Anthracene	ISO 17993: 2002	HPLC/Fluo	0,01	0,03	0,03	A	A
Atrazine	EN ISO 11369:1997	HPLC/UV	0,1	0,18	0,18	A	A
	EN ISO 10695: 2000	GC/NPD (MS for conf.)	0,05	0,18	0,18	A	A
Benzene	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01	3	2,4	A	A
	ISO 11423-1:1997	Headspace-GC/FID	2			A	A
Cadmium and its compounds	ISO 17294-2:2003	ICP-MS	0,5	0,024-0,075	0,06	C	C
Chlorfenvinphos	EN 12918:1999	GC	0,01	0,03	0,03	A	A
Chlorpyrifos (-ethyl, -methyl)	EN 12918:1999	GC	0,01	0,01	0,01	A	A
1,2-Dichloroethane	EN ISO 10301:1997	GC or Headspace-GC	5	3	3	B	B
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01			A	A
Dichloromethane	EN ISO 10301:1997	GC or Headspace-GC-ECD or other	50	6	6	B	B
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01			A	A
Di(2-ethylhexyl)phthalate (DEHP)[2]	ISO 18856:2004	GC/MS	0,02	0,390	0,390	C	C
Diuron	EN ISO 11369:1997	HPLC/UV	0,1	0,06	0,06	A	A
DDT (4 Isomers)[3]	EN ISO 6468:1996	GC/ECD	0,01	0,008	0,008	C	C
Fluoranthene	ISO 17993: 2002	HPLC/Fluo	0,01	0,03	0,03	A	A
Hexachlorobenzene[4]	EN ISO 6468	GC/ECD	0,01	0,003	0,003	C	C
Hexachlorobutadiene[1]	EN ISO 10301:1997	GC or Headspace-GC-ECD or other	0,01	0,03	0,03	A	A
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01			A	A
	EN ISO 6468:1996	GC/ECD				A	A
Hexachlorocyclohexane[5]	EN ISO 6468:1996	GC/ECD	0,01	0,006	0,0006	C	D
Isoproturon	EN ISO 11369:1997	HPLC/UV	0,1	0,1	0,1	A	A
Lead and its compounds	ISO 17294-2:2003	ICP-MS	0,1	2,2	2,2	A	A
	ISO 15586:2003	ET-AAS	10			B	B
Mercury and its compounds[4]	EN 12338:1998	CV-AAS with Amalgamation	0,01	0,015	0,015	A	A
	ISO 17582:2006	Atomic fluor. spectrometry	0,01			A	A
Naphthalene	ISO 17993: 2002	HPLC/Fluo	0,01	0,72	0,36	A	A
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01			A	A
Nickel and its compounds	ISO 17294-2:2003	ICP-MS	1	6	6	A	A
	EN ISO 11885:2007	ICP-AES	2-5*			A	A
	ISO 15586:2003	ET-AAS	7			A	A
Tetrachloromethane	EN ISO 10301:1997	GC or Headspace-GC-ECD or other	0,1	4	4	A	A
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,2			A	A
Trichlorobenzenes	EN ISO 6468:1996	GC/ECD	0,01	0,12	0,12	A	A
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01			A	A
Trichloroethene	EN ISO 10301:1997	GC or Headspace-GC-ECD or other	0,05	3	3	A	A
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,05			A	A
Trichloromethane	EN ISO 10301:1997	GC or Headspace-GC-ECD or other	0,05	0,75	0,75	A	A
	EN ISO 15680:2003	Purge/Trap + Therm. Desorp.	0,01			A	A
Trifluralin	EN ISO 10695: 2000	GC/MS or GC/ECD or GC/NPD	0,05	0,01	0,01	A	A
Pentabromodiphenyl Ether	No standard available						
C10-13-chloroalkanes	No standard available						
Endosulfan	Existing standard method not sensitive enough						
Pentachlorobenzene	Existing standard method not sensitive enough						
Benzo(ghi)perylene	Existing standard method not sensitive enough						
Indeno(1,2,3-cd)pyrene	Existing standard method not sensitive enough						
Tributyltin compounds	Existing standard method not sensitive enough						
Aldrin	Existing standard method not sensitive enough						
Endrin	Existing standard method not sensitive enough						
Isodrin	Existing standard method not sensitive enough						
Dieldrin	Existing standard method not sensitive enough						

NOTES:
* axial viewing
1) Alachlor and hexachlorobutadiene are not within the scope of the standard but national monitoring laboratories reported that EN6468 may be used for the determination of these compounds
2) Although the method is applicable to the analysis of DEHP in surface water and allows achieving sufficiently low LoQ to conduct compliance checking in principle, many laboratories have serious blank problems and are hence not able to meet the LoQ performance criterion
3) According to the results of the CMA survey LoQ low enough to allow compliance checking is difficult to achieve or even impossible for DDT due to the fact that 4 isomers have to be determined
4) Although the method is applicable to the analysis of mercury in surface water and allows achieving sufficiently low LoQ to conduct compliance checking in principle, some laboratories have difficulties in meeting the LoQ performance criterion due to problems with blanks and memory effects
5) According to the results of the CMA survey, a sufficiently low LoQ for compliance checking is difficult to achieve or even impossible for hexachlorocyclohexane and hexachlorobenzene.
6) Although the method is applicable to the analysis of NP in surface water and allows achieving sufficiently low LoQ to conduct compliance checking in principle, many laboratories have serious blank problems and are hence not able to meet the LoQ performance criterion
7) Although benzo(k)fluoranthene and benzo(b)fluoranthene (28) are mentioned in the scope LoQ low enough to allow compliance checking is difficult to achieve or even impossible
Category:
A = LLOA meets target LoQ criterion
B = LLOA does not meet target LoQ criterion but CMA survey indicated that laboratories are able to meet target LoQ criterion
C = LLOA does not meet target LoQ criterion, only well-equipped laboratories with highly qualified staff were able to meet target LoQ criterion, there are certain limitations in applicability of the standard
D = Standard not sensitive enough for the analysis of other surface waters For more detailed information see the main document

Estonian Environmental Research Centre lacked for analytical capability to analyse in full the hazardous substances surveyed in the project. Therefore, some of the analytes were sent to foreign laboratory for analysis – to GALAB Laboratories GmbH, Max-Planck-Strasse 1, Geesthacht, Germany. The activity of GALAB has been declared to be in line with the requirements of standards EN ISO/IEC 17025 and DAC-PL-0114-00-10 by the Accreditation Centre of Germany.

The limit of quantification of the analytical method (in-house method, GC-MSD) used by GALAB laboratories for measuring of polybrominated diphenyl ethers, particularly derivatives PBDE-28, PBDE-47, PBDE-99, PBDE-100, PBDE-153, PBDE-154, was not as low as needed. The annual average of validated quality of the environment limit value for the land surface water is 0.0005 µg/l, but the limit of quantification of the analytical method used by the laboratory was ten times higher – 0.005 µg/l. At the same time there is no standard method that is sensitive enough to fulfil the requirements given in the directives 2008/105/EC and 2009/90/EC (see 3.2.1, Table 1).

It would have been possible to find laboratories within the European Union that have the capability to analyse priority hazardous substances in full, but using their services would have limited the number of samples and analyses because of remarkably higher price for analyses.

3.2.2 Laboratory capacities in Estonia

In Estonian Environmental Research Centre the standard methods are preferably used for conducting the analyses. In case there is no standard method or the limit of quantification of standard method is inadequate, modified methods based on standard methods are used.

The list of substances analysed in Estonian Environmental Research Centre and the analytical methods used during the project are given in the Table 3.2.2.1.

The technical capability of the laboratories of Estonian Environmental Research Centre in 2009 did not allow analysing all the hazardous substances according to Directives 2008/105/EC and 2009/90/EC.

Table 3.2.2.2. presents the substances analysed in the laboratory of the Estonian Environmental Research Centre, limits of detection, limits of quantification and measurement uncertainties of the analytical methods, and the respective limit values of the quality of the environment for substances.

Table 3.2.2.1. The list of substances analysed in Estonian Environmental Research Centre and the used analytical methods until 2009.

Substance/substance group	Surface water/effluent	Sediment/sludge
Heavy metals, except Mercury	Inductively coupled plasma-mass spectrometry (ICP-MS), EVS EN ISO 17294-2:2004; flame atomic absorption spectrophotometry (FAAS), ISO 8288	Inductively coupled plasma-mass spectrometry (ICP-MS), STJ No. M/U 91 (EN ISO 11885)
Heavy metals: Mercury	Cold vapour method (EVS-EN 1483).	Cold vapour method (EVS – EN 1483).
Benzene	Gas chromatography, flame ionisation detector (GC-FID), ISO 11423-2. STJ No. U62B	-
Volatile Organic Compounds (VOC), including chlorobenzenes	Gas chromatogaphy, electron capture detector (GC-ECD), EVS-EN ISO 10301, STJ No. V75	-
Chloroorganic pesticides	Gas chromatography, electron capture detector (GC-ECD) (EN ISO 6468); STJ No. U63;	Gas chromatography, electron capture detector (GC-ECD); STJ No. U63;
Cyanide	Spectrophotometric method (ISO 6703-1), STJ No. V37.	-
Pentachlorophenol	Liquid chromatography, diode array detector (HPLC-DAD), STJ No. U12A	Liquid chromatography, diode array detector (HPLC-DAD), STJ No. U12A
Phenols	Liquid chromatography, electrochemical detector (HPLC-ECD), STJ No. U12	Liquid chromatography, electrochemical detector (HPLC-ECD), STJ No. U12

Table 3.2.2.2 Substances analysed in the laboratory of the Estonian Environmental Research Centre, limits of detection, limits of quantification and measurement uncertainties of the analytical methods, and the respective limit values of the quality of the environment for substances (according to Directives 2008/105/EC and 2009/90/EC).

CAS No.	Priority substances, priority hazardous substances and certain others pollutants	AA-EQS Land surface water (µg/l)	AA-EQS Other surface water bodies (µg/l)	Surface water LOQ (µg/l)	Criterion of 2009/90 LOQ/ AA-EQS %	Surface water LOD (µg/l)	MU %	Sediment LOQ (µg/kg)	Sediment LOD (µg/kg)	MU %
120-82-1	1,2,4-Trichlorobenzene	0.4	0.4	0.005	1.25	0.0025	18	1	0.8	20
67-66-3	Trichloromethane (chloroform)	2.5	2.5	0.1	4	0.08	35	-	-	-
71-43-2	Benzene	10	8	0.2	2/2.5	0.05	20	-	-	-
107-06-2	1,2-Dichloroethane	10	10	0.1	1	0.08	45	-	-	-
75-09-2	Dichloromethane	20	20	0.1	0.5	0.08	45	-	-	-
608-93-5	Pentachlorobenzene	0.007	0.0007	0.005	71/7140	0.0037	32	1	0.8	23
115-29-7	Endosulfan	0.005	0.0005	0.005	100	0.0037	10	1	0.8	25
118-74-1	Hexachlorobenzene (HCB)	0.01	0.01	0.005	50	0.0037	32	1	0.8	20
87-68-3	Hexachlorobutadiene	0.1	0.1	0.1	100	0.08	12	-	-	-
608-73-1	Hexachlorocyclohexane	0.02	0.002	0.005	25/250	0.0037	17	1	0.8	33
7439-92-1	Lead and its compounds	7.2	7.2	1	13.8	0.3	12	2000	1500	28
7439-97-6	Mercury and its compunds	0.05	0.05	0.05	100	0.02	29	20	10	17
7440-43-9	Cadmium and its compounds	≤0.08-0.25	0.2	0.1	40/50	0.007	11	1000	500	38.5
7440-02-0	Nickel and its compounds	20	20	1	5	0.3	12	1000	500	23
7440-66-6	Zinc and its compounds	10*	5*	10	50	4	9	1000	500	25
7440-38-2	Arsenic	10*	10*	1	10	0,02	12	2500	1250	42
7440-47-3	Chromium	5*	5*	0.1	2	0.05	16	1000	500	17.6
7440-38-2	Copper	15*	5*	1	6.6	0.2	13	1000	500	16.5
	p,m-cresol			2		0.4	20	100	50	20
95-48-7	o-cresol			2		0.4	25	100	50	20
309-00-2	Aldrin	0.01	0.005	0.005	500/100	0.0026	9	1	0.8	25
60-57-1	Dieldrin	0.01	0.005	0.005	500/100	0.003	12.3	1	0.8	25
72-20-8	Endrin	0.01	0.005	0.005	500/100	0.003	14.7	1	0.8	25
465-73-6	Isodrin	0.01	0.005	0.005	500/100	0.0022	19	1	0.8	23
56-23-5	Tetrachloromethane	12	12	0.1	0.8	0.08	35	-	-	-
127-18-4	Tetrachloroethylene	10	10	0.1	1	0.08	44	-	-	-
79-01-6	Trichloroethylene	10	10	0.1	1	0.081	30	-	-	-
57-12-5	Cyanides	100*	50*	3	3/6	1.6	25	0.5	0.25	0.25

NOTES:
AA-EQS – Annual Average; inland water bodies and other surface waters
* Estonian national AA-EQS (Regulation of the Minister of the Environment No. 49 from the 9th of September 2010)
LOQ – Limit of Quantification
LOD – Limit of Detection
MU – Measurement Uncertainty
Minimum criteria for the analytical methods: measurement uncertainty up to 50 % (k=2) and limit of quantification up to 30 % of the relevant standard for quality of the environment (Directive 2009/90/EC Article 4)

The technical capability was not sufficient for quantification of some organic halogen compounds (Pentachlorobenzene, Endosulfan, Hexachlorobenzene, **Hexachlorobutadiene, Cyclodienes) and some heavy metals (Mercury, Cadmium,** Zinc) according to **Directive 2009/90/EC.**

For the sake of updating the quantification limits and reconciliation with the valid requirements the analytical and sample preparation methods of Estonian Environmental Research Centre were improved.

For the analysis of heavy metals a new analyser RA915 plus Zeeman Mercury Spectrometer was put to use for mercury analysis. Furthermore, for the sake of updating the analysis of metals a more sensitive instrumentation – inductively coupled plasma mass-spectrometer (ICP-MS) was introduced.

To bring the analysis of **pesticides** into conformity with the requirements, the concentration of samples and a number of samples to extract were increased instead of acquiring new instrumentation. However, it is being planned to acquire new, more sensitive and economical up-to-date instrumentation in the near future.

For the analysis of **Pentachlorophenol** in conformity with the requirements, a new more sensitive device was introduced - a liquid chromatograph with diode array detector (HPLC-DAD).

For the analysis of **Pentachlorobenzene** in conformity with the requirements, the concentration of samples and a number of samples to extract were increased.

The method that was used so far for the analysis of Polyaromatic hydrocarbons (PAH) did not allow achieving the limits of quantification corresponding to EQS requirements. It is emphasized in the Annex of Directive 2008/105/EC, in the list of suitable reference methods for analysis that the quantification with the reference method at the required LOQ is difficult or impossible. Currently Estonian Environmental Research Centre is validating the new ISO 28540 (Quantification of 16 PAH in water. GS-MS method) for quantifying PAHs, where the given limit of quantification is 0.005 µg/l in ground water and 0.01 µg/l in surface water. Using the best possible technological devices it is possible to obtain even lower limits of quantification with the same method. The limits of quantification, given in the method do not satisfy the requirements of EQS, for example the limit of quantification of the sum of Benzo(g,h,i)perylene and Indeno(1,2,3-cd) pyrene is 0.002 µg/l according to EQS requirements.

As a result of developing and updating the methods, the reconciliation of the limits of quantification of several quantified indicators with the existing legislation has been successful. The data on updating the limits of quantification of the methods is presented in Table 3.2.2.3.

Table 3.2.2.3. Updating the limits of quantification of methods used at Estonian Environmental Research Centre – land surface water 2009–2011.

CAS No.	Priority substances, priority hazardous substances and certain others pollutants	AA-EQS Land surface water (µg/l)	Commission Directive 2009/90 criterion land surface water	EERC LOQ 2009 Land surface water (µg/l)	EERC LOQ 2011 Land surface water (µg/l)
608-93-5	Pentachlorobenzene	**0.007**	**0.0021**	**0.005**	**0.0014**
115-29-7	Endosulfan	**0.005**	**0.0015**	**0.005**	**0.0015**
118-74-1	Hexachlorobenzene (HCB)	**0.01**	**0.003**	**0.005**	**0.0015**
87-68-3	Hexachlorobutadiene	**0.1**	**0.03**	**0.1**	**0.0034**
608-73-1	Hexachlorocyclohexane	**0.02**	**0.006**	**0.005**	**0.002**
7439-97-6	Mercury and its compounds	**0.05**	**0.015**	**0.05**	**0.015**
7440-43-9	Cadmium and its compounds	**<0.08-0.25**	**0.024-0.075-**	**0.1**	**0.02**
7440-66-6	Zinc and its compounds	**10***	**3**	**10**	**1**
309-00-2	**Aldrin**	**0.01**	**0.003**	**0.005**	**0.002**
60-57-1	**Dieldrin**	**0.01**	**0.003**	**0.005**	**0.0028**
72-20-8	**Endrin**	**0.01**	**0.003**	**0.005**	**0.0022**
465-73-6	**Isodrin**	**0.01**	**0.003**	**0.005**	**0.0034**

NOTE:
* Estonian national AA-EQS (Regulation of the Minister of the Environment No. 49 from the 9th of September 2010)

Table 3.2.2.4. Updating the limits of quantification of methods used at Estonian Environmental Research Centre – other surface waters 2009–2011.

CAS No.	Priority substances, priority hazardous substances and certain others pollutants	AA-EQS Land surface water (µg/l)	Commission Directive 2009/90 criterion other surface waters	EKUK LOQ 2009 Land surface water (µg/l)	EKUK LOQ 2011 Land surface water (µg/l)
608-93-5	Pentachlorobenzene	0.0007	0.00021	0.005	0.0007
115-29-7	Endosulfan	0.0005	0.00015	0.005	0.0008
118-74-1	Hexachlorobenzene (HCB)	0.001	0.003	0.005	0.001
87-68-3	Hexachlorobutadiene	0.1	0.03	0.1	0.0025
608-73-1	Hexachlorocyclohexane	0.002	0.0006	0.005	0.0008
7439-97-6	Mercury and its compounds	0.05	0.015	0.05	0.015
7440-43-9	Cadmium and its compounds	0.2	0.06	0.1	0.02
7440-66-6	Zinc and its compounds	5*	1.5	10	1
309-00-2	**Aldrin**	0.005	0.0015	0.005	0.0006
60-57-1	**Dieldrin**	0.005	0.0015	0.005	0.0005
72-20-8	**Endrin**	0.005	0.0015	0.005	0.0007
465-73-6	**Isodrin**	0.005	0.0015	0.005	0.0014

NOTE:
* Estonian national AA-EQS (Regulation of the Minister of the Environment No. 49 from the 9th of September 2010)

For quantification of several priority substances there are no sufficiently sensitive reference methods to date. For example, there is no sufficiently sensitive reference method for quantifying Pentachlorobenzene in seawater. According to the quality of the environment standard the limit of quantification of Pentachlorobenzene has to be 30 % of relevant quality of the environment standard, or 0.2 ng/l.

It is emphasized in the Annex of Directive 2008/105/EC, in the list of suitable reference methods for analysis that the quantification of Hexachlorobenzene with the reference method at the required LOQ is difficult or impossible; nor is there sufficiently sensitive reference method for quantifying Pentachlorobenzene.

As a result of the BaltActHaz project, Estonian Environmental Research Centre has reviewed its analytical methods and conducted development procedures, in consequence of which the quantification of Pentachlorophenol, Polyaromatic hydrocarbons, Hexachlorobenzene, organic halogen pesticides and heavy metals content in different types of water has been in the majority of cases brought into conformity with the requirements of Directives 2008/105/EC and 2009/90/EC.

3.2.3. Plans for the future

Estonian Environmental Research Centre is planning to enhance its analytical capability with reference to several substance groups that could not be analysed in Estonia so far. Due to the lack of instrumentation of sufficient sensitivity it is currently not possible to quantify all the priority substances in accordance with valid EQS requirements.

In order to enhance its analytical capability Estonian Environmental Research Centre participates in an international project "Enhancing the capability of analysing priority hazardous substances". In the course of the project, instrumentation of sufficient sensitivity will be procured which will allow quantifying the content of hazardous substances in conformity with the requirements of Directives 2008/105/EC and 2009/90/EC.

In connection with procurement of new technological equipment it is being planned to construct corresponding laboratory facilities, procure suitable instrumentation for sample preparation and train qualified personnel. The listed activities have been planned for the year 2012; accreditation of methods has been planned for 2013.

4. Analyses of practices of monitoring and screening of hazardous substances in Estonia

Elaboration of the Estonian National Environmental Monitoring Programme was started in The Ministry of the Environment in 1993 [4.1] and the programme became operational in 1994 when it received funding from the state budget for the first time. The monitoring reports are available on Internet Web sites of the Ministry and the subordinate establishments in its area of government: Ministry of the Environment: (environment monitoring programme) – (http://eelis.ic.envir.ee:88/seireveeb/).

The monitoring programme of hazardous substances in the National Environmental Monitoring Program has three subprograms (monitoring of inland water bodies, monitoring of coastal sea waters and monitoring of ground water) which in turn have five subprograms (monitoring of hazardous substances in water bodies, surveillance monitoring of pollution loads, monitoring of hazardous substances in the Baltic Sea, heavy metals in the ground water in the industrial area of North-eastern Estonia, and monitoring of organic compounds in the ground water in industrial area of North-eastern Estonia).

In recent years the monitoring of hazardous substances has become more exhaustive as new substances have been included in the surveys on the ground of the new requirements of the EU; however, the state budget has not been capable of funding all monitoring surveys of hazardous substances for the aquatic environment, included in the national monitoring programme. For that reason the screening conducted in the framework of the BaltActHaz project was essential, as the content of such hazardous substances about which there was hitherto no data concerning aquatic environment were also quantified for the first time – most of alkylphenols and their ethoxylates, phtalates, polybrominated diphenyls, diphenylethers and polybrominated organic compounds, organotin compounds, short and medium chained chlorinated paraffins, perfluorinated substances, sodium tripolyphosphate and certain pesticides. In Table 4.1 are listed compounds about which there is data of monitoring and screening results in Estonia.

Table 4.1. Compounds quantified in the surface water of Estonian national monitoring as a result of enterprise monitoring and screening.

Hazardous substances	NEMP* until 2004	2005–2010	2010
Heavy metals	X	X	X
Phenols: -1-base -2-base	 X X	 X X	 X X
Volatile Organic Compounds	X	X	X
Chloroorganic pesticides	X	X	X
Polyaromatic hydrocarbons	X	X	X
Polychlorinated biphenyls	X	X	X
Alkylphenols and their ethoxylates	–	X**	X
Organotin compounds	–		X
Phtalates	–	X**	X
Polybrominated diphenyls, diphenylethers, and polybrominated organic compounds	–	–	X
Short- and medium-chained chlorinated paraffins		–	X
Perfluorinated compounds	–	X	X
Plant protection products (except chloroorganic pesticides)	–	X**	X
Pentachlorophenol	–	–	X
Sodium polyphosphate	–	–	X
Cyanides	X	X	X
Oil (C_{10}-C_{40} hydrocarbons)	X	X	X

NOTES:
NEMP* – National Environmental Monitoring Programme
X** – Quantified in part only:
- Alkylphenols and their ethoxylates (only 4-tert-octylphenol, 4-n-nonylphenol and iso-nonylphenol were quantified [4.2])
- Plant protection products (AMPA, Glyphosate, Glyphosate, Mecoprop (MCPP), Trifluralin, Chlorfenvinphos [4.2], Atrazine, Diuron, Simazin, Isoproturon [4.3] were quantified)
- Phtalates (di(2-ethyl-hexyl)phtalate (DEHP) were quantified)

National monitoring of hazardous substances until 2004

In the early years of monitoring of hazardous substances for aquatic environment attention was paid above all to those priority hazardous substances which could be quantified in Estonia and which have been by now included in the Water Framework Directive of the EU. Data about heavy metals (As, Zn, Sn, Cu, Cr, Cd, Pb, Hg, Ni) and organic compounds (petroleum products, polychlorinated biphenyls, monobasic and dibasic phenols benzene, fluoranthene, hexachlorobenzene, hexachlorocyclohexane, naphtalene, polyaromatic hydrocarbons, trichloromethane, DDT, dieldrin, endrin, isodrin, etc.) is presented until 2004, before Estonia's accession to the European Union. Alachlor, chlorfenvinphos, diuron and isopropturon were not quantified because the existing data gave reason to presume that they were not used in Estonia. Other substances such as pentabromodiphenyl ethers, chloroalkanes, methylene chloride, nonyl- and octylphenols were not quantified because of lack of relevant competence of laboratories. The nation-wide inventories of hazardous substances in Estonia in 1999–2001 gave only starting-points for organising effluent / wastewater audits. Samples were taken from 104 spots altogether. Computational total emissions into water (kg/year) of substances belonging to List 1 of Estonian hazardous substances. Petroleum products – 15,771 kg; Carbon tetrachloride – 304 kg; Perchloroethene (PER) – 21.9 kg; Trichloroethene (TRI) – 10.4 kg; 1.2-dichloro-ethane – 3.9 kg; Cd – 3.1 kg; Chloroform – 2.1 kg; Pentachlorophenol – 1.7 kg; Hg – 0.3 kg; Cyanide – 0.2 kg; Lindane – 0.02 kg;

Computational total emissions into water (kg/year) of substances belonging to List 2 of Estonian hazardous substances. Ba – 7 531 kg; Ni – 4 980 kg; Zn – 2 566 kg; Monobasic phenols – 866 kg; Cr – 451 kg; Cu – 398 kg; As – 104 kg; Co – 90,7 kg; Pb – 83,2 kg; Mo – 76,7 kg; Benzene – 19.7 kg; Sn – 2,1 kg; V –1,7 kg; Se – 0,6 kg; Polyaromatic hydrocarbons 0.09 kg.

In 2005 Estonian Environmental Research Centre prepared a report on the basis of the Directive 92/446/EEC about surveys of hazardous substances in Estonia in 2002–2004 [4.4], before Estonia's accession to the European Union. Data on average, minimum and maximum content of hazardous substances, as well as the number of analysis made annually both in surface water and the surface water sediments, and also biota were presented in the report.

The national monitoring and surveys of hazardous substances 2004–2010

In the years 2005–2010 the Estonian Environmental Research Centre, AS Maves, as well as the Estonian Marine Institute made several proposals for updating the water monitoring programme for hazardous substances. In the reports prepared by Estonian Environmental Research Centre in 2005 "Defining the priority hazardous substances for Estonian surface water bodies and creating a monitoring network" [4.5] ("Eesti pinnaveekogude jaoks prioriteetsete ohtlike ainete määramine ja seirevõrgu moodustamine") and "Updating the monitoring programme of hazardous substances" [4.6] ("Ohtlike ainete seireprogrammi uuendamine") recommendations were given about conducting the monitoring programme of hazardous substances in 2006–2008 concerning the substances, monitoring spots and monitoring frequency. In 2006 Estonian Marine Institute of The University of Tartu prepared recommendations for updating the monitoring programme of coastal waters [4.7].

In the framework of the international project "EU-Wide Monitoring Survey of Polar Persistent Pollutants in European Waters" 122 water samples of 27 EU member states were surveyed in 2008 [4.3]. In all, the concentration of 35 selected polar persistent organic compounds in 100 European rivers and other similar watercourses were surveyed. Three rivers from Estonia participated in the project: the rivers Narva, Purtse and Emajõgi. 10 % of the European rivers and wartercourses surveyed were classified as "very clean". **The cleanest water samples were obtained from Estonian, Lithuanian and Swedish water bodies**.

In the survey "Inventory and monitoring organisation analysis of priority substances with intent to fulfil the requirements of the Directive No. 2008/105/EC from 6. December 2008 of the European Parliament and the Council" ("Euroopa Parlamendi ja Nõukogu 6. detsembri 2008 direktiivi 200(/105/EÜ nõuete täitmiseks prioriteetsete ainete inventuur ning seirekorralduse analüüs."), ordered by The Ministry of the Environment in 2010 the existence of altogether 52 hazardous substances was monitored in 19 water monitoring stations (18 of them elements of surface water). Samples were collected from 15 sampling points in May and September 2010. Analyses were conducted at the accredited laboratory GBA Gesellschaft für Bioanalytic Hamburg mbH. **The majority of concentrations of hazardous substances quantified in inland water bodies remained below the limits of quantification**. In a few surveying spots cadmium, nickel and tin were detected above the limit of quantification. The concentration of nickel was above the limit value in Kroodi stream and the coastal waters of Narva and Kunda bay. The annual average concentration of cadmium was over the limit value in the Kuusiku river. The concentration of Iso-nonylphenol was over the annual average limit value in the waters of Kroodi stream, the river Emajõgi, Gulf of Pärnu and lake Peipus. This substance was detected over the limit of quantification in some other water bodies. The concentration of 4-tert-octylphenol was over the limit value in Kroodi stream. Alkylphenols were detected in many water bodies but the concentrations discovered did not exceed the allowed limit values [4.2].

5. Proposals for improvement of the Estonian monitoring programme based on BaltActHaz project findings

5.1. Summary of the main findings on hazardous substances occurrence in BaltActHaz project and other HS screening activities in Estonia

From the point of view of detection of substances previously not quantified the results of the given work were fresh and would provide material for designing further surveys and applying relevant measures for the purpose of improving the status of the environment. The results of screening and source survey are mainly addressed for the state agencies that are responsible for the elaboration of the policy and strategy for hazardous substances for aquatic environment and the inspection of corresponding substances. The information about hazardous substances allows developing monitoring programmes, making the inspection of emissions of hazardous substances more efficient; and it helps with the Estonian rendering accounts about hazardous substances both for the European Commission and HELCOM.

The concentration of several substances such as alkylphenols and their ethoxylates, organotin compounds, phthalates, polybrominated diphenyls, diphenyl ethers and polybrominated organic compounds, short and medium chain chlorinated paraffins, perfluorinated substances, some pesticides, and sodium polyphosphate were never quantified in Estonia before, as there was neither explicit obligation nor necessity to quantify them, and also the laboratories lacked for relevant capability.

Among the substances previously not quantified in Estonia, alkylphenols, organotin compounds and phthalates were detected both in surface water, effluent and waste water, as well as in ground water sediments and the waste water sediments. The highest concentrations were in the waste water sediments. The detected concentration of these compounds in surface water, however, were still below the limit values of the quality of the environment, laid down for those compounds by the legislation. For the ground water sediments and the waste water sediments, both the EU and Estonian limit values are absent.

Among hazardous substances and their groups all concentrations of polybrominated diphenyls, diphenylethers, polybrominated organic compounds, short and medium chain chlorinated paraffins, and perfluorinated compounds quantified in the water samples were below the quantification limit of the used analytical methods. The concentration of polybrominated diphenyls, diphenylethers and polybrominated organic compounds were analysed above the quantification limit in the waste water sediments of the waste water treatment plants. Based on the source survey the concentrations of polyaromatic hydrocarbons and volatile organic compounds also exceeded the quantification limit of the methods used.

The concentrations of 23 plant protection products quantified in the water of inland water bodies of the most intensive farming activity areas of Estonia remained below the quantification limits of the used analytical methods. However, the above-mentioned does not exclude their random test in a wider area and more gauging sections, in addition to surveys conducted in the framework of national monitoring.

Based on the screening results we can say that of those hazardous substances for aquatic environment that have been surveyed here until now, the concentration of heavy metals in our surface water bodies may still cause problems. Of those substances that were not quantified in Estonia earlier, organotin compounds and phthalates were identified both in land surface waters and bottom sediment; however, the concentrations in the surface water detected within the framework of screening **remained below the limit values of the quality of the environment, laid down for those compounds by the legislation**. During the screening extremely high volumes of organotin compounds were detected in the water and sediments of Tallinn Bay, in the adjacent area to the Tallinn Shipyard BLRT Grupp. The organotin compounds were formerly widely used as additives in antifouling (for avoiding the fastening of water organisms onto the hull) ship paints. The fact that such volumes of pollution exist in the coastal sea and in sediments probably indicates to the deficiency of work organisation of the company at cleaning the ships of old ship paint, or refers to the so-called historical load accumulated in the sediments in the course of time. Whether this is an abandoned or still on-going practice should be definitely found out. Unfortunately, hazardous pollution in the sea actually exists and goes on endangering the marine biota. Since there is an excessive quantity of TBT compounds also in surface water the activity of the company may cause the moving of the compounds from the so-called historically loaded sediments into the surface water, and as a consequence the contamination of surface water with organotin compounds.

Substances and substance groups detected in the highest concentrations both in surface water bodies and outlets of wastewater treatment plants during the screening, and which turned out to be the most alarming are given below.

The concentrations of hazardous substances and substance groups quantified in Estonian surface waters (rivers, Lake Peipus) remained in the majority of cases below the quantification limits of the used analytical methods and did not exceed the valid limit values of the quality of the environment. The concentrations of a few phthalates like diisobutyl phthalate, di(2-ethylhexyl)phthalate, and dimethyl phthalate were detected over the limit of quantification but they remained below the established standards. Among the so-called "old hazardous substances" high concentrations of monobasic phenols were detected in the gauging sections of the rivers Kohtla, Vasalemma, Narva and Keila, and in a water sample taken at the national monitoring point No. 38 on Lake Peipus. Mono- and dibutyltin were detected in the rivers Narva, Keila and Kasari. In the water of the rivers Kunda and Pühajõgi a high benzene concentration was quantified.

As for coastal waters **high concentrations of monobasic phenols and organotin compounds were detected**. Water samples taken at the coastal region of Sillamäe had high concentration of monobasic phenols. **Extremely high** concentrations of organotin compounds were detected in the water samples taken from the coastal area of Tallinn Bay, adjacent to Tallinn Shipyard of BLRT Grupp AS. Moreover, the concentration of tributyltin exceeded more than 6000 times the allowed maximum limit value of 0.0015 µg/l, set for this substance in respect to the quality of the environment.

The concentrations of hazardous substances and substance groups quantified in surface water sediments (rivers, Lake Peipus), **in the majority of cases remained below the limits of quantification of the used analytical methods**. However, relatively high quantities of heavy metals such as nickel, chromium, zinc and copper were detected in sediments of both the river Narva and Lake Peipus, and high concentrations of 2.5-dimethylresorcin in sediments of the rivers Keila, Narva and Pühajõgi.

In the surface water sediments of the coastal areas, **higher amounts** of heavy metals like nickel, zinc and arsenic were detected in the sediment samples taken at the coastal area of Sillamäe. Sediments of Tallinn Bay. Extremely high concentrations of accumulated organotin compounds like tributyltin, dibutyltin, monobutyltin, etc. were detected in the sediments of the adjacent area to Tallinn Shipyard BLRT Grupp.

Effluent from the waste water treatment plants contained **high quantities of some heavy metals like monobasic phenols, alkylphenols and their ethoxylates. In the effluent from the waste water treatment plants of Tallinn, Keila and Kohtla-Järve high concentrations of arsenic, lead, nickel , chromium, copper and zinc were quantified**. High concentrations of monobasic phenols were detected in the effluent from the waste water treatment plants of Kohtla-Järve, Keila, Tallinn and Narva. The effluent of the waste water treatment plant of Keila was highly contaminated with alkylphenols and their ethoxylates like 4-tert-octylphenol, 4-tert-butylphenol, iso-nonylphenol monoethoxylate, 4-t-octylphenol monoethoxylate, and 4-t-octylphenol diethoxylate. Also the effluent of the waste water treatment plants of Tallinn and Kuressaare contained remarkable amounts of 4-tert-octylphenol and 4-tert-butylphenol.

In the waste water sediments of waste water treatment plants high concentrations of heavy metals, organotin compounds, monobasic and dibasic phenols, alkylphenols and their ethoxylates, and phthalates were detected. Unfortunately the limit values set for waste water sediments are so far missing in the EU legislation. The concentration of chromium was very high in the waste water sediments of the waste water treatment plants of Keila and Narva. In the waste water sediments from most of the waste water treatment plants that were included in the screening very high tributyltin, dibutyltin, monobutyltin, monooctyltin and dioctyltin quantities were detected. The waste water sediment of the waste water treatment plant of Tallinn contained high quantities of iso-nonylphenol, iso-nonylphenol monoethoxylate, and 4-tert-octylphenol. In the waste water sediments of the waste water treatment plants of Kohtla-Järve and Keila very high quantities of monobasic and dibasic phenols were detected. The concentration of pentachlorophenol was high in the waste water sediments of the waste water treatment plants of Keila and Narva. As for phthalates, the concentration of di(2-ethylhexyl)phthalate (DEHP) was the highest, and detected at high concentrations in the waste water sediments of all the waste water treatment plants. Polybrominated diphenylethers and tetrabromobisphenyl A (PBB-52) were also detected in waste water sediments.

During the screening it became obvious that the effluent exiting the waste water treatment plants contaminates both the surface water as well as the coastal sea, as it contains in addition to heavy metals and phenols also high quantities of such other hazardous substances that have not yet been analysed in the process of monitoring in Estonia, like alkyl phenols, their ethoxylates and phthalates.

5.2. Proposals for improvement of the current monitoring programme

In the Water Framework Directive the monitoring of surface water is described as surveillance monitoring, operational monitoring and research monitoring. The monitoring of the effluent, however, belongs to the enterprise monitoring programme. In case of monitoring of priority substances the hazardous substances must be assessed both in the surface water and the effluent. As the list of hazardous substances is long, the analyses expensive and partly impossible due to the low capability of Estonian laboratories, is it important to find the most suitable way to assess the concentration of hazardous substances in the surface water and the effluent.

Improving the monitoring programme, introducing new substances or compounds and selecting matrices (water, sediments or biota) should be based on Guidance Document

No. 7 "Monitoring under the Water Framework Directive", Guidance Document No. 19 "Guidance on Surface Water Chemical Monitoring under the Water Framework Directive", European Union, 2009, Guidance Document No. 25 "Guidance on Chemical Monitoring of Sediment and Biota under the Water Framework Directive", European Union, 2010; and in case of monitoring of biota the recommendations of HELCOM COMBINE programme.

The screening results showed that the selection of the right matrix is of essential relevance. But in addition to water and sediments biota is the third important matrix. [4.10]. In the Baltic Sea countries first of all in fish – the Baltic herring and the bass. The monitoring of hazardous substances in biota should be based on the recommendations of HELCOM COMBINE programme.

The monitoring of biota and the bottom sediments should also be included in the National Environmental Monitoring Programme, whereby the selection of matrices (water, biota or sediment) should be based on the recommendations of the reports of the European Commission "Guidance document No. 19" (Guidance on surface water chemical monitoring under the Water Framework Directive) ja "Guidance document No. 25" (Guidance on chemical monitoring of sediment and biota under the Water Frame-work Directive) regarding the substances for which it is preferable to monitor the concentration of pollutants in water, and which substances should preferably be monitored in sediments or biota [4.9].

In case of monitoring of many priority hazardous substances such as polybrominated diphenylethers, polyaromatic hydrocarbons, chloroorganic pesticides, tributyltin and short chain chlorinated paraffins, sediments or biota is preferred as matrix instead of water samples. Based on following preferences on matrix options. The list of priority hazardous substances and the preferences on the matrices (W – water, S – sediments, B – biota) to be monitored in Directive 2008/105/EC:

1. Anthracene – W/S/B
2. Bromodiphenylethers – S/B
3. Cadmium – not applicable
4. Cloroalkanes, C10–13 – S/B
5. Endosulfan – W/S/B
6. Hexachlorocyclohexane – B
7. Hexachlorobutadiene – B
8. Hexachlorobenzene – S/B
9. Mercury – B
10. Nonylphenol – W/S
11. Pentachlorobenzene – S
12. Polyaromatic hydrocarbons – S/B
13. Tributyltin – B

For monitoring of biota in coastal waters and estuaries the bio indicators recommended by the Baltic Sea HELCOM should be applied. For offshore monitoring – the Baltic herring (12–15 two to three-year old female Baltic herrings in a sample, captured in August or September) and for the coastal and estuary monitoring – the bass (10–15 female bass in a sample, 15–20 cm long, captured in August or September) [4.10].

Bio tests that would allow easily determining the effluent toxicity and the interaction of hazardous substances should be used more often. Since the using of bio tests in monitoring has not been yet validated by legislation, introduction of the relevant amendments should be considered. It is important to use both acute and chronic tests as they describe the concentration of hazardous substances differently. Which bio tests should be used in which gauge sections needs additional research and getting acquainted with the experience of other countries. There are lots of possibilities and enough standard methods, just the most suitable options for Estonia have to be used.

In the national monitoring programme the assessment of chemical condition of water bodies should be based on toxicity of hazardous substances, determined with the help of chronic toxicity tests. These tests allow assessing the interactions and long-term changes in water bodies caused by hazardous substances. The most important are the rivers that flow into the sea and the border water bodies, Lake Peipus (border lake) and the coastal sea to keep an eye on the flowing of hazardous substances out from Estonia. The sediment that absorbs hazardous substances should also be tested. If the chronic toxicity test shows toxicity the chemical analysis of sediment and biota should be conducted to find out the substances / substance groups that cause this toxicity, making simplified source analysis to find out the possible cause of the waste. The chronic toxicity tests should be conducted as circular monitoring with a three-year cycle.

Bio tests for assessing the acute toxicity are usable as obligatory surveillance monitoring for the enterprises that use hazardous substances, for the waste water treatment plants of cities, and landfills, including closed ones. Monitoring of hazardous substances in effluent is part of the self-monitoring of an enterprise, implementing the principle " the polluter pays" more effectively.

The surveillance monitoring plan should include the testing of the toxicity of surface waters with the help of bio tests in those surface water bodies where the effluent of cities/settlements is discharged: more than 10,000 – bio test of effluent and sediment of the receiving water body at least four times per year; and more than 2,000 – bio test of sediment once/twice a year. When determining the sampling point, the impact of the enterprise to the receiving water body should be considered in addition, i.e. to test farther than at the end of the pipe taking into account the settling of hazardous substances into the sediment, and also the forward motion of that sediment.

If the sediment shows acute toxicity, a further survey of substances should be conducted, i.e. a full analysis of the hazardous substances should be made as part of the operational monitoring. Further investigation of substances detected by the sediment analysis, based on source survey would be the research monitoring.

The surveillance monitoring of the receiving water bodies conducted by the Environmental Board should involve the monitoring of hazardous substances. If no hazardous substances have been detected during earlier surveys, bio tests will be sufficient. If any of the substances has been detected it should be also quantified.

Among the new (not surveyed earlier in Estonia) substances relatively big volumes of organotin compounds, alkylphenols and their ethoxylates, phthalates and polyaromatic hydrocarbons were detected in different sample matrices. When updating the National Environmental Monitoring Programme, inclusion of some new compounds like organotin compounds, alkylphenols and their ethoxylates, phthalates, polyaromatic hydrocarbons, and polybrominated diphenyl ethers into the monitoring programme should be considered.

During the screening of BaltActHaz project very high concentrations of organotin compounds were detected both in the water and sediments of Tallinn Bay near the dockyard of BLRT Grupp AS. Organotin compounds, including tributyltin (TBT) should definitely be included in enterprise monitoring programme (monitoring of fish and sediments) both near to the dockyard of BLRT Grupp AS, as well as port areas (Tallinn Bay, Kunda Bay, the coast at Sillamäe). TBT has not been monitored yet in the biota of Tallinn Bay and the coast at Sillamäe.

For using the waste water sediment in agriculture, greenery and recultivation extra care must be taken, as in addition to the concentrations of earlier detected, quantified and legally regulated heavy metals, it may contain such hazardous substances as organotin compounds, alkylphenols and their ethoxylates, phthalates and decabromodiphenyl ether. In the future, an analysis of the afore-mentioned compounds should be made before using the waste water sediment in agriculture and greenery. The results of the analysis will show if the waste water sediment can be used, or it should be treated as hazardous waste.

Further surveys are needed to inspect the occurrence of some hazardous substances in the surface water that have not been detected in Estonia previously. If the occurrence of these substances in the surface water is further confirmed they must be included in the monitoring programme. The results of the work point out the need to conduct more thorough surveys to find out the pollution sources and the behaviour of some hazardous contaminants in the waste water treatment plants and the treatment processes.

The results of screening and source surveys of LIFE BaltActHaz project should henceforth be used as the basis for planning and conducting of surveillance, operational and research monitoring, whereby first and foremost for monitoring of those priority substances, priority hazardous substances and certain other contaminants in the natural environment, the concentrations of which in the water exceed the valid maximum allowed limit value of the quality of the environment both in land and other surface waters (MAC-EQS). As for the other compounds, a random inspection should be conducted once in two or three years, or when new hazardous compounds which endanger peoples' health have been detected in the natural environment.

When taking samples the high-water season should be avoided when the dilution is at its maximum. Samples should be taken during the low-water period of surface water – in summer.

Many legislative acts stipulate the obligation of enterprises to monitor the hazardous substances, which should also be reflected on the enterprise's environmental permit. However, as often as not the corresponding sections are missing due to the unawareness of both the issuing authority and the applicant. That is why it is very important to have a consistent training, to foster the awareness of the permit issuing authorities and the enterprises / applicants. One possible way of improvement would be the issue of guidelines for the scope of application and the environmental impact assessment of hazardous substances. This would facilitate the work of both sides and would help to find in the long list of hazardous substances exactly those substances which are involved in the work of a particular enterprise, and find out the eventual need of their monitoring.

For the elaboration of the national monitoring system the following amendments should be made in the near future:

The enhancement of capability of the Estonian laboratories regarding the instrumentation:

Lack of the sufficiently sensitive analysis equipment does not allow in Estonia to quantify all the priority substances with the limit of quantification in accordance with the valid requirements (2000/60/EC, 2008/105/EC, 2009/90 and 166/2006). The chemical analysis instrumentation base of the laboratories participating in the Estonian environmental monitoring will be supplemented with new devices and new analytical methods in the near future, so that all necessary analysis of hazardous substances could be conducted in Estonia. All hazardous substances which cannot be quantified in Estonia so far, and which endanger the health of people must become quantifiable in Estonia.

Requirements for the laboratories participating in the National Environmental Monitoring Programme:

When selecting the monitoring laboratories in the course of performing the National Monitoring Programme it is necessary to ensure that the selected laboratory fulfils the quality requirements for sample analysis (standard EVS-EN ISO/IEC 17025), laid down by the Regulation of the Minister of the Environment No. 57 from 25th of August 2011 "Requirements for a testing laboratory conducting surveys on physico-chemical and chemical parameters of water, for analyses conducted in the framework of those surveys ,and for ensuring the quality of operation of the testing laboratory, and the reference methods of analysis". It is important to observe the accredited laboratories and methods also when taking samples, as in the case of hazardous substances there are

different matrices and very low concentrations which make the accurate sample taking especially important

Using the results of screening of BaltActHaz project for the elaboration of the environmental monitoring programme:

Organotin compounds were surveyed in the waters of only three rivers (Narva, Keila and Kasari), the limit of quantification was exceeded by the concentration of mono- and dibutyltin in those rivers. The concentrations of organotin compounds in the surface water sediments of all 11 rivers remained below the limit of quantification of the used analytical method. From now on organotin compounds should be quantified in the framework of enterprise monitoring in the port areas of the coastal sea where the high concentrations of organotin compounds, especially mono-, di- and tributyltin were detected in the course of screening. Additional help should be obtained from the analysis of biota (the Baltic herring and the bass). The latter feeds both in the coastal sea and in the estuaries. The concentrations of organotin compounds in the sediments of the waste water treatment plants, detected in the course of screening to exceed the limits of quantification of the used analytical method indicate that the monitoring of organotin compounds should continue. Several organotin compounds were detected – in addition to mono-, di- and tributyltin also mono- and dioctyltin. Judging by the results of the given screening in-depth surveys should be conducted in the neighbourhood of the dockyard of BLRT Grupp, and in the water areas of ports in general, and in case of need measures should be envisaged. Hereby, the participation of the BLRT Grupp in financing such surveys and taking measures would be normal.

As for phthalates, the concentrations of diisobutylphthalate, di(2-ethylhexyl)phthalate and dimethylphthalate should be randomly monitored once every two or three years in 7 rivers of the 11 surveyed: the rivers Kohtla-Järve, Kunda, Vääna, Jägala, Vasalemma, Kasari and Pärnu. In addition to the above mentioned substances, dibutylphthalate and diisononylphthalate were detected in the waste water sediments.

Alkylphenols and their ethoxylates, and polybrominated biphenyls were detected only in the waste water sediments of the waste water treatment plants.

In the case of the waste water sediments as waste, an important aspect is the possibility of its recycling, i.e. leading it back to the nature or soil. Right now this is regulated by the European Union directive from 1986 and a corresponding regulation of the Minister of the Environment. Unfortunately they both are utterly behind the times and lay down the limit value only for the concentration of seven heavy metals in the waste water sediment (cadmium, copper, nickel, lead, zinc, mercury and chromium). The regulation should be amended in the near future. All organic contaminants are not regulated, as well as for example tin and all organotin compounds among heavy metals, which unfortunately often occur and in remarkable quantities in certain waste water sediments. Observing formalities, the mentioned seven metals would allow the sediment, under certain circumstances, to be outspread in the field or used in greenery planting. On the other hand, the large quantities of the other hazardous contaminants in the sediments, identified in the course of screening indicate that the regulation of the Minister of the Environment should be amended by adding limit values for new hazardous substances.

6. Management of emerging substances

6.1. Lists of emerging substances of concern in EU

Potential lists of emerging substances to analyse: **Annex III of 2008/105/EC:**

- AMPA
- Bentazon
- Bisphenol-A
- Dicofol
- EDTA
- Glyphosate
- Mecoprop (MCPP)
- Musk xylene
- Perfluorooctane sulphonic acid (PFOS)
- Quinoxyfen (5,7-dichloro-4-(p-fluorophenoxy)quinoline)

Substances proposed for EQS derivation (Draft substance impact reports March 2011: http://circa.europa.eu/Public/irc/env/wfd/library?l=/framework_directive/thematic_documents/priority_substances/supporting_substances/substance_impacts_2011&vm=detailed&sb=Title)

- Bifenox
- Terbutryn
- Cybutryne (Irgarol)
- Cypermethrin
- Dichlorvos
- Heptachlor epoxide
- PFOS and perfluorooctane sulfonic fluoride
- HBCDD
- Quinoxyfen
- Dicofol
- Aclonifen
- Diclofenac
- 17 alpha-ethibylestradiol
- Ibuprofen
- 17 beta-estradiol

NORMAN (Network of reference laboratories for monitoring of emerging environmental pollutants) list of emerging substances: http://www.norman-network.net/index_php.php?interface=1024&module=public/about_us/emerging&lang=en

6.2. Screening survey as a tool to identify emerging substances and an input to the monitoring programme

Many of the chemical substances found in society at large end up in sewers and treatment plants. The amounts of some substances are regularly monitored within the obligatory inspection programmes. However the numerous organic contaminants, on the other hand, are not analysed regularly because doing so would be both difficult and costly. In addition, new chemical substances are appearing all the time. Therefore a special programme with campaign-type sampling and analysis of new environmental pollutants and pharmaceutical residues, in particular, could be applied. This 'screening programme' makes it possible to carry out spot checks to see how far these substances occur in the environment, what their sources are and whether human beings are at risk of exposure to them. **In many countries screening programme is a central part of environmental monitoring programme for toxic pollutants**. The number of samples are limited in both space and time, why the results may be regarded as a first investigation of the release and environmental occurrence of a certain pollutant. Sludge, sediments and wastewater from industry and MWTPs are usually sampled, since they collect pollutants from many sources, but also water, air or fish could be analysed.

Objectives of the screening:

- Identify EU/WFD prioritized and nationally selected organic pollutants in aquatic environments near sources of discharge,
- Provide information to source identification – but not single pollution sources (impact monitoring, compliance checking => enterprises),
- To develop best practices, analytical methods and cooperation between laboratories,
- To identify substances which should enter the regular national monitoring networks.

Setting the screening survey

A proper screening study consists of 4 major closely related parts: substance selection, preliminary theoretical research, measurement study and survey follow-up.

1.Substance selection

- As early as the selection stage, consideration should be given to the question of **for what purpose the results will be used**.
  - Usually it is not primarily intended to support research on new environmental pollutants, but, rather, constitutes compliance with the requirements to report certain substances laid down in a number of EU directives and international conventions.
  - Many of such substances are of no relevance to country and screening in these cases might be a useful instrument in demonstrating this, so to avoid investing major resources into measuring them.
  - Another purpose is to follow up work in order to achieve a pollutant-free environment. Substances found in high concentrations can be followed up over a lengthy period of time.
  - Another one of the reasons for selecting a certain substance might be to use the results in order to plug data holes for the purposes of risk assessment or justifying a risk-reducing measure.
- Chemicals/ products registers can be also used for selection of substances, i.e. from among chemicals used in large volumes.

2. Preliminary theoretical research

- Certain information about the **properties, use and dispersal** of a substance is required in order to set up sampling and analysis.
- Theoretical research should also collect data on the **different types of impacts** the substance produces. That way, when performing the risk assessment, it can decided whether the concentrations being measured necessitate action or not.
- On the basis of substance properties, it can also be possible, for example, to compare concentrations to those of other known substances or groups of substances, such as PCB, in order to obtain an "estimated screening reference value", against which to make a comparison when a concentration is high.

3. Measurement study

- On the basis of what is known about the properties of substances and the information available on their dispersal conditions and routes, a type of matrix (i.e. the medium being analysed, such as water, sludge, air or fish), as well as a sampling strategy should be chosen. The objective of the survey and the reason why we have selected one substance in particular are relevant to the choice of location and matrix.
- When selecting samples, balance of choice between mapping as many different matrices as possible, disseminating measurement data across the country or detecting statistical differences should be considered. In this respect, too, the choice of substance informs the decision as to which substance should be given priority in a given study.
- There are many challenges in analytical development during screening studies. It is better to try to select substances for which analytical methods exist. Screening is not intended for the purpose of developing analytical methods, but since measurement studies often require new substances to be analysed, it can be more or less necessary to develop the analytical method partly (e.g. pre-treatment part different matrix, complex samples).
- Evaluation stage is very important in the measurement study step. What do the results actually tell? Are values high or low, based on the available knowledge of impacts? How accurate are values arrived at through analyses conducted in other countries and other measurements? Evaluation is performed on the basis of literature review and the rationale for the choice of substance to be submitted for screening. It is also important that all the information on samples, matrices, localities and methods considered relevant for future use is well-documented in the report.

4. Survey follow-up

- Evaluation is an important part of the screening survey. Can the screening be considered final or further data is needed to be able to draw conclusions and answer questions raised when selecting the substance? Whether obtained results are of a sufficient quantity and good quality.
- Drawing the ideas for actions, measures is an important follow-up step. There are a variety of measures, e.g. influence the choice of substance by companies (voluntary agreements), to include substance for a new screening e.g. in 5 years in order to delineate a trend, to include substance into regular monitoring programme etc.
- Disseminating the results is also important to draw up some proposals for further actions, therefore it should be decided to whom should the proposals put forward be addressed.
- The data should be easily available for all interested parties. For example in Sweden having long tradition of screening studies, the data host for screening is IVL, Swedish Environmental Research Institute Ltd. Data and reports can be downloaded from the following website of the screening data host: www.ivl.se

6.3. Ideas and proposals for screening surveys in Estonia

Screening surveys should be regarded as research monitoring, and here the pollution related to oil shale should be surveyed more closely – surveys of hazardous substances with reference to oil shale mining, energy and chemical industry should be conducted. The oil shale related waste forms the major part of our hazardous waste. It is difficult to find data even about the concentration of heavy metals in oil shale, let alone the concentration of other substances in the waste from the oil shale chemical industry, first of all of persistent organic compounds which form during such thermal processes. The latest fundamental surveys related to oil shale were conducted a long time ago, and in the meanwhile the capability of laboratories, as well as the scientific and technical level and their application possibilities have improved. As the relevant surveys are expensive, appropriate foundations for funding this work should be found if possible, but also the Estonian government, however, should find the possibility to share the costs.

Another essential approach to screening surveys would be a source-based monitoring – as the possible origin of hazardous substance / substance group is known a list of the respective enterprises should be prepared based on this, and a source-based survey conducted in co-operation with those enterprises. As an essential part of hazardous substances come from diffuse sources is it important to conduct more screening surveys also in closed landfills.

Although the majority of hazardous substances do not form the part of the natural cycle but are man-made chemicals, there are also some hazardous substances or their compounds (first of all heavy metals) which participate in the natural substance circulation. This means that these substances also have a natural background level. These values are low but we lack for relevant surveys. The same problem applies to the surveys of chemical composition of rainfall which may contribute as the natural background a substantial part for the formation of the concentration in the effluent of an enterprise. In earlier years it was not possible due to the incapacity of the laboratories but in the recent years new devices have been procured and the limit of quantification has been lowered. In order to find out the natural background level in our water bodies, and on the basis of that to assess later its contribution to the effluent, in-depth surveys for determination of natural background level of heavy metals and other naturally occurring hazardous substances should be conducted, including also different regions.

Annex III of Directive 2008/105/EC of the European Parliament and of the Council on quality of the environment standards in the field of water policy, amending and subsequently repealing Council Directives 82/176/EEC, 83/513/EEC, 84/156/EEC, 84/491/EEC and 86/280/EEC and amending Directive 2000/60/EC, contains the list of substances subject to review for possible identification as priority substances or priority hazardous substances (Table 6.5.1).

Table 6.3.1. Substances subject to review

CAS number	EU number	Substance Name
CAS number	EU number	**Substance Name**
1066-51-9	-	**AMPA**
25057-89-0	246-585-8	**Bentazone**
80-05-7		**Bisphenol A**
115-32-2	204-082-0	**Dicofol**
60-00-4	200-449-4	**EDTA**
57-12-5		**Cyanide**
1071-83-6	213-997-4	**Glyphosate**
7085-19-0	**230-386-8**	**Mecoprop (MCPP)**
81-15-2	**201-329-4**	**Musk xylene**
1763-23-1		**Perfluorooctanesulphonate (PFOS)**
124495-18-7	-	**Quinoxyfen**
		Dioxins
		Polychlorinated biphenyls

In the framework of LIFE + BaltActHaz project the concentrations of AMPA, Glyphosate, Mecoprop (MCPP) in eight water samples from agricultural area of Estonia, and the concentration of PFOS in the surface waters were surveyed. The concentrations of all compounds remained below the limit of quantification of the used analytical method.

As a part of the pan-European project "EU Wide Monitoring Survey of Polar Persistent Pollutants in European River Waters" the concentrations of the above-mentioned Bisphenol A, Mecoprop and PFOS were quantified in the waters of three Estonian rivers: Narva, Purtse and Emajõgi. All concentrations of Bisphenol A and Mecoprop remained below the quantification limit of the used analytical method. The concentrations of PFOS in the water sample from the Purtse river remained below the quantification limit of the used analytical method; small concentration of 1 ng/l was quantified in the waters of the rivers Narva and Emajõgi [4.3].

The concentrations of dioxins and polychlorinated biphenyls have been surveyed in Estonian foodstuff (including the fish from the Baltic Sea and Lake Peipus) since 2002 on the initiative of The Ministry of Agriculture (http://www.agri.ee/uuringud-statistika/) and the Veterinary and Food Board (http://www.vet.agri.ee/?op=body&id=821). Polychlorinated biphenyls have been surveyed both in the water samples and sediments, as well as in biota on the initiative of The Ministry of the Environment [4.10]; since 1974 also in the fish and the molluscs of the Baltic Sea.

7. List of abbreviations

AA-EQS	annual average value of environmental quality standard in surface water
AMPA	aminomethyl phosphonic acid, degradation compound of a plant protection product glyphosate
COMBINE	Cooperative Monitoring in the Baltic Marine Environment
DEHP	di(2-ethylhexyl)phthalate
EERC	Estonian Environmental Research Centre
EU	the European Union
HBCDD	hexabromocyclododecane
HCB	hexachlorobenzene
HCH	hexachlorocyclohexane
HELCOM	the Helsinki Commission
LOD	limit of detection
LOQ	limit of quantification
MAC-EQS	maximum allowable concentration of environmental quality standard in surface water
MCCP	medium-chain chlorinated paraffins
MCPP	mecoprop-p (plant protection product)
MU	measurement uncertainty
PAH	polycyclic aromatic hydrocarbons
PBDE	polbrominated diphenylethers
PFOS	perfluorooctane sulfonate
SCCP	short-chain chlorinated paraffins
TBT	tributyltin
TRI	trichloroethylene
WFD	the European Union Water Framework Directive (2000/60/EC)

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