



Qualitative review report describing the sediment laboratory methods' current status and problems in the DTP Countries

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The WP4 - Activity 4.1 is a qualitative review based on WP3 Questionnaires. Within the review report, current status is described for existing laboratory practices and experiences, inventory of laboratory methodologies and quality controls in the DTP countries, including both EU and non-EU members for sediment quality measurements of the water, biota, bottom sediment, suspended sediment and floodplain sediment.

Qualitative review report was made by the following criteria: protocols should be acceptable in all DTP countries, in-line with the ICPDR and the EU requirements, use the latest scientific knowledge and have to be sustainable.

The review laboratory methods and procedures (norms) of HSs and its compounds include HSs analysis from agricultural activities and industrial activities, determination of heavy metals and quality control procedures.

The review laboratory methods for analysis of **HSs from agricultural activities** contain following 39 substances: *Alachlor, Atrazine, Chlorfenvinphos, Chlorpyrifos (Chlorpyrifos-ethyl), Cyclodiene pesticides, Total DDT, Diuron, Endosulfan, Hexachlorobenzene, Hexachlorociklohexene, Isoproturon, Pentachlorophenol, Simazine, Trifluralin, Dicofol, Quinoxifen, Aclonifen, Bifenox, Cybutryne, Cypermethrin, Dichlorvos, Heptachlor and heptachlor epoxide, Terbutryn, Perfluorooctane sulfonic acid and its derivatives (PFOS), Anthracene, Benzene, Fluoranthene, Hexachloro-butadiene, Naphthalene, Pentachlorobenzene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(g,h,i)-perylene, Indeno(1,2,3-cd)-pyrene, Tributyltin compounds (Tributyltin-cation), Dioxins and dioxin-like compounds, PCB (total) and PAH (total)* (Appendix 1).

The review of organic pollutants predominantly released from the **industrial activates** include 13 HSs: *Perfluorooctane sulfonic acid and its derivatives (PFOS), Anthracene, Benzene, Brominated diphenylether, Carbon-tetrachloride, C10-13 Chloroalkanes, 1,2-Dichloroethane, Dichloromethane, Di(2-ethylhexyl)-phthalate (DEHP), Fluoranthene, Hexachloro-butadiene, Naphthalene, Nonylphenol (4-Nonylphenol), Octylphenol ((4-(1,1',3,3'-tetramethylbutyl)-phenol)), Pentachlorobenzene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(g,h,i)-perylene, Indeno(1,2,3-cd)-pyrene, Tetrachloro-ethylene, Trichloro-ethylene, Tributyltin compounds (Tributyltin-cation), Trichloro-benzenes, Trichloro-methane, Dioxins and dioxin-like compounds and Hexabromocyclododecanes (HBCDD)* (Appendix 2).

The laboratory procedures and methods in the DTP countries concerning to HSs from **agricultural and industrial** activities are various. In Austria are in usage standardized Austrian norms: ÖNORM M 6616, DIN 38404, DIN EN ISO 17294, DIN 38409 and DIN EN ISO 10304 for all HSs. In Bulgaria are applied two standards VIM and/or ISO for the most substances. In Croatia are mentioned all compounds from agricultural and industrial activities as analyzed but there are no data regards norms for particular compound. Federation Bosnia and Herzegovina (B&H) is provided no data.

In Hungary are obtained EPA, ISO and MZS standards and GC-MS or GC-MSD (SIM) methods. In Moldova are standards EPA, SM GOST and ISO and methods GC, LC or GC-MS in use. Montenegro carried out analyzes of the HSs in certificated laboratories. In Republic of Srpska are in usage EPA and ISO standards.

In Romania are implemented only ISO standards and for majority of HSs the GC-ECD and purge-and-trap with thermal desorption methods. In Slovakia are almost all compounds nominated, but there are no norms for any particular compound. Applied methods are GC-ECD, HPLC-UV and GC-MS.

In Slovenia laboratory analyzes are managed in accordance with ISO / IEC 17025 and meet the minimum efficiency criteria for the analytical methods set out in Article 16 of the Surface Water Surveillance Regulation. In Ukraine are the laboratory procedures and methods set by the two standards DSTU ISO 6468-2002 and DSTU ISO 17993: 2008.

The review of the laboratory methods and procedures (norms) for determination of **heavy metals and its compounds** includes following metals: *Cadmium and its compounds, Lead and its compounds, Mercury and its compounds, Nickel and its compounds, Arsenic and its compounds, Zinc and its compounds, Chromium and its compounds and Copper and its compounds* (Appendix 3).

In Austria is the norm DIN EN ISO 17294-2 and method ICP-MS 7500 (Agilent) in usage for heavy metals. Additionally, the XRF method is used for analyses of all metals with the exception of mercury. In Bulgaria laboratory analyses are doing in accordance with norm BSS EN ISO 17294/2004, only for mercury is another norm (VIM 1012/2010 (1 2.3.4)). The laboratory analyses for heavy metals in soils/ sediments are carried out according to ISO 11047. The methods ICP-MS and Flame AAS are used for the analyses of water.

In Croatia there are no data about norms, for methods is named ICP-MS. In Federation Bosnia and Herzegovina (B&H) are applied methods: for water USEPA-Method 245.1. and for sediments: EPA Method 7473, U.S. EPA Method 245.5(CVAAS), ASTM D6722 -thermal decomposition and ASTM D6414-99 (wet digestion). In Hungary is used the standard for sediment: MSZ 21470-50:2006 and methods AAS ETA and ICP-OES.

In Moldova are implemented the following norms: SM SR ISO 11047:2006, SM GOST R 51309:2006, SR EN ISO 5961:2002, SM SR ISO 8288:2006, SR EN 1233:2003 and SR ISO 9174-98. The methods in use are: for soil/sediments THGA-AAS and Flame AAS, for water THGA-AAS and Flame AAS. In Montenegro are employed standards: national for water/sediments EPA7473 and international for water: USEPA-Method 245.1.; for sediments: EPA Method 7473, U.S. EPA Method 245.5(CVAAS), ASTM D6722 - thermal decomposition and ASTM D6414-99 (wet digestion).

In Republic of Srpska (B&H) is used Standard Methods 3113-B, issued by APHA-AWWA-WEF 2005 and method Shimadzu FAAS AA6300. In Romania is mostly applied standards for solids: ISO 11047:1998 and for water: ISO 8288:1986 and usage of method flame and electrothermal atomic absorption spectrometric. Slovakia is provided no data of the norms. Regarding methods, in the most cases is performed for water: ICP-MS Agilent/ Brucker and for solid: ICP-MS Agilent/ Brucker and XRF. In Slovenia is for the analysis of water, sediment and biota samples, standardized analysis methods validated and documented in accordance with ISO / IEC 17025 and meet the minimum efficiency criteria for the analytical methods set out in Article 16 of the Surface Water Surveillance Regulation. In Ukraine are utilized norms DSTU ISO 15586: 2012; ISO 15586: 2003, IDT and methods AAS-ETA; AAS-Flame and Spectrophotometric.

In general, in Austria, Bulgaria, Moldova, Romania, Slovenia and Ukraine are adopted ISO standards for laboratory analyses. The most frequent method for determination of heavy metals is ICP-MS, then ICP-OES and rare XRF, in Moldova is common use of the THGA-AAS and Flame AAS. There are usually different norm and method for determination mercury and in some cases chromium from applied norms and methods for determination other heavy metals. Croatia, Federation of Bosnia and Herzegovina (B&H), Hungary, Montenegro, Republic of Srpska (B&H) and Slovakia were provided no data of the standards for the majority of heavy metals.

The review of **Quality Assurance / Quality control** procedures includes checking accuracy and precision protocols in the DTP countries.

In Bulgaria, Croatia, Hungary, Republic of Srpska (B&H) and Slovenia is adopted standard ISO / IEC 17025.

In Moldova are applied ten standards or procedures for control of accuracy and precision:

1. SM SR ISO 5725:1-2002. Exactitatea (justețea și fidelitatea) metodelor de măsurare și a rezultatelor măsurărilor. Partea 1. Principii generale și definiții;
2. ISO 8466:1990 Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics;
3. ISO 8466:2001 Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second order calibration functions;
4. ISO 11843-2:2000 Capability of detection — Part 2: Methodology in the linear calibration case;
5. EURACHEM. Quantifying Uncertainty in Analytical Measurement. LGC, 1995. ISBN 0-948926-08-2;
6. Mandel J., The statistical analysis of experimental data, Interscience Publ., J. Wiley & Sons,(1964), New York;
7. 2007 ALACC Guide "How to meet ISO 17025 requirements for method verification";
8. Test methodic validation. CTB 1436-2004. Gosstandard, Minsk, 2004 (in Russian);
9. K. Doerfeli. The statistics in analytical chemistry. "Mir", 1969 (in Russian);
10. A.G. Orlov. Calculation methods in quantitative spectral analysis."Nedra", 1977 (in Russian).

In Ukraine is in usage standard JMA 73.1-41-08.00.01: 2004. Slovakia does not provide any standard but description of the quality control procedures. Accuracy was expressed by average yield for individual analytes of ten repeated measurements of two calibration points. Accuracy represents the average yield calculated from the individual measurements by comparing the average of the measured values with the reference value. Precision of the calibration points for each analyte was expressed by the standard deviation under the repeatability conditions, respectively relative standard deviation of repeatability.

Austria, Federation Bosnia and Herzegovina (B&H), Montenegro and Romania are given no data.

The qualitative review report based on WP3 Questionnaires was made in the frame of the WP4 - Activity 4.1 This report will serve as one of the factor for WP4 Activity 4.2 – Development of the proposal of harmonized sediment sampling and laboratory analysis protocols for HSs in DTP Countries.

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Appendix 2

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Country	Austria	Bulgaria	Croatia	Federation BH (B&H)	Hungary	Moldova	Montenegro	Republic of Srpska (B&H)	Romania	Slovakia	Slovenia	Ukraine			
	Hss from industrial activities	Procedures(norms)	Methods	Procedures(norms)	Methods	Procedures(norms)	Methods	Procedures(norms)	Methods	Procedures(norms)	Methods	Procedures(norms)			
1.	Brominated diphenylether		Not analyzed												
2.	Carbon-tetrachloride		BSS EN ISO 15680			MSZ 1484-7-2009 MSZ 1484-4:1998 (visszavont szabány)2	GC-FID	EPA Method 524.3	GC-MS		ISO 15009:2016	Purge-and-trap method with thermal desorption			
3.	C10-13 Chloroalkanes		Not analyzed			MSZ 1484-7-2009 MSZ 1484-4:1998 (visszavont szabány)2	GC-FID				ISO 16558-1:2015	gas chromatography (static headspace method) GC-FID			
4.	1,2-Dichloroethane		BSS EN ISO 15680			MSZ 1484-5-1998 3.2. szakasz	HS-GC-ECD HS-GC/MSD (SIM)	EPA Method 524.3	GC-MS	StaticHeadspace/GC/Theory &Practice	ISO 15009:2016	Purge-and-trap method with thermal desorption			
5.	Dichloromethane		BSS EN ISO 15680			MSZ 1484-5-1998 3.2. szakasz	HS-GC-ECD	EPA Method 524.3	GC-MS	StaticHeadspace/GC/Theory &Practice	ISO 15009:2016	Purge-and-trap method with thermal desorption			
6.	Di(2-ethylhexyl)-phthalate (DEHP)	Hydrochemical analyses are standardized by Austrian norms: ÖNORM M 6616, DIN 38404, DIN EN ISO 17294, DIN 38409, DIN EN ISO 10304	Not analyzed			All compounds are mentioned, but there are no norms for particular compound.	GC-MS, GC-MS/MS and LC-MS/MS.	No data.		Hazardous substances are analyzed in external laboratories as follows: 1. Institute of Hydrometeorology and seismology of Montenegro, http://www.meteo.co.me/ekologija/Akreditacija.pdf 2. Institute for Public Health from Podgorica, 3. Center for Eco-Toxicology Research from Podgorica, http://eng.ceti.me/?page_id=3610	EPA 525.2:1994	ISO 13913:2014	GC-MS	Almost all compounds are mentioned, but there are no norms for particular compound.	For the analysis of water, sediment and biota samples, standardized analysis methods are validated and documented in accordance with ISO / IEC 17025 and meet the minimum efficiency criteria for the analytical methods set out in Article 16 of the Surface Water Surveillance Regulation. MM (measurement methodology) 99-12-98 (petroleum products); DSTU ISO 7875-1: 2012,
7.	Nonylphenol (4-Nonylphenol)		BSS EN ISO 18857-1						EPA 8040	ISO/TS 13907:2012	GC-MS				
8.	Octylphenol ((4-(1,1',3,3'-tetramethylbutyl)-phenol))		BSS EN ISO 18857-1						EPA 8040	ISO/TS 13907:2012	GC-MS				
9.	Tetrachloro-ethylene		BSS EN ISO 15680			MSZ 1484-5-1998 3.2. szakasz	HS-GC-ECD	EPA Method 524.3	GC-MS		ISO 15009:2016	Purge-and-trap method with thermal desorption			
10.	Trichloro-ethylene		BSS EN ISO 15680			MSZ 1484-5-1998 3.2. szakasz	HS-GC-ECD	EPA Method 524.3	GC-MS		ISO 15009:2016	Purge-and-trap method with thermal desorption			
11.	Trichloro-benzenes		BSS EN ISO 15680					EPA Method 8015C	GC		ISO 6468:1996	Gas chromatographic method after liquid-liquid extraction			
12.	Trichloro-methane		BSS EN ISO 15680			MSZ 1484-5-1998 (visszavont szabány)2 3.2. szakasz	HS-GC/ECD	EPA Method 524.3	GC-MS	StaticHeadspace/GC/Theory &Practice	ISO 15009:2016	Purge-and-trap method with thermal desorption			
13.	Hexabromocyclododecanes (HBCDD)														

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	Country / Heavy metals	Cadmium (Cd) and its compounds	Lead (Pb) and its compounds	Mercury (Hg) and its compounds	Nickel (Ni) and its compounds	Arsenic (As) and its compounds	Zinc (Zn) and its compounds	Chromium (Cr) and its compounds	Copper (Cu) and its compounds						
		Procedures(norms)	Methods	Procedures(norms)	Methods	Procedures(norms)	Methods	Procedures(norms)	Methods						
1.	Austria	DIN EN ISO 17294-2 XRF	ICP-MS 7500 (Agilent) XRF	DIN EN ISO 17294-2 XRF	ICP-MS 7500 (Agilent) XRF	DIN EN ISO 17294-2 XRF	ICP-MS 7500 (Agilent) XRF	DIN EN ISO 17294-2 XRF	ICP-MS 7500 (Agilent) XRF						
2.	Bulgaria	BSS EN ISO 17294/2004 BSS EN ISO 17294/2004	ICP-MS (water) Flame AAS (water)	BSS EN ISO 17294/2004 BSS EN ISO 17294/2004	ICP-MS (water) Flame AAS (water)	VIM 1012/2010 (1.2.3.4) VIM 1012/2010 (1.2.3.4)	ICP-MS (water) ICP-MS (water)	BSS EN ISO 17294/2004 BSS EN ISO 17294/2004	ICP-MS (water) Flame AAS (water)						
3.	Croatia		ICP-MS (water) ICP-MS (sediment)		ICP-MS (water) ICP-MS (sediment)	EPA Method 245.7- Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry	Atomic Fluorescence Spectrometry		ICP-MS (water) ICP-MS (sediment)						
4.	Federation BH (B&H)					Water: USEPA-Method 245.1. Sediments: EPA Method 7473 -U.S. EPA Method 245.5(CVAAS) -ASTM D6722 -thermal decomposition * -ASTM D6414-99 (wet digestion)									
5.	Hungary	Sediment: MSZ 21470-50:2006 4.2. szakasz 4.1. szakasz	Sediment: AAS ETA ICP-OES	Sediment: MSZ 21470-50:2006 4.2. szakasz 4.1. szakasz	Sediment: AAS láng ICP-OES	Sediment: MSZ 21470-50:2006 4.2. szakasz 4.1. szakasz	Sediment: AAS láng ICP-OES	Sediment: MSZ 21470-50:2006 4.2. szakasz 4.1. szakasz	Sediment: MSZ 18412: 2007 photometric method Sediment: MSZ 21470-50:2006 4.2. szakasz 4.1. szakasz						
6.	Moldova	SM SR ISO 11047:2006 SM GOST R 51309:2006 SR EN ISO 5961:2002 SM SR ISO 8288:2006 SR EN 1233:2003 SR ISO 9174-98	THGA-AAS (soil, sediments) Flame AAS (soil, sediments) THGA-AAS (water) SR EN ISO 11047:2006 SM GOST R 51309:2006 SR EN ISO 5961:2002 SM SR ISO 8288:2006 SR EN 1233:2003 SR ISO 9174-98	SM SR ISO 11047:2006 Flame AAS (soil, sediments) THGA-AAS (soil, sediments) Flame AAS (soil, sediments) THGA-AAS (water) SR EN ISO 5961:2002 ISO 20280:2007 (solid samples)	SM SR ISO 11047:2006 THGA-AAS (soil, sediments) Flame AAS (soil, sediments) SM GOST R 51309:2006 SR EN ISO 5961:2002 ISO 20280:2007 (solid samples)	SM GOST R 51309:2006 THGA-AAS (water) SR EN ISO 5961:2002 ISO 20280:2007 (solid samples)	SM GOST R 51309:2006 THGA-AAS (water) SR EN ISO 5961:2002 ISO 20280:2007 (solid samples)	SM SR ISO 11047:2006 Flame AAS (soil, sediments) SM GOST R 51309:2006 THGA-AAS (water) SR EN ISO 5961:2002 SM SR ISO 8288:2006 SR EN 1233:2003 SR ISO 9174-98	SM SR ISO 11047:2006 THGA-AAS (soil, sediments) Flame AAS (soil, sediments) SM GOST R 51309:2006 THGA-AAS (water) SR EN ISO 5961:2002 SM SR ISO 8288:2006 SR EN 1233:2003 SR ISO 9174-98	SM SR ISO 11047:2006 THGA-AAS (soil, sediments) Flame AAS (soil, sediments) SM GOST R 51309:2006 THGA-AAS (water) SR EN ISO 5961:2002 SM SR ISO 8288:2006 SR EN 1233:2003 SR ISO 9174-98					
7.	Montenegro					National: EPA7473 (water/sediments) International (water): USEPA-Method 245.1. Sediments: EPA Method 7473 -U.S. EPA Method 245.5(CVAAS) -ASTM D6722 -thermal decomposition * -ASTM D6414-99 (wet digestion)	direct mercury analyzer DMA								
		All water quality measurements are carried out in the Water Quality Laboratory, which is accredited for sampling and chemical analysis in accordance with MEST EN ISO / IEC 17025: 2011 (Institute for Hydrometeorology and Seismology of Montenegro). Dangerous substances are analyzed in external laboratories as follows: 1. Institute of Hydrometeorology and Seismology of Montenegro, http://www.meteo.co.me/ekologija/Akreditacija.pdf 2. Institute for Public Health from Podgorica, 3. Center for Eco-Toxicology Research from Podgorica, http://en.ceti.me/?page_id=3610													
8.	Republic of Srpska (B&H)	Standard Methods 3113-B, issued by APHA-AWWA-WEF 2005	Shimadzu FAAS AA6300	Standard Methods 3113-B, issued by APHA-AWWA-WEF 2005	Shimadzu FAAS AA6300	AMA 254, Advanced Mercury Analyser,Operating Manual	AMA 254	Standard Methods 3113-B, issued by APHA-AWWA-WEF 2005	Shimadzu FAAS AA6300	Standard Methods 3113-B, APHA-AWWA-WEF 2005	Shimadzu FAAS AA6300	BAS ISO 9174	Shimadzu FAAS AA6300	Standard Methods 3113-B, APHA-AWWA-WEF 2005	Shimadzu FAAS AA6300
9.	Romania	Solid (S): ISO 11047:1998 Water (W): ISO 5961:1994 ISO 8288:1986	Flame and electrothermal atomic absorption spectrometric methods Solid: XRF ICP-MS Agilent/ Brucker	S: ISO 11047:1998 W: ISO 8288:1986	Flame and electrothermal atomic absorption spectrometric methods W: ISO 12846:2012 ISO 17852:2006	S: ISO TS 16727:2013 ISO 16772:2004 W: ISO 12846:2012 ISO 17852:2006	Flame and electrothermal atomic absorption spectrometric methods W: ISO 8288:1986	S: ISO 11047:1998 W: ISO 17378-1:2014 ISO 17378-2:2014 ISO/TS 19620:2018 (HPL-ICP-MS or HG-AFS)	Flame and electrothermal atomic absorption spectrometric methods W: ISO 8288:1986	S: ISO 11047:1998 W: ISO 17378-1:2014 ISO 17378-2:2014 ISO/TS 19620:2018 (HPL-ICP-MS or HG-AFS)	Flame and electrothermal atomic absorption spectrometric methods W: ISO 8288:1986	S: ISO 11047:1998 W: ISO 17378-1:2014 ISO 17378-2:2014 ISO/TS 19620:2018 (HPL-ICP-MS or HG-AFS)	S: ISO 11047:1998 W: ISO 17378-1:2014 ISO 17378-2:2014 ISO/TS 19620:2018 (HPL-ICP-MS or HG-AFS)	Flame and electrothermal atomic absorption spectrometric methods W: ISO 8288:1986	S: ISO 11047:1998 W: ISO 17378-1:2014 ISO 17378-2:2014 ISO/TS 19620:2018 (HPL-ICP-MS or HG-AFS)
10.	Slovakia		Water: ICP-OES Agilent ICP-MS Agilent/ Brucker	Water: ICP-OES Agilent ICP-MS Agilent/ Brucker	Water: AAS AMA- 254	Water: ICP-OES Agilent ICP-MS Agilent/ Brucker	Water: AAS AMA- 254	Water: ICP-OES Agilent ICP-MS Agilent/ Brucker	Water: ICP-MS Agilent/ Brucker	Water: ICP-MS Agilent/ Brucker	Water: ICP-MS Agilent/ Brucker	Water: Cr ICP-OES Agilent ICP-MS Agilent/ Brucker Cr ⁶⁺ spectrophotometric method	Water: ICP-MS Agilent/ Brucker Cr ⁶⁺ spectrophotometric method	Water: ICP-MS Agilent/ Brucker Cr ⁶⁺ spectrophotometric method	
11.	Slovenia	For the analysis of water, sediment and biota samples, standardized analysis methods are validated and documented in accordance with ISO / IEC 17025 and meet the minimum efficiency criteria for the analytical methods set out in Article 16 of the Surface Water Surveillance Regulation.													
12.	Ukraine	DSTU ISO 15586: 2012; ISO 15586: 2003, IDT	AAS-ETA; AAS-Flame, Spectrophotometric	DSTU ISO 15586: 2012; ISO 15586: 2003, IDT	AAS-ETA; AAS-Flame, Spectrophotometric	DSTU ISO 16772: 2005 (ISO 16772: 2004, IDT)	AAS Coldvapors	DSTU ISO 15586: 2012; ISO 15586: 2003, IDT	AAS-ETA; AAS-Flame, Spectrophotometric	DSTU ISO 15586: 2012; ISO 15586: 2003, IDT	AAS-ETA; AAS-Flame, Spectrophotometric	DSTU ISO 15586: 2012; ISO 15586: 2003, IDT	AAS-ETA; AAS-Flame, Spectrophotometric	DSTU ISO 15586: 2012; ISO 15586: 2003, IDT	AAS-ETA; AAS-Flame, Spectrophotometric