Harmonization and Networking for contaminant assessment in the Ionian and Adriatic Seas

Protocol Review
Deliverable T1.1.1

Work Package T1 - Sharing best practices for transnational harmonization for EQSD implementation and link with UNEP/MAP MEDPOL Program and Offshore Protocol of Barcelona Convention

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1. Introduction

The deliverable T1.1.1 required defining a common protocol regarding the procedure of the analysis to evaluate the contamination of selected pollutants in water, sediment and biota according to EQSD requirements and definition.
A questionnaire was sent on 18th May 2018 to national institutes involved in the project with the following remarks for compilation:

1) Provide QA/QC data on contaminants that are actually monitored for research or standard monitoring program and not simply included in your laboratory catalogue of analytical analysis.
2) Whenever possible, provide information on single compound and not on group of compounds as the comparability analysis is more feasible on single compound.
3) Express your information with units that are specified, making conversion where necessary.
4) For pharmaceuticals, you will find 'Macrolides' and 'other', please specify, if possible, the single compound analyzed.
5) For any additional information use the last column "Remarks".
6) For any additional contaminant not listed, but that you consider relevant in research or standard monitoring program, use a new row under the last row.

2. Questionnaire

2.1. Structure of the Questionnaire

The questionnaire is subdivided in three different excel sheets according to the matrix monitored: water, sediment and biota.
For each matrix, a selected list of contaminants has been chosen after some preliminary discussions between partners, taking into account contaminants routinely monitored for WFD and MSFD assessment objectives and Environmental Impact Assessment (EIA) procedure for offshore platforms.
In particular, the following list has been selected with associated unit of measure for each matrix:

Seawater		
Group	**Examples**	**Units**
Pesticides and biocides	DDT	µg/l
	DDE	µg/l
	DDD	µg/l
	HCB	µg/l
Antifoulants	Tributyltin (TBT) (as cation)	µg/l
	Triphenyltin (TPhT) (as cation)	µg/l
Pharmaceuticals	Macrolides	µg/l
	other	µg/l
Heavy metals	Mercury	µg/l
	Cadmium	µg/l
	Lead	µg/l
Hydrocarbons	Anthracene	µg/l
	Fluoroanthene	µg/l
	Naphthalene	µg/l
	Acenaphthylene	µg/l
	Acenaphthene	µg/l
	Fluorene	µg/l
	Phenanthrene	µg/l
	Pyrene	µg/l
	Benzo(a)anthracene	µg/l
	Chrysene	µg/l
	Benzo(b)fluoranthene	µg/l
	Benzo(k)fluoranthene	µg/l
	Benzo(a)pyrene	µg/l
	Dibenzo(a,h)anthracene	µg/l
	Benzo(g,h,i)perylene	µg/l
	Indenopyrene	µg/l
	Total hydrocarbons	µg/l
	Aliphatic hydrocarbons C_6-C_{12}	µg/l
	Aliphatic hydrocarbons C_{10}-C_{40} (Specify range)	µg/l
	Benzene	µg/l
	Toluene	µg/l
	Ethylbenzene	µg/l
	o,m,p-Xylene	µg/l
Radionuclides	Cs^{137}, Pu^{239}	Bq/l
Physical parameters	Dissolved Oxygen	%
	Salinity	PSU
	Temperature	°C
	Transmittance	%
	Fluorescence	mg/m^3

Sediment		
Group	**Examples**	**Units (on dry weight)**
Pesticides and biocides	DDT	µg/kg
	DDE	µg/kg
	DDD	µg/kg
	HCB	µg/kg
Antifoulants	Tributyltin (TBT)	µg/kg (as cation)
	Triphenyltin (TPhT)	µg/kg (as cation)
Pharmaceuticals	Macrolides	µg/kg
	other	µg/kg
Heavy metals	Mercury	mg/kg
	Cadmium	mg/kg
	Lead	mg/kg
	Vanadium	mg/kg
	Chromium	mg/kg
	Barium	mg/kg
	Copper	mg/kg
	Iron	mg/kg
	Arsenic	mg/kg
	Zinc	mg/kg
	Nickel	mg/kg
	Aluminum	mg/kg
Hydrocarbons	Anthracene	µg/kg
	Fluoroanthene	µg/kg
	Naphthalene	µg/kg
	Acenaphthylene	µg/kg
	Acenaphthene	µg/kg
	Fluorene	µg/kg
	Phenanthrene	µg/kg
	Pyrene	µg/kg
	Benzo(a)anthracene	µg/kg
	Chrysene	µg/kg
	Benzo(b)fluoranthene	µg/kg
	Benzo(k)fluoranthene	µg/kg
	Benzo(a)pyrene	µg/kg
	Dibenzo(a,h)anthracene	µg/kg
	Benzo(g,h,i)perylene	µg/kg
	Indenopyrene	µg/kg
	Total hydrocarbons	µg/kg
	Aliphatic hydrocarbons C_6-C_{12}	µg/kg
	Aliphatic hydrocarbons C_{10}-C_{40} (Specify range)	µg/kg
	Benzene	µg/kg
	Toluene	µg/kg
	Ethylbenzene	µg/kg
	o,m,p-Xylene	µg/kg
Brominated compounds	Brominated diphenylether	µg/kg
Radionuclides	Cs^{137}, Pu^{239}	Bq/kg
Organic matter	Total organic carbon (TOC)	%
	Loss on ingnition (LOI)	%
Grain size analysis	Sand	%
	Silt	%
	Clay	%
Water	Water content	%

Biota		
Group	**Examples**	**Units**
Pesticides and biocides	DDT	µg/kg
	DDE	µg/kg
	DDD	µg/kg
	HCB	µg/kg
Antifoulants	Tributyltin (TBT)	µg/kg (as cation)
	Triphenyltin (TPhT)	µg/kg (as cation)
Pharmaceuticals	Macrolides	µg/kg
	other	µg/kg
Heavy metals	Mercury	mg/kg
	Cadmium	mg/kg
	Lead	mg/kg
	Vanadium	mg/kg
	Chromium	mg/kg
	Barium	mg/kg
	Copper	mg/kg
	Iron	mg/kg
	Arsenic	mg/kg
	Zinc	mg/kg
	Nickel	mg/kg
Hydrocarbons	Anthracene	µg/kg
	Fluoroanthene	µg/kg
	Naphthalene	µg/kg
	Acenaphthylene	µg/kg
	Acenaphthene	µg/kg
	Fluorene	µg/kg
	Phenanthrene	µg/kg
	Pyrene	µg/kg
	Benzo(a)anthracene	µg/kg
	Chrysene	µg/kg
	Benzo(b)fluoranthene	µg/kg
	Benzo(k)fluoranthene	µg/kg
	Benzo(a)pyrene	µg/kg
	Dibenzo(a,h)anthracene	µg/kg
	Benzo(g,h,i)perylene	µg/kg
	Indenopyrene	µg/kg
	Total hydrocarbons	µg/kg
	Aliphatic hydrocarbons C_6-C_{12}	µg/kg
	Aliphatic hydrocarbons C_{10}-C_{40} (Specify range)	µg/kg
	Benzene	µg/kg
	Toluene	µg/kg
	Ethylbenzene	µg/kg
	o,m,p-Xylene	µg/kg
Brominated compounds	Brominated diphenylethers	µg/kg
Lipid	Lipid content	%
Water	Water content	%

Information requested, common to all matrices, regarded: QA/QC analytical performance such as LOQ/LOD, accuracy and reproducibility, storage, method of analysis, use of reference material and participation to intercalibration round. For each matrix, specific information was requested (seawater: total, dissolved or particulate phase and filtration; sediment: grain size, type of sampler and thickness of sampled sediment; biota: dry/wet weight, species, tissue and replicates).
The following table summarizes information requested:

Seawater	Sediment	Biota
Sample type (total, dissolved or particulate phase)	Grain size (Unsieved; <63um;<2mm)	Dry weight or wet weight (specify)
Sampling method (ex: no filtration; filtration 0,2; 0,45; 0,7µm); Filter type (GF/F, etc…)	Sampling method (type of sampler, ex: box corer)	Species (ex: *Mytilus galloprovincialis* , etc..)
	Thickness of sampled sediment (cm)	Tissue (ex: muscle, liver, etc…)
		Number of replicates (1,2,3)
Storage (ex: method, temperature, addition of preservative etc..)	Storage (ex: method, temperature , etc…)	Storage (ex: method, temperature, etc..)
Method of Analysis	Method of Analysis	Method of Analysis
Limit of detection (LOD)	Limit of detection (LOD)	Limit of detection (LOD)
Limit of quantification (LOQ)	Limit of quantification (LOQ)	Limit of quantification (LOQ)
Reference Materials (ex: NRC-CNRC, etc..)	Reference Materials (ex: NRC-CNRC, etc…)	Reference Materials (ex: NRC-CNRC, etc.)
Intercalibration round (ex: Quasimeme, etc..)	Intercalibration round (ex: Quasimeme, etc..)	Intercalibration round (ex: Quasimeme, etc.)
Accuracy (% wrt certified value)	Accuracy (% wrt certified value)	Accuracy (% wrt certified value)
Reproducibility (RSD %)	Reproducibility (RSD %)	Reproducibility (RSD %)
Remarks	Remarks	Remarks

2.2. Analysis of results

The institutions that replied to questionnaire were:

- Hellenic Centre for Marine Research (HCMR), Greece
- Rudjer Boskovic Institute (RBI), Croatia
- University of Montenegro – Institute of Marine Biology (UoM-IMBK), Montenegro
- Slovenian Environment Agency (ARSO), Slovenia
- Agriculture University of Tirana (AUT), Albania
- Center for Ecotoxicological Research (CETI), Montenegro
- Italian National Institute for Environmental Protection and Research (ISPRA), Italy
- National Institute of Biology (NIB), Slovenia
- Institute of Oceanography and Fisheries (IOF), Croatia.

Different analytes were evaluated considering the water, sediment and biota matrices.

The QA-QC Questionnaires received from six countries (represented by nine Institutes) involved in the project HarmoNIA have been compared. The comparative analyzes of the questionnaires highlighted the results described below.

Notes regarding Attachements (1, 2, 3): In the tables, when N.A. cell is filled with X, means that for the given analyte no information has been provided.
Furthermore, when N.A. cell and Parameter cell are simultaneously empty means that no information was given only for this specific parameter.
By mistake, in the questionnaires related to the three matrices, the "naphtalene" parameter was repeated twice. HCMR Institute was the only one to introduce for this entry the different values regarding LOD, the accuracy and the reproducibility in the water matrix. In this case, the range of declared values was presented in the report.

WATER MATRIX

For the "WATER" matrix (Attachment 1) the following GENERAL OBSERVATIONS were made:

- The IOF-Croatia did not provide any information regarding measurements in the seawater matrix.
- For pesticides and biocides only CETI-Montenegro and ISPRA-Italy laboratory analyzed all required parameters, while AUT-Albania and RBI-Croatia reported only information for DDT. However, RBI-Croatia reported information for some additional parameters, in particular: cybutryne, terbutryn, endosulfan, hexacholorocyclohexan, penthachlorbenzene, brominated diphenylethers. ARSO-Slovenia reported information for this group of pollutants, but they considered p,p and o,p isomers for DDT and DDD, while for DDE only p,p isomer was considered. NIB-Slovenia did not provide any information on pesticides and biocides in water.
- Also for antifoulants CETI-Montenegro was the only laboratory to give information for all the required compounds, while AUT-Albania and RBI-Croatia filled only the TBT cell in the questionnaire. ARSO-Slovenia gave further information regarding dibutyltin (DBT) as an additional parameter. ISPRA-Italy presented only LOQ for TBT. Other laboratories did not present any information.
- Analysis of the concentrations of pharmaceutical products has not been carried out by any laboratory.
- UoM-IMBK-Montenegro and NIB-Slovenia laboratories did not reported the data for the required heavy metals, while HCMR-Greece did not reported information on mercury. RBI-Croatia reported additional information for zinc, copper and nickel.
- Considering hydrocarbons, only CETI-Montenegro and ISPRA-Italy laboratories reported data for all the required substances. UoM-IMBK-

Montenegro reported any information, RBI-Croatia and AUT-Albania analyzed only anthracene, fluoranthene and benzo(a)pyrene. HCMR-Greece analyzed almost all compounds, except for aliphatic hydrocarbons C_6-C_{12}, benzene, toluene, ethylbenzene, o,m,p xylene, while ARSO-Slovenia did not reported data for total hydrocarbons, aliphatic hydrocarbons C_6-C_{12}, ethylbenzene and o,m,p xylene. It must be taken into account that for xylene ARSO-Slovenia reported data for o and m,p isomers as additional parameters. Among all the required parameters, NIB-Slovenia reported only the information concerning the polycyclic hydrocarbons. This institute did not specify the sample type and expressed the results in ng/l instead of µg/l.

- None of the nine laboratories reported information about radionuclides.
- Only ISPRA-Italy analyzed all the required physical parameters, while HCMR-Greece, RBI-Croatia AUT-Albania and NIB-Slovenia did not reported any data. ARSO-Slovenia and both Montenegro laboratories analyzed only dissolved oxygen, salinity and temperature.
- Following additional parameters were added to the questionnaires: ISPRA-Italy reported alcohols, glycols, 1,2,4 trimethylbenzene, amines, glutaraldehyde, benzalkonium chloride; RBI-Croatia included cybutryn, terbutryn, endosulfan, hexacholorocyclohexan, pentachlorobenzene, brominated diphenylethers, nickel, copper and zinc; ARSO-Slovenia reported data regarding analysis of DDT (p,p), DDT (o,p), DDE (o,p), DDD (o,p), DDD (p,p), Dibutyltin (as cation), m,p-Xylen and o-Xylen.

Most of the laboratories reported the UNITS OF MEASUREMENT as indicated in the basic questionnaire. If expressed in other units than those indicated in the basic questionnaire, the results shown in Attachment 1 have been transformed into units of measure requested (µg/l), in order to facilitate comparison with other participants.

Regarding the indication of SAMPLE TYPE:

- Laboratories analyzed dissolved or total samples. In particular, for pesticides and biocides, antifoulants and hydrocarbons the laboratories analyzed total samples. NIB-Slovenia did not report any information regarding the sample type.
- For heavy metals RBI-Croatia, AUT-Albania and CETI-Montenegro considered total samples for mercury analysis, while ARSO-Slovenia reported results for dissolved samples. For other heavy metals, all the laboratories analyzed dissolved fraction except for CETI-Montenegro that provided results for total samples. ISPRA-Italy takes into account dissolved plus reactive particulate <0.4/0.45um.
- For physical parameters, only total samples were analyzed, except for UoM-IMBK-Montenegro that reported dissolved samples for dissolved oxygen.

- All the additional parameters reported by RBI-Croatia, ARSO-Slovenia and ISPRA-Italy, were analyzed in total samples except for nickel, copper and zinc analyses of RBI-Croatia in dissolved samples.

Considering SAMPLING METHOD:

- All laboratories used non-filtered samples for pesticides and biocides, antifoulants, and hydrocarbons, where reported.
- Filtered samples (at 0.45 µm) were indicated for heavy metals analyses. At this regards RBI-Croatia reported the use of cellulose acetate filter, while HCMR-Greece reported membrane filters. Only ISPRA-Italy took into account dissolved plus reactive particulate <0.4/0.45um.
- For physical parameters "no filtration" was indicated by CETI-Montenegro, while UoM-IMBK-Montenegro reported "*in situ measurement*".

Considering STORAGE, different details were reported in the questionnaire by the eight laboratories, which provided data.

- For pesticides and biocides only ARSO-Slovenia referred to SIST EN ISO 5667-3 for HCB, while RBI-Croatia, ISPRA-Italy and AUT-Albania reported storage at 4°C in dark condition for DDT. CETI-Montenegro reported storage conditions at 1-5 °C, without addition of preservation and the use of glass container.
- Considering antifoulants, in particular for TBT, RBI-Croatia and AUT-Albania reported acidification and storage at 4 °C in dark condition, ARSO-Slovenia reported the use of SIST EN ISO 5667-3 method. CETI-Montenegro reported storage conditions at 1-5 °C, without addition of preservation and the use of glass container for both antifoulants.
- For heavy metals HCMR-Greece reported the use of acidification with HNO_3 followed by an SPE preconcentration step, RBI-Croatia and AUT-Albania reported acidification and storage at 4 °C in dark condition, ARSO-Slovenia used SIST EN ISO 5667-3 method and CETI-Montenegro reported storage conditions at 1-5 °C, without addition of preservation and the use of glass container.
- Regarding hydrocarbons HCMR-Greece reported extraction with n-hexane onboard. RBI-Croatia, AUT-Albania and ISPRA-Italy reported storage at 4 °C in dark condition, while ARSO Slovenia used SIST EN ISO 5667-3 method. CETI-Montenegro reported storage conditions at 1-5 °C, “without addition of preservation” and the use of glass container except for monocycles, which were stored at 1-5 °C with the use of H_2SO_4 or HCl (pH=1-2) for preservation in glass container. Only for aliphatic hydrocarbons C_{10}-C_{40}, CETI-Montenegro reported the same condition with the use of H_2SO_4 or HCl (pH=1-2) for preservation in glass container, without indication about the storage temperature. NIB-Slovenia referred to store their samples at 5 °C in the dark.

- For physical parameters, UoM-IMBK-Montenegro indicated the use of B.O.D. bottles for Winkler method and storage in dark place for dissolved oxygen analyses, while CETI-Montenegro analysed physical parameters *in situ*.
- Regarding additional parameters, ISPRA-Italy reported the addition of NaCl for all analytes except for benzalkonium chloride, which was stored at 4 °C without preservation. RBI-Croatia stored at 4°C in the dark the samples for analysis of cybutryn, terbutryn, endosulfan, hexacholorocyclohexane, and penthachlorbenzene, while the acidification and 4 °C storage were used for remaining analytes. For additional parameters reported by ARSO-Slovenia, the SIST EN ISO 5667-3 method was employed.

The METHOD OF ANALYSIS varied among countries and contaminants.

- For pesticides, the use of the analytical technique was referred by RBI-Croatia (GC-MS), AUT-Albania (GC-MS) and ISPRA-Italy (HPLC-FLC), while CETI-Montenegro reported the use of EPA 8080 A. ARSO-Slovenia referred to the use of ISO 6468-modified method (GC-ECD) for HCB.
- For antifoulants, considering TBT analysis RBI-Croatia and AUT-Albania reported GC-ICP-MS, while ARSO-Slovenia reported ISO 17353-modified method (GC-ICP-MS). CETI-Montenegro reported the use of ISO 17353:2004 method for both compounds.
- Regarding heavy metals, in particular cadmium and lead, HCMR-Greece and ISPRA-Italy reported the use of ICP-MS technique, ARSO-Slovenia used ISO 17294-2:2003 (ICP-MS), RBI-Croatia and AUT-Albania used voltammetry, while CETI-Montenegro reported MEST EN ISO 17294-2:2013 method. Mercury was analyzed by CV-AAS in RBI-Croatia, AUT-Albania and ISPRA-Italy, while CETI-Montenegro employed Solid AAS-Mercury and ARSO-Slovenia referred to SIST EN ISO 17852 mod.: 2008 (AFS) method.
- Considering hydrocarbons, for polycyclic compounds HCMR-Greece, RBI-Croatia and AUT-Albania used GC-MS, while ARSO-Slovenia used ISO 17993-modified method (HPLC) and CETI-Montenegro EPA 8270C. ISPRA-Italy reported the use of HPLC-FLD. For total hydrocarbons, HCMR-Greece used GC-FID, CETI-Montenegro ISO 9377-2/EPA 5021A and ISPRA-Italy refers to GC-FID (ISO9377-2:2000). NIB-Slovenia indicates "UNEP, IOC, 1984", without indicating the specific number of the method. Regarding aliphatic hydrocarbons C_6-C_{12}, CETI-Montenegro refers to the EPA 5021A method, while ISPRA-Italy used GC-MS. In relation to aliphatic hydrocarbons C_{10}-C_{40}, HCMR-Greece and ISPRA-Italy reported the use of GC-FID, ARSO-Slovenia IM/GC/MS/ECNi-MS (GC/MS/NCI) and CETI-Montenegro EN ISO 9377-2. For monocyclic compounds ARSO-Slovenia referred to ISO 15680 (GC/MS/PT), CETI-Montenegro reported EPA 5021A and ISPRA-Italy used GC-MS (EPA5030C-2003 and EPA8260C-2006).
- Considering physical parameters, in particular dissolved oxygen, CETI-Montenegro reported polarographic method, ARSO-Slovenia used

multiparametric probe and calculation, UoM-IMBK-Montenegro reported the Winkler titration (Parsons T.R., Maita Y., Lalli C.M., 1985). Regarding salinity, CETI-Montenegro reported conductivity conversion, UoM-IMBK-Montenegro used CTD probe and ARSO-Slovenia used multiparametric probe or SIST EN 2788. For temperature, CETI-Montenegro reported thermistor method, UoM-IMBK-Montenegro used CTD probe, while ARSO-Slovenia made use of multiparametric probe or SIST DIN 38404-6 method. ISPRA-Italy analyzed all requested parameters by the use of CTD probe.

The LIMITS OF DETECTION (LOD) were not reported for all the compounds analyzed.

- The lowest LOD for pesticides and biocides was obtained by CETI-Montenegro laboratory (0.0005 µg/l w.w.), while ARSO-Slovenia reported only for HCB LOD of 0.001 µg/l. Furthermore, ARSO-Slovenia obtained LOD in the range 0.002 µg/l to 0.003 µg/l for DDT, DDD and DDE isomers that they reported as additional parameters indicating the different isomers analyzed.
- For antifoulants, the lowest limit was reported by ARSO-Slovenia for TBT (0.000049 µg/l), while CETI-Montenegro reported limits of 0.001 µg/l for both TBT and TPhT.
- Considering heavy metals, ARSO-Slovenia reached the best LOD for mercury (0.005 µg/l), followed by CETI-Montenegro (0.1 µg/l). For cadmium higher performance was obtained by HCMR-Greece (0.01 µg/l), followed by ARSO-Slovenia (0.02 µg/l) and CETI-Montenegro (1 µg/l). Also for lead higher performance were obtained by HCMR-Greece (0.026 µg/l), followed by ARSO-Slovenia (0.2 µg/l) and CETI- Montenegro (2 µg/l).
- Regarding hydrocarbons, for polycyclic compounds the lowest LOD values were reported by HCMR-Greece (range from 0.000003 µg/l to 0.000009 µg/l), followed by ARSO-Slovenia (from 0.0002 µg/l to 0.005 µg/l) and then both CETI-Montenegro and NIB-Slovenia (from 0.001 µg/l to 0.002 µg/l). No LOD was reported by ISPRA-Italy regarding polycyclic compounds. For total hydrocarbons, HCMR-Greece reported a LOD of 0.16 µg/l, while ISPRA-Italy reported 10 µg/l. For aliphatic hydrocarbons, C_6-C_{12} CETI-Montenegro reported a LOD of 10 µg/l, while ISPRA-Italy reached LOD of 0.005 µg/l. For aliphatic hydrocarbons C_{10}-C_{40} very different LOD were reported by the laboratories, with ARSO-Slovenia having the best performance (0.01 µg/l), followed by HCMR-Greece (0.16 µg/l) and CETI-Montenegro (100 µg/l). For all monocyclic compounds the best LODs were those of ISPRA-Italy (0.005 µg/l), followed by ARSO-Slovenia (0.1 µg/l) and CETI-Montenegro (1 µg/l). The information is missing for total hydrocarbons from CETI-Montenegro and for DDT, anthracene, fluoranthene, benzo(a)pyrene from RBI-Croatia and AUT-Albania.

- For physical parameters, CETI-Montenegro reported 0.01 µg/l and 0.1 ppt for dissolved oxygen and salinity respectively, while for temperature they indicated a range from -10 to 50 °C.

Values of the QUANTIFICATION LIMITS (LOQ) have been provided for almost all the parameters analyzed, with the exception of total hydrocarbons from CETI-Montenegro and the physical parameters of ARSO-Slovenia, ISPRA-Italy and the two Montenegro laboratories. Where possible, the LOQ values were compared with the Annual Average Environmental Quality Standard (AA-EQS) of the Directive 2013/39/EU (where non-applicable, Maximum Admissible Concentration Environmental Quality Standard, MAC-EQS, was used). Furthermore, it was evaluated whether the LOQs presented by different Institutes meet the requirements of Directive 2009/90/EU, according to which the LOQ value of the method must be equal to or less than 30% of the EQS value.

- The best LOQ for DDT (0.0005 µg/l) was obtained by RBI-Croatia and AUT-Albania, followed by CETI-Montenegro reporting for all the required pesticides and biocides LOQ of 0.001 µg/l, better than that of ARSO-Slovenia for HCB (0.002 µg/l). ISPRA-Italy reported LOQ for DDT, DDD, DDE and hexachlorobenzene of 0.01 µg/l. All four laboratories analyzing DDT were able to determine concentrations at AA-EQS (0.025 µg/l for Total DDT and of 0.01 µg/l for p,p DDT). Only ISPRA-Italy had LOQ higher than 30 % of AA-EQS (30 % of Total DDT EQS = 0.0075 µg/l; 30 % of p,p DDT EQS = 0.003 µg/l), and thus would not be able to satisfy the Directive 2009/90/EU. Both ARSO-Slovenia and CETI-Montenegro had LOQs at least equal to AA-EQs for hexachlorobenzene (0.002 µg/l), while ISPRA-Italy would not be able to determine such concentration for HCB. None of the laboratories analyzing HCB could satisfy the requirements of the Directive 2009/90/EU (30 % of HCB EQS = 0.0006 µg/l).
- Regarding antifoulants, the same LOQ vales were reported for TBT (0.0002 µg/l) by RBI-Croatia, ARSO-Slovenia and AUT-Albania. This LOQ was equal to AA-EQS for TBT cation. CETI-Montenegro reported higher LOQ (0.003 µg/l) for both compounds. Also ISPRA-Italy reported the LOQ value for TBT (0.01 µg/l) too high to meet the legal requirements. All LOQs presented for TBT were higher than 30 % of EQS, thus all methods should be improved to fulfill the requirements of the Directive 2009/90/EU.
- Regarding heavy metals, RBI-Croatia and AUT-Albania showed the best LOQ (0.000005 µg/l) for mercury, followed by ARSO-Slovenia and ISPRA-Italy (0.01 µg/l) and CETI-Montenegro (0.15 µg/l). For cadmium the best LOQ was obtained by RBI-Croatia and AUT-Albania (0.002 µg/l), followed by HCMR-Greece (0.004 µg/l), ARSO-Slovenia (0.06 µg/l), ISPRA-Italy (0.1 µg/l) and CETI-Montenegro (1.5 µg/l). Concerning lead, RBI-Croatia and AUT-Albania showed the best LOQ (0.003 µg/l), followed by HCMR-Greece (0.088 µg/l), ARSO-Slovenia and ISPRA-Italy (1 µg/l) and CETI-Montenegro (2.5 µg/l). Only

CETI-Montenegro laboratory would not be able to fulfill the requirements of the Directive 2013/39/EU (MAC-EQS for mercury is 0.07 µg/l (no AA-EQS has been proposed); AA-EQS for cadmium: 0.2 µg/l, for lead: 1.3 µg/l). According to the Directive 2009/90/EU, methods for analysis of mercury were all performing enough, except for the one employed by CETI-Montenegro (30 % of Hg EQS = 0.021 µg/l). Only CETI-Montenegro and ISPRA-Italy need to improve their methods for cadmium analysis, as their methods could not determine the concentrations lower than 30 % of Cd EQS (0.06 µg/l). Only HCMR-Greece, RBI-Croatia and AUT-Albania presented LOQs lower than 30 % of lead EQS (0.39 µg/l).

- Considering hydrocarbons, for the polycyclic compounds the best LOQ values were obtained by HCMR-Greece (0.00002 µg/l), followed by RBI-Croatia and AUT-Albania (0.0001 µg/l), ISPRA-Italy (0.0005 µg/l), CETI-Montenegro (LOQ ranging from 0.002 µg/l to 0.006 µg/l), NIB-Slovenia (0.003 - 0.005 µg/l) and ARSO-Slovenia (LOQ ranging from 0.004 µg/l to 0.07 µg/l). For total hydrocarbons HCMR-Greece provided a value of LOQ (0.5 µg/l), while ISPRA-Italy reported 110 µg/l. For aliphatic hydrocarbons, C_6-C_{12} ISPRA-Italy obtained a LOQ of 0.02 µg/l and CETI-Montenegro reported a LOQ of 25 µg/l. For aliphatic hydrocarbons C_{10}-C_{40} the best LOQ was obtained by ARSO-Slovenia (0.04 µg/l for specific range C_{10}-C_{13}), followed by HCMR-Greece (0.5 µg/l), ISPRA-Italy (100 µg/l) and CETI-Montenegro (250 µg/l). For monocyclic hydrocarbons the best LOQ values were those reported by ISPRA-Italy (0.01-0.1 µg/l) followed by ARSO-Slovenia (0.2 µg/l) and by CETI-Montenegro (3 µg/l). The Directive 2013/39/EU have imposed the following AA-EQS: 0.1 µg/l for anthracene, 0.0063 µg/l for fluoranthene, 2 µg/l for naphthalene and 8 µg/l for benzene. For these compounds, all participating Institutes were able to determine concentrations at EQS level. Considering the values of 30 % of these EQS, it emerged that for all Institutes the methods used for anthracene and naphthalene are able to satisfy the requirements of the Directive 2009/90/EU. On the contrary, only HCMR-Greece, RBI-Croatia, AUT-Albania and ISPRA-Italy had LOQs of fluoranthene lower than 30 % of ESQ. Only ARSO-Slovenia and ISPRA-Italy reached LOQs low enough for benzene. Regarding PAHs such as benzo(a)pyrene, benzo(a)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene and indenopyrene, only HCMR-Greece, RBI-Croatia and AUT-Albania had methods that were able to determine EQS concentration (1.7×10^{-4} µg/l); nevertheless, none of these Institutes reached LOQ lower than 30 % of EQS (0,000051 µg/l).

Most of the participants reported different REFERENCE MATERIALS used for quality control (QC) of analytical performance. For some analyses reference materials were not used.

- Considering pesticides and biocides, the only information was provided by ARSO-Slovenia that reported the use of in house reference material for HCB and additional parameters.

- For heavy metals, regarding mercury RBI-Croatia referred to use BCR-579, while ARSO-Slovenia used NIST SRM 1641d. For cadmium and lead NRC_CNRC were used by HCMR-Greece, CASS-5 by RBI-Croatia, NRC-CNRC SLEW-3 by ARSO-Slovenia, and NASS-6 by CETI-Montenegro. RBI-Croatia reported CASS-5 also for the other heavy metals, which were reported as additional parameters.
- Regarding hydrocarbons, where reported, HCMR-Greece used spiked material, while ARSO-Slovenia reported the use of in house reference material, also for the additional parameters not required by the questionnaire. ISPRA-Italy reported the use of the laboratory certified material.
- No information were obtained for physical parameters.

Most of the laboratories (except for IMBK-Montenegro, CETI-Montenegro, NIB-Slovenia), participated in INTERCALIBRATION ROUND exercises for at least one group of parameters analysed in water matrix. Only ARSO-Slovenia laboratory has subjected almost all methods to the intercalibration exercise.

- For pesticides and biocides and antifoulants ARSO-Slovenia reported the use of Aquacheck for HCB analysis and PT-WFD for Organotins in Surface Water 2010, respectively.
- Considering heavy metals, only ARSO-Slovenia gave information about participation to intercalibration test "Ielab" for mercury and lead, while for cadmium this Institute reported Aquacheck S11. HCMR-Greece participated to Quasimeme for both cadmium and lead.
- For polycyclic hydrocarbons, RBI-Croatia and AUT-Albania indicated Qualitycheck circuit, ARSO-Slovenia reported Aquacheck and ISPRA-Italy participated in QUASIMEME. Regarding aliphatic hydrocarbons C_6-C_{12} ISPRA-Italy indicated UNICHEM-ERA. For hydrocarbons C_{10}-C_{40} ARSO-Slovenia participated in AGLAE Association intercalibration test (for specific range C_{10}-C_{13}). Only ISPRA-Italy participated to UNICHEM-ERA circuit for monocyclic hydrocarbons.
- No information has been provided regarding physical parameters.

Little information has been provided regarding the ACCURACY of analytical methods.

- For antifoulants ARSO-Slovenia indicated accuracy as the recovery of the spike experiment of 90-103 % for TBT. The same value was reported for DBT (as additional parameter).
- Regarding heavy metals, the only information available for mercury was provided by ARSO-Slovenia that reported a recovery value of 100.6 %. The same laboratory reported for cadmium and lead values of 96 % and 100 % respectively. HCMR-Greece reported accuracy of 19 % and 33 % respectively for cadmium and lead. In the latter case, we suppose that the accuracy was

expressed as the % difference between the obtained results and the reference value, and the method doesn't seem to be very performant in terms of accuracy (if meant as the % difference, it should normally be lower than 20%).

- Only HCMR-Greece provided information on accuracy for hydrocarbons in the range of 4.6 - 30.4 %, probably expressed as the difference between the obtained result and the reference value, showing rather good performance except for acenaphthene (26.1 %), benzo(k)fluoranthene (25.6 %), dibenzo(a,h)anthracene (21.8 %) and benzo(g,h,i)perylene (30.4%).
- No information was available for physical parameters.

As already observed for other parameters, REPRODUCIBILITY information was also incomplete.

- It was difficult to compare the performance of the methods used, as it was not clear at what level of concentration the reproducibility reported by the laboratories was assessed (only the RBI-Croatia and AUT-Albania laboratories indicated that the RSD % was calculated at the LOQ concentration). Furthermore, these two laboratories gave the same reproducibility values except for heavy metals (not reported by AUT-Albania). Regarding heavy metals, the best reproducibility was reported by CETI-Montenegro with values ranging from 1.40 % to 2.10 %, followed by ARSO-Slovenia (3.6-8.7 %), HCMR-Greece (4.3-5.6 %) and RBI-Croatia (20 % at LOQ).
- The reproducibility of hydrocarbons analyses reported by HCMR-Greece was ranging from 5.8 % to 15.9 %, while RBI-Croatia and AUT-Albania declared 40 % at LOQ concentration.

No information has been reported in REMARKS section of questionnaire by any laboratory.

An additional parameter, measurement uncertainty (%), has been reported in the questionnaire by the ARSO-Slovenia laboratory and was inserted in the Attachment 1 as Remarks.

SEDIMENT MATRIX

Data QA-QC questionnaire vs data reported

The comparison between the requested data reported on QA-QC Questionnaire and the data presented by the participating institutes for the sediment matrix are shown below (see **Attachment 2**).

In relation to pesticides and biocides (DDT, DDE, DDD and HCB), in sediments:

- HCMR-Greece, ARSO-Slovenia, ISPRA-Italy, CETI-Montenegro and IOF-Croatia reported information on all the analytes requested in the questionnaire;
- only ARSO-Slovenia specified that isomers (p,p) and (o,p) of DDT, DDE and DDD have been analyzed;
- RBI-Croatia analyzed only HCB, and introduced extra compounds (hexachlorbenzene, hexachlorocyclohexan, penthachlorbenzene, hexachlorobutadien, PFOS and PFOA); this institute reported the data related to hexachlorbenzene in an additional row at the end of the contaminants list requested by Questionnaire and these data have been reported in HCB row;
- AUT-Albania did not report information on the required analytes in the questionnaire, but added to contaminant's list aldrin, dieldrin and methoxiclor. However, for all of these compounds the only information available was the use of the IAEA 383 (certified sediment sample for chlorinated pollutants) for method validation, inserted into the "remarks" column. For this reason in the corresponding cells of the N.A. column the X mark was used;
- analyses were not performed by UoM-IMBK-Montenegro, ISPRA-Italy and NIB-Slovenia.

In relation to antifoulants (tributyltin (TBT), triphenyltin (TPhT)), in sediments:

- RBI-Croatia, CETI-Montenegro and NIB-Slovenia performed the analysis on both the required parameters, while ARSO-Slovenia and ISPRA-Italy only analyzed TBT;
- HCMR-Greece, UoM-IMBK-Montenegro, AUT-Albania and IOF-Croatia did not analyze these compounds.

In relation to pharmaceuticals (macrolides and others) and radionuclides (Cs^{137}, Pu^{239}) in sediments:

- none of the nine institutes reported information.

In relation to heavy metals (mercury, cadmium, lead, vanadium, chromium, barium, copper, iron, arsenic, zinc, nickel and aluminum) in sediments:

- only CETI-Montenegro analyzed all contaminants from the questionnaire, while ISPRA-Italy lacked only aluminum;
- only RBI-Croatia did not analyze any heavy metals;
- AUT-Albania analyzed only vanadium and barium required by the questionnaire;
- ARSO-Slovenia did not analyze arsenic and vanadium;
- UoM-IMBK-Montenegro did not analyze cadmium, vanadium, barium, arsenic, aluminum;
- HCMR-Greece did not analyze mercury, cadmium, vanadium, barium, aluminum, whereas cobalt analysis was added;

- NIB-Slovenia did not perform analyses of vanadium, barium and aluminum, but cobalt and manganese analyses were introduced as additional parameters;
- IOF-Croatia analysed only mercury, cadmium, lead, chromium, copper and zinc.

In relation to hydrocarbons (anthracene, fluoroanthene, naphthalene, acenaphthylene, acenaphthene fluorene, phenanthrene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene, indenopyrene, total hydrocarbons, aliphatic hydrocarbons C_6-C_{12}, aliphatic hydrocarbons C_{10}-C_{40} (specify range) benzene, toluene, ethylbenzene, o,m,p-xylene) in sediments:

- CETI-Montenegro and ISPRA-Italy have analyzed all the hydrocarbons from the questionnaire;
- RBI-Croatia and AUT-Albania analyzed only anthracene, fluoroanthene and benzo(a)pyrene;
- HCMR-Greece reported information for all contaminants, except for aliphatic hydrocarbons C_6-C_{12}, benzene, toluene, ethylbenzene, o,m,p-xylene;
- ARSO-Slovenia did not analyze total hydrocarbons, aliphatic hydrocarbons C_6-C_{12}, benzene, toluene, ethylbenzene, o,m,p-xylene; for aliphatic hydrocarbons C_{10}-C_{40}, ARSO-Slovenia was the only Institute which specified the range (C_{10}-C_{13}) as required in the questionnaire;
- only polycyclic hydrocarbons were analysed by NIB-Slovenia; this laboratory reported the results as mg/kg instead of µg/kg and in the Attachment 2 their results (LOD and LOQ) were transformed in units requested (µg/kg);
- UoM-IMBK-Montenegro and IOF-Croatia did not analyze any of hydrocarbons required in questionnaire.

In relation to brominated compounds (brominated diphenylether) in sediments:

- only RBI-Croatia, ARSO-Slovenia, AUT-Albania reported the information on these compounds;
- UoM-IMBK-Montenegro, CETI-Montenegro, HCMR-Greece, ISPRA-Italy, NIB-Slovenia and IOF-Croatia did not report any information.

In relation to organic matter (total organic carbon (TOC), loss on ignition (LOI)) in sediments:

- only ISPRA-Italy and NIB-Slovenia analyzed TOC;
- only UoM-IMBK-Montenegro and CETI-Montenegro reported information on analysis for LOI.

In relation to grain size analysis (sand, silt, clay) in sediments:

- only ISPRA-Italy and NIB-Slovenia performed the complete analysis, while UoM-IMBK-Montenegro conduced grain size analysis only on sandy fraction.

In relation to water (water content) in sediments:

- HCMR-Greece, RBI-Croatia, ISPRA-Italy, NIB-Slovenia and IOF-Croatia did not report information;
- UoM-IMBK-Montenegro, CETI-Montenegro, ARSO-Slovenia, AUT-Albania performed the analysis.

NIB-Slovenia introduced following PCBs (polychlorobiphenyls) as additional parameters: (2,4,4'-trichlorobiphenyl, 2,2',5,5'-tetrachlorobiphenyl, 2,2',4,5,5'-pentachlorobiphenyl, 2,2',3,4,4',5'-hexachlorobiphenyl, 2,2',4,4',5,5'-hexachlorobiphenyl, 2,2',3,4,4',5'-hexachlorobiphenyl).

Units in sediments

Almost all the Institutes reported units as requested in Questionnaire. Only NIB-Slovenia presented their results for hydrocarbons as mg/kg and not as requested (µg/kg). The results in the Attachment 2 were transformed to the unit adopted by other participants for the comparison.

Grain size

The grain size parameter could have been expressed as unsieved, <63 µm, <2 mm.

- ARSO-Slovenia reported <63 µm for all analyzed parameters;
- UoM-IMBK-Montenegro reported <63 µm for all analyzed parameters, except for LOI and water content where unsieved sediment was analysed;
- CETI-Montenegro reported <2 mm for most of analytes and only for aliphatic hydrocarbons C_6-C_{12}, monocyclic hydrocarbons, LOI and water content unsieved sediment was employed;
- HCMR-Greece reported <0.5 mm for most compounds, except for heavy metals, which were analyzed unsieved;
- both AUT-Albania and RBI-Croatia wrote unsieved, where reported;
- ISPRA-Italy used fraction <2 mm for all analyses, except for grain size, that was analysed on unsieved sediment;
- NIB-Slovenia analysed antifoulants and polycyclic hydrocarbons on <63 µm fraction. For heavy metals analysis, this Institute applied two methods: method 1 (used for Hg, Cd, Pb, Cr, Cu, Zn, Ni, Fe, As, Co) employed <63 µm fraction while method 2 (Hg, Cd, Pb, Cr, Cu, Zn, Ni, Mn) used <500 µm fraction; NIB-Slovenia also reported different fractions for grain size analysis: >63 µm for sand, 2-63 µm for silt and <2 µm for clay;
- IOF-Croatia used sediments sieved under 250 µm for pesticides and biocides analyses, while unsieved samples were used for heavy metals.

Sampling method (type of sampler, ex: box corer)

- HCMR-Greece, AUT-Albania, RBI-Croatia, ISPRA-Italy and IOF-Croatia used box corer as sampling method for all analysed compounds;

- NIB-Slovenia used gravity corer to collect samples for all the performed analyses;
- CETI-Montenegro used Van Veen grab for all analytes;
- ARSO-Slovenia sampled with the use of Van Veen grab, refering to ISO 5667-19 for all of analytes reported, except for TBT for which SIST ISO 5667-12 was employed;
- UoM-IMBK-Montenegro used Ponar grab for all analytes.

Thickness of sampled sediment

- AUT-Albania and RBI-Croatia used a thickness of 5 cm for all analytes where reported;
- ARSO-Slovenia and ISPRA-Italy sampled 10 cm for all reported analytes (ISPRA-Italy did not refer the thickness for pesticides and for antifoulants), while CETI-Montenegro reported "~10 cm" for all analytes; also NIB-Slovenia used 10 cm for all analyzed parameters, including the method 1 for heavy metals (while method 2 used 5 cm thickness);
- UoM-IMBK-Montenegro used 5 cm for all analytes with the exception of LOI, grain size (sand) and water contents where <10 cm were sampled;
- HCMR-Greece used thickness of 1 cm for pesticides and biocides, hydrocarbons, while 2 cm were used for heavy metals;
- IOF-Croatia sampled a superficial layer of 0-3 cm for determinations of pesticides and biocides and 0-2 cm for heavy metals analyses.

Storage (method, temperature , etc.)

- HCMR-Greece used -20 °C as a storage method;
- AUT-Albania and RBI-Croatia used dry sediment stored at 4 °C;
- UoM-IMBK-Montenegro used freeze-dried sediment stored at 4 °C, except for: LOI and water content samples (which were sampled at 4°C and stored deep-frozen at -20 °C) and grain size (sand samples were air dried or freeze-dried and stored in dark, cool place at 20 °C);
- CETI-Montenegro stored the samples at 1 - 5 °C in glass container;
- ARSO-Slovenia used the SIST ISO 5667-15 method; also NIB-Slovenia applied SIST ISO 5667-15 for antifoulants, heavy metals (method 1) and hydrocarbons, while for grain size analysis and heavy metals (method 2) the analyses were performed on freeze dried sediment;
- ISPRA-Italy stored at -20 °C sediments for the analysis of pesticides, heavy metals, polycyclic hydrocarbons, total and aliphatic hydrocarbons C_{10}-C_{40} and TOC, while environmental temperature and ammonium bisulphate addiction was reported for aliphatic hydrocarbons C_6-C_{12} and monocyclic hydrocarbons. Samples for grain size analysis were stored at environmental temperature;
- IOF-Croatia stored sediment samples at -40°C: for pesticides and biocides freeze-dried samples were employed, while heavy metals were determined in untreated sediment.

Method of Analysis

In relation to pesticides and biocides in sediments:

- GC-ECD methods were used by HCMR-Greece, ARSO-Slovenia (ISO 10382 modified method) and IOF-Croatia (UNEP 1996);
- ISPRA-Italy and RBI-Croatia used GC-MS (RBI-Croatia only for HCB);
- CETI-Montenegro used the EPA 8080A method.

Regarding antifoulants in sediments:

- RBI-Croatia used GC-PFPD;
- ARSO-Slovenia used the ISO 17353 modified method (GC-ICP-MS) for TBT; NIB-Slovenia applied ISO 17353:2004 method for both TBT and TPhT;
- CETI-Montenegro used the ISO 23161:2009 method.
- ISPRA-Italy used an internal method (extraction with dichloromethane and tin determination on total extracted).

In relation to Heavy metals in sediments:

for mercury the various institutes reported the following methods:

- Five laboratories employed atomic absorption spectrometry: UoM-IMBK-Montenegro (HVG AAS), ARSO-Slovenia (EPA 7473 (CV-AAS)) CETI-Montenegro (Solid AAS-Mercury) and IOF-Croatia (CV AAS); NIB-Slovenia reported ISO 5666, modified 1999 as method 1 and AAS CV as method 2;
- ISPRA-Italy employed EPA 3050B-1996 + EPA 7474-2007 (CVAFS).

For other metals, where reported:

- Four laboratories applied ICP-MS-based methods: HCMR-Greece, ARSO-Slovenia (ISO 17294-2, modified ICP/MS method) and ISPRA-Italy (EPA 3050B-1996 + EPA 6020B-2014 (ICP/MS)); NIB-Slovenia used IS0 17294-2, modified: 2003 as method 1 and AAS as method 2;
- UoM-IMBK-Montenegro used AAS-Flame;
- GF-AAS was employed by IOF-Croatia;
- CETI-Montenegro employed the BS EN 13656:2002 method.

In relation to hydrocarbons in sediments, various gas chromatographic methods have been employed by participating laboratories:

- HCMR-Greece (GC-MS for polycyclic hydrocarbons and GC-FID for total hydrocarbons and aliphatic hydrocarbons C_{10}-C_{40}),
- RBI-Croatia and AUT-Albania（GC-MS),
- ARSO-Slovenia and NIB-Slovenia（IM/GC MSD/SOP 055 (GC/MS),
- CETI-Montenegro (EPA 8270 C for polycyclic hydrocarbons, EN ISO 16703:2011/EPA 5021A method for total hydrocarbons, EPA 5021A for aliphatic hydrocarbons C_6-C_{12} and monocyclic compounds, and EN ISO 16703:2011 for aliphatic hydrocarbons C_{10}-C_{40})

- ISPRA-Italy (EPA 3550C-2007+ EPA8270D-2007 (GC-MS-SIM) for polycyclic compounds, GC-FID for total hydrocarbons, EPA 5035+ EPA 8260 for aliphatic hydrocarbons C_6-C_{12} and monocyclic compounds and ISO 16703:2004 (GCFID) for aliphatic hydrocarbons C_{10}-C_{40}).

In relation to brominated compounds in sediments:

- RBI-Croatia and AUT-Albania used GC-ICPMS;
- ARSO-Slovenia applied the EPA 1614 (RGC/HRMS) method.

In relation to organic matter in sediments:

- ISPRA-Italy analysed TOC by means of CHN analyser;
- LOI was analysed by UoM-IMBK-Montenegro (UNEP(DEC)/MED WG.282/Inf.5/Rev.1 method) and by CETI-Montenegro (EN 12879 method).

In relation to grain size analysis in sediments:

- UoM-IMBK-Montenegro applied the UNEP/IOC/IAEA (1995) Reference Methods for Marine Pollution Studies No 63;
- ISPRA-Italy performed the analysis by means of mechanical sieving;
- NIB-Slovenia used FRITSCH particle Sizer.

In relation to water content in sediments:

- UoM-IMBK-Montenegro reported to use the UNEP/IOC/IAEA (1995) Reference Methods for Marine Pollution Studies No 63;
- ARSO-Slovenia used the ISO 11465:1993 (GR);
- CETI-Montenegro reported oven drying.

Limit of detection (LOD)

In relation to pesticides and biocides in sediments:

- for DDT, DDE and DDD, the lowest LOD was presented by HCMR-Greece (0.03 µg/kg), followed by IOF-Croatia (0.06-0.09 µg/kg), CETI-Montenegro (0.5 µg/kg), and ARSO-Slovenia with 5 µg/kg;
- for HCB, the lowest LOD (0.003 µg/kg) was obtained by ARSO-Slovenia, while other Institutes reported values of: 0.03 µg/kg (HCMR-Greece), 0.05 µg/kg (IOF-Croatia) and 0.5 µg/kg (CETI-Montenegro);
- ISPRA-Italy did not report LOD for pesticides and biocides.

In relation to antifoulants in sediments:

- ARSO-Slovenia and NIB-Slovenia indicated LOD of 1 µg/kg (as cation) for TBT, CETI-Montenegro reported the same value for both indicated contaminants.
- RBI-Croatia did not provide any information.

In relation to heavy metals in sediments:

- HCMR-Greece, AUT-Albania and NIB-Slovenia did not report any LOD;

- for mercury, ISPRA-Italy achieved LOD of 0.0003 mg/kg, IOF-Croatia presented the value of 0.0005 mg/kg, CETI-Montenegro reported 0.001 mg/kg, while ARSO-Slovenia reported 0.01 mg/kg;
- for cadmium, ISPRA-Italy provided LOD of 0.005 mg/kg, followed by ARSO-Slovenia (0.01 mg/kg), IOF-Croatia (0.015 mg/kg) and CETI-Montenegro (0.07 mg/kg);
- for lead, IOF-Croatia reached LOD of 0.02 mg/kg, while ISPRA-Italy referred 0.03 mg/kg, CETI-Montenegro reported 0.25 mg/kg and ARSO-Slovenia reported 2 mg/kg;
- for vanadium, ISPRA-Italy provided LOD of 0.02 mg/kg and CETI-Montenegro obtained LOD of 0.25 mg/kg;
- for chromium, ISPRA-Italy reported 0.01 mg/kg, IOF-Croatia achieved 0.035 mg/kg, CETI-Montenegro 0.5 mg/kg, UoM-IMBK-Montenegro 1.5 mg/kg and ARSO-Slovenia 2 mg/kg;
- for barium, the following LOD values were reported: 0.02 mg/kg by ISPRA-Italy, 0.8 mg/kg by CETI-Montenegro and 5 mg/kg by ARSO-Slovenia;
- for copper, IOF-Croatia referred 0.005 mg/kg, ISPRA-Italy reported 0.04 mg/kg, CETI-Montenegro 0.25 mg/kg, UoM-IMBK-Montenegro 1.5 mg/kg and ARSO-Slovenia 2 mg/kg;
- for iron, the lowest LOD of 0.2 mg/kg was declared by ISPRA-Italy, followed by CETI-Montenegro (1 mg/kg), UoM-IMBK-Montenegro (4.5 mg/kg) and ARSO-Slovenia (20 mg/kg);
- for arsenic, ISPRA-Italy reported 0.02 mg/kg, while CETI-Montenegro indicated LOD of 1 mg/kg;
- for zinc, IOF-Croatia reported LOD of 0.05 mg/kg, UoM-IMBK-Montenegro indicated 0.3 mg/kg, ISPRA-Italy 0.4 mg/kg, CETI-Montenegro 1 mg/kg, ARSO-Slovenia 5 mg/kg;
- for nickel, the following LODs were reported: 0.05 mg/kg by ISPRA-Italy, 0.3 mg/kg by CETI-Montenegro, 2 mg/kg by ARSO-Slovenia and 3 mg/kg by UoM-IMBK-Montenegro;
- for aluminum CETI-Montenegro reported 1 mg/kg, while ARSO-Slovenia had LOD of 20 mg/kg.

In relation to hydrocarbons in sediments, only HCMR-Greece, CETI-Montenegro ARSO-Slovenia and ISPRA-Italy provided for LOD (NIB-Slovenia presented only for naphtalene the LOD of 0.01 mg/kg, equal to 10 µg/kg):

- comparing polycyclic hydrocarbons in the list, the lowest LOD values belong to HCMR-Greece (0.05-0.26 µg/kg) followed by CETI-Montenegro and ISPRA-Italy (0.5-1 µg/kg), ARSO-Slovenia (5-10 µg/kg) and NIB-Slovenia (10 µg/kg for naphtalene);
- regarding total hydrocarbons, only HCMR-Greece provided a LOD of 50 µg/kg;

- for aliphatic hydrocarbons C_6-C_{12} CETI-Montenegro reported a LOD of 20 µg/kg;
- for aliphatic hydrocarbons C_{10}-C_{40} (specify range C_{10}-C_{13}) ARSO-Slovenia had the lowest LOD (0.2 µg/kg), followed by HCRM-Greece (50 µg/kg) and CETI-Montenegro (5000 µg/kg);
- for monocyclic hydrocarbons, only CETI-Montenegro reported LOD equal to 1 µg/kg.

In relation to brominated compounds in sediments:

- ARSO-Slovenia indicated a LOD equal to 0.05 µg/kg.

In relation to organic matter in sediments:

- CETI-Montenegro reported a LOD equal to 0.01 % for LOI analyses.

In relation to grain size analysis in sediments, any information on LOD was reported.

In relation to water content in sediments:

- CETI-Montenegro reported LOD of 0.01 %, while ARSO-Slovenia reported a LOD of 0.1 %

Limit of quantification (LOQ). Where possible, the LOQ values were compared with the Annual Average Environmental Quality Standard of the Italian Legislative Decree n° 172 13/10/2015 (Table 2/A), which is the executive decree for the Directive 2013/39/UE. In addition, the LOQ values have been evaluated in accordance with Directive 2009/90/EU, which states that LOQ must be equal to or less than 30% of the EQS value.

In relation to pesticides and biocides in sediments:

- for DDT, DDE, DDD the lowest LOQ was reported by HCMR-Greece and ISPRA-Italy (0.1 µg/kg), followed by IOF-Croatia (0.2-0.3 µg/kg), CETI-Montenegro (1 µg/kg) and ARSO-Slovenia (10 µg/kg); according to these data, only HCMR-Greece, ISPRA-Italy and IOF-Croatia would be able to determine concentrations at EQS level for all pesticides requested (1 µg/kg for DDT, 0.8 µg/kg for DDD and 1.8 µg/kg for DDE), with LOQs satisfying the Directive 2009/90/EU;
- regarding HCB, ARSO-Slovenia reached the lowest LOQ (0.005 µg/kg), followed by HCMR-Greece, ISPRA-Italy and RBI-Croatia (0.1 µg/kg), IOF-Croatia (0.167 µg/kg) and CETI-Montenegro (1 µg/kg).

In relation to antifoulants in sediments:

- for TBT, the lowest LOQ was that of RBI-Croatia and ISPRA-Italy (1 µg/kg) followed by CETI-Montenegro (3 µg/kg) and both ARSO-Slovenia and NIB-Slovenia (3.33 µg/kg), showing that all these laboratories manage to fulfill legislative requirements as EQS for TBT in sediments is 5 µg/kg; on the

contrary, only the LOQ presented by RBI-Croatia and ISPRA-Italy were low enough to fulfill the requirements of the Directive 2009/90/EU (30 % of TBT EQS = 1.5 µg/kg);
- for triphenyltin, CETI-Montenegro had the lowest LOQ (3 µg/kg), followed by RBI-Croatia (5 µg/kg); NIB-Slovenia did not provide the LOQ for TPhT.

In relation to heavy metals in sediments, the values were provided by ARSO-Slovenia, NIB-Slovenia, CETI-Montenegro, ISPRA-Italy and IOF-Croatia:
- for mercury, ISPRA-Italy indicated the lowest LOQ of 0.0005 mg/kg, followed by IOF-Croatia (0.00167 mg/kg), CETI-Montenegro (0.005 mg/kg) and ARSO-Slovenia (0.05 mg/kg); NIB-Slovenia did not provide the LOQ for mercury; all the presented values were lower than EQS (0.3 mg/kg) and were sufficiently low according to the Directive 2009/90/EU;
- for cadmium, the following LOQ values were reported: 0.05 mg/kg by ISPRA-Italy, NIB-Slovenia and IOF-Croatia, 0.1 mg/kg by both ARSO-Slovenia and CETI-Montenegro; EQS concentrations (0.3 mg/kg) were determinable for all five Institutes, but LOQs presented by ARSO-Slovenia and CETI-Montenegro were too high to fulfill the specifications of the Directive 2009/90/EU;
- for lead, NIB-Slovenia reported LOQ of 0.05 mg/kg, IOF-Croatia referred 0.067 mg/kg, CETI-Montenegro declared LOQ of 0.5 mg/kg, ISPRA-Italy had 1 mg/kg and ARSO-Slovenia 5 mg/kg; all five laboratories were able to quantificate lead concentrations at EQS level (30 mg/kg), satisfying also the requirements of the Directive 2009/90/EU;
- for vanadium, CETI-Montenegro achieved the LOQ of 0.5 mg/kg and ISPRA-Italy 1 mg/kg;
- for chromium, NIB-Slovenia had LOQ of 0.05 mg/kg, IOF-Croatia reached 0.1167 mg/kg, CETI-Montenegro reported 0.75 mg/kg, ISPRA-Italy indicated 1 mg/kg and ARSO-Slovenia reported 5 mg/kg;
- for barium, ISPRA-Italy and CETI-Montenegro reported a LOQ of 1 mg/kg, while ARSO-Slovenia reached 10 mg/kg;
- for copper, IOF-Croatia reported LOQ of 0.0167 mg/kg, NIB-Slovenia achieved LOQ of 0.05 mg/kg, CETI-Montenegro reported a LOQ of 0.5 mg/kg, ISPRA-Italy of 1 mg/kg and ARSO-Slovenia of 5 mg/kg;
- for iron, NIB-Slovenia reported LOQ of 0.05 mg/kg, CETI-Montenegro presented LOQ equal to 3 mg/kg, ISPRA-Italy declared 10 mg/kg and ARSO-Slovenia reported a value of 50 mg/kg;
- for arsenic, NIB-Slovenia reached the lowest LOQ of 0.001 mg/kg, ISPRA-Italy reported 1 mg/kg and CETI-Montenegro indicated 3 mg/kg;
- for zinc, NIB-Slovenia obtained LOQ of 0.001 mg/kg, IOF-Croatia referred 0.167 mg/kg, CETI-Montenegro reported LOQ of 3 mg/kg, ISPRA-Italy indicated 5 mg/kg and ARSO-Slovenia reported 10 mg/kg;

- for nickel, the following values were presented: 0.05 mg/kg by NIB-Slovenia, 0.5 mg/kg by CETI-Montenegro, 1 mg/kg by ISPRA-Italy and 5 mg/kg by ARSO-Slovenia;
- for aluminum, CETI-Montenegro reported LOQ of 3 mg/kg and ARSO-Slovenia declared 50 mg/kg.

In relation to hydrocarbons in sediments, the LOQ values were indicated for almost all the analysed compounds:

- regarding polycyclic aromatic hydrocarbons, AUT-Albania and RBI-Croatia presented the lowest LOQ for anthracene, fluoroanthene, benzo(a)pyrene, corresponding to 0.1 µg/kg, HCMR-Greece reported values between 0.16-0.87 µg/kg, followed by CETI-Montenegro (1-3 µg/kg), ISPRA-Italy (2 µg/kg), ARSO-Slovenia (10 µg/kg with the exception of naphtalene with LOQ of 50 µg/kg) and NIB-Slovenia (50 µg/kg for all polycyclic hydrocarbons); the Italian Legislative Decree n° 172 13/10/2015 (Table 2/A) imposes EQS of 24 µg/kg and 35 µg/kg for anthracene and naphtalene, respectively – with the exception of NIB-Slovenia and ARSO-Slovenia (only for naphtalene), all Institutes were able to determine EQS concentrations; LOQ of naphthalene and anthracene referred by HCMR-Greece, RBI-Croatia, AUT-Albania, CETI-Montenegro and ISPRA-Italy fulfill the specifications of the Directive 2009/90/EU;
- for total hydrocarbon, HCMR-Greece reported the LOQ of 150 µg/kg and ISPRA-Italy declared 5000 µg/kg;
- for aliphatic hydrocarbons C_6-C_{12}, ISPRA-Italy and CETI-Montenegro reported LOQs of 0.25 and 50 µg/kg, respectively;
- for aliphatic hydrocarbons C_{10}-C_{40}, ARSO-Slovenia reported the lowest LOQ (0.5 µg/kg for specific range C_{10}-C_{13}) followed by HCMR-Greece (150 µg/kg), ISPRA-Italy (5000 µg/kg) and CETI-Montenegro (10000 µg/kg);
- for monocyclic aromatic hydrocarbons, the lowest range of LOQ values (0.03-0.1 µg/kg) was presented by ISPRA-Italy, while CETI-Montenegro reported the LOQ value of 3 µg/kg for all monocyclic compounds.

In relation to brominated compounds in sediments:

- RBI-Croatia, ARSO-Slovenia and AUT-Albania declared the same LOQ value (0.1 µg/kg).

In relation to organic matter in sediments:

- ISPRA-Italy indicated the LOQ of 0.1 % for TOC analysis;
- CETI-Montenegro reported the LOQ of 0.01 % for LOI determination.

In relation to grain size analysis in sediments, no information has been included.

In relation to water content in sediments:

- CETI-Montenegro and ARSO-Slovenia reported LOQ values of 0.01 % and 1 %, respectively.

Reference Materials (ex: NRC-CNRC, etc.)

In relation to pesticides and biocides in sediments:

- HCRM-Greece reported to use the NIST SRM 1941b;
- CETI-Montenegro made use of the IAEA 459 and IAEA 159;
- IOF-Croatia employed IAEA-408;
- ARSO-Slovenia reports to use an in-house reference material;
- ISPRA-Italy did not provide any information.

In relation to antifoulants in sediments:

- for TBT, RBI-Croatia and ARSO-Slovenia used PACS-2 (NRCC, sea sediment), while CETI-Montenegro analysed BCR-646;
- for TPhT, RBI-Croatia and CETI-Montenegro used the same reference material, BCR-646;
- no information on reference material was provided by NIB-Slovenia and ISPRA-Italy.

In relation to heavy metals in sediments:

- HCRM-Greece employed IAEA 458 and IAEA 433 (all the other numbers reported in the questionnaire are typing errors);
- UoM-IMBK-Montenegro reported to use the IAEA 158, marine sediment;
- ARSO-Slovenia employed the IRMM ERM-CC141;
- CETI-Montenegro used the NIST 2702;
- IOF-Croatia referred to use MESS-1;
- ISPRA-Italy declared the use of internal reference material;
- AUT-Albania and NIB-Slovenia did not present any information.

In relation to hydrocarbons in sediments, where reported:

- regarding polycyclic hydrocarbons, HCMR-Greece used NIST SRM 1941b, CETI-Montenegro made use of IAEA 459 and IAEA 159, ISPRA-Italy employed River sediment PAHs - LGC 6188; RBI-Croatia, AUT-Albania, ARSO-Slovenia and NIB-Slovenia did not report anything;
- only ISPRA-Italy referred to use the reference material for the analysis of total hydrocarbons (River sediment LGC PAH LCG 6188) and aliphatic hydrocarbons C_6-C_{12} (internal reference material);
- regarding aliphatic hydrocarbons C_{10}-C_{40}, ARSO-Slovenia (specified range: C_{10}-C_{13}) reported to use in-house reference materials, while ISPRA-Italy utilized ERM-CC016 and ERM-CC017;
- for monocyclic aromatic hydrocarbons, RTC-CRM631-030 was employed by CETI-Montenegro and internal reference material by ISPRA-Italy.

In relation to brominated compounds in sediments:

- only ARSO-Slovenia reported to use an in-house reference material.

In relation to organic matter, ISPRA-Italy declared to use the CRM B2152 material for TOC analysis.

In relation to grain size analysis and water in sediments, none of the Institutes reported information.

Intercalibration round (ex: Quasimeme, etc.)

Only UoM-IMBK-Montenegro and NIB-Slovenia did not participate in any intercalibration exercise (for any of the required parameters).

In relation to pesticides and biocides in sediments:

- HCMR-Greece participated in the Quasimeme circuit;
- ARSO-Slovenia took part in the SETOC intercalibration;
- CETI-Montenegro participated in the IAEA circuit;
- IOF-Croatia reported to perform the IAEA/MEL/MEDPOL intercalibration proficiency test;
- ISPRA-Italy did not provide information on participation in intercalibration for pesticides and biocides.

In relation to antifoulants in sediments:

- Any Institute participated in the intercalibration circuits for these compounds.

In relation to heavy metals in sediments:

- both HCMR-Greece and CETI-Montenegro reported the participation in the IAEA intercalibration, ARSO-Slovenia in addition to IAEA was also present in MEDPOL and SETOC circuits; ISPRA-Italy took part in UNICHIM intercalibration; no information was reported by IOF-Croatia.

In relation to hydrocarbons in sediments, where reported:

- regarding polycyclic hydrocarbons: HCMR-Greece and ARSO-Slovenia took part in the Quasimeme proficiency testing circuit, RBI-Croatia and AUT-Albania participated in WEPAL-SETOC, CETI-Montenegro participated in IAEA circuit, and ISPRA-Italy took part in the UNICHIM intercalibration;
- only CETI-Montenegro participated in IAEA circuit with the analyses of total hydrocabons and aliphatic hydrocarbons C_{10}-C_{40}.

In relation to brominated compounds, to organic matter, to grain size analysis and water in sediments:

- no information available.

Accuracy (% wrt certified value)

In relation to pesticides and biocides in sediments:

- only HCMR-Greece and IOF-Croatia reported the data on accuracy (19.1 - 22.5 % wrt certified value and 0.81 - 6.84 % wrt certified value, respectively); both laboratories expressed the accuracy as a measure of the difference between the test result and the accepted reference value and the method employed by IOF-Croatia resulted to be more performing.

In relation to antifoulants in sediments:

- only ARSO-Slovenia reported the accuracy for TBT analysis as recovery % (90-103 %), and specifies that it is accuracy of the spike experiment.

In relation to heavy metals in sediments, where reported:

- HCMR-Greece reported good accuracy with values between 1.04-11.2 %, as the % difference between the test result and the reference value;
- IOF-Croatia reported the accuracy of 95 % (as recovery) for all analysed metals;
- UoM-IMBK-Montenegro reports accuracy as recovery between 80-115 % (wrt certified value);
- ARSO-Slovenia reports accuracy as recovery between 92-104 % wrt certified;
- CETI-Montenegro, ISPRA-Italy and NIB-Slovenia did not report anything.

In relation to hydrocarbons in sediments:

- only HCMR-Greece reported values of accuracy for polycyclic hydrocarbons (2.5-28.3 % wrt certified value, as the difference between the test result and the certified value);
- ISPRA-Italy presented the accuracy values as recovery % only for total hydrocarbons (93 %), aliphatic hydrocarbons C_6-C_{12} (100-132 %), aliphatic hydrocarbons C_{10}-C_{40} (91-93 %) and monocyclic hydrocarbons (107-116 %).

In relation to brominated compounds, to organic matter, to grain size analysis and water in sediments:

- no information was available.

Reproducibility (RSD %)

No data on reproducibility was presented by UoM-IMBK-Montenegro and NIB-Slovenia.

In relation to pesticides and biocides in sediments:

- for DDT,DDE, DDD CETI-Montenegro reported a reproducibility of 12.5 % for all compounds, while HCMR-Greece presented values between 5.1-8.2 % and IOF-Croatia obtained the lowest RSD % range of 3.25-5.39 %;

- RBI-Croatia reported reproducibility of 40 % at LOQ concentration only for HCB;
- ISPRA-Italy did not provide any information on RSD %.

In relation to antifoulants in sediments:

- RBI-Croatia reported the reproducibility of 20 % at LOQ concentration for both compounds;
- ARSO-Slovenia presented RSD of 6 % for TBT;
- ISPRA-Italy did not provide any information on RSD %.

In relation to heavy metals in sediments:

- HCRM-Greece, IMBK-Montenegro and AUT-Albania did not provide any information on reproducibility of analysed metals;
- IOF-Croatia presented a low RSD of 5 % for all analysed metals, ARSO-Slovenia reported values of reproducibility between 3.5-7.9 %, CETI-Montenegro had RSD between 9.4-12.7 % and ISPRA-Italy presented values in the range 0.9-15.5 %.

In relation to hydrocarbons in sediments, where reported:

- RBI-Croatia and AUT-Albania reported values of reproducibility equal to 40 % at LOQ;
- HCMR-Greece presented values of reproducibility between 2.6-12.7 %, while CETI-Montenegro reported 12.5 %for all compounds;
- ISPRA-Italy did not provide any information on RSD %.

In relation to brominated compounds in sediments:

- RBI-Croatia and AUT-Albania reported the same value of reproducibility (40 % at LOQ).

In relation to organic matter, the method adopted by ISPRA-Italy for TOC analyses presented the RSD of 0.16%.

Regarding grain size analysis and water in sediments, no information was available.

Remarks

- Regarding pesticides and biocides (including added compounds aldrine, dieldrine and methoxyclor), AUT-Albania reported that "IAEA 383 certified sediment sample for chlorinated pollutants was used for method validation";
- ARSO-Slovenia presented values of measurement uncertainty (%) for all the compounds analyzed; ARSO-Slovenia specified that “Indeno(1,2,3-cd)piren” was intended as indenopyrene;
- NIB-Slovenia and ARSO-Slovenia specified that grain size analyses were not included in the monitoring program.

BIOTA MATRIX

For the "BIOTA" matrix (Attachment 3) the following GENERAL OBSERVATIONS were made:

- The HCMR-Greece, CETI-Montenegro and IOF-Croatia laboratories analyzed all pesticides and biocides requested, while in ARSO-Slovenia and RBI-Croatia only HCB was analyzed. On the contrary, ISPRA-Italy analyzed DDT, DDE and DDD (no information on HCB was reported). AUT-Albania reported only the method of analysis for this group of contaminants (no other information was provided). UoM-IMBK-Montenegro and NIB-Slovenia did not analyze this group of contaminants.
- Both TBT and TPhT were analyzed in the laboratories of RBI-Croatia and CETI-Montenegro, while ARSO-Slovenia analyzed only TBT. HCMR-Greece, UoM-IMBK-Montenegro, AUT-Albania, ISPRA-Italy, NIB-Slovenia and IOF-Croatia did not analyze this group of contaminants.
- Analysis of the concentrations of pharmaceutical products has not been carried out by any laboratory.
- Only the CETI-Montenegro laboratory carried out analyses of all the heavy metals required, while only a part of the analytes was analyzed in HCMR-Greece (Cd, Cr, Cu, Fe, Zn and Ni), ARSO-Slovenia (Hg, Cd, Pb, Cr, Cu, Zn and Ni), IOF-Croatia (Hg, Cd, Pb, Cr, Cu and Zn) and UoM-IMBK-Montenegro (for Hg, Cd, Cu, Fe, Zn most of the required information was provided, while for Pb, Cr, As and Ni only the specification about dry weight was added to the questionnaire). ISPRA-Italy analyzed all required metals except for vanadium. None of the heavy metals was analyzed by AUT-Albania, RBI-Croatia and NIB-Slovenia.
- Only CETI-Montenegro and ISPRA-Italy laboratories carried out analyses of all the hydrocarbons required, while only part of the requested list was considered by HCMR-Greece (aliphatic hydrocarbons C_6-C_{12} and monocyclic aromatic hydrocarbons, i.e.: benzene, toluene, ethylbenzene and o,m,p-Xylene, were not analyzed) and both ARSO-Slovenia and NIB-Slovenia (not analyzed total hydrocarbon, aliphatic hydrocarbons C_6-C_{12} and monocyclic hydrocarbons). RBI-Croatia and AUT-Albania have provided information only on fluoranthene and benzo(a)pyrene. UoM-IMBK-Montenegro and IOF-Croatia did not analyze any of the hydrocarbons.
- Analysis of brominated compounds was carried out only in RBI-Croatia, ARSO-Slovenia and AUT-Albania.
- The lipid content was analysed only by CETI-Montenegro and ISPRA-Italy laboratories.
- The water content was analyzed only by CETI-Montenegro.

- Some additional contaminants were introduced in the questionnaires: AUT-Albania added aldrin, dieldrin and methoxiclor to the group of pesticides and biocides (but the only information available was the method of analysis); manganese was added by HCMR-Greece to the group of heavy metals; ISPRA-Italy added the analyses of manganese, selenium and cobalt; The RBI-Croatia laboratory added hexachlorobenzene (which was copied to the row "HCB" of the original questionnaire in the comparative tables), hexachlorobutadiene, hexabromocyclododecane, PFOS and PFOA.

All the laboratories reported the UNITS OF MEASUREMENT as indicated in the basic questionnaire, also for the analytes that were not analyzed.

Regarding the indication of WET or DRY WEIGHT:

- The HCMR-Greece, ARSO-Slovenia, RBI-Croatia, ISPRA-Italy and CETI-Montenegro laboratories specified that the results for pesticides and biocides analyzed have been reported as wet weight, while IOF-Croatia reported their results as dry-weight.
- The data on antifoulants reported by RBI-Croatia were expressed as dry weight, while CETI-Montenegro and ARSO-Slovenia used wet weight.
- Results of heavy metal analyses were reported as wet weight by the CETI-Montenegro and ARSO-Slovenia laboratories, while HCMR-Greece, UoM-IMBK-Montenegro, ISPRA-Italy and IOF-Croatia reported the dry weight for the considered analytes.
- All laboratories that carried out analyzes of hydrocarbons expressed the results as wet weight, with the exception of ISPRA-Italy and NIB-Slovenia, that expressed results as dry weight.
- All laboratories that have carried out analyzes of the brominated compounds have reported the results as wet weight.
- The lipid content determined by CETI-Montenegro has been reported with respect to wet weight, while ISPRA-Italy did not provide this information.
- The water content was expressed as wet weight by CETI-Montenegro.

As for the analyzed SPECIES:

- *Mytilus galloprovincialis* has been used for the analysis of antifoulants, heavy metals and hydrocarbons and pesticides and biocides by all institutes, where reported (with the exception of RBI-Croatia, which analyzed HCB in fish, but species was not specified).
- The brominated compounds were determined in samples of *Mytilus galloprovincialis* in the ARSO-Slovenia laboratory, while fish tissue was analyzed in the laboratories of RBI-Croatia and AUT-Albania.
- *Mytilus galloprovincialis* was used for the analysis of lipid and water content determined by CETI-Montenegro.

Some inconsistencies have been observed in reporting the name of the TISSUE used for the analyzes, and the need to standardize the naming of tissues is evident.

- For pesticides and biocides, the HCMR-Greece and CETI-Montenegro laboratories reported "muscle", whereas ARSO-Slovenia called it "mussel tissue"; "total sample" was analysed in ISPRA-Italy and "whole soft tissue" was used in IOF-Croatia; RBI-Croatia analyzed "fish fillet".
- For the analysis of antifoulants, RBI-Croatia reported "all soft tissues", ARSO-Slovenia "mussel tissue" and CETI-Montenegro "muscle".
- Six different terms appeared for tissues employed for heavy metals analysis: "whole body tissue" in HCMR-Greece, "soft tissue" in UoM-IMBK-Montenegro, "mussel tissue" in ARSO-Slovenia, "muscle" in CETI-Montenegro, "total sample" in ISPRA-Italy and "whole tissue" in IOF-Croatia.
- Also for hydrocarbons the following denominations were observed: "muscle" in HCMR-Greece and CETI-Montenegro, "all soft tissues" in RBI-Croatia and AUT-Albania, "mussel tissue" in both ARSO-Slovenia and NIB-Slovenia, and "total sample" in ISPRA-Italy.
- Brominated compounds were determined on fish fillet in RBI-Croatia and AUT-Albania, and on mussel tissue in ARSO-Slovenia.
- Lipid and water content reported by CETI-Montenegro have been analyzed on muscle.

The NUMBER OF REPLICATES varied between countries and contaminants.

- Pesticides and biocides were analyzed in one replicate by HCMR-Greece and IOF-Croatia, in two replicates by CETI-Montenegro; ISPRA-Italy, RBI-Croatia and ARSO-Slovenia did not report the number of replicates.
- For the analysis of antifoulants the number of replicates was not reported by RBI-Croatia, while ARSO-Slovenia laboratory made 3 replicates and CETI-Montenegro analyzed 2 of them.
- Heavy metals were analyzed in two replicates in ARSO-Slovenia, CETI-Montenegro and UoM-IMBK-Montenegro, while in HCMR-Greece 5 replicates were used. IOF-Croatia made 1-5 replicates. ISPRA-Italy did not specify the number of replicates.
- For analysis of hydrocarbons CETI-Montenegro, ARSO-Slovenia and NIB-Slovenia made two replicates, while HCMR-Greece conduced the determination in one replicate. No information concerning the number of replicates was provided by RBI-Croatia, AUT-Albania and ISPRA-Italy.
- No information was provided on the number of replicates of brominated compounds from RBI-Croatia, ARSO-Slovenia and AUT-Albania.
- The lipid and water content determinations carried out in CETI-Montenegro were performed in two replicates. ISPRA-Italy did not provide the information on the number of replicates analysed for lipid content determination.

Sample STORAGE was uniform for all contaminant groups in most of laboratories (except ISPRA-Italy and IOF-Croatia), but there were differences between laboratories:

- HCMR-Greece, RBI-Croatia, ARSO-Slovenia, NIB-Slovenia and AUT-Albania stored samples of all groups of determinants at -20 °C. In UoM-IMBK-Montenegro samples were frozen at -20°C, freeze-dried and stored at 4 °C. Laboratory of CETI-Montenegro stored all samples at the temperature of 1-5 °C.
- ISPRA-Italy kept at -20 °C samples for the analyses of heavy metals, polycyclic hydrocarbons, total hydrocarbons and aliphatic hydrocarbons C_{10}-C_{40}, while samples for the analyses of aliphatic hydrocarbons C_6-C_{12} and monocyclic hydrocarbons were stored at 4 °C with ammonium bisulphate addition.
- IOF-Croatia used freeze-dried samples stored at -20 °C for pesticides and biocides analyses, while untreated tissue frozen at -40 °C was used for heavy metals analyses.

There were several differences regarding the ANALYTICAL METHODS applied in different laboratories, considering that in some cases, only the name of analytical instrumentation was reported and in other cases, the name/number of the official method was provided.

- Pesticides and biocides have been determined by GC-ECD in HCMR-Greece and in ARSO-Slovenia (modified EN 1528/1-4 method); RBI-Croatia and ISPRA-Italy used GC-MS; CETI-Montenegro reported to apply the AOAC 983.21 method. IOF-Croatia referred only to use UNEP/IOC/IAEA 1996 method, without specifying the method number or analytical technique.
- Antifoulants were determined by gas chromatographic methods in RBI-Croatia (GC-PFPD), in ARSO-Slovenia (modified ISO 17353 (GC-ICP-MS)) and in CETI-Montenegro (modified ISO 23161:2009).
- Methods to determine heavy metals differed not only between laboratories, but also between different metals:
  - mercury determinations were conduced with HVG (AAS) in UoM-IMBK-Montenegro, with the EPA 7473:2007 (DMA) method in ARSO-Slovenia, with Solid AAS-Mercury Analyser in CETI-Montenegro, by means of EPA7473 (AAS-DMA) in ISPRA-Italy and with CV-AAS in IOF-Croatia;
  - cadmium was determined with GF AA in HCMR-Greece and IOF-Croatia, AAS-Flame in UoM-IMBK-Montenegro, the SIST EN 15763 (ICP/MS) method in ARSO-Slovenia, the MEST EN 14084:2009 method in CETI-Montenegro and with the UNI EN ISO 17294-2 in ISPRA-Italy;
  - arsenic was analyzed in CETI-Montenegro using the AOAC 986.15 method and in ISPRA-Italy according to UNI EN ISO 17294-2;
  - remaining metals were analyzed by Flame AA in HCMR-Greece and in UoM-IMBK-Montenegro (Flame AAS), by GF AAS in IOF-Croatia, by the SIST EN

15763 modified (ICP/MS) method in ARSO-Slovenia, by the MEST EN 14084:2009 modified method in CETI-Montenegro and by EPA 3052 + EPA6010C modified method (ICP-OES) in ISPRA-Italy.

- Polycyclic aromatic hydrocarbons were analyzed by means of GC-MS in HCMR-Greece, RBI-Croatia and AUT-Albania (in the last two only fluoranthene and benzo(a)pyrene), using IM/GC-MSD/SOP 112 (GC-MS) in ARSO-Slovenia and NIB-Slovenia, with EPA 8270D-2007+internal method in ISPRA-Italy, with QuEChERS-PAH method in CETI-Montenegro.
  Total hydrocarbons have been determined in HCMR-Greece (by GC-FID), ISPRA-Italy (HPLC fluorescence) and in CETI-Montenegro (no analytical method was indicated).
  Aliphatic hydrocarbons C_6-C_{12} and monocyclic hydrocarbons were determined in CETI-Montenegro (headspace method) and in ISPRA-Italy (EPA 5021A-2003 + EPA 8260C-2006 (GCMS)).
  Aliphatic hydrocarbons C_{10}-C_{40} have been analyzed by means of GC-FID in HCMR-Greece and in ISPRA-Italy (ISO 16703-2004 (GC FID)), by means of IM/GC MSD (GC/MS/NCI) in ARSO-Slovenia and NIB-Slovenia, and by the use of an "in-house" method (no specifications provided) in CETI-Montenegro.
- Brominated compounds were determined in RBI-Croatia and AUT-Albania by the use of GC-ICPMS, as well as in ARSO-Slovenia where the EPA 1614 (HRGC/HRMS) method was applied.
- In order to determine the lipid content, CETI-Montenegro laboratory used AOAC method 948.15, and ISPRA-Italy applied EPA 8290A-2007 method.
- The water content was analysed in CETI-Montenegro by AOAC method 952.08.

The LIMITS OF DETECTION (LOD) were not reported for all the requested analytes, in particular information on LOD is missing in the questionnaires of RBI-Croatia and AUT-Albania.

- The lowest LOD for pesticides and biocides was obtained in the HCMR-Greece (0.01 µg/kg w.w. for all compounds), followed by CETI-Montenegro (0.5 µg/kg w.w. for all compounds) and ARSO-Slovenia (1 µg/kg w.w. only for HCB). IOF-Croatia reported values of 0.05-0.09 µg/kg on dry weight, which, if converted to wet weight would be about 0.01-0.02 µg/kg. No LOD was reported by ISPRA-Italy.
- As regards antifoulants, the ARSO-Slovenia laboratory recorded a LOD of 0.17 µg/kg w.w. (TBT only), while the CETI-Montenegro laboratory was able to detect both TBT and TPhT at a concentration of 1 µg/kg w.w.
- No LOD has been reported for heavy metals by HCMR-Greece and ISPRA-Italy, whereas only LOD for some of the analyzed metals was reported by UoM-IMBK-Montenegro (0.12 mg/kg d.w. for Cd, 0.4 mg/kg d.w. for Cu, 1.2 mg/kg d.w. for Fe and 0.08 mg/kg d.w. for Zn), ARSO-Slovenia (0.002 mg/kg w.w. for Hg, 0.005 mg/kg w.w. for Cd, 0.01 mg/kg w.w. for Pb) and IOF-

Croatia (0.0005 mg/kg d.w. for Hg, 0.015 mg/kg d.w. for Cd, 0.02 mg/kd d.w. for Pb, 0.03 mg/kg d.w. for Cr, 0.005 mg/kg d.w. for Cu and 0.05 mg/kg d.w. for Zn). The complete list of LOD values was provided by CETI-Montenegro (in the range of 0.001-0.1 mg/kg w.w.), and in almost all cases the reported LODs were lower than those presented by ARSO-Slovenia and UoM-IMBK-Montenegro and higher than those of IOF-Croatia (except for Cd).

- The highest LOD values for policyclic aromatic hydrocarbons were provided by ARSO-Slovenia (1 µg/kg w.w.) and CETI-Montenegro (0.2 µg/kg w.w.), whereas the lowest LODs were obtained by HCRM-Greece (0.01-0.07 µg/kg w.w.). ISPRA-Italy and NIB-Slovenia reported LODs of 1.5 µg/kg and 1 µg/kg, respectively, expressed as dry weight, which as wet weight would be about 0.3-0.4 µg/kg and 0.2-0.25 µg/kg, respectively (usually the dry weight/wet weight ratio in *Mytilus galloprovincialis* is 0.2-0.25). RBI-Croatia and AUT-Albania did not provide LOD for fluoranthene and benzo(a)pyrene.
  HCMR-Greece was the only one to report LOD for total hydrocarbons (12.5 µg/kg w.w.).
  LOD for aliphatic hydrocarbons C_6-C_{12} was provided only from CETI-Montenegro (30 µg/kg w.w.). For the aliphatic hydrocarbons C_{10}-C_{40} it was presented by all five laboratories, with the best performance of 0.2 µg/kg d.w. presented by NIB-Slovenia (≈ 0.04 - 0.05 µg/kg w.w.), followed by ARSO-Slovenia (0.2 µg/kg w.w.), HCMR-Greece (12.5 µg/kg w.w.), ISPRA-Italy (350 µg/kg d.w., that would be about 70-90 µg/kg w.w.) and CETI-Montenegro (6000 µg/kg w.w.).
  CETI-Montenegro obtained a LOD of 1 µg/kg w.w. for monocyclic hydrocarbons, whereas ISPRA-Italy did not provide this information.
- Only ARSO-Slovenia reported the LOD for brominated compounds (0.00005 µg/kg w.w.).
- LOD for the lipid and water content analyzed in CETI-Montenegro were of 0.01 %.

The values of the QUANTIFICATION LIMITS (LOQ) have been provided for almost all the parameters analyzed.

- The lowest value of the LOQ for pesticides and biocides was obtained in the HCMR-Greece laboratory (0.03 µg/kg w.w. for all compounds), followed by IOF-Croatia (0.167 - 0.3 µg/kg d.w. for all four compounds), RBI-Croatia (0.1 µg/kg w.w. for HCB) and ISPRA-Italy (0.1 µg/kg w.w. for DDT, DDE and DDD), CETI-Montenegro (1 µg/kg w.w. for all compounds) and ARSO-Slovenia (3 µg/kg w.w. for HCB). Among the requested pesticides, the European Directive 2013/39/EU established biota EQS only for hexachlorobenzene (HCB) (10 µg/kg w.w.) and DDT (50 µg/kg w.w.), even if referred to fish. All laboratories that analyzed HCB and DDT were able to quantify the concentrations under EQS, with LOQs lower than 30 % of EQS, thus satisfying the Directive 2009/90/EU.

- With regard to antifoulants, the RBI-Croatia laboratory obtained LOQ of 5 µg/kg d.w. and 10 µg/kg d.w., respectively for TBT and TPhT. ARSO-Slovenia and CETI-Montenegro provided LOQs expressed as wet weight (ARSO-Slovenia: 0.59 µg TBT /kg w.w., CETI-Montenegro: 3 µg/kg w.w. for both TBT and TPhT). Considering that the wet weight/dry weight ratio in the organism analyzed (*Mytilus galloprovincialis*) is usually in the range of 0.2-0.25, if converted to dry weight these LOQ values would be at least four times higher than the values expressed on wet weight. Considering the transformation of the wet weight values, the best performance in terms of the LOQ was achieved by the ARSO-Slovenia laboratory, followed by the RBI-Croatia and CETI-Montenegro.
- Neither HCMR-Greece nor UoM-IMBK-Montenegro reported LOQ referring to heavy metals that were analyzed in their laboratories. CETI-Montenegro, ISPRA-Italy and IOF-Croatia provided the LOQ of all metals analyzed. ARSO-Slovenia reported LOQs only for three of analysed metals: 0.005 mg/kg w.w. for Hg, 0.01 mg/kg w.w. for Cd and 0.02 mg/kg w.w for Pb. ISPRA-Italy reported LOQ values expressed as dry weight in the range 0.0005-1 mg/kg, lower than those referred by IOF-Croatia (except for Cu). Considering the approximate transformation of the dry weight results in wet weight, the ISPRA-Italy method appeared to be most performing, for all the analysed metals (except for Cu, for which IOF-Croatia had more performing method). Considering that European Directive 2013/39/EU established an EQS of 20 µg/kg w.w. (equal to 0.02 mg/kg w.w.) for mercury and its compounds in biota (even if referred to fish), all the above mentioned laboratories were able to quantify the concentrations of Hg lower than EQS, and LOQs presented were lower than 30 % of EQS, fulfilling the requirements of the Directive 2009/90/EU.
- Fluoranthene and benzo(a)pyrene were the two hydrocarbons analysed by most of participants (UoM-IMBK-Montenegro and IOF-Croatia did not provide any information on the analyses of hydrocarbons). HCMR-Greece achieved the best results in terms of LOQ for most of policyclic aromatic hydrocarbons (0.07-0.22 µg/kg w.w.), with exception of benzo(a)pyrene, which was quantifiable at lower concentration (0.1 µg/kg w.w.) in RBI-Croatia and AUT-Albania laboratories. For all polycyclic aromatic hydrocarbons CETI-Montenegro reported LOQ of 0.6 µg/kg w.w., while ARSO-Slovenia obtained for these substances the LOQ of 2 µg/kg w.w.. ISPRA-Italy and NIB-Slovenia were the only laboratories that expressed the results as dry weight, reporting LOQ values of 3-5 µg/kg d.w. (ISPRA) and 2 µg/kg d.w. (NIB). These values, if transformed as wet weight, would result in about 0.7-1.2 µg/kg w.w. (ISPRA) and 0.4-0.5 µg/kg w.w. (NIB). The European Directive 2013/39/EU considers fluoranthene and benzo(a)pyrene as priority substances and their EQS were established (30 µg/kg w.w. for fluoranthene and 5 µg/kg w.w. for benzo(a)pyrene). All LOQs reported for these compounds (if expressed as wet weight) were lower than the required EQS. With the exception of ARSO-

Slovenia regarding benzo(a)pyrene, all presented LOQ values were sufficiently low to satisfy the Directive 2009/90/EU.
The HCMR-Greece laboratory was the only one to supply the LOQ for the total hydrocarbons (37.5 µg/kg w.w.).
The LOQ for aliphatic hydrocarbons C_6-C_{12} was reported by CETI-Montenegro (90 µg/kg w.w.) and ISPRA-Italy (5 µg/kg d.w.), while the LOQ for aliphatic hydrocarbons C_{10}-C_{40} was provided by NIB-Slovenia (0.5 µg/kg d.w.), ARSO-Slovenia (0.5 µg/kg w.w.), HCRM-Greece (37.5 µg/kg w.w.), ISPRA-Italy (500 µg/kg d.w.) and CETI-Montenegro (12000 µg/kg w.w.). Monocyclic aromatic hydrocarbons were analyzed and characterized with LOQ only by ISPRA-Italy (1-2 µg/kg d.w.) and CETI-Montenegro (3 µg/kg w.w.).

- The LOQ value for brominated diphenylethers of 0.0001 µg/kg w.w. has been reported by ARSO-Slovenia, while RBI-Croatia and AUT-Albania referred 0.1 µg/kg w.w.. Only ARSO-Slovenia method was performing enough to determine concentrations at the EQS level of 0.0085 µg/kg w.w., with LOQ satisfying the Directive 2009/90/EU.
- The LOQ of 0.1 %, related to the analysis of lipid and water content, was reported by CETI-Montenegro.

Most of the participants reported different REFERENCE MATERIALS used for quality control (QC) of analytical performance.

- For the control of pesticides and biocides, CETI-Montenegro used IAEA-451, while HCB determined in ARSO-Slovenia was controlled using an in-house reference material (without specifications). IOF-Croatia made use of IAEA-406. HCMR-Greece, RBI-Croatia and ISPRA-Italy did not provide a description of the reference materials.
- Regarding the antifoulants, ERM-CE477 mussel tissue was employed for QC of tributyltin (TBT) in RBI-Croatia and ARSO-Slovenia. None of the laboratories that carried out the TPhT analyzes (RBI-Croatia and CETI-Montenegro) reported information on the reference material used for quality control.
- Six different reference materials have been used in the analysis of heavy metals. NIST 2976 was used by UoM-IMBK-Montenegro (Hg, Cd, Cu, Fe and Zn), HCRM-Greece (Cd, Fe, Mn and Zn), CETI-Montenegro (Cr), ISPRA-Italy (Hg, Pb, Cr, Ba, Fe, Zn and Ni) and IOF-Croatia (Cd, Pb, Cr, Cu and Zn).
  IAEA 407 was employed by HCMR-Greece for Cr, Cu and Ni.
  NRC-CNRC DORM-4 was used in ARSO-Slovenia for Hg, Cd and Pb analysis.
  DORM-2 was emploed by ISPRA-Italy for Cd and As.
  CETI-Montenegro used SRM 1566 (oyster tissue) for almost all metals (except for Cr).
  Mussel tissue N°278 IRMM was used by ISPRA-Italy for Hg, Pb, Cr, Ba, Fe, Zn, Ni.

- Only CETI-Montenegro provided information on the use of reference material for polycyclic aromatic hydrocarbons (IAEA-451).
  ARSO-Slovenia and NIB-Slovenia reported only that an in-house reference material was used for aliphatic hydrocarbons C_{10}-C_{40}.
  No information was available on reference materials for total hydrocarbons, aliphatic hydrocarbons C_6-C_{12}, and monocyclic aromatic hydrocarbons.
- The brominated compounds were subjected to QC by means of an in-house reference material in ARSO-Slovenia, while AUT-Albania and RBI-Croatia did not provide information.
- No details on references for the analysis of lipid and water content were presented in this section, but CETI-Montenegro specified in the section INTERCALIBRATION EXERCISE that Matrix Meat Reference Material SMRD 2000 was employed for lipid and water content.

Five laboratories (ARSO-Slovenia, NIB-Slovenia, CETI-Montenegro, ISPRA-Italy and IOF-Croatia) participated in some INTERCALIBRATION EXERCISES. CETI-Montenegro laboratory has subjected almost all methods to the intercalibration in the IAEA circuit, with the exception of the following parameters: antifoulants, aliphatic hydrocarbons C_6-C_{12}, monocyclic aromatic hydrocarbons, brominated compounds. Matrix Meat Reference Material SMRD 2000 was employed for lipid and water content (this information should have been reported in REFERENCE MATERIALS).
ARSO-Slovenia participated in FAPAS intercalibration for heavy metals and EU-RL proficiency tests for polycyclic aromatic hydrocarbons, aliphatic hydrocarbons C_{10}-C_{40} and brominated compounds. NIB-Slovenia reported the participation in EU-RL for polycyclic hydrocarbons and aliphatic hydrocarbons C_{10}-C_{40}. ISPRA-Italy took part in QUASIMEME circuit for heavy metals. IOF-Croatia took part in IAEA/MEL/Medpol intercalibration proficiency test for pesticides and biocides.

Little information has been provided regarding the ACCURACY of analytical methods.

- No details were given on the accuracy of methods for the analysis of hydrocarbons, brominated compounds and lipid and water content.
- For pesticides and biocides, only IOF-Croatia provided results on accuracy as the % difference between the test result and the reference value, in the range of 3.89 – 10.01 %.
- For antifoulants, a good accuracy (as recovery %) of TBT analysis was reported by ARSO-Slovenia (90-103 % wrt certified value), while RBI-Croatia and CETI-Montenegro did not provide any information.
- The methods for determining heavy metals were characterized with good accuracy, based on information provided by UoM-IMBK-Montenegro (92-102 % wrt certified value), IOF-Croatia (95 % wrt certified value) and ARSO-Slovenia (96-106 % wrt certified value). Data provided by HCMR-Greece were

expressed as the % difference between the test result and the reference value, and are in the range of 1 – 12 %.

- RBI-Croatia, AUT-Albania, CETI-Montenegro, ISPRA-Italy and NIB-Slovenia did not provide any information on accuracy.

As already observed for other parameters, REPRODUCIBILITY information is not complete. It was difficult to compare the performance of the methods used, as it was not clear at what level of concentration the reproducibility reported by the laboratories was assessed (only the RBI-Croatia and AUT-Albania indicated that the RSD % was calculated at the LOQ concentration).

- A good reproducibility for pesticides and biocides methods was obtained by IOF-Croatia (0.83 – 8.33 % for all parameters) and HCMR-Greece (6.8 - 8.4 % for all parameters), followed by CETI-Montenegro (RSD of 12.5% for compounds) and RBI-Croatia (RSD = 40% at LOQ only for HCB). ARSO-Slovenia and ISPRA-Italy did not present the reproducibility values.
- CETI-Montenegro did not present the data on the reproducibility for antifoulants. RBI-Croatia has declared RSD of 20 % at LOQ concentration. A good performance in terms of reproducibility for TBT was obtained by ARSO-Slovenia (RSD=6 %).
- HCMR-Greece and UoM-IMBK-Montenegro did not provide the RSD % for the analysis of heavy metals, whereas all data reported by ARSO-Slovenia, CETI-Montenegro and IOF-Croatia were below 10 %, indicating a good performance of the methods in terms of reproducibility. ISPRA-Italy indicated RSD of 5-15% for each heavy metal analysed.
- The methods employed for analysis of polycyclic aromatic hydrocarbons and aliphatic hydrocarbons C_{10}-C_{40} in HCMR-Greece and CETI-Montenegro were characterized by a good reproducibility (RSD < 15 %). RBI-Croatia and AUT-Albania, which only analyzed fluoranthene and benzo(a)pyrene, reported that the reproducibility at LOQ concentration was 40 %. ARSO-Slovenia, NIB-Slovenia and ISPRA-Italy did not report any information on RSD % in hydrocarbons. No information was available about aliphatic hydrocarbons C_6-C_{12} and monocyclic aromatic hydrocarbons.
- The precision of methods used by the RBI-Croatia and AUT-Albania laboratories for the analysis of brominated compounds was of 40 % at LOQ concentrations. ARSO-Slovenia did not report the value of RSD %.
- The determinations of lipid and water content carried out by CETI-Montenegro were characterized by reproducibility of 2 % and 1.8 %, respectively.

In REMARKS section, ARSO-Slovenia and NIB-SLovenia specified that "Indeno(1,2,3-cd)piren" was intended as indenopyrene.

An additional parameter "UNCERTAINTY" has been included in the questionnaire by ARSO-Slovenia and NIB-Slovenia laboratories (the same values were presented by both Institutes for hydrocarbons), but it has not been specified whether it is the combined standard uncertainty or the extended uncertainty.

Data Gaps

As pointed out by the staff of the NIB-Slovenia, the data presented in that Institute's questionnaire do not come from monitoring, but from scientific research. Many of the parameters included in the questionnaire were analyzed for NIB in external laboratories, which did not provide all the information on the methods used (such as LOD, LOQ, intercalibration exercises and reference materials), thus not allowing NIB to provide complete information.

Conclusion

On the basis of the outcomes of the protocol review, and with the aim to define a Methodological proposal for harmonized sampling procedure, analytical methodologies, the following main targets of harmonization are proposed:

- Matrix characteristics
- Measurement units
- Sampling procedures
- Indications on sample storage and methods of analysis
- Indications on LOQ.

3. Annex:

Analysis of questionnaires (zip file)

4. References:

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