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JRC TECHNICAL REPORT

EMBLAS Plus
Joint Black Sea Survey 2019
"JRC Chemical Contaminant Measurements"

Sampling, analytical methodologies and results of ultra-trace organic contaminants monitoring

Mariani, G., Tavazzi, S., Skejo, H., Comero, S., Oswald, P., Litvinova, M., Gawlik, B.M., Hanke, G.

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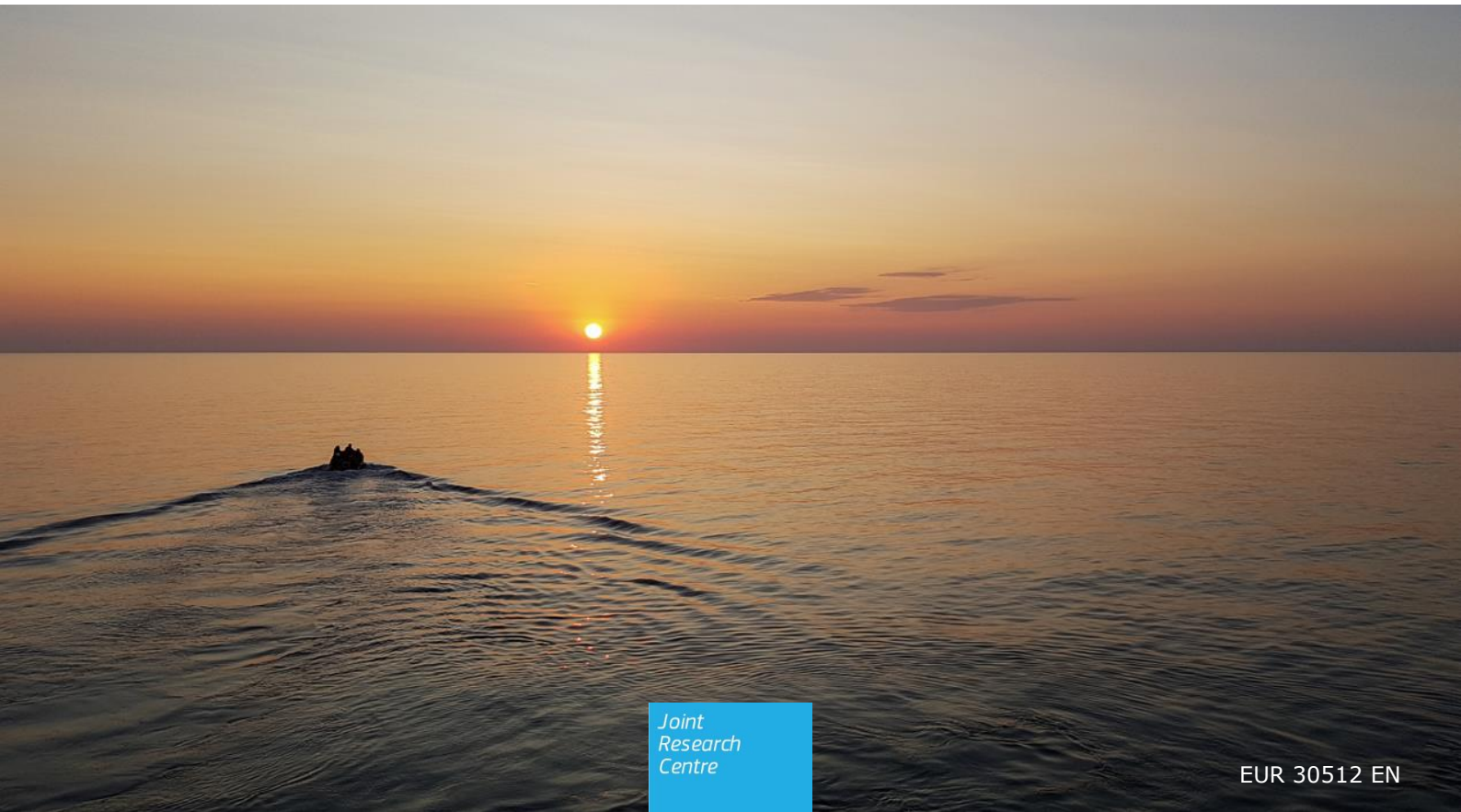

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Abstract

JRC provided sampling support and ultra-trace organic analytical measurements of marine contaminants in the framework of the support to DG NEAR (C2 – Neighbourhood East) for the EMBLAS_Plus project. Aim is to improve the monitoring and the availability of analytical data for the Black Sea. This report compiles the analytical results together with information on sampling, sample preparation, analytical instrumentation, analytical conditions and Quality Assurance/Quality Control information. On 4 transects and 21 spot sampling points a total of 39 samples were collected and 112 substances analysed. Contaminants selected from the so-called WFD EU-Watch list, from the Danube specific pollutants list (DRSPs), from the priority substances list of the Directive 2013/39/EU on priority substances in the field of water policy as well as from a list of flame retardants. The analytical measurements provide 4088 individual results as contribution to the environmental assessment of the Black Sea.

1. Introduction

The European Commission Joint Research Centre JRC provides support to the implementation of the Marine Strategy Framework Directive MSFD (EU 2008), aiming at achieving or maintaining good environmental status of the European Seas. The marine pollution by chemical contaminants is addressed by Descriptors D8 and D9 of the MSFD. Criteria and methodological standards as well as approaches for monitoring and assessment are specified in the Commission Decision 2017/848/EU (EU 2017). The protection of the European Seas requires a close collaboration across borders and with EU neighbouring countries in the shared marine basins. Therefore, scientific collaboration and the application of agreed approaches are needed in order to derive comparable assessments results for marine pollution issues. The JRC is providing specific technical information for these harmonization processes, e.g. on the selection and prioritization of chemical substances in the marine environment (Tornero 2016, Tornero 2017, Tornero 2018). Further information can be found on the website of the JRC MSFD Competence Centre (http://mcc.jrc.ec.europa.eu/).

The work presented in this report is aiming at improved chemical pollution monitoring of the Black Sea environment, enhancing the regional cooperation in the Black Sea area. Furthermore the aim is to increase the alignment with MSFD principles in a shared sea and at an improved collaboration with EU associated and neighbouring countries, in order to provide the basis for measures against chemical contaminants, including emerging substances. The analyses complement other work done during EMBLAS-Plus in different environmental matrices and on different substances. The water matrix, including the contained particulate suspended matter, represents the mobile fraction of marine chemical contaminants, links to riverine sources and reflects the exposure of pelagic wildlife to trace pollution. While this technical report is providing analytical results and technical considerations about the methodologies and their application, the interpretation of the results, the assessment of contamination levels and identification of sources is not in the scope of this report.

The DG NEAR project EMBLAS-Plus (http://emblasproject.org/) aims at improving the protection of the Black Sea environment. It continues the work of EMBLAS and EMBLAS II projects, providing comparable results from multiannual surveys. The project is addressing the overall need for support in protection and restoring the environmental quality and sustainability of the Black Sea. The availability and quality of data on the chemical and biological status of the Black Sea should be improved, in line with the Marine Strategy Framework Directive MSFD and expected Black Sea Strategic Action Plan needs.

DG JRC, Directorate for Sustainable Resources, through the Water and Marine Resources Unit provided support to this project by chemical analysis of selected organic trace contaminants in sea water samples and for monitoring of marine litter.

2. Activities

Following analytical support work to EMBLAS and EMBLAS II in 2016 and 2017 (EMBLAS, Mariani et al, 2017 and 2018), JRC provided, besides continued collaboration for the monitoring of marine litter, extended support in 2019. In close collaboration with the EMBLAS Plus coordination team and the Slovak Environmental Institute, sampling strategies and work planning have been agreed.

The samples for organic trace contaminant analysis have been taken during the summer 2019 EMBLAS Plus Joint Black Sea Survey and during dedicated field campaigns in Ukraine and Georgia.

Peter Oswald a scientist from the Slovak Environmental Institute visited JRC in order to prepare sampling material for the cruise. The material was transported by the Slovak Environmental Institute to the departure port, Costanta, Romania and the sampling/ extraction devices were installed on Mare Nigrum on 26.7.2019.

The sampling on board was performed by Peter Oswald and supported by Georg Hanke, JRC, who joined the cruise for the first 1 day leg from Constanta, Romania to Odessa, Ukraine.

The sampling activities included collecting 20 L spot samples, taken with 20 L stainless steel tanks on the open sea water surface from a small boat in a distance from the research vessel. Further 20 L samples have been taken in coastal areas of Ukraine and Georgia. 3 samples have been taken in the coastal area of Georgia as well as 3 in Ukrainain coastal area, 3 outside the Danube delta area, 12 samples in open sea and 5 samples for QA/QC and as replicates. The 20 L samples have been filtered and extracted on-board of Mare Nigrum with a JRC developed manifold (Mariani 2017, Mariani 2017a, Mariani 2018) onto filtration/extraction disks.

A second set of samples was collected during ships transect, providing large scale integrated sampling. The large volume seawater sampling (Large Volume Transect Sampling, LV-TS) system has been installed on board the Mare Nigrum. This system provided water samples (typically of 180-400 L volume) during transects of the moving ship for on-line filtration and extraction with two subsequent cartridges for later instrumental analysis at JRC.

A total of 5 transects was sampled across the Black Sea between Georgia and Ukraine with the Large Volume Transect Sampling (LV-TS) method. The first transect was sampled in collaboration for non-target analysis by the University of Athen, while the other four were sampled for analysis in the JRC laboratory.

The resulting filtration/extraction disks and cartridges have been transported by the SK Environmental Institute to JRC in Ispra, Italy. After the cruise, the visiting scientists Peter Oswald and Marina Litvinova in collaboration with Helle Skejo (JRC), prepared samples for instrumental analysis in the JRC Ispra laboratory facility. All the samples were extracted in the JRC laboratory and the extracts of LV-TS sampled for the partners were delivered to the laboratories of the University of Athens.

The analyses of the samples at JRC were performed by Gas Chromatography-High Resolution Mass Spectrometry (HRGC-HRMS) by Giulio Mariani, and by High Performance Liquid Chromatography-Mass Spectrometry, performed by Simona Tavazzi.

In the context of the fourth Joint Danube Survey (JDS-4), which took place at the end of June - beginning of July 2019, in agreement with ICPDR, several contaminants were selected from the so-called WFD EU-Watch list, from the Danube specific pollutants list (DRSPs), from the priority substances of the Directive 2013/39/EU as well as from a list of flame retardants.

Since the Danube River is the main tributary of the Black Sea, in order to take advantage of the concomitance of the monitoring campaigns of JDS4 and Emblas-Plus, it was decided to measure the same compounds in the two projects.

In the following list all chemicals analysed in the EMBLAS Plus project are reported; compounds written in blue are those also analysed in JDS4 (60 out of 112).

Polar Chemicals	Chlorinated Pesticides	Phosphate Flame Retardants	Polycyclic Aromatic Hydrocarbons (PAHs)	Polychlorobiphenyls Indicator-PCBs
10,11-dihydro-10,11-dihydroxy-carbamazepine	PeCBz	TEP	Phenanthrene	PCB 28
	HCB	TNPP	Anthracene	PCB 52
Acetamiprid	a-HCH	TIBP	Fluoranthene	PCB 101
Amoxicillin	b-HCH	TNBP	Pyrene	PCB 118
Atrazine	g-HCH	TCEP	Benz(a)anthracene	PCB 138
Atrazine-desethyl	d-HCH	TCPP	Chrysene	PCB 153
Atrazine-desisopropyl (desethyl-simazine)	e-HCH	TDCPP	Sum Benzo(b,j,k)fluoranthene	PCB 180
Azithromycin	Heptachlor	TBOEP	Benzo(e)pyrene	
Benzotriazole	Heptachlor-exo-epoxide	TPhP	Benzo(a)pyrene	
Bezafibrate	Heptachlor-endo-epoxide	EHDP	Perylene	
Bromacil	Aldrin	TEHP	Indeno(123-cd)pyrene	
Carbamazepine	Dieldrin	TMPP	Benzo(ghi)perylene	
Chloroxuron	Endrin	TIPPP	Dibenz(ah)anthracene	
Ciprofloxacin	Isodrin	T35DMPP	Coronene	
Clarithromycin	trans-chlordane			
Desethylterbutylazine	cis-chlordane		**Others**	
Diazinon	Oxychlordane			
Dicamba	trans-nonachlor		BHT	
Diclofenac	cis-nonachlor		EHMC	
Dimethenamid	Endosulfane-alpha			
E1	Endosulfane-beta			
E2	Endosulfane-sulphate			
EE2	op-DDE			
Fipronil	pp-DDE			
Ibuprofen	op-DDD			
Imidacloprid	pp-DDD			
Linuron	op-DDT			
Metaflumizone	pp-DDT			
Metazachlor	Methoxychlor			

Polar Chemicals	Chlorinated Pesticides	Phosphate Flame Retardants	Polycyclic Aromatic Hydrocarbons (PAHs)	Polychlorobiphenyls Indicator-PCBs
Metolachlor	Mirex			
Naproxen				
Simazine	**Other pesticides:**			
Sulfamethoxazole				
Tebuconazole	HCBD			
Terbutryn	Dichlorvos			
Terbutylazin-desethyl	Trifluralin			
Terbutylazine	Triallate			
	Chlorpyriphos			
	Chlorfenvinphos			
	Dicofol			
	Cypermethrins			
	Chlorotalonin			

The samples have been analysed with multi-compound methodologies including 112 substances selected from the following categories: corrosion inhibitor, antioxidants, anti-epileptic drug and metabolite, hypolipidemic agent, nonsteroidal anti-inflammatory drugs (NSAID), sunscreen, antibiotic, insecticide, herbicide, algicide, dielectric and coolant fluid, products by incomplete combustion of matter, plasticizers, phosphate flame retardants.

The sampling and analytical work provides 4088 final individual results.

3. Sampling and sample extraction

3.1. Mariani Box spot samples

During the cruise on the research vessel Mare Nigrum, 20 L water samples were collected from the sea surface using a glass fiber boat in distance from the research vessel, (Figure 1).

Figure 1. *Research vessel Mare Nigrum and the operations of 20L open sea spot sampling with a boat*

Coastal and open sea surface water spot samples were collected in 20 L steel tanks. The containers were previously cleaned with acetone and rinsed with Milli-Q water. Field blanks, reproducibility tests and break-through samples (BT) were collected in order to evaluate the efficiency of the extraction procedure.

A total of 21 different spots were sampled: 3 on the coast of Georgia (provided by Georgian partners), 3 (provided by Ukrainian partners) on the coast of the Ukraine, 3 outside the Danube delta and 12 samples in the open sea. In Table 1 samples and sampling conditions are reported.

Figures 2, 3, 4 and 5 show the maps of the sampling points. Figure 6 shows all the samples arrived in the laboratory after the cruise. Figures 7-13 depict filters as they appeared after extraction.

Table 1. *20L Spot samples and sampling description*

Sample Code	Latit.	Long.	Date	Water sampling Depth	Site Depth (meters)	Container Type	Filtration volume (L)	Internal Standard mix add (yes or no)	Notes
JBSS_GE_UA-1A	45º 15′ 00.0"	30º 15′ 00.0"	28/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.65	Y	
JBSS_GE_UA-2A	46º 20′ 00.0"	31º 00′ 00.0"	28/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.34	Y	
JBSS_GE_UA-3A	46º 29′ 00.0"	30º 49′ 00.0"	28/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.55	Y	
JBSS-GE_UA-1_1	46º 26′ 00.0"	31º 01′ 00.0"	29/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.30	Y	**Validation study**
JBSS-GE_UA-1_1BT			29/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank		Y	
JBSS-GE_UA-1_2			29/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.05	Y	
JBSS-GE_UA-1_2BT			29/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank		Y	
JBSS-GE_UA-2	45º 13′ 00.0"	31º 14′ 00.0"	30/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.89	Y	
JBSS-GE_UA-3	44º 51′ 00.0"	31º 18′ 00.0"	30/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.35	Y	
JBSS-GE_UA-4	44º 07′ 00.0"	31º 34′ 00.0"	30/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.15	Y	
JBSS-GE_UA-5	43º 24′ 00.0"	31º 50′ 00.0"	31/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.55	Y	
JBSS-GE_UA-6	43º 25′ 00.0"	32º 52′ 00.0"	31/7/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.80	Y	
JBSS-GE_UA-7	43º 22′ 00.0"	34º 46′ 00.0"	8/1/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.15	Y	

Sample Code	Latit.	Long.	Date	Water sampling Depth	Site Depth (meters)	Container Type	Filtration volume (L)	Internal Standard mix add (yes or no)	Notes
JBSS-GE_UA-8	43º 32′ 00.0"	36º 04′ 00.0"	8/1/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.15	Y	
JBSS-GE_UA-9	42º 14′ 00.0"	39º 53′ 00.0"	2/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.45	Y	
JBSS-GE_UA-10	42º 06′ 00.0"	40º 20′ 00.0"	3/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	18.90	Y	
JBSS-GE_UA-11	41º 56′ 00.0"	40º 50′ 00.0"	3/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.85	Y	
JBSS-GE_UA-12	41º 47′ 00.0"	41º 13′ 00.0"	3/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.22	Y	
JBSS_UA_10	46.295°	30.662°	4/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	20.00	Y	provided by Ukrainian partners
JBSS_UA_11	46.068°	30.463°	4/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.30	Y	provided by Ukrainian partners
JBSS_UA_15	46.607°	31.539°	4/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.55	Y	provided by Ukrainian partners
JBSS_GE_1	41°41'30.45''	41°42'12.64''	7/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.85	Y	provided by Georgian partners
JBSS_GE_2	41°39'3.28"	41°38'40.42''	7/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	20.05	Y	provided by Georgian partners
JBSS_GE_4	41°39'23.66''	41°37'59.1''	7/8/2019	Surface	0.5	20 Liter Sea Water samples in steel tank	19.47	Y	provided by Georgian partners
JBSS_Blank				Surface	0.5	20 Liter Sea Water samples in steel tank	19.28	Y	

Figure 2. *Danube delta and Coastal sampling points*

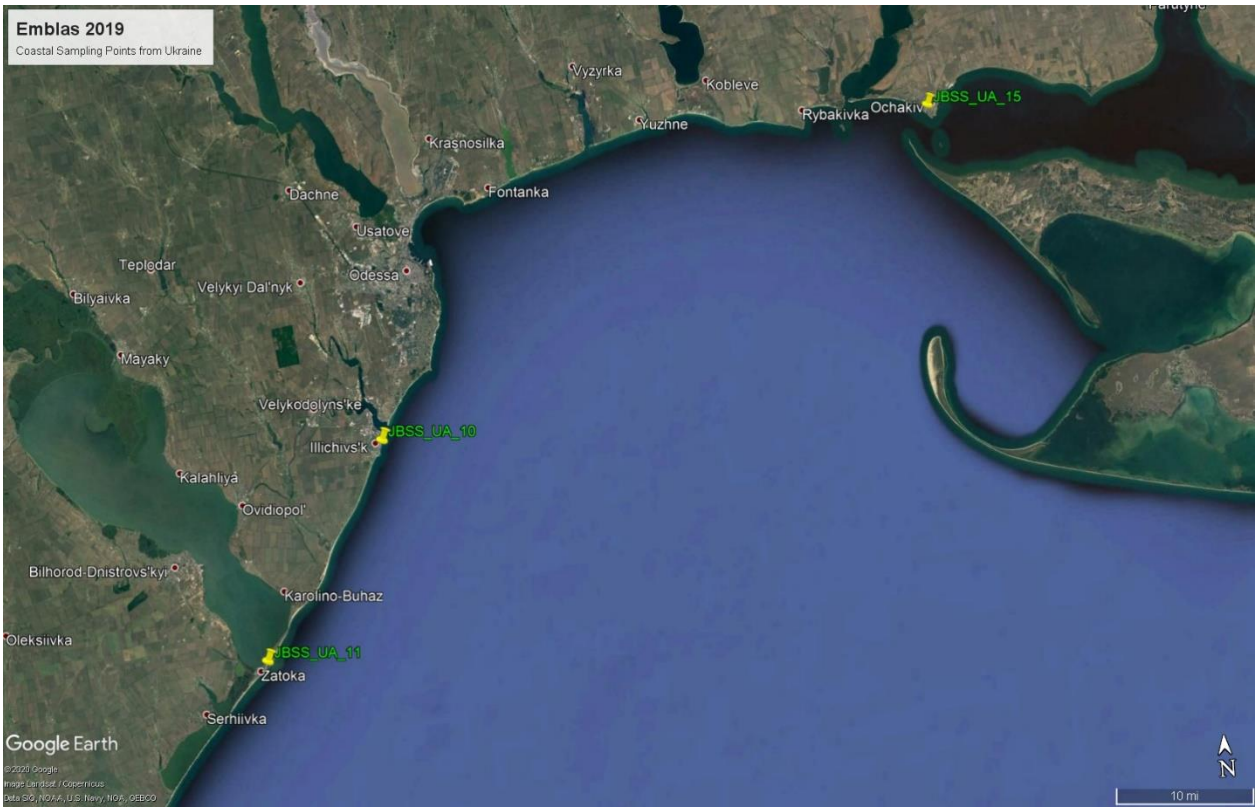


Figure 3. *Sampling points on coast of Ukraine, provided by Ukrainian partners*

Figure 4. *Sampling points on coast of Ukraine, provided by Georgian partners*

Figure 5. *12 Sampling points in open sea*

Figure 6. *Large volume transect and filter spot samples arrived in the laboratory after the cruise*

Figure 7. *HLB disks used for Field blanks*

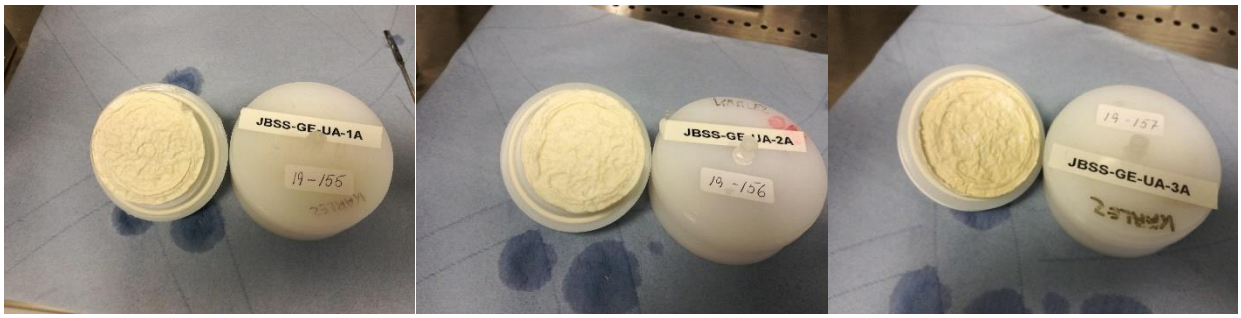


Figure 8. *HLB disks used for the coastal sampling in Ukraine and for the sampling outside the Danube delta (JBSS_GE_UA-1A)*

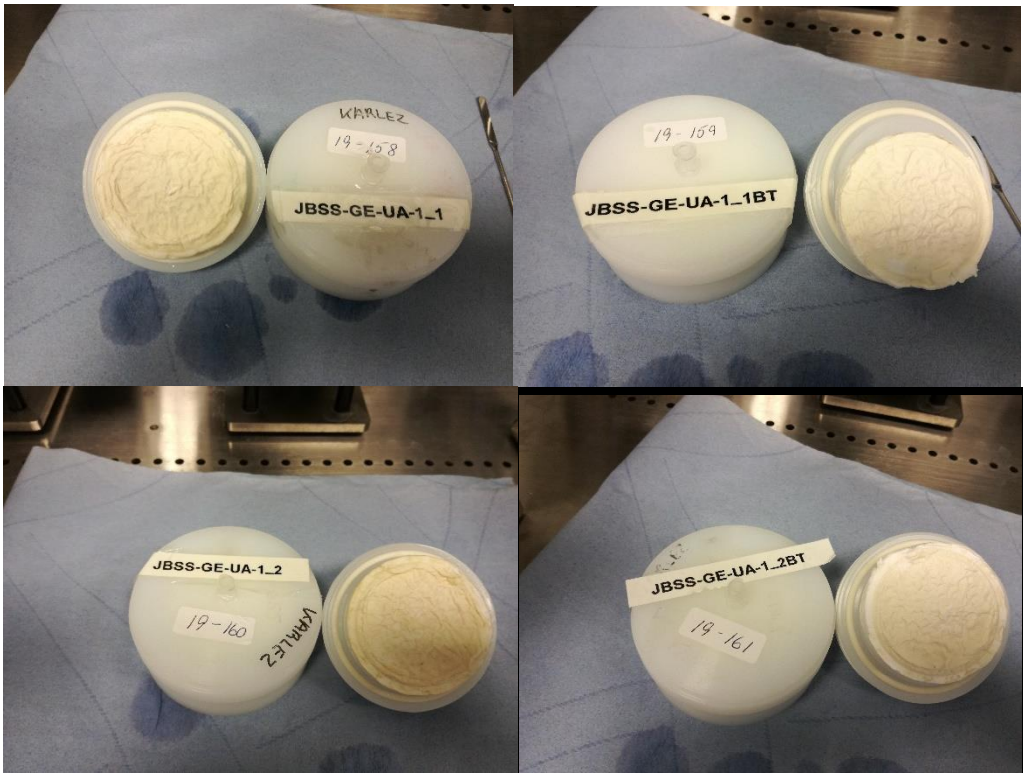


Figure 9. *HLB disks used for reproducibility tests and for breakthrough evaluation at the open sea sampling point JBSS-GE-UA-1*

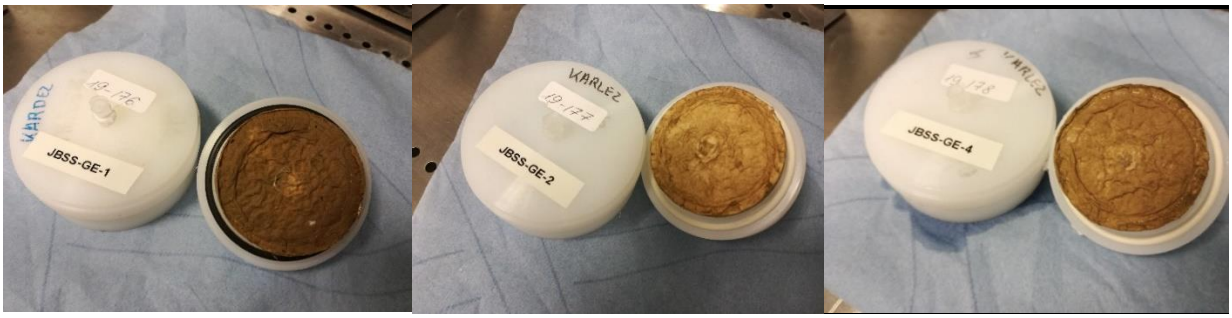


Figure 10. *HLB disks used for the coastal sampling in Georgia, provided by Georgian partners*

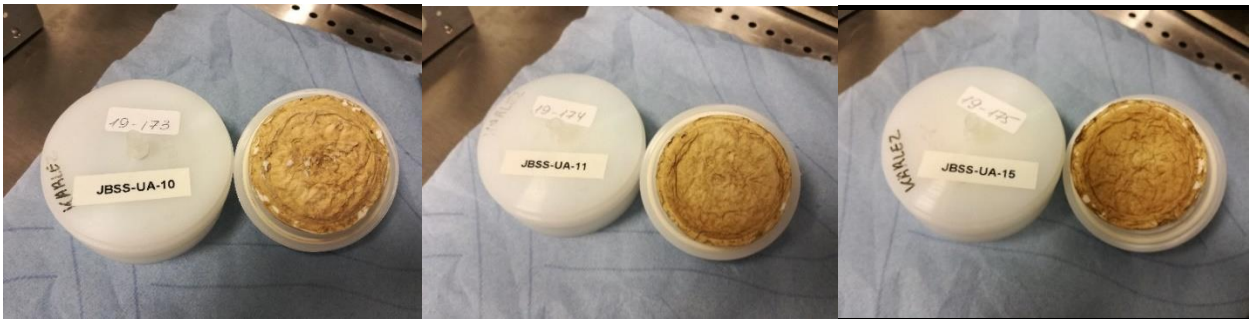


Figure 11. *HLB disks used for the coastal sampling in Ukraine, provided by Ukrainian partners*

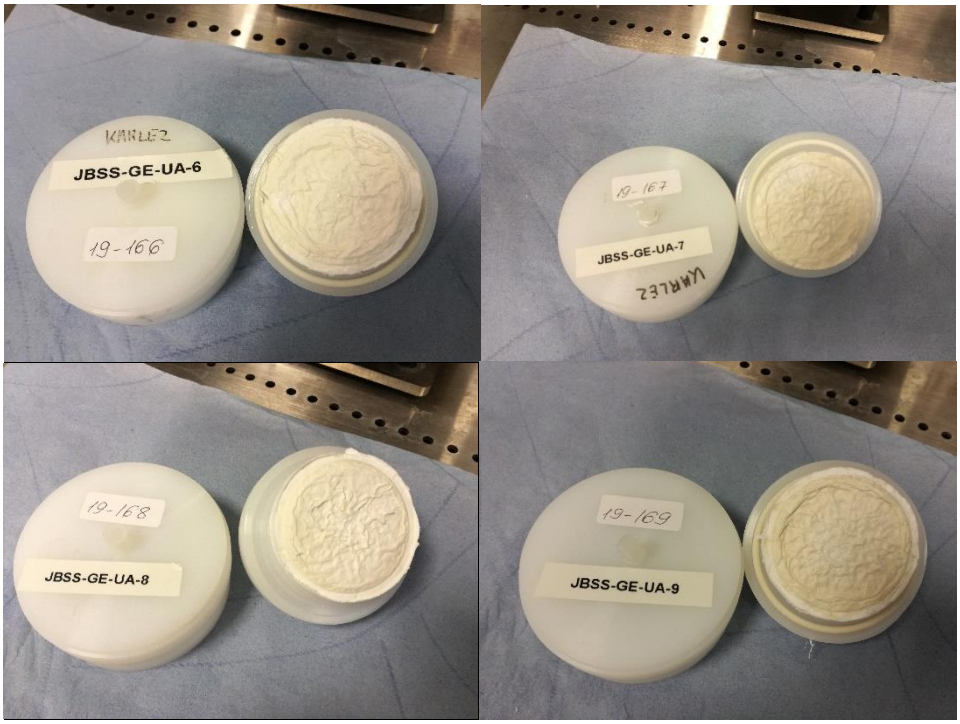


Figure 12. *HLB disks used in open sea for samples from JBSS-GE-UA-2 to JBSS-GE-UA-9*

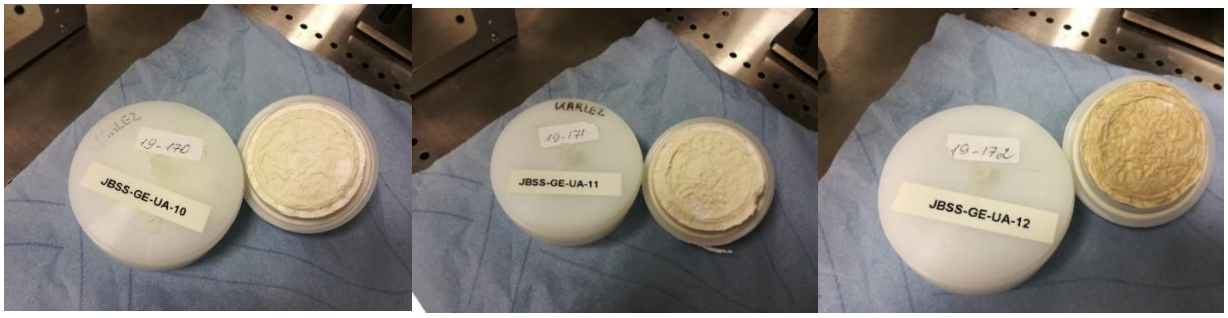


Figure 13. *HLB disks used in open sea for samples from JBSS-GE-UA-10 to JBSS-GE-UA-12*

3.2. Mariani Box Extraction method

The extraction was performed with a manifold combining filtration and extraction in a single field-portable box (Mariani 2017 + Mariani 2017a). The device consists of a Teflon holder for a 47mm SPE Disk, a membrane pump, a digital flowmeter and a battery (12V-9Ah). All parts are assembled in an aluminum box, as shown in Figure 14.

Quality assurance and control measures included analytical blanks, reproducibility test, field blanks and break-through samples. HLB SPE Disk (Hydrophilic/Lipophilic Balanced - Atlantic™ HLB-H, Horizon Technology) filtration/adsorption cartridges, previously cleaned and conditioned, were used for sample extraction.

Water samples collected in containers were spiked with a mix of isotope-labelled internal standards and filtered/extracted on site at an average flow of 0.14 l/min.

Figure 14. *Sampling device used for 20L spot samples*

HLB disk activation, drying and elution were performed using an automatic extractor (J2 Scientific, Figure 15).

SPE experimental conditions are summarized in Table 2.

Table 2. *SPE experimental conditions*

Atlantic™ HLB disk	Volume (ml)	Solvent
Conditioning and pre-cleaning	3 x 20	Ethyl acetate
Conditioning and pre-cleaning	3 x 20	Methanol
Conditioning	1 x 20	Water
Water Sample Filtration after Labelled Internal Standards spiking		
Drying	Under N_2 for 30 min at 20 ml/min	
Labelled Syringe Standard spiking		

Atlantic™ HLB disk	Volume (ml)	Solvent
Elution	3 x 20 ml	Ethyl acetate
Elution	3 x 20 ml	Methanol

A two fractions sequential elution was performed with 3 x 20 ml ethyl acetate (1st fraction) followed by 3 x 20 ml methanol (2nd fraction). All used solvents were Pesticide Analysis grade. The ethyl acetate fraction was divided into two portions, for the apolar and polar compounds analysis, respectively.

Figure 15. *Filter elution on automatic extractor*

The portion dedicated to apolar compound analysis was concentrated under gentle nitrogen flow to 100 µl and submitted to HRGC-HRMS analysis. The portion dedicated to polar compound analysis was added to the methanolic eluate, mixed and evaporated to dryness. The sample was reconstituted in 0.5 ml of reconstituting solution and analysed by UHPLC-MS/MS.

3.3. Large Volume Transect Sampling

A total of 5 transects was sampled across the Black Sea between Georgia and Ukraine with the Large Volume Transect Sampling (LV-TS) method.

The first transect was sampled for non-target analysis by the University of Athens, while the other four were sampled for processing in the JRC laboratory.

A 10 mm o.d./8 mm i.d. teflon tube has been mounted alongside a steel protection tube on the right side of the ship's hull in the aft working deck area, secured with steel cables, in order to collect sea water during navigation (Figure 16). The open sampling tube inlet was directed towards the navigation direction at ca. 1 m water depth. A Teflon membrane pump (KNF-FLODOS) pumped the water at a rate of ca. 0.7 L/min to the laboratory container on the ships main deck into an overflow container.

Figure 16. *Sampling metal probe mounted on the hull of the ship and the main pump*

From the overflow container a sub-sample (approx. 1/5) of the pumped water, using 0.8 o.d./0.6 i.d. mm Teflon tubes, was pumped with a second Teflon membrane pump (KNF-FLODOS) to a glass fiber filter cartridge (Filterite G1A4SE, nominal 1 µm) and then through a set of two extraction cells (ASE 100 ml extraction cells, Thermo Fisher Scientific Dionex, with adapters) connected in series. Sampling flow rate through the filtration/extraction system (see Figure 17) was kept at 220 ml/min, controlled with a digital flowmeter. The first cell was the primary extraction cartridge, while the second one was used for breakthrough evaluation. Both cells were filled with Amberlite XAD-2 as a stationary phase, mainly suitable for hydrophobic compounds.

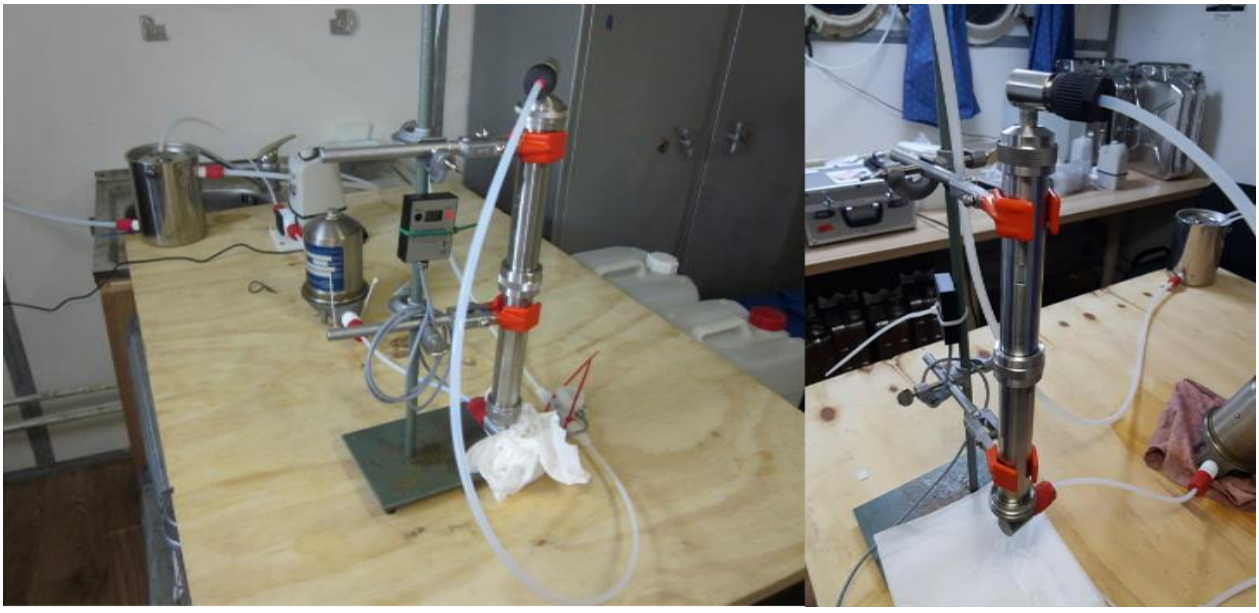


Figure 17: Sampling set-up used for LV-TS

All the cells as well as the glass fiber filter cartridges used for the sampling campaign were pre-cleaned, extracted and blanks analysed for several contaminants before use, in order to evaluate and confirm low background contamination values appropriate for the purpose.

The transect coordinates and the sampled volume of each transect are reported in table 3:

Table 3. *Large Volume Transect Samplings (LV-TS) dates, coordinates and volumes*

		Sampling Data:		
JBSS_XL_UoA-1		7/28/2019		
Start Point			**End Point**	
Longitude	29°48'32''		Longitude	30°46'14''
Latitude	44°35'41''		Latitude	46°29'55''
Total volume sampled:		303 L		
JBSS_XL_LVE-1		29-Jul-19		
Start Point			**End Point**	
Longitude	30°46'14''		Longitude	31°14'22''
Latitude	46°29'55''		Latitude	45°13'22''
Total volume sampled:		307 L		
JBSS_XL_LVE-2		30-Jul-19		
Start Point			**End Point**	
Longitude	31°14'22''		Longitude	34°46'29''
Latitude	45°13'22''		Latitude	43°22'24''
Total volume sampled:		266 L		
JBSS_XL_LVE-3		1-Aug-19		
Start Point			**End Point**	
Longitude	34°46'29''		Longitude	41°13'00''
Latitude	43°22'24''		Latitude	41°47'01''
		corrected		
Total volume sampled:		408 L		
JBSS_XL_LVE-4		8-Aug-19		
Start Point			**End Point**	
Longitude	30°46'14''		Longitude	30°12'23''
Latitude	46°29'55''		Latitude	45°12'36''
Total volume sampled:		180 L		

The map of transects is shown in Fig.18. Figure 19 shows the overlapping between LV Transect Sampling and the 12 spot samples on the open sea.

Figure 20 shows the LV filters arrived in the laboratory after the cruise.

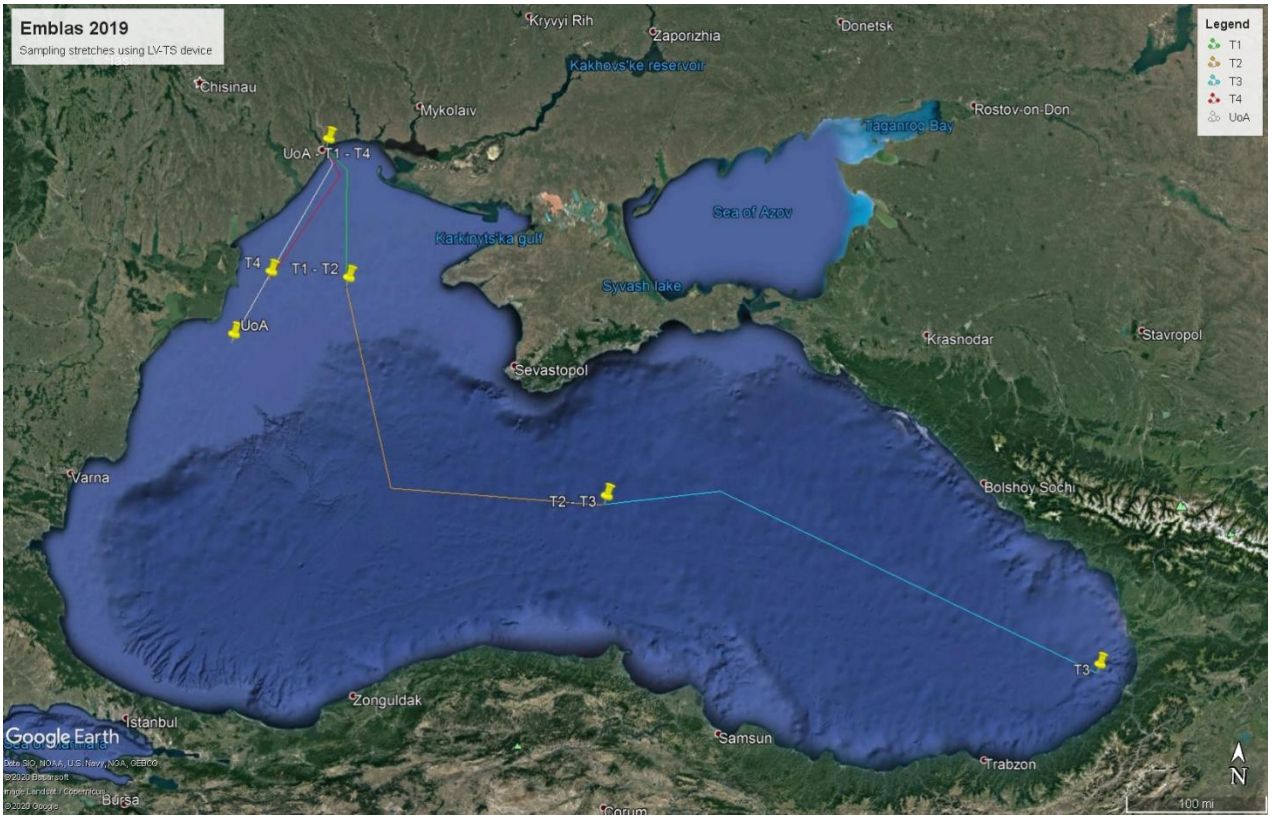


Figure 18. *LV transects sampling on the open sea*

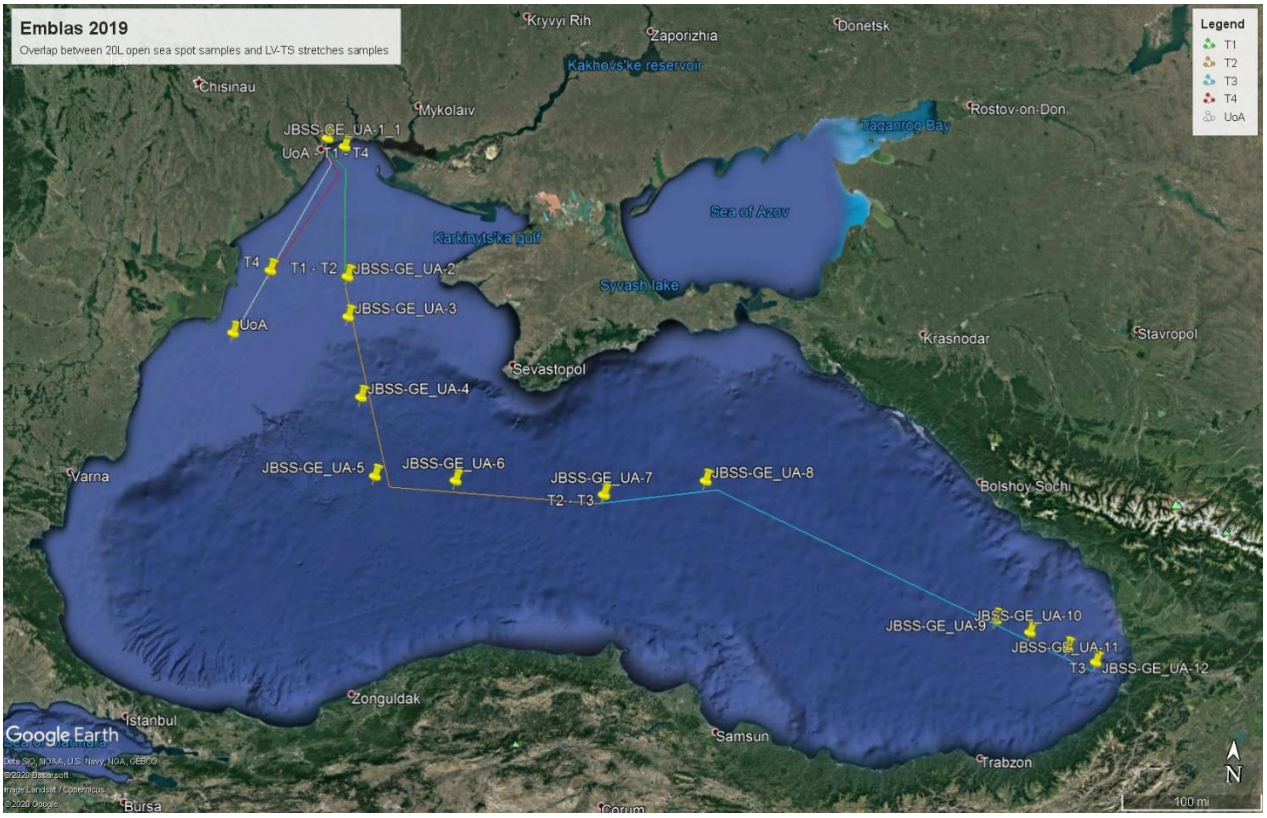


Figure 19. *Overlapping between LV transects and the 12 spot samples in open sea*

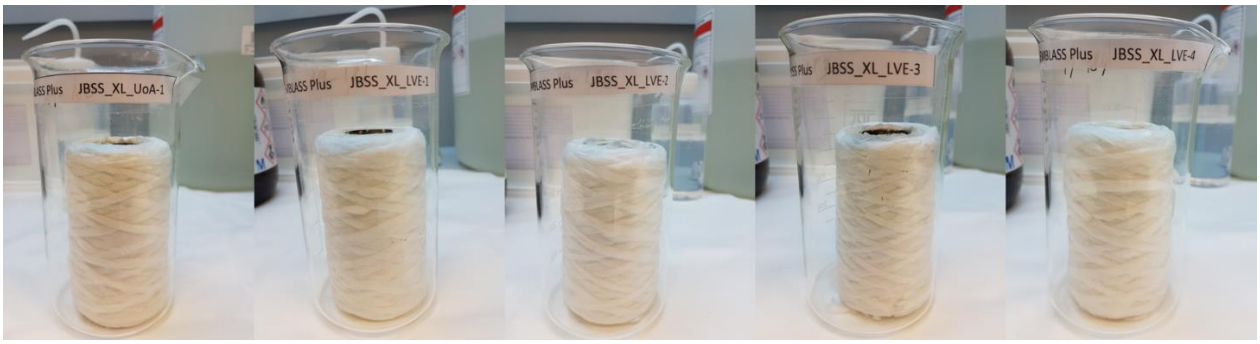


Figure 20. *LV filters arrived in the laboratory after the cruise*

3.4. Large Volume Transect Sampling extraction method

Each transect sample consists of a filter cartridge and two extraction cells. All the cells filled with XAD-2 phase were cleaned using an Accelerated Solvent Extraction system (ASE) and analysed before sampling (Figure 21). After suitable results of blank tests, the cells were conditioned in MilliQ water/methanol 80/20 and delivered for the sampling campaign. The background results are not reported. The ASE method parameters are reported in Table 4.

Figure 21. *ASE cells preparation by filling and conditioning with XAD-2 phase*

Table 4. *ASE method parameters for cells cleaning*

Extraction cells	Method/Activity	Cycles	Solvent
Cleaning	ASE	2	Methanol 100%
Cleaning	ASE	2	Acetone-Hexane 50% - 50%
Cleaning	ASE	2	Acetone 100%
	The acetone was spiked with both apolar internal and syringe mixture of standards	2	
Acetone evaporation	N_2 evaporation	2	Final volume 100 µl in Toluene
	Submitted to HRGC-HRMS analysis for background contamination determination		
Pre- Conditioning	ASE	2	Methanol 100%
Conditioning	ASE/Ready to use	2	M.Q. Water-Methanol 80% - 20%

Extraction cells as well as those used for the breakthrough and field blank evaluation were extracted using ASE (Figure 22). They were first extracted with methanol and then with

hexane. The two extracts were combined and 100ml MilliQ water were added. A back extraction of the methanolic phase was carried out by liquid-liquid extraction (LLE) using Hexane. The Hexane was evaporated under gentle nitrogen flow to 100 µl and submitted to HRGC-HRMS analysis.

Working conditions are summarized in Table 5.

Figure 22. *Cells extraction on ASE system and filter extraction on USE system*

Table 5. *Sample extraction protocol used for LV-TS cells*

Extraction cells	Method/Activity	Cycles	Solvent
Extraction	ASE	2	Methanol 100%
Extraction	ASE	2	Hexane 100%
Pooled extracts from ASE	Spiking with apolar internal standards		
Pooled extracts from ASE	LLE	3	Hexane 100%
Extracts evaporation after LLE	N_2 evaporation	1	Final volume 10 ml in Hexane
Extracts	Spiking with apolar syringe standards		
Extracts evaporation	N_2 evaporation		Volume 100 µl in toluene

Extraction cells	Method/Activity	Cycles	Solvent
	Submitted to HRGC-HRMS analysis		

Filter cartridges (Figure 20) including those used for the field blank evaluation, were extracted using an ultra-sonic extraction method (USE - Figure 22). As for the cells, the filters were cleaned, extracted and blanks analysed before sampling. The pre-cleaning and conditioning method for the filters is summarized in Table 6.

Table 6. *Filters pre-cleaning and conditioning parameters.*

Filters	Method/Activity	Cycles	Solvent
pre-cleaning	USE	1	Hexane 100%
pre-cleaning	USE	1	Acetone 100%
pre-cleaning	USE	1	Methanol 100%
pre-cleaning	USE	1	MilliQ Water 100%
pre-cleaning	USE	1	Methanol 100%
Extraction – 20 min	USE	2	Hexane 100%
Each extract of Hexane	Spiking with apolar internal mixture of standards		
Extracts evaporation	N_2 evaporation	1	Volume 100 µl in Toluene
	Submitted to HRGC-HRMS analysis for background contamination determination		
Conditioning	USE	1	Acetone 100%
Conditioning	USE	1	Methanol 100%
Conditioning	USE	1	MilliQ Water 100%

Filters	Method/Activity	Cycles	Solvent
Conditioning	USE/Ready to use	1	MilliQ Water 80% - Methanol 20%

After the sampling, the filters were first extracted with methanol and then with Hexane for 20 min. in ASE respectively. The two extracts were pooled and 100ml of MilliQ water were added. A back extraction of methanolic phase was carried out by liquid-liquid extraction (LLE) method using hexane. The Hexane was evaporated under gentle nitrogen flow to 100 µl and submitted to HRGC-HRMS analysis.

Filters were processed separately, all working conditions are summarized in Table 7.

Table 7. *Filters extraction condition*

Filters	Method/Activity	Cycles	Solvent
Extraction	USE	2	Methanol 100%
Extraction	USE	2	Hexane 100%
Extraction	USE	2	Hexane 100%
Pooled extracts from USE	Spiking with apolar internal standards		
Pooled extracts from USE	LLE	3	Hexane 100%
Extracts evaporation after LLE	N_2 evaporation	1	Final volume 10 ml in hexane
Extracts	Spiking with apolar syringe standards	1	
Extracts evaporation	N_2 evaporation	1	Volume 100 µl in Toluene

4. Analytical methods

4.1. QA/QC

Quantification of selected analytes was performed using an isotopic dilution method, employing isotopically labelled analogues for polar, semi-polar and apolar compounds.
The concept based on the use of identification points (IPs) proposed by the EU Commission Decision 2002/657/EC, both for GC-MS and LC-MS/MS analysis was used to identify and confirm the selected analytes in real samples.
The concept, originally defined for the determination of organic contaminants in food, has been widely used in a huge range of matrices, including environmental samples. It proposes a minimum number of IPs for the confirmation of a positive finding in real samples. Furthermore, the decision requests that the deviation of the relative intensity (ion ratio) of recorded ions/MRM transitions must be within a certain percentage value compared to the reference standard and the chromatographic retention time must not deviate more than 2.5%.

In the present report, the compounds were identified and confirmed based on:

- retention time comparison of the corresponding standard;
- ratios between two ions/MRM transitions (for all compounds analysed excepted for PAHs were just one ion was recorded).

Levels of analytical and field blanks were controlled during all processes (sampling and extraction process) for all studied compounds. The blank level, when positive, was not subtracted. Positive blanks are reported in the table of results.

LODs and LOQ have been calculated on each compound on the basis of a signal/noise ratio of 3:1 and 10:1 respectively.

4.2. UHPLC-MS/MS for polar compound analysis

4.2.1. UHPLC Chromatographic conditions

The UHPLC experimental conditions for polar compounds analysed in positive and negative ion MRM modes are reported in Tables 8 and 9 and Tables 10 and 11, respectively.

Table 8. *UHPLC experimental conditions for polar compounds chromatographic separation in positive ion MRM mode*

Pumps:	Binary Solvent Manager, Model UPB, Waters (Milford, MA, USA).
Autosampler:	Sample Manager, Model UPA, Waters (Milford, MA, USA).
Detector:	QTRAP 5500, Applied Biosystems MDS SCIEX, (Foster City, CA, U.S.A) equipped with Turbo V™ ion source.
Flow rate:	500 µL/min

Injection volume:	10 µL
Analytical column:	CSH C18, 2.1 x 50mm, 1.7 µm
Mobile phase:	A: CH_3COONH_4 5 mM B: Methanol
Reconstituting solution	CH_3COOH 5 mM:AcN:MeOH, 9:0.5:0.5, (% v/v)

The chromatography was performed in gradient mode according to the scheme reported in Table 9.

Table 9. *UHPLC gradient scheme for positive ion MRM mode*

Time (min)	Mobile phase (A%)	Mobile Phase B (%)
0	90	10
1.5	90	10
4	40	60
8	30	70
11	0	100
12	0	100
12.01	90	10
15	90	10

Table 10. *UHPLC experimental conditions for polar compounds chromatographic separation for negative ion MRM mode*

Pumps:	Binary Solvent Manager, Model UPB, Waters (Milford, MA, USA).
Autosampler:	Sample Manager, Model UPA, Waters (Milford, MA, USA).
Detector:	QTRAP 5500, Applied Biosystems MDS SCIEX, (Foster City, CA, U.S.A) equipped with Turbo V™ ion source.
Flow rate:	400 µL/min

Injection volume:	10 µL
Analytical column:	Hypersil GOLD, 2.1x100 mm, 1.9 µm, Thermo Scientific
Mobile phase:	A: 0.1% NH4OH B: Acetonitrile
Reconstituting solution	CH_3COOH 5 mM:AcN:MeOH, 9:0.5:0.5, (% v/v)

The chromatography was performed in gradient mode according to the scheme reported in Table 11.

Table 11. *UHPLC gradient scheme for negative ion MRM mode*

Time (min)	Mobile phase (A%)	Mobile Phase B (%)
0	90	10
0.5	90	10
1	60	40
5	10	90
6	10	90
6.5	90	10
12	90	10

4.2.2. QTRAP 5500 MS/MS operative conditions

An ABSciex QTRAP5500 mass spectrometer equipped with Turbo V™ ion source was used for polar compounds analysis. The instrument was previously tuned and calibrated in electrospray mode using PPG's. Prior to analysis all the specific parameters were optimized, infusing a 1 µg/mL standard solution of analytes and I.S.s.
The eluate from the column was introduced directly into the ion source. The rapid desolvatation and vaporization of the droplets minimizes thermal decomposition and preserves the molecular identity of the analytes.
The data were collected using the software program Analyst 1.6.2
All calculations were based on chromatographic peak area ratios for the MRM precursor-product ion transitions for analytes versus I.S.s.

The general operating conditions were as follows:

Scan Type:	Scheduled MRM
Polarity:	Positive / Negative
Ion Source:	Turbo Spray
Resolution Q1:	Unit
Resolution Q3:	Unit
MR Pause:	5.0000 msec
Curtain gas (CUR):	25.00
Collision Gas (CAD):	Medium
Temperature (TEM):	550.00
IonSpray Voltage (IS):	± 4500.00
Ion Source Gas 1 (GS1)	55
Ion Source Gas 2 (GS2)	45
Target Scan Time	0.3 sec
MRM detection window	60 sec

In Table 12 the QTrap ESI MRM parameters for both positive and negative ionization are reported for each compound.

Table 12. *QTRAP MS/MS parameters*

Q1	Q3	RT	ID	Internal Standard	DP	EP	CE	CXP
271	180	2.77	10,11-dihydro-10,11-dihydroxy-carbamazepine	Carbamazepine D10	80	10	47	13
271	210	2.77	10,11-dihydro-10,11-dihydroxy-carbamazepine 1	Carbamazepine D10	80	10	19	13
271	253	2.77	10,11-dihydro-10,11-dihydroxy-carbamazepine 2		80	10	10	13
342	121	0.85	ACEBUTOLOL-D5		150	10	26	13
342	324	0.85	ACEBUTOLOL-D5 1		150	10	23	13

Q1	Q3	RT	ID	Internal Standard	DP	EP	CE	CXP
223	126	2.83	Acetamiprid.1	Acetamiprid-d3	68	10	29	8
223	99	2.83	Acetamiprid.2	Acetamiprid-d3	68	10	53	6
226	126	2.83	Acetamiprid-d3		80	10	27	13
226	73	2.83	Acetamiprid-d3 1		80	10	80	13
226	190	2.83	Acetamiprid-d3 2		80	10	19	13
366.3	114	0.4	Amoxicillin	Amoxicillin 13C6	70	10	28	13
366.3	86	0.4	Amoxicillin 1	Amoxicillin 13C6	70	10	65	13
366.3	349	0.4	Amoxicillin 2		70	10	24	13
372	114	0.4	Amoxicillin 13C6		70	10	28	13
372	214	0.4	Amoxicillin 13C6 1		70	10	25	13
306	237	3.58	ANASTROZOLE-D12		150	10	30	13
306	118	3.57	ANASTROZOLE-D12 1		150	10	70	13
216	174	3.68	Atrazine	Atrazine-(triazyl-13C3,15N3)	258	10	25	13
216	104	3.68	Atrazine 1	Atrazine-(triazyl-13C3,15N3)	258	10	40	13
221	179	3.68	Atrazine-(triazyl-13C3,15N3)		120	10	24	13
221	137	3.88	Atrazine-(triazyl-13C3,15N3) 1		120	10	35	13
188	146	2.52	Atrazine-desethyl	Atrazine-(triazyl-13C3,15N3)	60	10	12	13
188	104	2.52	Atrazine-desethyl 1	Atrazine-(triazyl-13C3,15N3)	60	10	10	13
174	104	1.29	Atrazine-desisopropyl	Atrazine-(triazyl-13C3,15N3)	223	10	33	13
174	68	1.29	Atrazine-desisopropyl 1	Atrazine-(triazyl-13C3,15N3)	223	10	25	13
750	573	3.13	Azithromycin	Carbamazepine d10	200	10	47	13
750	158	3.13	Azithromycin 1	Carbamazepine d10	200	10	40	13
120	65	1.4	Benzotriazole	Benzotriazole d4	209	10	29	13
120	92	1.4	Benzotriazole 1	Benzotriazole d4	209	10	24	13
124	69	1.4	Benzotriazole d4		56	10	35	13
261	205	3.27	Bromacil	Atrazine-(triazyl-13C3,15N3)	50	10	20	13
261	188	3.27	Bromacil 1	Atrazine-(triazyl-13C3,15N3)	50	10	40	13
261	162	3.27	Bromacil 2		50	10	40	13
237	194	3.46	Carbamazepine	Carbamazepine d10	250	10	28	13
237	165	3.46	Carbamazepine 1	Carbamazepine d10	250	10	60	13
247	204	3.46	Carbamazepine d10		234	10	31	13
410	222	2.99	Carvedilol-D3		120	10	37	13
410	139	2.99	Carvedilol-D3 1		120	10	115	13
291	72	4.26	Chloroxuron	Linuron D6	81	10	47	4
291	218	4.26	Chloroxuron 1	Linuron D6	81	10	33	14
332	231	0.67	Ciprofloxacin	Ciprofloxacin 13C3, 15N	150	10	53	13

Q1	Q3	RT	ID	Internal Standard	DP	EP	CE	CXP
332	314	0.67	Ciprofloxacin 1	Ciprofloxacin 13C3, 15N	150	10	35	13
336	235	0.67	Ciprofloxacin 13C3, 15N		180	10	50	13
336	318	0.67	Ciprofloxacin 13C3, 15N 1		180	10	30	13
336	291	0.67	Ciprofloxacin 13C3, 15N 2		180	10	25	13
748.5	590.5	3.13	Clarythromycin	Ciprofloxacin 13C3, 15N	100	10	28	13
748.5	558.5	3.13	Clarythromycin 1	Ciprofloxacin 13C3, 15N	100	10	31	13
202	146	3.38	Terbutylazine-desethyl	Terbutylazine D5	78	10	23	13
202	79	3.38	Terbutylazine-desethyl 1	Terbutylazine D5	78	10	37	13
305	159	4.85	Diazinon	Diazinon D10	100	10	30	13
305	97	4.85	Diazinon 1	Diazinon D10	100	10	55	13
315.3	170	4.85	Diazinon D10		70	10	35	13
315.3	154.2	4.85	Diazinon D10 1		100	10	5	13
276	244	4.2	Dimethenamid	Carbamazepine d10	60	10	20	13
276	168	4.2	Dimethenamid 1	Carbamazepine d10	60	10	30	13
276	111	4.2	Dimethenamid 2		60	10	40	13
437.1	368	4.78	Fipronil.1		96	10	23	9
437.1	290	4.78	Fipronil.2		96	10	37	7
256	209.1	2.58	Imidacloprid.1	Imidacloprid-d4	61	10	21	14
256	175.1	2.58	Imidacloprid.2	Imidacloprid-d4	61	10	25	10
260	213	2.58	Imidacloprid-d4		60	10	26	13
260	179	2.58	Imidacloprid-d4 1		60	10	29	13
249.1	182.1	4.2	Linuron.1	Linuron D6	Linuron D6	10	21	12
249.1	160	4.2	Linuron.2	Linuron D6	66	10	25	34
255	133	4.2	Linuron D6		120	10	50	13
255	160	4.2	Linuron D6 1		120	10	25	13
255	185	4.2	Linurion d6 2		120	10	30	13
507.1	178.1	5.83	Metaflumizone.1	Anastrozole-D12	101	10	33	10
507.1	287.1	5.83	Metaflumizone.2	Anastrozole-D12	101	10	33	6
278	134	3.9	Metazachlor 1		60	10	10	13
278	105	3.9	Metazachlor		60	10	50	13
284	252	4.5	Metolachlor		200	10	22	13
284	148	4.5	Metolachlor 1		200	10	35	13
202	104	3.24	Simazine	Simazine-d10	253	10	34	13
202	132	3.24	Simazine 1	Simazine-d10	253	10	26	13
212	184	3.24	Simazine-d10		120	10	30	35
212	134	3.24	Simazine-d10 1		120	10	35	30

Q1	Q3	RT	ID	Internal Standard	DP	EP	CE	CXP
254	156	2.79	Sulfamethoxazole	Sulfamethoxazole D4	150	10	22	13
254	92	2.79	Sulfamethoxazole 1	Sulfamethoxazole D4	150	10	38	13
258	96	2.79	Sulfamethoxazole D4		80	10	30	13
308.2	70	4.42	Tebuconazole.1	Tebuconazole-(tert-butyl-d4)	86	10	51	10
308.2	125	4.42	Tebuconazole.2	Tebuconazole-(tert-butyl-d4)	86	10	55	8
312	70	4.42	Tebuconazole-(tert-butyl-d4)		100	10	70	13
312	129	4.42	Tebuconazole-(tert-butyl-d4) 1		100	10	50	13
242.1	186.1	3.43	Terbutryn.1	Terbutylazine D5	36	10	25	12
242.1	68.1	3.43	Terbutryn.2	Terbutylazine D5	36	10	61	4
230	174	4.12	Terbutylazine	Terbutylazine D5	219	10	26	13
230	132	4.12	Terbutylazine 1	Terbutylazine D5	219	10	35	13
235	179	4.12	Terbutylazine D5		130	10	30	13
235	137	4.12	Terbutylazine D5 1		130	10	35	13
188	105	2.52	Atrazine-desethyl 2		60	10	12	13
188	169	2.52	Atrazine-desethyl 3		60	10	10	13
276	145	2.71	17β-Estradiol-D5		-150	-10	-70	-11
276	147	2.71	17β-Estradiol-D5 1		-150	-10	-52	-11
276	187	2.85	17β-Estradiol-D5 1		-150	-10	-53	-11
360	274	1.72	Bezafibrate	Bezafibrate D4	-100	-10	-24	-11
360	154	1.72	Bezafibrate 1	Bezafibrate D4	-100	-10	-39	-11
364	278	1.72	Bezafibrate D4		-165	-10	-24	-11
264	178	0	Chlorotalonil		-270	-10	-40	-11
264	231	0	Chlorotalonil 1		-270	-10	-10	-11
221	177	0.51	Dicamba	Dicamba-(phenyl-13C6)	-50	-10	-5	-11
219	175	0.51	Dicamba 1	Dicamba-(phenyl-13C6)	-50	-10	-5	-11
227	183	0.51	Dicamba-(phenyl-13C6)		-50	-10	-5	-11
225	181	0.51	Dicamba-(phenyl-13C6) 1		-50	-10	-5	-11
294	250	1.78	Diclofenac	Diclofenac 13C6	-42	-10	-16	-11
294	214	1.78	Diclofenac 1	Diclofenac 13C6	-42	-10	-29	-11
300	256	1.78	Diclofenac 13C6		-173	-10	-15	-11
269	145	2.92	Estrone	Estrone D4	-100	-10	-53	-11
269	143	2.92	Estrone 1	Estrone D4	-100	-10	-74	-11
273	147	2.92	Estrone D4		-100	-10	-88	-11
273	187	2.92	Estrone D4 1		-100	-10	-50	-11
271	145	2.71	17β-Estradiol	17β-Estradiol-D5	-83	-10	-60	-11
271	143	2.71	17β-Estradiol 1	17β-Estradiol-D5	-83	-10	-78	-11
295	145	2.85	17α-Ethinyl-Estradiol	17α-Ethinyl-Estradiol D4	-100	-10	-70	-11

Q1	Q3	RT	ID	Internal Standard	DP	EP	CE	CXP
295	143	2.85	17α-Ethinyl-Estradiol	17α-Ethinyl-Estradiol D4	-100	-10	-50	-11
299	145	2.85	17α-Ethinyl-Estradiol D4		-100	-10	-60	-11
299	187	2.85	17α-Ethinyl-Estradiol d4 1		-100	-10	-45	-11
205	161	1.74	Ibuprofen	Ibuprofen-methyl-13C,d3	-132	-10	-10	-11
205	159	1.74	Ibuprofen 1	Ibuprofen-methyl-13C,d3	-132	-10	-10	-11
208	164	1.74	Ibuprofen-methyl-13C,d3		-80	-10	-10	-11
208	161	1.74	Ibuprofen-methyl-13C,d3 1		-80	-10	-11	-11
229	169	1.05	Naproxen	Naproxen 13C3	-100	-10	-47	-11
229	185	1.05	Naproxen 1	Naproxen 13C3	-100	-10	-10	-11
233	169	1.11	Naproxen 13C3		-42	-10	-46	-11

DP: Declustering Potential; EP: Entrance Potential; CE: Collision Energy; CXP: Collision Cell Entrance Potential.

4.3. HRGC-HRMS for semi-polar and apolar compound analysis

The extracts were analysed by HRGC-HRMS using an isotopic dilution method for all semi-polar and apolar compounds.

EC-7 PCBs, Pesticides, Triazine, HCBD, PAHs, EHMC, BHT and OPCs were analysed on double HRGC (Thermo Trace GC Ultra, Thermo Electron, Bremen, Germany), coupled with a DFS high resolution mass spectrometer HRMS (Thermo Electron, Bremen, Germany) operating in the EI-mode at 45 eV with a resolution of 8000-10000.

For EC7-PCBs the two most abundant ions of the isotopic molecular cluster were recorded for both native and labelled congeners.

For chlorinated pesticides (OCPs) and Triazine, two ions of the isotopic cluster from the fragmentation were chosen on the basis of close elution of different OCPs and the dynamic mass range of the HRMS. For non-chlorinated pesticides, the two most abundant ions were selected from the fragmentation products and chosen on the basis of close elution with other pesticides.

For PAHs the single molecular ion was recorded both for native and labelled compounds. For BHT the molecular ion and -15 m/z ion were recorded. For EHMC the two most abundant ions were recorded.

For OPCs two most abundant ions after fragmentation were chosen on the basis of close elution of different OPCs and the dynamic mass range of the HRMS.

4.3.1. Organophosphate Compounds OPCs

OPCs (Phosphate flame retardants and plasticizers) *were separated on a HP-5ms UI 60 m long column with 0.25 mm i.d. (inner diameter) and 0.25 µm film* (Agilent J&W, USA).

Gas chromatographic conditions for OPCs were:

PTV injector with temperature program from 100 to 300 °C at 14.5 °C/s, splitless time 1 min., split flow 50 ml/min., constant flow at 1.5 ml min^{-1} of He, GC-MS interface at 300 °C and a GC program rate: 80 °C for 1 min., 10 °C min^{-1} to 250 °C for 5 min., then 5 °C min^{-1} to 300 °C for a final isotherm of 1 min.

In Table 13 the exact recorded masses and retention times in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

Table 13. *HRGC-HRMS experimental conditions for OPCs analysis*

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
1	135.0657	167.1221	6.98	TEP-d15	TEP-d15	TPhP-d15
	127.0155	155.0468	7.11	TEP		
2	131.0375	151.0939	10.38	TNPP-d21	TNPP-d21	
	122.9842	141.0311	10.56	TNPP		
3	131.0375	167.1221	13.63	TNBP-d27		
	139.0155	155.0468	12.25	TNBP	TNBP-d27	
	124.9998	155.0468	13.84	TIBP		
4	261.0598	263.0568	15.09	TCEP-d12		
	248.9845	250.9786		TCEP	TCEP-d12	
	277.0158	279.0128	15.65	TCPP-1		
			15.78	TCPP-2		
			15.89	TCPP-3		
	244.1969		19.87	p-terphenyl-d14		
5	393.9775	395.9746	21.38	TDCPP-d15		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	380.8939	382.9746	21.62	TDCPP	TDCPP-d15	
6	299.1618	300.1652	22.42	TBOEP-$^{13}C_6$		TPhP-d15
	303.1752	304.1785	22.43	TBOEP	TBOEP-$^{13}C_6$	
	343.1228	344.1306	22.69	TPhP-$^{13}C_{18}$		
	339.1503	341.1644	22.56	TPhP-d15		
	325.0624	326.0702	22.70	TPhP	TPhP-$^{13}C_{18}$	
7	250.0389	251.0468	23.03	EHDP		
8	98.9842	113.1325	23.44	TEHP		
9	403.1893	419.2206	30.27	T35DMPP-d9		
	367.1094	368.1172	26.85	TMPP-1	T35DMPP-d9	
			27.35	TMPP-2		
			27.86	TMPP-3		
			28.37	TMPP-4		
	452.2111	453.2145	29.02	TIPPP		
	395.1407	410.1641	30.33	T35DMPP		

4.3.2. Chlorinated Pesticides

Pesticides and Chlorinated Flame Retardants were separated on a 60 m long HP-5ms UI column with 0.25 mm i.d. (inner diameter) and 0.25 µm film (Agilent J&W, USA).

Gas chromatographic conditions for chlorinated pesticides were:

PTV injector with temperature program from 100 to 245 °C at 14.5 °C/s, splitless time 1 min., split flow 50 ml/min., constant flow at 1.0 ml min^{-1} of He, GC-MS interface at 250 °C and a GC program rate: 100 °C for 1 min., 10 °C min^{-1} to 270 °C for 5 min., then 30 °C min^{-1} to 300 °C for a final isotherm of 9 min.

In Table 14 exact recorded masses and retention times in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

Table 14. *HRGC-HRMS experimental conditions for chlorinated pesticides analysis*

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
1	191.0142	193.0112	8.39	Dichlorvos-d6		β-HCH-$^{13}C_6$
	184.9765	186.9735	8.44	Dichlorvos	Dichlorvos-d6	
	230.8512	232.8483	8.26	HCBD $^{13}C_4$		
	222.8408	224.8408	8.26	HCBD	HCBD $^{13}C_4$	
2	255.8693	257.8663	12.15	PeCBz$^{13}C_6$		
	249.8491	251.8462	12.15	PeCBz	PeCBz$^{13}C_6$	
	264.0227	306.0696	13.54	Trifluralin	γ-HCH-$^{13}C_6$	
3	222.9341	224.9312	14.21	α-HCH-$^{13}C_6$		
	216.9140	218.9110	14.21	α-HCH	α-HCH-$^{13}C_6$	
	222.9341	224.9312	14.75	β-HCH-$^{13}C_6$		
	222.9341	224.9312	14.92	γ-HCH-$^{13}C_6$	γ-HCH-$^{13}C_6$	
	216.9140	218.9110	14.75	β-HCH		
	216.9140	218.9110	14.92	γ-HCH		
	216.9140	218.9110	15.39	δ-HCH		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	268.0324	270.0295	15.41	Triallate		
	263.8810	265.8781	15.54	Chlorothalonil		
	216.9140	218.9110	15.63	ε-HCH		
	289.8297	291.8268	14.42	HCB-$^{13}C_6$		
	283.8096	285.8067	14.42	HCB	HCB-$^{13}C_6$	
4	276.8264	278.8234	16.43	Heptachlor-$^{13}C_{10}$		
	271.8096	273.8067	16.44	Heptachlor	Heptachlor-$^{13}C_{10}$	
	324.0196	326.0167	16.98	Chlorpyriphos-d10		
	313.9539	315.9539	17.07	Chlorpyriphos	Chlorpyriphos-d10	
	269.8799	271.8769	17.18	Aldrin-$^{13}C_{12}$		
	262.8564	264.8535	17.19	Aldrin	Aldrin-$^{13}C_{12}$	
	333.0629	335.0599	17.77	Chlorfenvinphos-d10		
5	323.0001	324.9972	17.85	Chlorfenvinphos	Chlorfenvinphos-d10	
	375.9125	377.9095	17.79	Isodrin-$^{13}C_{12}$		
	363.8722	365.8693	17.80	Isodrin	Isodrin-$^{13}C_{12}$	
	362.8772	364.8743	17.95	Cis-Heptachlor-epoxide-$^{13}C_{10}$		
	352.8437	354.8407	17.95	Cis-Heptachlor-epoxide	Cis-Heptachlor-epoxide-$^{13}C_{10}$	p,p'-DDD-d8
	352.8437	354.8407	18.04	Trans-Heptachlor-epoxide		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	396.8382	398.8353	17.97	Oxychlordane-$^{13}C_{10}$	Cis-Heptachlor-epoxide-$^{13}C_{10}$	
	386.8047	388.8017	17.97	Oxychlordane	Oxychlordane-$^{13}C_{10}$	
	327.9777	329.9748	18.40	o,p-DDE-$^{13}C_{12}$		
6	315.9375	3179345	18.40	o,p-DDE	o,p-DDE-$^{13}C_{12}$	
	347.9027	349.8997	18.73	α-Endosulfan-$^{13}C_{9}$		
	338.8725	340.8695	18.73	α-Endosulfan	α-Endosulfan-$^{13}C_{9}$	
	382.8590	384.8560	18.42	Trans-Chlordane-$^{13}C_{10}$		
	372.8254	374.8225	18.42	Trans-Chlordane	Trans-Chlordane-$^{13}C_{10}$	
	372.8254	374.8225	18.74	Cis-Chlordane	Trans-Chlordane-$^{13}C_{10}$	
	416.8200	418.8170	18.83	Trans-Nonachlor-$^{13}C_{10}$		
	406.7864	408.7835	18.83	Trans-Nonachlor	Trans-Nonachlor-$^{13}C_{10}$	
	327.9777	329.9748	19.00	p,p'-DDE-$^{13}C_{12}$		
7	315.9375	3179345	19.01	p,p'-DDE	p,p'-DDE-$^{13}C_{12}$	
	247.0481	249.0449	19.23	o,p-DDD-$^{13}C_{12}$		
	235.0076	237.0046	19.24	o,p-DDD	o,p-DDD-$^{13}C_{12}$	
	269.8799	271.8769	19.28	Dieldrin-$^{13}C_{12}$		
	262.8564	264.8535	19.28	Dieldrin	Dieldrin -$^{13}C_{12}$	

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	269.8799	271.8769	19.79	Endrin-$^{13}C_{12}$		
	262.8564	264.8535	19.81	Endrin	Endrin -$^{13}C_{12}$	
	243.0578	245.0550	19.91	p,p'-DDD-d8		
	235.0076	237.0046	19.96	p,p'-DDD	o,p-DDD-$^{13}C_{12}$	
	239.8605	341.8575	19.99	β-Endosulfan-$^{13}C_9$		
	234.8437	236.8408	20.00	β-Endosulfan	β-Endosulfan-$^{13}C_9$	
	247.0481	249.0449	20.09	o,p-DDT-$^{13}C_{12}$		
	235.0076	237.0046	20.10	o,p-DDT	o,p-DDT-$^{13}C_{12}$	
	239.8605	341.8575	20.24	Cis-Nonachlor-$^{13}C_{10}$		
	234.8437	236.8408	20.25	Cis-Nonachlor	Cis-Nonachlor-$^{13}C_{10}$	
8	247.0481	249.0449	20.09	p,p'-DDT-$^{13}C_{12}$		
	235.0076	237.0046	20.10	p,p'-DDT	p,p'-DDT-$^{13}C_{12}$	
	276.8264	278.8234	21.02	Endosulfan-sulfate-$^{13}C_9$		
9	271.8096	273.8067	21.03	Endosulfan-sulfate	Endosulfan-sulfate-$^{13}C_9$	
	239.1475	240.1508	22.44	Methoxychlor-$^{13}C_{12}$		
	227.1067	228.1106	22.45	Methoxychlor	Methoxychlor-$^{13}C_{12}$	
10	258.0527	261.0498	22.44	Dicofol-d8	Methoxychlor-$^{13}C_{12}$	
	251.0025	252.9995	22.68	Dicofol		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	276.8264	278.8234	24.14	Mirex-$^{13}C_{10}$		
11	271.8096	273.8067	24.15	Mirex	Mirex-$^{13}C_{10}$	
	169.0452	171.0423	25.93	Trans-Cypermehtrin-d6 (2 isomers)		
12	169.0452 163.0081	171.0423 165.0052	26.08	Trans-Cypermehtrin-d6 (2 isomers)		
			25.88	Mix solution of Trans and Cis-Cypermehtrin (4 isomers)	Trans-Cypermehtrin-d6	
	163.0081	165.0052	25.98	Mix solution of Trans and Cis-Cypermehtrin (4 isomers)	Trans-Cypermehtrin-d6	
			26.09			

4.3.3. Triazines pesticides

Triazines were separated on a 60 m long HP-5ms UI column with 0.25 mm i.d. (inner diameter) and 0.25 µm film (Agilent J&W, USA).

Gas chromatographic conditions for triazines were:

Splitless injector with temperature 280°C, splitless time 1 min., split flow 50 ml/min., constant flow at 1.0 ml min^{-1} of He, GC-MS interface at 280 °C and a GC program rate: 100 °C for 1 min., 7 °C min^{-1} to 160 °C for 6 min., then 30 °C min^{-1} to 320 °C for a final isotherm of 4 min.

In Table 15 exact recorded masses and retention times in HRGC-HRMS for native compounds and internal labelled standards are reported.

HRGC-HRMS was used for triazines pesticides analysis both for the LV transect samples and 20L spot samples. Spot samples were also analysed using UPLC-MS/MS.

Table 15. *HRGC-HRMS experimental conditions for triazine pesticides analysis*

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
1	173.0463	175.0433	18.69	Desisopropyl-Atrazine	Terbuthylazine-d5	---
	172.0390	174.0360	18.83	Desethyl-Atrazine		
	186.0541	188.0511	19.04	Desethyl-Terbuthylazine		
	211.1403	213.1374	19.52	Simazine-d10		
	201.0776	203.0746	19.60	Simazine	Simazine-d10	
	220.1246	222.1217	19.64	Atrazine-d10		
	215.0932	217.0903	19.68	Atrazine	Atrazine-d10	
	219.1168	221.1138	19.88	Terbuthylazine-d5		
	214.0854	216.0824	19.90	Terbuthylazine	Terbuthylazine-d5	

4.3.4. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

PAHs, EHMC and BHT were separated on a 60 m long HP-5ms UI column with 0.25 mm i.d. (inner diameter) and 0.25 µm film (Agilent J&W, USA).

Gas chromatographic conditions were:

PTV injector with temperature program from 100 to 300 °C at 14.5 °C/s, splitless time 1 min., split flow 100 ml/min., constant flow at 1.0 ml min^{-1} of He, GC-MS interface at 320 °C and a GC program rate: 100 °C for 1 min., 10 °C min^{-1} to 320 °C for a final isotherm of 17 min.

In Table 16 exact recorded mass and retention time in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

Table 16. *HRGC-HRMS experimental conditions for PAHs analysis*

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
1	136.1123		7.84	Naphthalene-d8		Biphenyl-d10
	128.0621		7.87	Naphthalene	Naphthalene-d8	
2	160.1123		10.94	Acenaphthylene-d8		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	152.0621		10.97	Acenaphthylene	Acenaphthylene-d8	
	164.1405		9.91	Biphenyl-d10		
	164.1405		11.31	Acenaphthene-d10		
	154.0777		11.39	Acenaphthene	Acenaphthene-d10	
	222.2638	240.3062	11.28	BHT-d21		
	205.1587	220.1822	11.43	BHT	BHT-d21	
3	176.1405		12.50	Fluorene-d10		
	166.0777		12.58	Fluorene	Fluorene-d10	
	188.1405		14.81	Phenanthrene-d10		p-terphenyl-d14
	178.0777		14.87	Phenanthrene	Phenanthrene-d10	
	188.1405		14.93	Anthracene-d10		
	178.0777		14.98	Anthracene	Anthracene-d10	
4	212.1405		17.72	Fluoranthene-d10		
	202.0777		17.77	Fluoranthene	Fluoranthene-d10	
	212.1405		18.27	Pyrene-d10		
	202.0777		18.31	Pyrene	Pyrene-d10	
	161.0597	178.0624	19.58	EHMC		
	244.1969		18.69	p-terphenyl-d14		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
5	240.1687		21.16	Benzo(a)anthracene-d12		Benzo(a)anthracene-d12
	228.0934		21.21	Benzo(a)anthracene	Chrysene-d12	
	240.1687		21.25	Chrysene-d12		
	228.0934		21.31	Chrysene	Chrysene-d12	
6	264.1687		23.68	Benzo(b)fluoranthene-d12		Benzo(k)fluoranthene-d12
	252.0934		23.71	Benzo(b)fluoranthene	Benzo(b)fluoranthene-d12	
	264.1687		23.72	Benzo(k)fluoranthene-d12		
	252.0934		23.76	Benzo(k+j)fluoranthene	Benzo(k)fluoranthene-d12	
	264.1687		24.30	Benzo(e)pyrene-d12		
	252.0934		24.37	Benzo(e)pyrene	Benzo(e)pyrene-d12	
	264.1687		24.43	Benzo(a)pyrene-d12		
	252.0934		24.49	Benzo(a)pyrene	Benzo(a)pyrene-d12	
	264.1687		24.62	Perylene-d12		
	252.0934		24.69	Perylene	Perylene-d12	
	288.1687		27.44	Indeno(123-cd)pyrene-d12		
	276.0934		27.52	Indeno(123-cd)pyrene	Indeno(123-cd)pyrene-d12	

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	288.1687		28.24	Benzo(ghi)perylene-d12		
	276.0934		28.33	Benzo(ghi)perylene	Benzo(ghi)perylene-d12	
	292.1969		27.43	Dibenzo(ah)anthracene-d12		
	278.1090		27.53	Dibenzo(ah)anthracene	Dibenzo(ah)anthracene-d12	

4.3.5. Indicator Polychlorinated Biphenyls (EC-7 PCBs)

EC7-PCBs were separated on a HT-8 capillary column, 60 m long with 0.25 mm i.d.(inner diameter) and 0.25 µm film (SGE, Victoria, Australia).

Gas chromatographic conditions were: Split/splitless injector at 280 °C, constant flow at 1.5 ml min^{-1} of He, GC-MS interface at 280 °C and a GC program rate: Starting from 120 °C with 20 °C min^{-1} to 180 °C, 2 °C min^{-1} to 260 °C, and 5 °C min^{-1} to 300 °C isotherm for 4 min.

In Table 17 exact recorded mass and retention time in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

Table 17. *HRGC-HRMS experimental conditions for PCBs analysis*

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
1	268.0016	269.9986	23.22	PCB-31 $^{13}C_{12}$		PCB-31 $^{13}C_{12}$
	268.0016	269.9986	23.58	PCB-28 $^{13}C_{12}$		
	255.9613	257.9584	23.61	PCB-28	PCB-28 $^{13}C_{12}$	
	301.9626	303.9597	25.76	PCB-52 $^{13}C_{12}$		
	289.9224	291.9194	25.78	PCB-52	PCB-52 $^{13}C_{12}$	
2	337.9207	339.9178	33.61	PCB-101 $^{13}C_{12}$		PCB-111 $^{13}C_{12}$

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	325.8804	327.8775	33.63	PCB-101 $^{13}C_{12}$	PCB-101 $^{13}C_{12}$	
	337.9207	339.9178	36.40	PCB-111 $^{13}C_{12}$		
	337.9207	339.9178	40.34	PCB-118 $^{13}C_{12}$		
	325.8804	327.8775	40.37	PCB-118	PCB-118 $^{13}C_{12}$	
	371.8817	373.8788	41.76	PCB-153 $^{13}C_{12}$		
	359.8415	361.8385	41.79	PCB-153	PCB-153 $^{13}C_{12}$	
	371.8817	373.8788	44.46	PCB-138 $^{13}C_{12}$		
	359.8415	361.8385	44.49	PCB-138	PCB-138 $^{13}C_{12}$	
3	405.8428	407.8398		PCB-180 $^{13}C_{12}$		PCB-170 $^{13}C_{12}$
	393.8025	395.7995		PCB-180	PCB-180 $^{13}C_{12}$	
	405.8428	407.8398		PCB-170 $^{13}C_{12}$		

5. QA/QC Results

5.1. QA/QC Mariani Box

Recovery, sampling efficiency, limits of detection/quantitation and reproducibility data have been obtained for 20 L spot samples (Filtration/Extraction Manifold, Mariani Box).

Recovery and sampling efficiency evaluations were calculated based on the labelled internal standards added to the water samples before filtration/extraction using the Mariani Box. These parameters were calculated only for the chemicals detected by HRGC-HRMS because this analytical technique allows the use of labelled syringe standards and suffers of a minor signal suppression/ enhancement compared to LC-MS/MS.

A plausible reason for the observed increased interferences present in the samples can be the different season of the current campaign, resulting in a higher concentration of organic matrix substances, compared to the previous campaigns.

5.1.1. Polar compounds

Table 18 report methods' LOD (calculated as blank value plus 3 standard deviation) and LOQ (calculated as blank value plus 10 standard deviation).

The samples JBSS_UA_GE-1_1 and JBSS_UA_GE-1_2 were sampled for the reproducibility test. Table 19 reports the concentrations of detected compounds and their relative calculated coefficients of variation; the reproducibility data are illustrated in graph 1.

Sampling, extraction and storage condition of the samples were not suitable for the stability of Amoxicillin, therefore the results are not reported (Not Available - N.A.).

Table 18. *LOD/LOQ of Polar Compounds*

	LOD	LOQ
	Blank +3sd	Blank +10sd
	ng/L	**ng/L**
Bezafibrate	0.004	0.009
Dicamba	6	15
Diclofenac	0.01	0.03
E1	0.007	0.01
E2	0.02	0.03
EE2	0.04	0.11
Ibuprofen	0.10	0.28
Metaflumizone	0.01	0.02

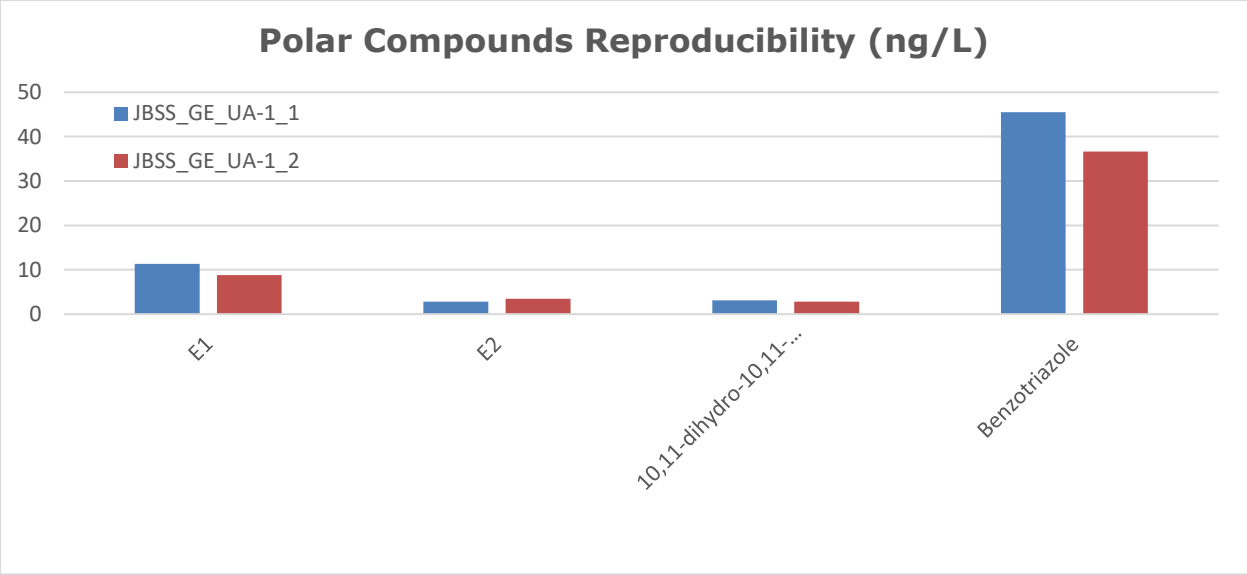
	LOD	LOQ
	Blank +3sd	Blank +10sd
	ng/L	**ng/L**
Naproxen	0.16	0.45
10,11-dihydro-10,11-dihydroxy-carbamazepine	0.17	0.48
Acetamiprid	0.003	0.01
Amoxicillin	N.A.	N.A.
Azithromycin	0.21	0.59
Benzotriazole	0.04	0.07
Bromacil	0.15	0.43
Carbamazepine	0.10	0.28
Chloroxuron	0.13	0.37
Ciprofloxacin	6	11
Clarithromycin	0.34	0.98
Diazinon	0.03	0.08
Dimethenamid	0.0007	0.001
Fipronil	0.01	0.02
Imidacloprid	0.06	0.16
Linuron	0.13	0.38
Metazachlor	0.006	0.011
Metolachlor	0.003	0.01
Sulfamethoxazole	0.004	0.01
Tebuconazole	0.001	0.003
Terbutryn	0.09	0.26

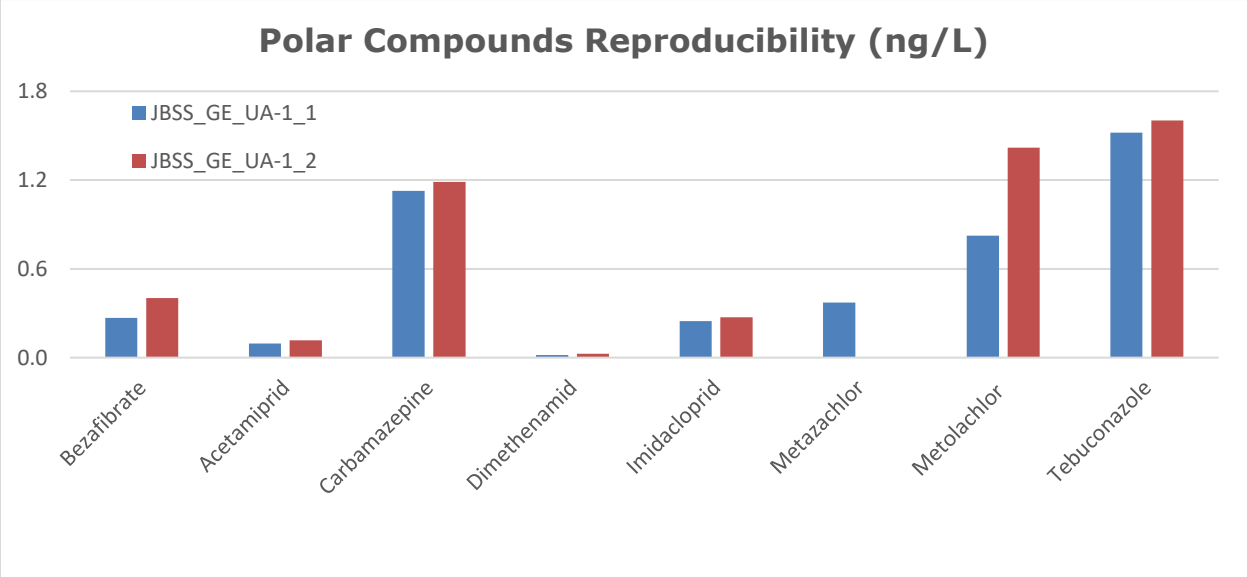
Table 19. *Reproducibility data of Polar Compounds*

Lab. Code:	OPC-EMB-19-158	OPC-EMB-19-160		
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2		
Volume sampled (L):	18.30	18.05		
Concentration	**ng/L**	**ng/L**	**Average**	**Cv %**
Bezafibrate	0.3	0.4	0.34	28.29
Dicamba	<LOD	<LOD	---	---
Diclofenac	<LOD	<LOD	---	---
E1	11.3	8.8	10.04	17.62

Lab. Code:	OPC-EMB-19-158	OPC-EMB-19-160		
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2		
Volume sampled (L):	18.30	18.05		
Concentration	ng/L	ng/L	Average	Cv %
E2	2.8	3.5	3.14	14.33
EE2	<LOD	<LOD	---	---
Ibuprofen	<LOD	<LOD	---	---
Metaflumizone	<LOD	<LOD	---	---
Naproxen	<LOD	<LOD	---	---
10,11-dihydro-10,11-dihydroxy-carbamazepine	3.1	2.8	2.97	7.59
Acetamiprid	0.10	0.12	0.11	13.75
Amoxicillin	N.A	N.A	---	---
Azithromycin	<LOD	<LOD	---	---
Benzotriazole	45.5	36.6	41.05	15.29
Bromacil	<LOD	<LOD	---	---
Carbamazepine	1.1	1.2	1.16	3.79
Chloroxuron	<LOD	<LOD	---	---
Ciprofloxacin	<LOD	<LOD	---	---
Clarithromycin	<LOD	<LOD	---	---
Diazinon	<LOD	<LOD	---	---
Dimethenamid	0.019	0.027	0.02	25.67
Fipronil	<LOD	<LOD	---	---
Imidacloprid	0.2	0.3	0.26	6.86
Linuron	<LOD	<LOD	---	---
Metazachlor	0.4	<LOD	0.37	---
Metolachlor	0.8	1.4	1.12	37.52
Sulfamethoxazole	<LOD	<LOD	---	---
Tebuconazole	1.5	1.6	1.56	3.78
Terbutryn	<LOD	<LOD	---	---

Graph 1. *Reproducibility of Polar Compounds*





5.1.2. Semi-polar and apolar compounds

Tables 20, 23, 28 and 31 report: average recovery of internal standards and relative coefficients of variation calculated in real samples, sampling efficiency evaluated in real samples JBSS_UA_GE-1_1 and JBSS_UA_GE-1_2, for which the break-through tests were available. The data for triazines are not reported because no syringe standard was available.

The sampling efficiency evaluation was calculated based on the labeled internal standards added to the water samples before filtration, using the Mariani Box, comparing their presence in the first and break-through filters. The following formula was applied:

$$Sampling\ Efficiency\ (\%) = 100 * \frac{X\ Rec\ F1}{X\ Rec\ F1 + X\ Rec\ F - BT}$$

where:

X Rec F1: Recovery (%) of analyte X calculated in the first filter

X Rec F-BT: Recovery (%) of analyte X calculated in the break-through filter

Tables 21, 24, 26, 29 and 32 report method LODs (calculated as signal to noise 3:1) and LOQs (calculated as signal to noise 10:1) respectively for OPCs, Chlorinated and Triazines pesticides, PAHs and PCBs.

The environmental samples JBSS_UA_GE-1_1 and JBSS_UA_GE-1_2 were sampled for the reproducibility test. In the tables 22, 25, 27, 30 and 30 the concentrations of compounds detected, their relative coefficients of variation calculated are reported. Graphs 2, 3, 4, 5 and 6 show results respectively for OPCs, pesticides, Triazines, PAHs and PCBs.

5.1.2.1. *Organophosphate Compounds OPCs*

Table 20. *Recovery and Sampling efficiency of Organophosphate Compounds OPCs*

	20L real samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	*JBSS_GE_UA-1_1*	*JBSS_GE_UA-1_2*
TEP-D9	22.8	51.5	64.3	43.5
TNPP-D21	44.4	54.7	66.9	47.0
TNBP-D27	65.7	55.0	66.8	53.4
TCEP-D12	44.1	43.9	65.6	44.1
TDCPP-D15	41.1	41.5	61.8	56.7
TBOEP-13C6	83.6	49.3	64.3	54.1
TPhP-13C18	59.3	46.9	64.7	57.7
T35DMPP-D9	27.6	46.3	63.3	75.8

Table 21. *LOD/LOQ of Organophosphate Compounds OPCs*

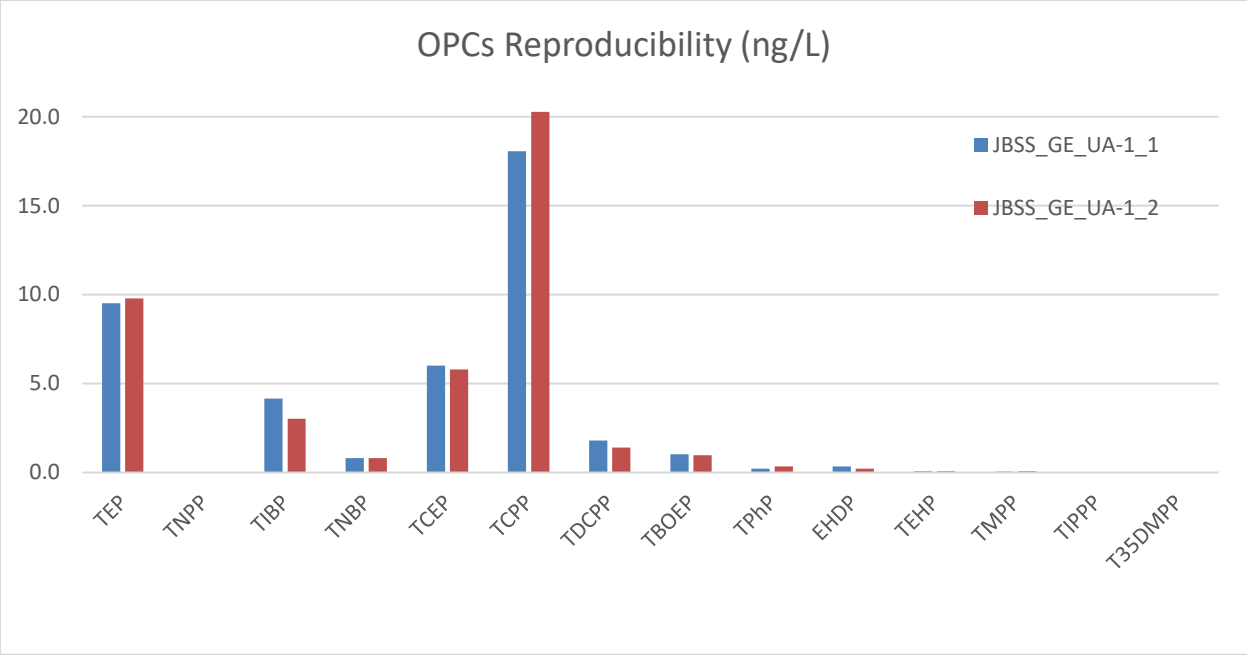
	LOD 3/1	LOQ 10/1
	ng/L	ng/L
TEP	0.05	0.167
TNPP	0.05	0.167
TIBP	0.015	0.05

	LOD 3/1	LOQ 10/1
TNBP	0.02	0.067
TCEP	0.03	0.1
TCPP	0.015	0.05
TDCPP	0.005	0.017
TBOEP	0.3	1
TPhP	0.05	0.167
EHDP	0.003	0.01
TEHP	0.001	0.003
TMPP	0.02	0.067
TIPPP	0.12	0.4
T35DMPP	0.01	0.033

Table 22. *Reproducibility data of Organophosphate Compounds (OPCs)*

Lab. Code:	**OPC-EMB-19-158**	**OPC-EMB-19-160**		
Sample name:	**JBSS_GE_UA-1_1**	**JBSS_GE_UA-1_2**		
Type of sample:	**MB Black Sea water**	**MB Black Sea water**		
Volume sampled (L):	**18.30**	**18.05**		
Sampling period:	**29/07/2019**	**29/07/2019**		
Analysis date:	**10/30/2019**	**10/30/2019**		
Concentration	**ng/L**	**ng/L**	**Average**	**Cv %**
TEP	9.5	9.8	9.65	1.86
TNPP	**<LOD**	**<LOD**	---	---
TIBP	4.1	3.0	3.58	22.41
TNBP	0.81	0.80	0.80	0.45
TCEP	6.0	5.8	5.90	2.51
TCPP	18	20	19.17	8.21
TDCPP	1.8	1.4	1.60	17.78
TBOEP	1.0	0.98	1.00	2.81
TPhP	0.22	0.33	0.28	30.15
EHDP	0.33	0.22	0.28	28.29
TEHP	0.07	0.08	0.08	6.08
TMPP	0.05	0.08	0.06	34.55
TIPPP	**<LOD**	**<LOD**	---	---
T35DMPP	**<LOD**	**<LOD**	---	---

Graph 2. *Reproducibility of Organophosphate Compounds (OPCs)*



5.1.2.2. ***Chlorinated Pesticides***

Dicofol has been analysed but not reported in the recovery table and in the results. The labeled internal standard was introduced in the methodology in order to reduce the result variability due to the compound instability. The experiments showed that the degradation of dicofol-d8 occurred completely, so was not possible to evaluate Dicofol concentration in all samples.

The low compatibility of the Chlorfenvinphos-d10 with the syringe standards adopted and the presence of interferences for C13-Endrin can lead to an overestimation or underestimation of the recoveries, thus their recoveries are considered as indicative.

Low molecular weight of HCBD and Dichlorvos and their high volatility makes the methodology not suitable for this substances. Therefore the concentrations of HCBD and Dichlorvos are not reported.

Table 23. *Recovery and Sampling efficiency of Chlorinated Pesticides*

	20L real samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	*JBSS_GE_UA-1_1*	*JBSS_GE_UA-1_2*
C13 HCBD	---	---	---	---
D6 Dichlorvos	---	---	---	---
C13_PeCBz	24.9	81.0	62.9	62.8
D14 Trifluralin	36.6	79.2	38.7	75.9
13C HCB	39.9	92.6	43.4	65.6

	20L real samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	*JBSS_GE_UA-1_1*	*JBSS_GE_UA-1_2*
13C a-HCH	62.0	69.4	66.4	56.5
13C_g-HCH	68.6	66.1	63.8	52.0
13C_Heptachlor	46.7	66.8	43.0	72.1
D10 Chlorpyriphos	97.3	87.8	56.6	77.7
C13_Aldrin	30.2	63.2	52.3	73.2
D10 Chlorfenvinphos (*)	208.7	65.0	67.1	52.4
C13 Isodrin	35.1	75.3	59.3	72.1
C13_Oxychlordane	35.4	63.1	40.3	74.5
C13-Endosulfane-alpha	95.0	70.2	53.5	61.0
C13_Heptachlor-exo-epoxide	52.3	66.8	50.2	62.1
C13_trans-chlordane	32.8	63.0	42.0	76.9
C13_trans-nonachlor	29.4	57.0	44.2	79.3
C13_op-DDE	50.6	63.4	56.1	67.3
C13_pp-DDE	40.6	63.9	62.3	75.8
C13_op-DDD	43.1	65.1	51.5	76.8
C13_op-DDT	53.8	60.4	60.7	75.6
C13_pp-DDT	58.1	58.0	60.6	76.0
C13_Dieldrin	58.8	65.4	48.4	64.2
C13_Endrin (*)	155.4	68.8	51.0	64.3
C13_Endosulfane-beta	71.8	69.3	54.5	63.2
C13_cis-nonachlor	36.0	57.7	43.8	79.4
C13 Endosulfane-sulphate	54.6	64.6	52.3	54.1
C13 Methoxychlor	95.3	49.3	50.4	67.0
C13_Mirex	42.0	64.3	62.8	76.8
D6 Cypermethrin	43.4	61.4	53.5	73.2
(*): Recoveries considered as indicative				

Table 24. *LOD and LOQ of Chlorinated Pesticides*

	LOD 3/1	LOQ 10/1
	pg/L	pg/L
PeCBz	0.50	1.67
HCB	0.50	1.67
a-HCH	3.00	10.0
b-HCH	3.00	10.0
g-HCH	3.00	10.0

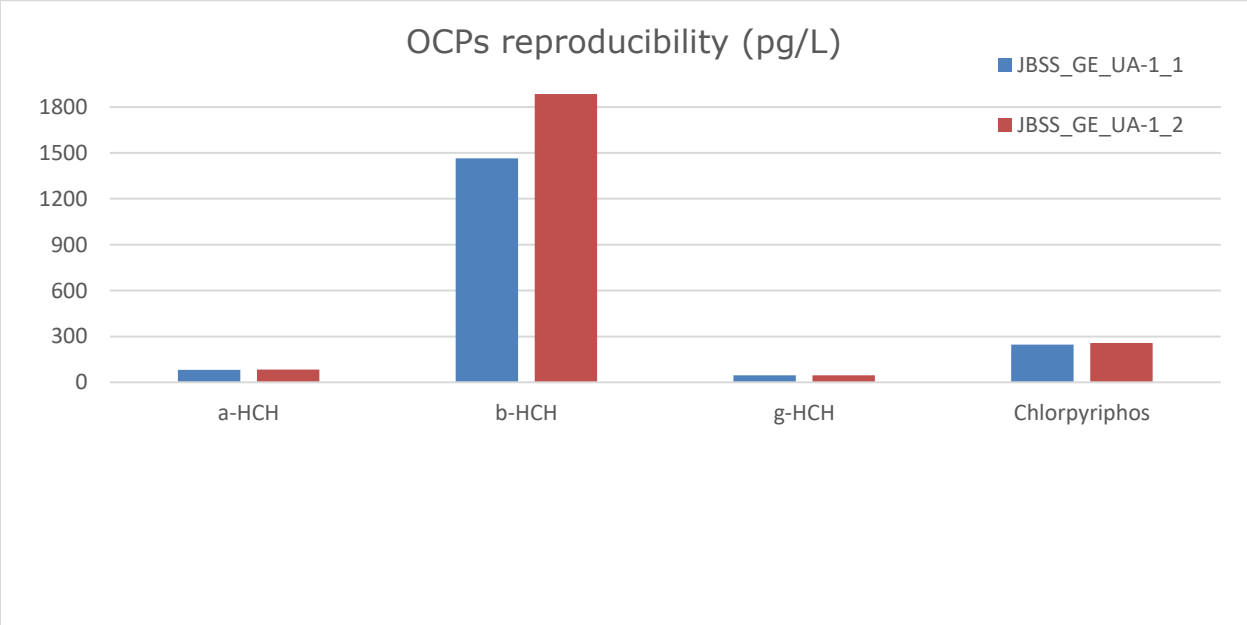
	LOD 3/1	LOQ 10/1
d-HCH	3.00	10.0
e-HCH	3.00	10.0
Heptachlor	0.80	2.67
Heptachlor-exo-epoxide	1.50	5.00
Heptachlor-endo-epoxide	10.0	33.3
Aldrin	3.50	11.7
Dieldrin	2.50	8.33
Endrin	2.00	6.67
Isodrin	15.0	50.0
trans-chlordane	5.00	16.7
cis-chlordane	5.00	16.7
Oxychlordane	3.00	10.0
trans-nonachlor	0.50	1.67
cis-nonachlor	3.00	10.0
Endosulfane-alpha	15.0	50.0
Endosulfane-beta	2.00	6.67
Endosulfane-sulphate	0.50	1.67
op-DDE	2.00	6.67
pp-DDE	2.00	6.67
op-DDD	1.50	5.00
pp-DDD	1.50	5.00
op-DDT	3.00	10.0
pp-DDT	3.00	10.0
Methoxychlor	12.0	40.0
Mirex	0.50	1.67
Others;		
HCBD	2.00	6.67
Dichlorvos	15.0	50.0
Trifluralin	1.00	3.33
Triallate	7.00	23.3
Chlorpyriphos	3.50	11.7
Chlorfenvinphos	25.0	83.3
Dicofol	180	600
Cypermethrins	35.0	117
Chlorothalonil	3.00	10.0

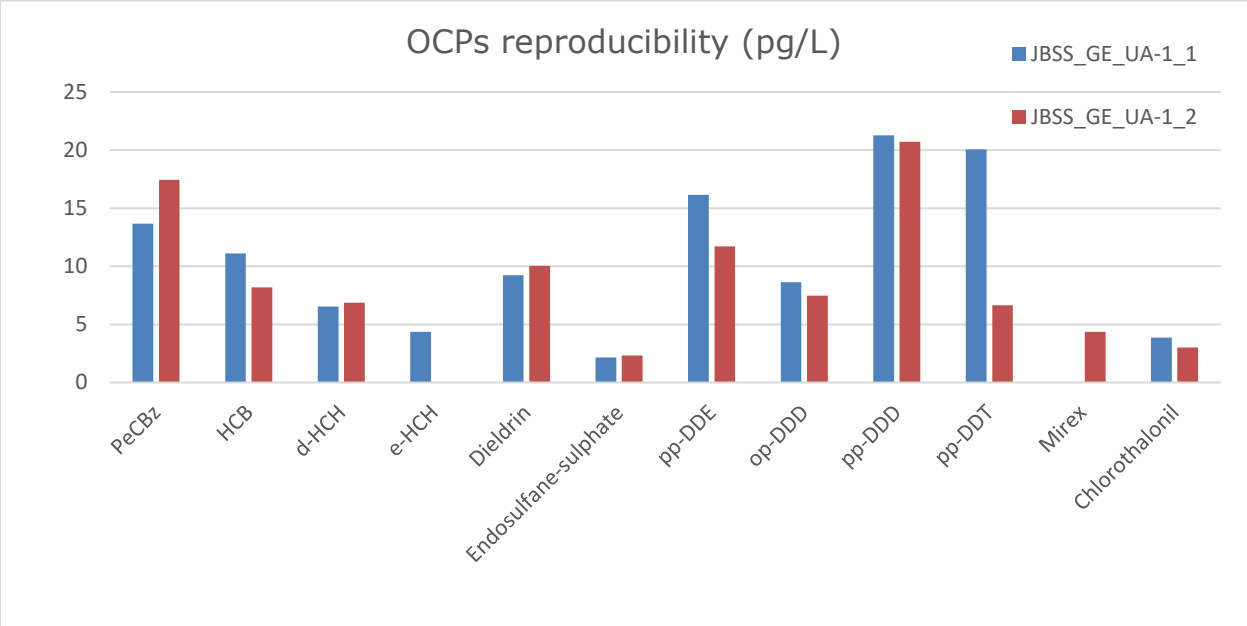
Table 25. *Reproducibility data of Chlorinated Pesticides*

Lab. Code:	OCP-EMB-19-158	OCP-EMB-19-160		
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	18.30	18.05		
Sampling period:	29/07/2019	29/07/2019		
Analysis date:	12/2/2019	12/2/2019		
Concentration	pg/L	pg/L	Average	Cv %
PeCBz	14	17	16	17.16
HCB	11	8.2	9.6	21.38
a-HCH	81	84	83	2.55
b-HCH	1464	1885	1675	17.77
g-HCH	46	45	45	1.23
d-HCH	6.5	6.9	6.7	3.56
e-HCH	4.4	**<LOD**	4.4	---
Sum-HCHs	1602	2021	1811	16.36
Heptachlor	**<LOD**	**<LOD**	---	---
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	---	---
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	---	---
Sum-Heptachlorepoxides	---	---	---	---
Aldrin	**<LOD**	**<LOD**	---	---
Dieldrin	9.2	10	9.6	5.83
Endrin	**<LOD**	**<LOD**	---	---
Isodrin	**<LOD**	**<LOD**	---	---
Sum-Drins	9.2	10	9.6	5.83
trans-chlordane	**<LOD**	**<LOD**	---	---
cis-chlordane	**<LOD**	**<LOD**	---	---
Sum-Chlordane	---	---	---	---
Oxychlordane	**<LOD**	**<LOD**	---	---
trans-nonachlor	**<LOD**	**<LOD**	---	---
cis-nonachlor	**<LOD**	**<LOD**	---	---
Sum-nonachlor	---	---	---	---
Endosulfane-alpha	**<LOD**	**<LOD**	---	---
Endosulfane-beta	**<LOD**	**<LOD**	---	---
Sum-Endosulfanes	---	---	---	---
Endosulfane-sulphate	2.2	2.3	2.3	5.20
op-DDE	**<LOD**	**<LOD**	---	---
pp-DDE	16	12	14	22.53
op-DDD	8.6	7.5	8.0	10.33
pp-DDD	21	21	21	1.79
op-DDT	**<LOD**	**<LOD**	---	---
pp-DDT	20	6.7	13	70.89
Sum-DDTtotal	66	47	56	24.52

Lab. Code:	OCP-EMB-19-158	OCP-EMB-19-160		
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	18.30	18.05		
Sampling period:	29/07/2019	29/07/2019		
Analysis date:	12/2/2019	12/2/2019		
Concentration	**pg/L**	**pg/L**	**Average**	**Cv %**
Methoxychlor	**<LOD**	**<LOD**	---	---
Mirex	**<LOD**	4.4	4.4	---
Others;				
HCBD	**n.r.**	**n.r.**	**---**	**---**
Dichlorvos	**n.r.**	**n.r.**	**---**	**---**
Trifluralin	**<LOD**	**<LOD**	---	---
Triallate	**<LOD**	**<LOD**	---	---
Chlorpyriphos	246	257	252	2.89
Chlorfenvinphos	**<LOD**	**<LOD**	---	---
Dicofol	**n.r.**	**n.r.**	---	---
Cypermethrins	**<LOD**	**<LOD**	---	---
Chlorothalonil	3.9	3.0	3.4	17.62
n.r.: not recovered				

Graph 3. *Reproducibility graphs of Chlorinated Pesticides*





5.1.2.3. *Triazine pesticides*

Recovery and break-through tests data for triazines are not reported because no syringe standard was available.

Triazine pesticides in 20L spot samples were analysed both by HRGC-HRMS and UHPLC-MS/MS techniques.

HRGC-HRMS and UHPLC-MS/MS analytical results of JBSS_UA_GE-1_1 and JBSS_UA_GE-1_2 on reproducibility are reported and compared in table 27 and in the following graphs. The obtained results showed a good agreement.

In the report only the results obtained with HRGC-HRMS are reported because it is the same technique used for LV-TS samples in order to provide a better comparability between the two different sampling methodologies.

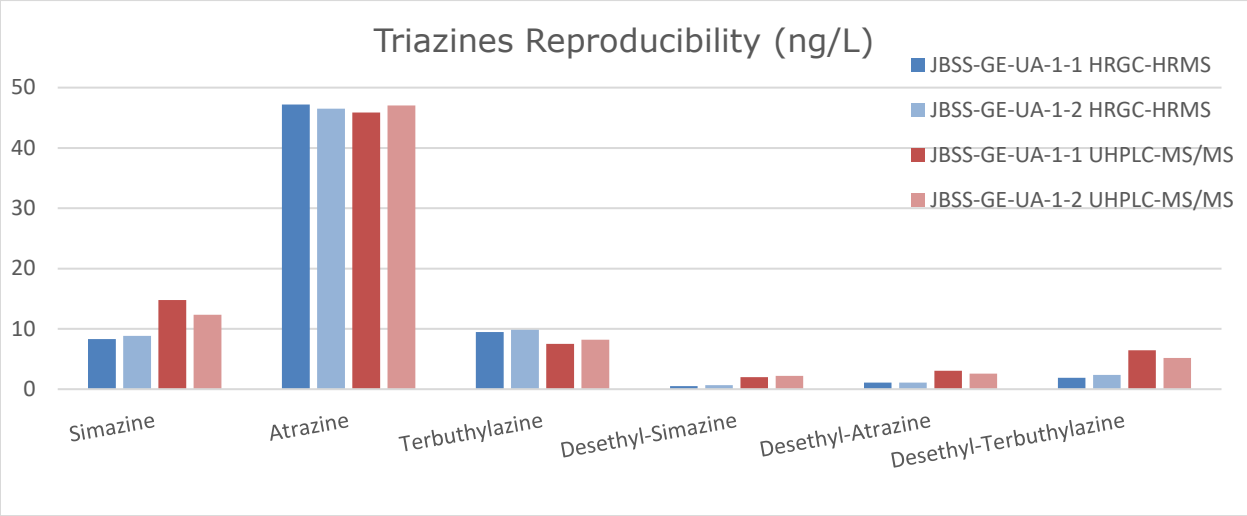
Table 26. *LOD and LOQ of Triazine Pesticides by HRGC-HRMS and UHPLC-MS/MS*

	HRGC-HRMS			UHPLC-MS/MS	
	LOD 3/1	LOQ 10/1		LOD 3/1	LOQ 10/1
	ng/L	ng/L		ng/L	ng/L
Simazine	0.05	0.12		0.1	0.2
Atrazine	0.04	0.11		0.07	0.1
Terbuthylazine	0.01	0.03		0.1	0.2
Desethyl-Simazine	0.01	0.01		0.1	0.3
Desethyl-Atrazine	0.01	0.03		0.4	1.2
Desethyl-Terbuthylazine	0.01	0.03		0.1	0.3

Table 27. *Triazine pesticides HRGC-HRMS and UHPLC-MS/MS reproducibility data*

Lab. Code:	TRIAZ-EMB-19-158	TRIAZ-EMB-19-160		
Sample name:	JBSS-GE-UA-1-1	JBSS-GE-UA-1-2		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	18.30	18.05		
Sampling period:	28/07/2019	28/07/2019		
Analytical Technique	HRGC-HRMS			
			Average	Cv %
Concentration	**ng/L**	**ng/L**	**ng/L**	
Simazine	8.3	8.9	8.6	4.7
Atrazine	47	47	47	1.0
Terbuthylazine	9.5	9.9	9.7	2.7
Desethyl-Simazine	0.50	0.66	0.6	19.2
Desethyl-Atrazine	1.1	1.1	1.1	0.2
Desethyl-Terbuthylazine	1.9	2.3	2.1	15.7
Analytical Technique	UHPLC-MS/MS			
			Average	Cv %
Concentration	**ng/L**	**ng/L**	**ng/L**	
Simazine	15	12	14	12.6
Atrazine	46	47	46	1.8
Terbuthylazine	7.5	8.2	7.8	6.2
Desethyl-Simazine	2.0	2.2	2.1	6.9
Desethyl-Atrazine	3.0	2.6	2.8	11.4
Desethyl-Terbuthylazine	6.4	5.2	5.8	15.1
	Average HRGC-HRMS	Average UHPLC-MS/MS	HRGC-HRMS + UHPLC-MS/MS	
			Average	Cv %
Concentration	**ng/L**	**ng/L**	**ng/L**	
Simazine	8.6	14	11	31.9
Atrazine	47	46	47	0.6
Terbuthylazine	9.7	7.8	8.8	14.8
Desethyl-Simazine	0.6	2.1	1.3	80.6
Desethyl-Atrazine	1.1	2.8	1.9	62.8
Desethyl-Terbuthylazine	2.1	5.8	4.0	66.1

Graph 4. *Triazine pesticides HRGC-HRMS and UHPLC-MS/MS reproducibility*



5.1.2.4. ***Polycyclic Aromatic Hydrocarbons (PAHs), EHMC, BHT***

PAHs: Low molecular weight PAH (Naphthalene, Acenaphthylene, Acenapthene and Fluorene) have been analysed but not reported, as the methodology is not suitable for these substances, due to their higher volatility.

EHMC is a sunscreen product, thus the interpretation of its elevated concentrations, also in blank samples, should take into account eventual contamination on-site and/or during sample handling and preparation.

Table 28. *Recovery and sampling efficiency for PAHs and BHT*

	20L real samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	*JBSS_GE_UA-1_1*	*JBSS_GE_UA-1_2*
Phenanthrene-d10	63.8	69.2	87.0	50.2
Anthracene-d10	56.1	68.6	86.4	50.6
Fluoranthene-d10	39.1	50.3	69.3	53.4
Pyrene-d10	38.7	51.2	71.0	53.8
Chrysene-d12	28.4	47.0	69.8	51.6
Benzo(b)fluoranthene-d12	25.7	47.3	70.2	52.2
Perylene-d12	17.4	44.0	68.3	53.8
Benzo(a)pyrene-d12	15.0	44.4	69.7	55.1
Benzo(e)pyrene-d12	21.1	44.4	66.5	51.8
Indeno(123-cd)pyrene-d12	16.5	44.3	64.8	53.5
Benzo(ghi)perylene-d12	17.4	44.5	58.9	52.4
Dibenzo(ah)anthracene-d12	17.4	44.7	64.2	52.9
BHT-21	15.8	58.1	56.3	63.7

Table 29. *LOD and LOQ of PAHs, EHMC and BHT*

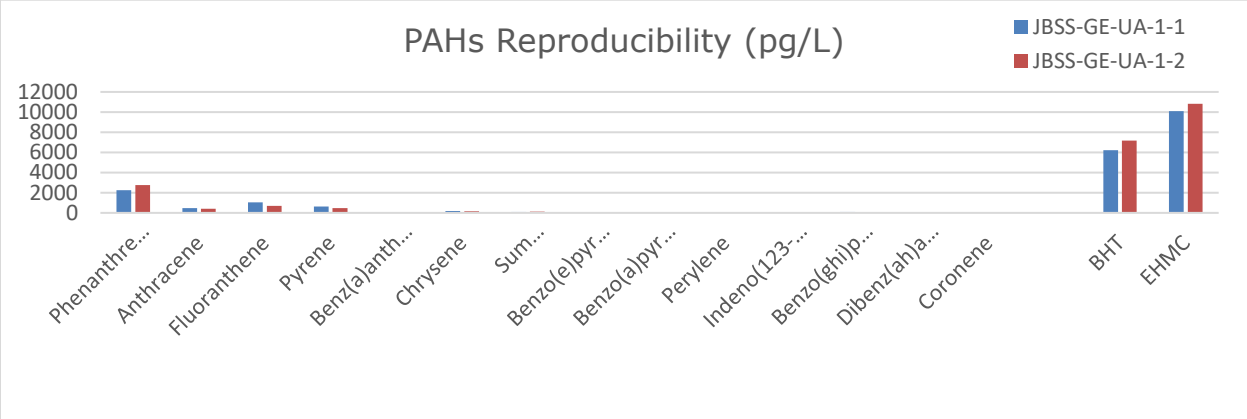
	LOD 3/1	LOQ 10/1
	pg/L	pg/L
Phenanthrene	10	33.3
Anthracene	10	33.3
Fluoranthene	8	26.7
Pyrene	8	26.7
Benz(a)anthracene	2	6.7
Chrysene	2	6.7
Sum Benzo(b,j,k)fluoranthene	1	3.3
Benzo(e)pyrene	3	10
Benzo(a)pyrene	3	10
Perylene	3.18	10.6
Indeno(123-cd)pyrene	4	13.3
Benzo(ghi)perylene	4	13.3
Dibenzo(ah)anthracene	2	6.7
Coronene	4	13.3
BHT	60	200
EHMC	60	200

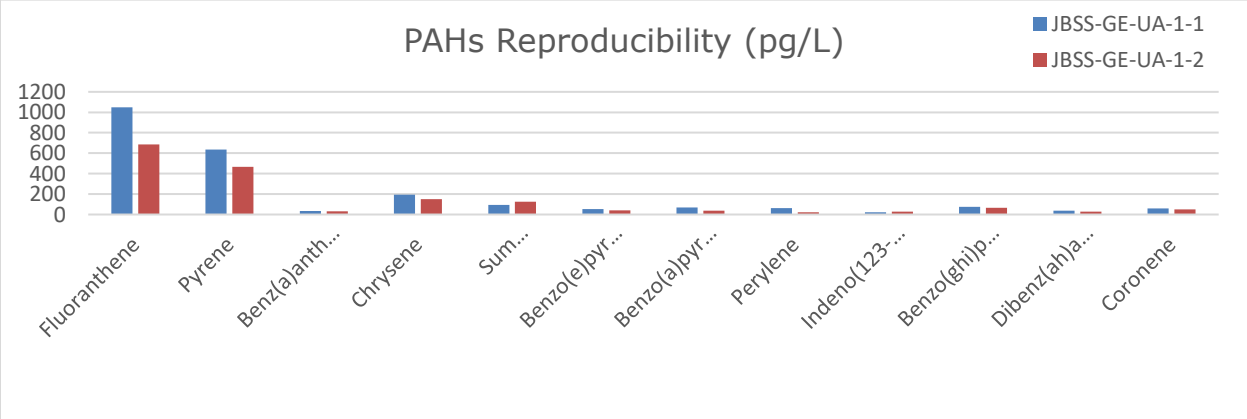
Table 30. *Reproducibility data of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT*

Lab. Code:	PAH-EMB-19-158	PAH-EMB-19-160		
Sample name:	JBSS-GE-UA-1-1	JBSS-GE-UA-1-2		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	18.30	18.05		
Sampling period:	29/07/2019	29/07/2019		
Analysis date:	11/21/2019	11/21/2019		
Concentration	pg/L	pg/L	Average	Cv %
Phenanthrene	2241	2745	2493	14.30
Anthracene	476	417	447	9.35
Fluoranthene	1050	685	867	29.76
Pyrene	636	467	551	21.66
Benz(a)anthracene	33	30	32	6.45
Chrysene	194	149	172	18.50
Sum Benzo(b,j,k)fluoranthene	93	126	110	21.02
Benzo(e)pyrene	53	42	47	16.04
Benzo(a)pyrene	69	39	54	40.08
Perylene	64	23	44	67.13

Lab. Code:	PAH-EMB-19-158	PAH-EMB-19-160		
Sample name:	JBSS-GE-UA-1-1	JBSS-GE-UA-1-2		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	18.30	18.05		
Sampling period:	29/07/2019	29/07/2019		
Analysis date:	11/21/2019	11/21/2019		
Concentration	pg/L	pg/L	Average	Cv %
Indeno(123-cd)pyrene	23	27	25	10.18
Benzo(ghi)perylene	77	64	70	12.41
Dibenzo(ah)anthracene	38	29	34	18.61
Coronene	58	50	54	10.23
BHT	6222	7183	6703	10.14
EHMC	10095	10839	10467	5.03

Graph 5. *Reproducibility of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT*





5.1.2.5. *Polychlorinated Biphenyls (PCBs)*

Table 31. *Recovery and sampling efficiency for PCBs*

	20L real samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	*JBSS_GE_UA-1_1*	*JBSS_GE_UA-1_2*
13C12 PCB 28	62.9	51.27	61.1	67.5
13C12 PCB 52	47.6	46.42	58.6	73.0
13C12 PCB 101	37.4	46.16	61.3	76.8
13C12 PCB 118	30.1	45.00	65.1	74.6
13C12 PCB 138	38.5	44.79	69.8	74.8
13C12 PCB 153	39.4	45.74	69.6	76.0
13C12 PCB 180	45.2	46.04	74.8	73.8

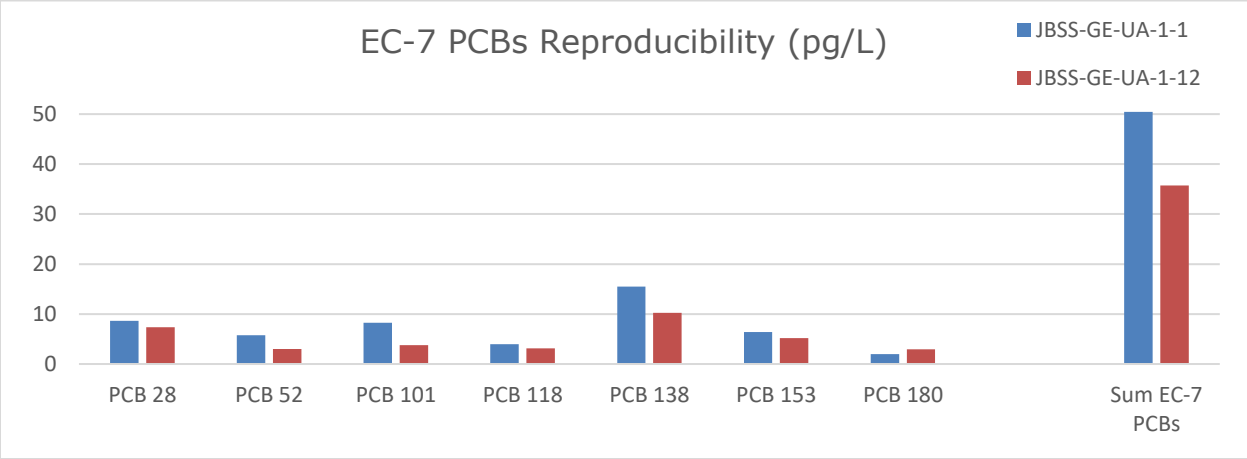
Table 32. *LOD and LOQ of PCBs*

	LOD 3/1	LOQ 10/1
	pg/L	pg/L
PCB 28	0.5	1.67
PCB 52	0.5	1.67
PCB 101	0.5	1.67
PCB 118	0.5	1.67
PCB 138	0.5	1.67
PCB 153	0.5	1.67
PCB 180	1	3.33

Table 33. *Reproducibility data of Polychlorinated Biphenyls (PCBs)*

Lab. Code:	P-EMB-19-158	P-EMB-19-160		
Sample name:	JBSS-GE-UA-1-1	JBSS-GE-UA-1-2		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	18.30	18.05		
Sampling period:	29/07/2019	29/07/2019		
Analysis date:	1/10/2020	1/10/2020		
Concentration	**pg/L**	**pg/L**	Average	Cv %
EC-7				
PCB 28	8.6	7.4	8.0	11.34
PCB 52	5.8	3.0	4.4	44.22
PCB 101	8.3	3.8	6.0	52.23
PCB 118	4.0	3.2	3.6	15.96
PCB 138	15	10	13	28.68
PCB 153	6.4	5.2	5.8	14.92
PCB 180	2.0	2.9	2.4	28.84
Sum EC-7 PCBs	50.4	35.7	43.1	24.17

Graph 6. *Reproducibility of Polychlorinated Biphenyls (PCBs)*



5.2. QA/QC Large Volume Transect Sampling,

Recovery, sampling efficiency, limits of detection and quantitation, obtained for transect samples using LV-Transect Sampling have been tested.

Tables 34, 37, 40, 43 and 46 report averages of analytical recovery of internal standards and their relative coefficients of variation obtained in filter and cells samples respectively for OPCs, Chlorinated and Triazine pesticides, PAHs and PCBs.

Tables 35, 38, 41, 44 and 47 report method LOD (calculated as blank value plus 3 standard deviations) and LOQ (calculated as blank value plus 10 standard deviations) for all three different volumes sampled respectively for OPCs, Chlorinated and Triazine pesicides, PAHs and PCBs.

Tables 36, 39, 42, 45 and 48 report the sampling efficiency obtained in LV transect samples during the cruise, respectively for OPCs, Chlorinated and Triazine pesicides, PAHs and PCBs. Evaluation of sampling efficiency was possible only for detectable compounds. For chemicals detected in both cells (cell 1 for chemicals trapping and cell 2 for break-through control) the following formula was applied:

$$Sampling\ Efficiency\ (\%) = 100 * \frac{X\ Cell\ 1}{X\ Cell\ 1 + X\ Cell\ 2}$$

where:

X Cell 1: concentration of analyte X detected in Cell 1

X Cell 2 : concentration of analyte X detected in Cell 2

Where the analyte was detected only in cell 1, in the tables >99 % is reported, for the compounds under LOD in both cell the sampling efficiency was not evaluable (n.e.).

5.2.1. Organophosphate Compounds OPCs

Interferences on Triphenyl phosphate-d15 syringe standard did not allow the calculation of the labeled phosphate flame retardants recoveries, therefore their recovery values are not reported.

Table 34. *Filters and cells analytical recovery of Organophosphate Compounds OPCs*

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
TEP-D9	n.r.	---	n.r.	---
TNPP-D21	n.r.	---	n.r.	---

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
TNBP-D27	n.r.	---	n.r.	---
TCEP-D12	n.r.	---	n.r.	---
TDCPP-D15	n.r.	---	n.r.	---
TBOEP-13C6	n.r.	---	n.r.	---
TPhP-13C18	n.r.	---	n.r.	---
T35DMPP-D9	n.r.	---	n.r.	---
n.r.: not reported				

Table 35. *LOD and LOQ of Organophosphate compounds at different sampled volumes*

	Transects 1 and 2		Transect 3		Transect 4	
Sampled volume	300L		408L		180L	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd
	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
TEP	0.028	0.072	0.020	0.053	0.046	0.120
TNPP	0.029	0.072	0.021	0.053	0.048	0.121
TIBP	0.021	0.053	0.015	0.039	0.034	0.088
TNBP	0.004	0.008	0.003	0.006	0.007	0.013
TCEP	0.001	0.002	0.001	0.002	0.002	0.004
TCPP	0.004	0.006	0.003	0.004	0.006	0.009
TDCPP	0.001	0.003	0.001	0.003	0.002	0.006
TBOEP	0.004	0.008	0.003	0.006	0.007	0.014
TPhP	0.001	0.002	0.001	0.001	0.002	0.003
EHDP	0.002	0.003	0.001	0.002	0.003	0.005
TEHP	0.0003	0.001	0.0002	0.0004	0.001	0.001
TMPP	0.0001	0.0002	0.0001	0.0001	0.0002	0.0003
TIPPP	0.0003	0.001	0.0002	0.001	0.0005	0.001
T35DMPP	0.0002	0.0004	0.0001	0.0003	0.0003	0.001

Table 36. *Sampling efficiency in 4 transect samplings of Organophosphate Compounds OPCs*

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
TEP	60.59	84.12	58.39	52.77
TNPP	---	---	---	---
TIBP	65.10	77.04	65.33	85.32
TNBP	74.49	82.13	75.37	86.00
TCEP	79.03	83.82	76.44	87.63
TCPP	83.40	71.45	67.75	89.30
TDCPP	86.43	77.93	78.65	90.94
TBOEP	62.49	>99	---	>99
TPhP	67.95	57.46	57.15	78.49
EHDP	60.06	57.38	53.49	64.85
TEHP	61.42	59.69	59.99	65.04
TMPP	>99	>99	84.81	>99
TIPPP	>99	---	---	---
T35DMPP	---	---	---	---

5.2.2. Chlorinated Pesticides

Dicofol has been analysed but not reported in the recovery table and in the results, because of compound degradation, see paragraph 5.1.2.2.

Table 37. *Filters and cells analytical recovery of Chlorinated Pesticides*

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
C13 HCBD	66.2	38.1	53.4	13.4
D6 Dichlorvos	8.6	17.3	15.1	20.9
C13_PeCBz	73.0	17.7	75.9	13.0
D14 Trifluralin	102.8	14.9	134.7	14.8
13C HCB	76.2	15.1	62.6	19.0
13C a-HCH	82.0	11.8	87.0	14.1

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
13C_g-HCH	80.3	11.0	86.2	14.7
13C_Heptachlor	101.1	15.8	107.2	15.8
D10 Chlorpyriphos	111.0	15.9	95.8	18.3
C13_Aldrin	91.0	12.5	86.8	19.6
D10 Chlorfenvinphos	65.7	19.5	87.9	23.0
C13 Isodrin	89.2	10.1	89.5	16.1
C13_Oxychlordane	81.5	10.7	73.3	18.9
C13-Endosulfane-alpha	87.6	9.3	64.3	32.5
C13_Heptachlor-exo-epoxide	78.3	6.6	77.7	17.0
C13_trans-chlordane	77.9	12.4	63.8	20.1
C13_trans-nonachlor	77.8	12.2	65.2	18.7
C13_op-DDE	87.1	11.7	76.2	16.7
C13_pp-DDE	88.0	11.0	81.3	13.9
C13_op-DDD	87.1	14.0	82.1	14.1
C13_op-DDT	97.7	10.6	97.9	15.0
C13_pp-DDT	97.6	14.4	100.4	17.2
C13_Dieldrin	86.9	10.9	79.2	15.6
C13_Endrin	111.2	14.0	131.9	13.4
C13_Endosulfane-beta	74.8	11.0	55.0	33.7
C13_cis-nonachlor	82.4	11.9	78.2	13.6
C13 Endosulfane-sulphate	69.3	20.7	56.5	35.1
C13 Methoxychlor	108.8	14.7	142.0	18.0
C13_Mirex	80.1	7.0	74.8	13.3
D6 Cypermethrin	134.9	10.2	66.7	24.8

Table 38. *LOD and LOQ of Chlorinated Pesticides at different sampled volumes*

	Transects 1 and 2		Transect 3		Transect 4	
Sampled volume	300L		408L		180L	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd
	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L
PeCBz	0.10	0.19	0.07	0.14	0.17	0.32
HCB	0.16	0.30	0.12	0.22	0.26	0.49
a-HCH	0.11	0.31	0.08	0.23	0.19	0.52
b-HCH	3.23	9.42	2.38	6.93	5.39	15.70
g-HCH	0.12	0.26	0.09	0.19	0.20	0.43
d-HCH	0.05	0.11	0.03	0.08	0.08	0.19
e-HCH	0.07	0.18	0.05	0.13	0.12	0.30
Heptachlor	0.03	0.07	0.02	0.05	0.05	0.12
Heptachlor-exo-epoxide	0.09	0.22	0.06	0.16	0.14	0.37
Heptachlor-endo-epoxide	0.49	1.35	0.36	0.99	0.82	2.24
Aldrin	0.12	0.30	0.09	0.22	0.19	0.49
Dieldrin	0.08	0.19	0.06	0.14	0.14	0.31
Endrin	0.10	0.20	0.07	0.14	0.16	0.33
Isodrin	0.60	1.46	0.44	1.08	0.99	2.44
trans-chlordane	0.02	0.04	0.01	0.03	0.03	0.06
cis-chlordane	0.02	0.04	0.01	0.03	0.03	0.07
Oxychlordane	0.06	0.17	0.05	0.12	0.11	0.28
trans-nonachlor	0.02	0.06	0.02	0.05	0.04	0.11
cis-nonachlor	0.03	0.05	0.02	0.04	0.04	0.08
Endosulfane-alpha	0.38	0.62	0.28	0.45	0.63	1.03
Endosulfane-beta	0.06	0.11	0.04	0.08	0.10	0.19
Endosulfane-sulphate	0.03	0.07	0.02	0.05	0.05	0.12
op-DDE	0.07	0.17	0.05	0.12	0.12	0.28

	Transects 1 and 2		Transect 3		Transect 4	
Sampled volume	300L		408L		180L	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd
	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L
pp-DDE	0.10	0.21	0.07	0.15	0.17	0.35
op-DDD	0.13	0.33	0.09	0.25	0.21	0.56
pp-DDD	0.03	0.06	0.02	0.05	0.05	0.10
op-DDT	0.10	0.14	0.07	0.10	0.17	0.24
pp-DDT	0.10	0.25	0.08	0.19	0.17	0.42
Methoxychlor	0.53	1.37	0.39	1.01	0.89	2.28
Mirex	0.01	0.02	0.01	0.02	0.02	0.04
Others;						
HCBD	3.47	7.91	2.55	5.82	5.78	13.19
Dichlorvos	0.57	1.36	0.42	1.00	0.95	2.27
Trifluralin	0.03	0.07	0.02	0.05	0.05	0.12
Triallate	0.07	0.16	0.05	0.11	0.11	0.26
Chlorpyriphos	0.25	0.62	0.19	0.46	0.42	1.04
Chlorfenvinphos	2.58	6.04	1.90	4.44	4.30	10.07
Dicofol	0.11	0.22	0.08	0.16	0.19	0.36
Cypermethrins	0.37	0.85	0.27	0.62	0.61	1.41
Chlorothalonil	0.20	0.67	0.15	0.49	0.33	1.11

Table 39. *Sampling efficiency in 4 transect samples of Chlorinated Pesticides*

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
PeCBz	>99	>99	>99	66.87
HCB	>99	>99	75.81	>99
a-HCH	91.36	87.12	81.76	95.27
b-HCH	90.81	86.73	82.25	95.28
g-HCH	88.64	92.42	77.33	95.03
d-HCH	95.89	86.81	84.94	96.51

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
e-HCH	92.89	84.32	83.39	>99
Heptachlor	>99	>99	>99	---
Heptachlor-exo-epoxide	89.82	82.95	81.09	91.82
Heptachlor-endo-epoxide	---	---	---	---
Aldrin	---	---	---	---
Dieldrin	87.79	82.89	80.90	92.06
Endrin	>99	>99	>99	>99
Isodrin	---	---	---	---
trans-chlordane	>99	>99	71.78	79.99
cis-chlordane	65.15	>99	82.78	>99
Oxychlordane	---	---	---	---
trans-nonachlor	>99	>99	68.25	>99
cis-nonachlor	73.86	75.72	>99	---
Endosulfane-alpha	---	---	---	---
Endosulfane-sulphate	90.61	85.65	79.50	>99
op-DDE	>99	>99	>99	67.79
pp-DDE	95.62	>99	80.39	93.77
op-DDD	>99	>99	>99	99.03
pp-DDD	97.60	>99	90.64	98.82
op-DDT	>99	94.87	>99	>99
pp-DDT	91.77	>99	>99	94.92
Methoxychlor	---	---	---	---
Mirex	46.79	---	---	49.83
Others;				
HCBD	>99	>99	>99	>99

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
Dichlorvos	---	---	---	---
Trifluralin	>99	---	---	72.11
Triallate	90.55	>99	86.30	>99
Chlorpyriphos	96.97	97.79	84.11	98.64
Chlorfenvinphos	---	---	---	---
Dicofol	---	---	---	---
Cypermethrins	---	---	>99	---
Chlorothalonil	>99	>99	>99	>99

5.2.3. Triazine Pesticides

Recovery data for triazines are not reported because no syringe standard was available.

Table 40. *Filters and cells analytical recovery of Triazine Pesticides*

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
Simazine-d10	n.r.	---	n.r.	---
Atrazine-d10	n.r.	---	n.r.	---
Terbuthylazine-d5	n.r.	---	n.r.	---
n.r.: not reported				

Table 41. *LOD and LOQ of Triazine Pesticides at different sampled volumes*

	Transects 1 and 2		Transect 3		Transect 4	
Sampled volume	300L		408L		180L	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd
	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
Simazine	0.0023	0.0061	0.0017	0.0045	0.0038	0.010
Atrazine	0.0048	0.0115	0.0036	0.0085	0.0081	0.019
Terbuthylazine	0.0068	0.019	0.0050	0.014	0.011	0.032
Desethyl-Simazine	0.0052	0.013	0.0038	0.0093	0.0087	0.021
Desethyl-Atrazine	0.0028	0.0078	0.0020	0.0057	0.0046	0.013
Desethyl-Terbuthylazine	0.0026	0.0070	0.0019	0.0052	0.0043	0.012

Table 42. *Sampling efficiency in 4 transect samples of Triazine Pesticides*

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
Simazine	70.64	88.36	52.87	75.83
Atrazine	75.19	82.78	61.18	81.00
Terbuthylazine	85.05	78.00	71.02	87.00
Desethyl-Simazine	---	---	---	---
Desethyl-Atrazine	49.42	79.41	41.32	46.05
Desethyl-Terbuthylazine	68.26	89.71	60.08	76.09

5.2.4. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

Table 43. *Filter and extraction cells analytical recovery of Polycyclic Aromatic Hydrocarbons (PAHs) and BHT*

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
Phenanthrene-d10	48.9	16.4	47.2	15.8
Anthracene-d10	48.8	18.2	46.9	15.0
Fluoranthene-d10	48.4	16.6	45.6	13.0
Pyrene-d10	48.8	17.2	46.7	15.4
Chrysene-d12	74.8	8.9	49.6	16.4
Benzo(b)fluoranthene-d12	74.6	4.2	49.9	19.2
Perylene-d12	73.5	9.9	52.9	14.8
Benzo(a)pyrene-d12	68.0	4.0	49.3	17.2
Benzo(e)pyrene-d12	81.0	2.8	55.3	14.0
Indeno(123-cd)pyrene-d12	62.5	9.7	39.8	25.4
Benzo(ghi)perylene-d12	54.6	10.9	29.9	27.1
Dibenz(ah)anthracene-d12	60.7	10.6	39.2	23.8
Coronene-12	23.6	30.6	11.9	58.4
BHT	45.3	51.6	115.3	68.6

Table 44. *LOD and LOQ of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT at different sampled volumes*

	Transects 1 and 2		Transect 3		Transect 4	
Sampled volume	300L		408L		180L	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd
	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L
Phenanthrene	12	26	8.96	19	20	43
Anthracene	3.34	7.12	2.46	5.23	5.57	12
Fluoranthene	2.38	4.80	1.75	3.53	3.97	8.01
Pyrene	7.40	18	5.44	13	12	31
Benz(a)anthracene	0.67	1.87	0.49	1.37	1.11	3.12
Chrysene	1.48	3.88	1.09	2.86	2.46	6.47
Sum Benzo(b,j,k)fluoranthene	0.55	1.12	0.41	0.83	0.92	1.87
Benzo(e)pyrene	0.12	0.28	0.09	0.21	0.20	0.47
Benzo(a)pyrene	0.09	0.22	0.07	0.16	0.16	0.37
Perylene	0.13	0.27	0.09	0.20	0.21	0.44
Indeno(123-cd)pyrene	0.11	0.29	0.08	0.21	0.19	0.49
Benzo(ghi)perylene	0.18	0.41	0.13	0.30	0.30	0.68
Dibenz(ah)anthracene	0.15	0.39	0.11	0.29	0.26	0.65
Coronene	0.40	1.04	0.29	0.77	0.67	1.74
BHT	117	305	86	224	195	508
EHMC	21	34	16	25	36	56

Table 45. *Sampling efficiency in 4 transect samples of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT*

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
Phenanthrene	85.83	94.86	88.17	94.72
Anthracene	>99	>99	>99	>99
Fluoranthene	74.61	87.41	72.57	82.69
Pyrene	88.45	93.32	87.78	91.88
Benz(a)anthracene	80.65	>99	>99	>99
Chrysene	94.33	>99	90.97	>99

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
Sum Benzo(b,j,k)fluoranthene	88.23	83.75	87.45	86.95
Benzo(e)pyrene	88.05	>99	89.01	91.83
Benzo(a)pyrene	>99	61.10	>99	>99
Perylene	77.33	>99	57.61	80.83
Indeno(123-cd)pyrene	81.78	66.35	78.79	76.83
Benzo(ghi)perylene	72.03	69.04	69.54	76.33
Dibenz(ah)anthracene	71.75	75.68	>99	72.33
Coronene	---	58.26	61.65	>99
BHT	---	---	---	>99
EHMC	69.62	95.37	89.86	97.80

5.2.5. Polychlorinated biphenyls (PCBs)

Table 46. *Filter and extraction cells analytical recovery of Polychlorinated biphenyls (PCBs)*

	LV-TS Filter		LV-TS Cells	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %
13C12 PCB 28	90.5	11.3	70.1	6.1
13C12 PCB 52	94.2	8.0	78.5	12.0
13C12 PCB 101	101.6	12.7	76.8	11.8
13C12 PCB 118	97.8	12.5	75.6	12.5
13C12 PCB 138	110.0	10.8	78.7	10.4
13C12 PCB 153	112.2	10.7	78.9	8.2
13C12 PCB 180	107.7	10.1	76.1	12.9

Table 47. *LOD and LOQ of Polychlorinated biphenyls (PCBs) at different sampled volumes*

	Transects 1 and 2		Transect 3		Transect 4	
Sampled volume	300L		408L		180L	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd	Blank +3sd	Blank +10sd
	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L
EC-7						
PCB 28	0.049	0.073	0.036	0.053	0.081	0.12
PCB 52	0.068	0.13	0.050	0.092	0.11	0.21
PCB 101	0.062	0.085	0.045	0.062	0.10	0.14
PCB 118	0.099	0.22	0.073	0.16	0.16	0.36
PCB 138	0.18	0.31	0.13	0.23	0.29	0.52
PCB 153	0.19	0.29	0.14	0.22	0.31	0.49
PCB 180	0.096	0.15	0.070	0.11	0.16	0.24

Table 48. *Sampling efficiency in 4 transect samples of Polychlorinated biphenyls (PCBs)*

	Transects			
	JBSS_XL_LVE-1	JBSS_XL_LVE-2	JBSS_XL_LVE-3	JBSS_XL_LVE-4
	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)	Sampling Efficiency (%)
EC-7				
PCB 28	97.84	92.08	90.04	81.05
PCB 52	92.61	70.36	64.68	67.57
PCB 101	88.45	68.65	62.84	49.98
PCB 118	87.00	>99	>99	35.75
PCB 138	83.36	>99	>99	48.58
PCB 153	83.24	>99	>99	37.91
PCB 180	>99	---	---	33.29

6. Analytical results

The concentrations of the selected compounds using different sampling devices (Mariani Box and LV transects) are reported in the following paragraphs.

6.1. Mariani Box 20L spot samples

6.1.1. Polar compounds

In Tables from 49 to 56 the results of Polar Compounds obtained with 20L spot samples are reported.

Table 49. *Polar Compounds concentrations in Blank samples*

Lab. Code:	OPC-LBLK-240919	OPC-LBLK-250919	OPC-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	JBSS_Field Blank
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2020	25/09/2020	28/07-08/08/2019
Concentration	ng/L	ng/L	ng/L
Bezafibrate	<LOD	<LOD	<LOD
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	<LOD	<LOD	<LOD
E2	<LOD	<LOD	<LOD
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	<LOD	<LOD	18
Acetamiprid	<LOD	<LOD	0.023
Amoxicillin	N.A.	N.A.	N.A.
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	<LOD	<LOD	0.43
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	<LOD	<LOD	1.03
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD

Lab. Code:	OPC-LBLK-240919	OPC-LBLK-250919	OPC-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	JBSS_Field Blank
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2020	25/09/2020	28/07-08/08/2019
Concentration	ng/L	ng/L	ng/L
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	<LOD	<LOD	<LOD
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	<LOD	<LOD	<LOD
Linuron	<LOD	<LOD	<LOD
Metazachlor	<LOD	<LOD	0.69
Metolachlor	<LOD	<LOD	0.035
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	<LOD	<LOD	0.55
Terbutryn	<LOD	<LOD	<LOD
N.A.: Not Available			

Table 50. *Polar Compounds concentrations in samples from the coast of Ukraine and from outside the Danube delta (JBSS_GE_UA-1A)*

Lab. Code:	19-155	19-156	19-157
Sample name:	JBSS_GE_UA-1A	JBSS_GE_UA-2A	JBSS_GE_UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Concentration	ng/L	ng/L	ng/L
Bezafibrate	0.2	0.7	0.7
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	15	5.0	3.3
E2	4.6	2.8	2.3
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD

Lab. Code:	19-155	19-156	19-157
Sample name:	JBSS_GE_UA-1A	JBSS_GE_UA-2A	JBSS_GE_UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	3.3	2.6	2.4
Acetamiprid	0.11	0.11	0.07
Amoxicillin	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	52.9	36.0	26.2
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	1.8	1.1	0.9
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	0.025	0.011	0.007
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	1.8	6.4	0.3
Linuron	<LOD	<LOD	<LOD
Metazachlor	0.4	0.6	0.4
Metolachlor	1.4	0.6	0.8
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	1.9	1.5	1.1
Terbutryn	<LOD	<LOD	<LOD

N.A.: Not Available

Table 51. *Polar Compounds concentrations in samples from the coast of Georgia, provided by Georgian partners*

Lab. Code:	19-176	19-177	19-178
Sample name:	JBSS_GE-1	JBSS_GE-2	JBSS_GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	07/08/2019	07/08/2019	07/08/2019
Concentration	ng/L	ng/L	ng/L
Bezafibrate	<LOD	<LOD	2.3
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	<LOD	<LOD	1.2
E2	<LOD	<LOD	<LOD
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	7.9	2.1	1.2
Acetamiprid	0.08	0.11	0.08
Amoxicillin	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	19	29	14
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	1.0	1.1	0.9
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	6.9	<LOD
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	<LOD	<LOD	<LOD
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	0.1	0.1	<LOD
Linuron	<LOD	<LOD	<LOD
Metazachlor	<LOD	<LOD	<LOD
Metolachlor	0.0	0.1	0.0
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	0.7	0.4	0.5
Terbutryn	<LOD	<LOD	<LOD

N.A.: Not Available

Table 52. *Polar Compounds concentrations in samples from the coast of Ukraine, provided by Ukrainian partners*

Lab. Code:	19-173	19-174	19-175
Sample name:	JBSS_UA-10	JBSS_UA-11	JBSS_UA-15
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	19.30	19.55
Sampling period:	04/08/2019	04/08/2019	04/08/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
Bezafibrate	<LOD	<LOD	<LOD
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	2.1	0.6	1.1
E2	0.7	<LOD	<LOD
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	2.4	9.1	4.5
Acetamiprid	0.03	0.06	0.06
Amoxicillin	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	21	22	32
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	1.0	2.5	1.8
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	0.016	<LOD	<LOD
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	0.9	0.2	0.5
Linuron	<LOD	<LOD	<LOD
Metazachlor	<LOD	<LOD	<LOD
Metolachlor	5.0	0.1	0.4
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	1.0	0.8	1.5
Terbutryn	<LOD	<LOD	<LOD

N.A.: Not Available

Table 53. *Polar Compounds concentrations in open sea samples*

Lab. Code:	19-158	19-160	19-162
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2	JBSS_GE_UA-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.30	18.05	18.89
Sampling period:	29/07/2019	29/07/2019	30/07/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
Bezafibrate	0.3	0.4	8.1
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	11	8.8	4.7
E2	2.8	3.5	<LOD
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	3.1	2.8	1.8
Acetamiprid	0.10	0.12	0.07
Amoxicillin	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	45	37	21
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	1.1	1.2	0.4
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	0.019	0.027	<LOD
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	0.2	0.3	<LOD
Linuron	<LOD	<LOD	<LOD
Metazachlor	0.4	<LOD	<LOD
Metolachlor	0.8	1.4	0.1
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	1.5	1.6	0.8
Terbutryn	<LOD	<LOD	<LOD

N.A.: Not Available

Table 54. *Polar Compounds concentrations in open sea samples*

Lab. Code:	19-163	19-164	19-165
Sample name:	JBSS_GE_UA-3	JBSS_GE_UA-4	JBSS_GE_UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.35	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	31/07/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
Bezafibrate	<LOD	<LOD	<LOD
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	2.5	4.1	3.3
E2	<LOD	1.0	0.7
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	2.3	1.7	1.5
Acetamiprid	0.09	0.05	0.07
Amoxicillin	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	17	17	16
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	0.5	0.4	0.4
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	<LOD	0.002	<LOD
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	0.1	0.1	0.1
Linuron	<LOD	<LOD	<LOD
Metazachlor	<LOD	<LOD	<LOD
Metolachlor	0.0	0.0	0.1
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	0.8	0.7	0.6
Terbutryn	<LOD	<LOD	<LOD

N.A.: Not Available

Table 55. *Polar Compounds concentrations in open sea samples*

Lab. Code:	19-166	19-167	19-168
Sample name:	JBSS_GE_UA-6	JBSS_GE_UA-7	JBSS_GE_UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.80	19.15	19.15
Sampling period:	31/07/2019	01/08/2019	01/08/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
Bezafibrate	<LOD	<LOD	<LOD
Dicamba	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD
E1	3.8	0.8	4.2
E2	<LOD	<LOD	1.3
EE2	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	1.5	1.7	1.4
Acetamiprid	0.05	0.05	0.06
Amoxicillin	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD
Benzotriazole	18	19	18
Bromacil	<LOD	<LOD	<LOD
Carbamazepine	0.3	0.4	0.4
Chloroxuron	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD
Clarithromycin	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD
Dimethenamid	<LOD	<LOD	<LOD
Fipronil	<LOD	<LOD	<LOD
Imidacloprid	<LOD	0.1	<LOD
Linuron	<LOD	<LOD	<LOD
Metazachlor	<LOD	<LOD	<LOD
Metolachlor	0.1	0.1	0.1
Sulfamethoxazole	<LOD	<LOD	<LOD
Tebuconazole	0.7	0.6	0.6
Terbutryn	<LOD	<LOD	<LOD
N.A.: Not Available			

Table 56. *Polar Compounds concentrations in open sea samples*

Lab. Code:	19-169	19-170	19-171	19-172
Sample name:	JBSS_GE_UA-9	JBSS_GE_UA-10	JBSS_GE_UA-11	JBSS_GE_UA-12
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.45	18.90	19.85	19.22
Sampling period:	02/08/2019	03/08/2019	03/08/2019	03/08/2019
Concentration	**ng/L**	**ng/L**	**ng/L**	**ng/L**
Bezafibrate	0.2	<LOD	0.3	<LOD
Dicamba	<LOD	<LOD	<LOD	<LOD
Diclofenac	<LOD	<LOD	<LOD	<LOD
E1	1.6	2.3	2.0	1.7
E2	0.7	1.0	1.0	0.8
EE2	<LOD	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD	<LOD
Metaflumizone	<LOD	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD	<LOD
10,11-dihydro-10,11-dihydroxy-carbamazepine	1.3	1.6	1.8	1.6
Acetamiprid	0.06	0.12	0.06	0.11
Amoxicillin	N.A	N.A	N.A	N.A
Azithromycin	<LOD	<LOD	<LOD	<LOD
Benzotriazole	12	16	16	15
Bromacil	<LOD	<LOD	<LOD	<LOD
Carbamazepine	0.3	0.4	0.5	0.4
Chloroxuron	<LOD	<LOD	<LOD	<LOD
Ciprofloxacin	<LOD	<LOD	<LOD	<LOD
Clarithromycin	<LOD	<LOD	<LOD	<LOD
Diazinon	<LOD	<LOD	<LOD	<LOD
Dimethenamid	<LOD	0.003	0.003	0.002
Fipronil	<LOD	<LOD	<LOD	<LOD
Imidacloprid	<LOD	<LOD	<LOD	<LOD
Linuron	<LOD	<LOD	<LOD	<LOD
Metazachlor	<LOD	<LOD	<LOD	<LOD
Metolachlor	0.1	0.2	0.4	0.3
Sulfamethoxazole	<LOD	<LOD	<LOD	<LOD
Tebuconazole	0.6	0.6	0.6	0.6
Terbutryn	<LOD	<LOD	<LOD	<LOD

N.A.: Not Available

6.1.2. Semi-polar and Apolar Compounds

6.1.2.1. *Organophosphate Compounds (OPCs)*

In Tables from 57 to 64 the results of Organophosphate Compounds obtained with 20L spot samples are reported.

Table 57. *Organoposphate Compounds (OPCs) concentrations in blank samples*

Lab. Code:	OPC-LBLK-240919	OPC-LBLK-250919	OPC-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	JBSS_Field Blank
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2020	25/09/2020	28/07-08/08/2019
Analysis date:	10/30/2019	10/30/2019	10/30/2019
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.10	7.98
TNPP	<LOD	<LOD	<LOD
TIBP	0.79	1.76	81.31
TNBP	0.07	0.10	6.01
TCEP	<LOD	0.08	0.89
TCPP	<LOD	0.25	11.21
TDCPP	0.09	0.20	0.63
TBOEP	<LOD	<LOD	2.71
TPhP	<LOD	<LOD	0.30
EHDP	0.02	0.04	61.54
TEHP	0.0027	0.0035	3.24
TMPP	<LOD	<LOD	<LOD
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 58. *Organophosphate Compounds (OPCs) concentrations in samples from the coast of Ukraine and from outside the Danube delta (JBSS_GE_UA-1A)*

Lab. Code:	OPC-EMB-19-155	OPC-EMB-19-156	OPC-EMB-19-157-C
Sample name:	JBSS_GE_UA-1A	JBSS_GE_UA-2A	JBSS_GE_UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Analysis date:	10/30/2019	10/30/2019	10/31/2019
Concentration	ng/L	ng/L	ng/L
TEP	18	10	8.53
TNPP	<LOD	<LOD	<LOD
TIBP	4.56	52.99	4.09
TNBP	1.07	2.53	0.57
TCEP	6.04	11	4.63
TCPP	26	37	10
TDCPP	2.12	14	2.25
TBOEP	0.75	2.14	0.56
TPhP	0.29	1.75	0.34
EHDP	0.59	3.90	0.38
TEHP	0.17	0.50	0.11
TMPP	0.15	0.06	0.10
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 59. *Organophosphate Compounds (OPCs) concentrations* in samples from the coast of Georgia, provided by Georgian partners

Lab. Code:	OPC-EMB-19-176	OPC-EMB-19-177	OPC-EMB-19-178
Sample name:	JBSS_GE-1	JBSS_GE-2	JBSS_GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	07/08/2019	07/08/2019	07/08/2019
Analysis date:	10/31/2019	10/31/2019	10/31/2019
Concentration	ng/L	ng/L	ng/L
TEP	7.18	16	7.07
TNPP	<LOD	<LOD	<LOD
TIBP	5.50	78	8.17
TNBP	2.58	4.91	3.29
TCEP	4.86	14	4.96
TCPP	25	53	12
TDCPP	3.42	10	3.53
TBOEP	1.52	27	<LOD
TPhP	1.73	3.38	0.77
EHDP	4.94	6.54	0.70
TEHP	1.02	7.30	0.31
TMPP	0.11	0.21	0.13
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 60. *Organophosphate Compounds (OPCs)* concentrations in samples from the coast of Ukraine, provided by Ukrainian partners

Lab. Code:	OPC-EMB-19-173	OPC-EMB-19-174	OPC-EMB-19-175
Sample name:	JBSS_UA-10	JBSS_UA-11	JBSS_UA-15
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	19.30	19.55
Sampling period:	04/08/2019	04/08/2019	04/08/2019
Analysis date:	10/31/2019	10/31/2019	10/31/2019
Concentration	ng/L	ng/L	ng/L
TEP	7.73	8.89	7.60
TNPP	<LOD	<LOD	<LOD
TIBP	5.95	4.73	15
TNBP	0.71	1.13	3.09
TCEP	4.42	4.80	10
TCPP	30	21	21
TDCPP	2.41	2.43	7.24
TBOEP	2.21	<LOD	<LOD
TPhP	1.93	1.43	1.50
EHDP	1.68	1.07	3.01
TEHP	1.23	0.71	1.02
TMPP	0.12	0.07	0.07
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 61. *Organophosphate Compounds (OPCs) concentrations in open sea samples*

Lab. Code:	OPC-EMB-19-158	OPC-EMB-19-160	OPC-EMB-19-162
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2	JBSS_GE_UA-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.30	18.05	18.89
Sampling period:	29/07/2019	29/07/2019	30/07/2019
Analysis date:	10/30/2019	10/30/2019	10/30/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
TEP	9.53	9.78	8.75
TNPP	**<LOD**	**<LOD**	**<LOD**
TIBP	4.15	3.01	6.88
TNBP	0.81	0.80	0.56
TCEP	6.00	5.79	4.08
TCPP	18	20	7.39
TDCPP	1.80	1.40	1.72
TBOEP	1.02	0.98	0.45
TPhP	0.22	0.33	0.16
EHDP	0.33	0.22	0.22
TEHP	0.07	0.08	0.06
TMPP	0.05	0.08	**<LOD**
TIPPP	**<LOD**	**<LOD**	**<LOD**
T35DMPP	**<LOD**	**<LOD**	**<LOD**

Table 62. *Organophosphate Compounds (OPCs) concentrations in open sea samples*

Lab. Code:	OPC-EMB-19-163	OPC-EMB-19-164	OPC-EMB-19-165
Sample name:	JBSS_GE_UA-3	JBSS_GE_UA-4	JBSS_GE_UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.35	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	31/07/2019
Analysis date:	10/31/2019	10/31/2019	10/31/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
TEP	9.24	10	7.87
TNPP	**<LOD**	**<LOD**	**<LOD**
TIBP	4.70	8.46	9.70
TNBP	0.35	0.55	0.68
TCEP	4.17	4.38	4.67
TCPP	9.52	8.85	7.89
TDCPP	0.96	3.23	3.42
TBOEP	**<LOD**	**<LOD**	**<LOD**
TPhP	0.16	0.21	0.32
EHDP	0.20	0.49	0.37
TEHP	0.05	0.11	0.10
TMPP	**<LOD**	**<LOD**	**<LOD**
TIPPP	**<LOD**	**<LOD**	**<LOD**
T35DMPP	**<LOD**	**<LOD**	**<LOD**

Table 63. *Organophosphate Compounds (OPCs) concentrations in open sea samples*

Lab. Code:	OPC-EMB-19-166	OPC-EMB-19-167	OPC-EMB-19-168
Sample name:	JBSS_GE_UA-6	JBSS_GE_UA-7	JBSS_GE_UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.80	19.15	19.15
Sampling period:	31/07/2019	01/08/2019	01/08/2019
Analysis date:	10/31/2019	10/31/2019	10/31/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
TEP	8.99	18	8.57
TNPP	**<LOD**	**<LOD**	**<LOD**
TIBP	7.67	34	4.22
TNBP	0.38	1.97	0.31
TCEP	4.22	7.02	3.73
TCPP	6.67	22	9.72
TDCPP	1.82	8.99	1.02
TBOEP	**<LOD**	**<LOD**	0.57
TPhP	0.27	0.83	0.17
EHDP	0.30	1.39	0.14
TEHP	0.14	0.36	0.04
TMPP	**<LOD**	0.07	0.04
TIPPP	**<LOD**	**<LOD**	**<LOD**
T35DMPP	**<LOD**	**<LOD**	**<LOD**

Table 64. *Organophosphate Compounds (OPCs) concentrations in open sea samples*

Lab. Code:	OPC-EMB-19-169	OPC-EMB-19-170	OPC-EMB-19-171	OPC-EMB-19-172
Sample name:	JBSS_GE_UA-9	JBSS_GE_UA-10	JBSS_GE_UA-11	JBSS_GE_UA-12
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.45	18.90	19.85	19.22
Sampling period:	02/08/2019	03/08/2019	03/08/2019	03/08/2019
Analysis date:	10/31/2019	10/31/2019	10/31/2019	10/31/2019
Concentration	**ng/L**	**ng/L**	**ng/L**	**ng/L**
TEP	7.14	9.01	9.02	8.91
TNPP	**<LOD**	**<LOD**	**<LOD**	**<LOD**
TIBP	6.30	3.24	1.78	4.46
TNBP	0.58	0.46	0.47	0.32
TCEP	3.07	4.10	3.56	4.55
TCPP	8.58	7.87	8.75	10
TDCPP	1.61	0.83	0.92	1.56
TBOEP	1.38	**<LOD**	0.44	**<LOD**
TPhP	0.24	0.19	0.11	0.24
EHDP	0.29	0.23	0.20	0.24
TEHP	0.09	0.03	0.04	0.04
TMPP	0.06	0.05	**<LOD**	**<LOD**
TIPPP	**<LOD**	**<LOD**	**<LOD**	**<LOD**
T35DMPP	**<LOD**	**<LOD**	**<LOD**	**<LOD**

6.1.2.2. *Chlorinated Pesticides*

In the Tables from 65 to 72 the results of Chlorinated Pesticides obtained with 20L spot samples are reported.

Table 65. *Chlorinated Pesticides concentrations in blank samples*

Lab. Code:	OCP-LBLK-240919	OCP-LBLK-250919	OCP-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	JBSS_Field Blank
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2019	25/09/2019	28/07-08/08/2019
Analysis date:	12/2/2019	12/2/2019	12/2/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	0.30	0.51	34
HCB	0.31	0.78	30
a-HCH	**<LOD**	**<LOD**	0.80
b-HCH	**<LOD**	**<LOD**	2.44
g-HCH	**<LOD**	**<LOD**	5.03
d-HCH	**<LOD**	**<LOD**	**<LOD**
e-HCH	**<LOD**	**<LOD**	**<LOD**
Sum-HCHs	**---**	**---**	8.27
Heptachlor	**<LOD**	**<LOD**	2.12
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	**---**	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	**<LOD**	**<LOD**	**<LOD**
Endrin	**<LOD**	**<LOD**	2.58
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	**---**	**---**	2.58
trans-chlordane	**<LOD**	**<LOD**	0.85
cis-chlordane	**<LOD**	**<LOD**	0.63
Sum-Chlordane	**---**	**---**	1.48
Oxychlordane	**<LOD**	**<LOD**	**<LOD**

Lab. Code:	OCP-LBLK-240919	OCP-LBLK-250919	OCP-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	JBSS_Field Blank
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2019	25/09/2019	28/07-08/08/2019
Analysis date:	12/2/2019	12/2/2019	12/2/2019
Concentration	pg/L	pg/L	pg/L
trans-nonachlor	<LOD	<LOD	0.29
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	0.29
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	<LOD	1.71
Sum-Endosulfanes	---	---	1.71
Endosulfane-sulphate	<LOD	<LOD	1.41
op-DDE	<LOD	<LOD	0.87
pp-DDE	<LOD	<LOD	13
op-DDD	<LOD	<LOD	<LOD
pp-DDD	<LOD	<LOD	2.99
op-DDT	<LOD	<LOD	<LOD
pp-DDT	<LOD	<LOD	19
Sum-DDTtotal	---	---	36
Methoxychlor	<LOD	<LOD	<LOD
Mirex	0.06	<LOD	0.18
Others;			
Trifluralin	<LOD	<LOD	55
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	<LOD	<LOD	14
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	<LOD	1.69
HCBD and Dichlorvos are not reported because very low recovery			

Table 66. Chlorinated Pesticides concentrations in samples from the coast of Ukraine and from outside the Danube delta (JBSS_GE_UA-1A)

Lab. Code:	OCP-EMB-19-155	OCP-EMB-19-156	OCP-EMB-19-157
Sample name:	JBSS_GE_UA-1A	JBSS_GE_UA-2A	JBSS_GE_UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Analysis date:	12/3/2019	12/2/2019	12/2/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	11	38	11
HCB	15	36	12
a-HCH	81	73	64
b-HCH	1218	824	1380
g-HCH	43	39	37
d-HCH	21	16	**<LOD**
e-HCH	24	24	**<LOD**
Sum-HCHs	1388	975	1481
Heptachlor	6.35	**<LOD**	**<LOD**
Heptachlor-exo-epoxide	5.43	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	5.43	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	13	6.76	23
Endrin	7.27	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	20	6.76	23
trans-chlordane	8.46	**<LOD**	**<LOD**
cis-chlordane	8.91	**<LOD**	**<LOD**
Sum-Chlordane	17	**---**	**---**
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	12	**<LOD**	**<LOD**
cis-nonachlor	11	**<LOD**	**<LOD**
Sum-nonachlor	23	**---**	**---**

Lab. Code:	OCP-EMB-19-155	OCP-EMB-19-156	OCP-EMB-19-157
Sample name:	JBSS_GE_UA-1A	JBSS_GE_UA-2A	JBSS_GE_UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Analysis date:	12/3/2019	12/2/2019	12/2/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	4.30	7.50	<LOD
op-DDE	6.62	<LOD	2.13
pp-DDE	12	10	19
op-DDD	7.04	<LOD	<LOD
pp-DDD	16	22	21
op-DDT	8.22	<LOD	<LOD
pp-DDT	14	4.54	<LOD
Sum-DDTtotal	65	37	43
Methoxychlor	<LOD	<LOD	<LOD
Mirex	25.29	<LOD	<LOD
Others;			
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	84	574	191
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	9.36	21	<LOD
HCBD and Dichlorvos are not reported because very low recovery			

Table 67. *Chlorinated Pesticides concentrations in samples from the coast of Georgia,provided by Georgian partners*

Lab. Code:	OCP-EMB-19-176	OCP-EMB-19-177	OCP-EMB-19-178
Sample name:	JBSS_GE-1	JBSS_GE-2	JBSS_GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	07/08/2019	07/08/2019	07/08/2019
Analysis date:	12/5/2019	12/5/2019	12/5/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	43	386	11
HCB	56	98	24
a-HCH	140	30	55
b-HCH	1425	472	1497
g-HCH	35	46	19
d-HCH	**<LOD**	**<LOD**	**<LOD**
e-HCH	**<LOD**	**<LOD**	**<LOD**
Sum-HCHs	1600	548	1571
Heptachlor	**<LOD**	**<LOD**	2.60
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	**---**	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	**<LOD**	**<LOD**	**<LOD**
Endrin	**<LOD**	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	---	---	---
trans-chlordane	**<LOD**	8.70	**<LOD**
cis-chlordane	**<LOD**	8.80	**<LOD**
Sum-Chlordane	**---**	17	**---**
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	**<LOD**	4.75	3.14
cis-nonachlor	**<LOD**	**<LOD**	**<LOD**
Sum-nonachlor	**---**	4.75	3.14

Lab. Code:	OCP-EMB-19-176	OCP-EMB-19-177	OCP-EMB-19-178
Sample name:	JBSS_GE-1	JBSS_GE-2	JBSS_GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	07/08/2019	07/08/2019	07/08/2019
Analysis date:	12/5/2019	12/5/2019	12/5/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	<LOD	17	5.14
op-DDE	<LOD	11	7.36
pp-DDE	103	67	55
op-DDD	27	24	53
pp-DDD	84	68	91
op-DDT	43	26	18
pp-DDT	126	96	181
Sum-DDTtotal	383	292	405
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	3.09	1.83
Others;			
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	416	236	21
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	<LOD	<LOD
HCBD and Dichlorvos are not reported because very low recovery			

Table 68. Chlorinated Pesticides concentrations in samples from the coast of Ukraine,provided by Ukrainian partners

Lab. Code:	OCP-EMB-19-173	OCP-EMB-19-174	OCP-EMB-19-175
Sample name:	JBSS_UA-10	JBSS_UA-11	JBSS_UA-15
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	19.30	19.55
Sampling period:	04/08/2019	04/08/2019	04/08/2019
Analysis date:	12/4/2019	12/4/2019	12/4/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	9.26	254	112
HCB	13	26	52
a-HCH	98	59	65
b-HCH	848	1700	907
g-HCH	33	23	28
d-HCH	**<LOD**	**<LOD**	**<LOD**
e-HCH	**<LOD**	**<LOD**	**<LOD**
Sum-HCHs	978	1782	1000
Heptachlor	**<LOD**	**<LOD**	**<LOD**
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	**---**	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	9.49	7.88	**<LOD**
Endrin	**<LOD**	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	9.49	7.88	---
trans-chlordane	**<LOD**	**<LOD**	**<LOD**
cis-chlordane	**<LOD**	**<LOD**	**<LOD**
Sum-Chlordane	**---**	**---**	**---**
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	**<LOD**	**<LOD**	**<LOD**
cis-nonachlor	**<LOD**	**<LOD**	**<LOD**
Sum-nonachlor	**---**	**---**	**---**

Lab. Code:	OCP-EMB-19-173	OCP-EMB-19-174	OCP-EMB-19-175
Sample name:	JBSS_UA-10	JBSS_UA-11	JBSS_UA-15
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	19.30	19.55
Sampling period:	04/08/2019	04/08/2019	04/08/2019
Analysis date:	12/4/2019	12/4/2019	12/4/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	3.68	<LOD	<LOD
op-DDE	7	4.20	9.88
pp-DDE	39	43	37
op-DDD	5.56	4.79	<LOD
pp-DDD	24	23	<LOD
op-DDT	<LOD	<LOD	<LOD
pp-DDT	34	31	<LOD
Sum-DDTtotal	110	105	46
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	2.67
Others;			
Trifluralin	1.70	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	112	98	285
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	<LOD	<LOD
HCBD and Dichlorvos are not reported because very low recovery			

Table 69. *Chlorinated Pesticides concentrations in open sea samples*

Lab. Code:	OCP-EMB-19-158	OCP-EMB-19-160	OCP-EMB-19-162
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2	JBSS_GE_UA-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.30	18.05	18.89
Sampling period:	29/07/2019	29/07/2019	30/07/2019
Analysis date:	12/2/2019	12/2/2019	12/3/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	14	17	15
HCB	11	8.18	14
a-HCH	81	84	67
b-HCH	1464	1885	1910
g-HCH	46	45	39
d-HCH	6.54	6.88	**<LOD**
e-HCH	4.37	**<LOD**	**<LOD**
Sum-HCHs	1602	2021	2016
Heptachlor	**<LOD**	**<LOD**	**<LOD**
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	**---**	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	9.23	10	**<LOD**
Endrin	**<LOD**	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	9.23	10	---
trans-chlordane	**<LOD**	**<LOD**	**<LOD**
cis-chlordane	**<LOD**	**<LOD**	**<LOD**
Sum-Chlordane	**---**	**---**	**---**
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	**<LOD**	**<LOD**	**<LOD**
cis-nonachlor	**<LOD**	**<LOD**	**<LOD**
Sum-nonachlor	**---**	**---**	**---**
Endosulfane-alpha	**<LOD**	**<LOD**	**<LOD**

Lab. Code:	OCP-EMB-19-158	OCP-EMB-19-160	OCP-EMB-19-162
Sample name:	JBSS_GE_UA-1_1	JBSS_GE_UA-1_2	JBSS_GE_UA-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.30	18.05	18.89
Sampling period:	29/07/2019	29/07/2019	30/07/2019
Analysis date:	12/2/2019	12/2/2019	12/3/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	2.17	2.33	<LOD
op-DDE	<LOD	<LOD	<LOD
pp-DDE	16	12	6.62
op-DDD	9	7.46	<LOD
pp-DDD	21	21	8.42
op-DDT	<LOD	<LOD	<LOD
pp-DDT	20	6.66	<LOD
Sum-DDTtotal	66	47	15
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	4.37	1.20
Others;			
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	246	257	118
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	3.86	3.00	<LOD
HCBD and Dichlorvos are not reported because very low recovery			

Table 70. *Chlorinated Pesticides concentrations in open sea samples*

Lab. Code:	OCP-EMB-19-163	OCP-EMB-19-164	OCP-EMB-19-165
Sample name:	JBSS_GE_UA-3	JBSS_GE_UA-4	JBSS_GE_UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.35	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	31/07/2019
Analysis date:	12/2/2019	12/3/2019	12/2/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	10	22	11
HCB	10	13	17
a-HCH	60	61	55
b-HCH	1608	1936	2416
g-HCH	31	23	49
d-HCH	**<LOD**	**<LOD**	**<LOD**
e-HCH	**<LOD**	**<LOD**	**<LOD**
Sum-HCHs	1699	2019	2520
Heptachlor	**<LOD**	**<LOD**	**<LOD**
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	**---**	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	**<LOD**	14	12
Endrin	**<LOD**	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	---	14	12
trans-chlordane	**<LOD**	**<LOD**	**<LOD**
cis-chlordane	**<LOD**	**<LOD**	**<LOD**
Sum-Chlordane	**---**	**---**	**---**
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	**<LOD**	**<LOD**	**<LOD**
cis-nonachlor	**<LOD**	**<LOD**	**<LOD**
Sum-nonachlor	**---**	**---**	**---**
Endosulfane-alpha	**<LOD**	**<LOD**	**<LOD**

Lab. Code:	OCP-EMB-19-163	OCP-EMB-19-164	OCP-EMB-19-165
Sample name:	JBSS_GE_UA-3	JBSS_GE_UA-4	JBSS_GE_UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.35	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	31/07/2019
Analysis date:	12/2/2019	12/3/2019	12/2/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	<LOD	<LOD	<LOD
op-DDE	<LOD	<LOD	0.88
pp-DDE	4.56	5.00	7.81
op-DDD	<LOD	<LOD	<LOD
pp-DDD	<LOD	<LOD	<LOD
op-DDT	<LOD	<LOD	<LOD
pp-DDT	<LOD	<LOD	<LOD
Sum-DDTtotal	4.56	5.00	8.69
Methoxychlor	<LOD	<LOD	<LOD
Mirex	1.73	<LOD	<LOD
Others;			
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	227	141	42
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	<LOD	<LOD
HCBD and Dichlorvos are not reported because very low recovery			

Table 71. *Chlorinated Pesticides concentrations in open sea samples*

Lab. Code:	OCP-EMB-19-166	OCP-EMB-19-167	OCP-EMB-19-168
Sample name:	JBSS_GE_UA-6	JBSS_GE_UA-7	JBSS_GE_UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.80	19.15	19.15
Sampling period:	31/07/2019	01/08/2019	01/08/2019
Analysis date:	12/2/2019	12/2/2019	12/3/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	8.80	not recovered	12
HCB	9.42	not recovered	7.94
a-HCH	60	101	57
b-HCH	1591	4639	2596
g-HCH	38	22	34
d-HCH	**<LOD**	**<LOD**	**<LOD**
e-HCH	**<LOD**	**<LOD**	**<LOD**
Sum-HCHs	1689	4762	2687
Heptachlor	**<LOD**	**<LOD**	**<LOD**
Heptachlor-exo-epoxide	**<LOD**	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	**---**	**---**	**---**
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	6.51	21	11
Endrin	**<LOD**	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	6.51	21	11
trans-chlordane	**<LOD**	**<LOD**	**<LOD**
cis-chlordane	**<LOD**	**<LOD**	**<LOD**
Sum-Chlordane	**---**	**---**	**---**
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	2.45	2.30	**<LOD**
cis-nonachlor	**<LOD**	**<LOD**	**<LOD**
Sum-nonachlor	2.45	2.30	**---**
Endosulfane-alpha	**<LOD**	**<LOD**	**<LOD**

Lab. Code:	OCP-EMB-19-166	OCP-EMB-19-167	OCP-EMB-19-168
Sample name:	JBSS_GE_UA-6	JBSS_GE_UA-7	JBSS_GE_UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.80	19.15	19.15
Sampling period:	31/07/2019	01/08/2019	01/08/2019
Analysis date:	12/2/2019	12/2/2019	12/3/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	<LOD	<LOD	<LOD
op-DDE	2.11	<LOD	<LOD
pp-DDE	9.02	8.33	4.48
op-DDD	<LOD	<LOD	<LOD
pp-DDD	<LOD	<LOD	2.62
op-DDT	<LOD	<LOD	<LOD
pp-DDT	<LOD	<LOD	<LOD
Sum-DDTtotal	11	8.33	7.11
Methoxychlor	<LOD	<LOD	<LOD
Mirex	1.00	1.07	1.21
Others;			
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	28	89	29
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	<LOD	<LOD
HCBD and Dichlorvos are not reported because very low recovery			

Table 72. Chlorinated *Pesticides concentrations in open sea samples*

Lab. Code:	OCP-EMB-19-169	OCP-EMB-19-170	OCP-EMB-19-171	OCP-EMB-19-172
Sample name:	JBSS_GE_UA-9	JBSS_GE_UA-10	JBSS_GE_UA-11	JBSS_GE_UA-12
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.45	18.90	19.85	19.22
Sampling period:	02/08/2019	03/08/2019	03/08/2019	03/08/2019
Analysis date:	12/3/2019	12/3/2019	12/4/2019	12/4/2019
Concentration	pg/L	pg/L	pg/L	pg/L
PeCBz	8.93	6.36	82	4.80
HCB	10	6.56	9.58	5.48
a-HCH	44	50	48	48
b-HCH	1832	1899	2188	1434
g-HCH	49	38	21	25
d-HCH	<LOD	<LOD	<LOD	<LOD
e-HCH	<LOD	<LOD	<LOD	<LOD
Sum-HCHs	1925	1987	2257	1507
Heptachlor	<LOD	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	<LOD	<LOD	<LOD
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD	<LOD
Sum-Heptachlorepoxides	---	---	---	---
Aldrin	<LOD	<LOD	<LOD	<LOD
Dieldrin	<LOD	<LOD	<LOD	10.32
Endrin	<LOD	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD	<LOD
Sum-Drins	---	---	---	10
trans-chlordane	<LOD	<LOD	<LOD	<LOD
cis-chlordane	<LOD	<LOD	<LOD	<LOD
Sum-Chlordane	---	---	---	---
Oxychlordane	<LOD	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD	<LOD

Lab. Code:	OCP-EMB-19-169	OCP-EMB-19-170	OCP-EMB-19-171	OCP-EMB-19-172
Sample name:	JBSS_GE_UA-9	JBSS_GE_UA-10	JBSS_GE_UA-11	JBSS_GE_UA-12
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.45	18.90	19.85	19.22
Sampling period:	02/08/2019	03/08/2019	03/08/2019	03/08/2019
Analysis date:	12/3/2019	12/3/2019	12/4/2019	12/4/2019
Concentration	pg/L	pg/L	pg/L	pg/L
Endosulfane-beta	<LOD	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---	---
Endosulfane-sulphate	<LOD	<LOD	<LOD	<LOD
op-DDE	<LOD	<LOD	<LOD	<LOD
pp-DDE	6.28	4.21	2.73	5.90
op-DDD	<LOD	<LOD	<LOD	<LOD
pp-DDD	<LOD	4.47	2.31	2.31
op-DDT	<LOD	<LOD	<LOD	<LOD
pp-DDT	<LOD	<LOD	<LOD	<LOD
Sum-DDTtotal	6.28	8.68	5.05	8.21
Methoxychlor	<LOD	<LOD	<LOD	<LOD
Mirex	1.38	1.42	<LOD	<LOD
Others;				
Trifluralin	<LOD	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD	<LOD
Chlorpyriphos	26	19	17	30
Chlorfenvinphos	<LOD	<LOD	<LOD	<LOD
Dicofol	Not stable	Not stable	Not stable	Not stable
Cypermethrins	<LOD	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	<LOD	10.09	<LOD
HCBD and Dichlorvos are not reported because of very low recovery rates				

6.1.2.3. *Triazine Pesticides*

In the tables 73-80 the results of Triazine Pesticides obtained with 20L spot samples are reported.

Table 73. *Triazine Pesticides concentrations in blank samples*

Lab. Code:	TRIAZ-LBLK-240919	TRIAZ-LBLK-250919	TRIAZ-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	FIELD BLANK
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2020	25/09/2020	28/07-08/08/2019
Analysis date:	11/11/2019	11/11/2019	11/11/2019
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	<LOD	<LOD
Atrazine	<LOD	<LOD	<LOD
Terbuthylazine	<LOD	<LOD	0.050
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	<LOD	<LOD
Desethyl-Terbuthylazine	<LOD	<LOD	0.020

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 74. Triazine Pesticides concentrations in samples from the coast of Ukraine and from outside the Danube delta (JBSS_GE_UA-1A)

Lab. Code:	TRIAZ-EMB-19-155	TRIAZ-EMB-19-156	TRIAZ-EMB-19-157
Sample name:	JBSS-GE-UA-1A	JBSS-GE-UA-2A	JBSS-GE-UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Analysis date:	11/11/2019	11/11/2019	11/11/2019
Concentration	ng/L	ng/L	ng/L
Simazine	4.14	0.41	13
Atrazine	39	7.78	33
Terbuthylazine	13	9.40	4.99
Desethyl-Simazine	0.82	2.49	0.96
Desethyl-Atrazine	1.73	0.67	1.84
Desethyl-Terbuthylazine	2.75	1.24	1.43

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 75. *Triazine Pesticides concentrations in samples from Georgia Coast provided by Georgian partners*

Lab. Code:	TRIAZ-EMB-19-176	TRIAZ-EMB-19-177	TRIAZ-EMB-19-178-B
Sample name:	JBSS-GE-1	JBSS_GE-2	JBSS-GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	07/08/2020	07/08/2019	07/08/2020
Analysis date:	11/12/2019	11/12/2019	11/12/2019
Concentration	ng/L	ng/L	ng/L
Simazine	10	17	8.20
Atrazine	33	41	32
Terbuthylazine	1.01	1.83	1.26
Desethyl-Simazine	0.34	1.61	1.20
Desethyl-Atrazine	0.59	2.96	1.78
Desethyl-Terbuthylazine	0.40	2.73	0.82

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 76. *Triazine Pesticides concentrations in samples from the coast of Ukraine, provided by Ukrainian partners*

Lab. Code:	TRIAZ-EMB-19-173	TRIAZ-EMB-19-174	TRIAZ-EMB-19-175-B
Sample name:	**JBSS-UA-10**	**JBSS-UA-11**	**JBSS-UA-15**
Type of sample:	**MB Black Sea water**	**MB Black Sea water**	**MB Black Sea water**
Volume sampled (L):	**20.00**	**19.30**	**19.55**
Sampling period:	**04/08/2020**	**04/08/2020**	**04/08/2020**
Analysis date:	**11/12/2019**	**11/12/2019**	**11/12/2019**
Concentration	ng/L	ng/L	ng/L
Simazine	11	9.48	3.40
Atrazine	42	41	19
Terbuthylazine	4.56	5.38	5.03
Desethyl-Simazine	0.12	0.12	0.30
Desethyl-Atrazine	0.44	0.58	0.71
Desethyl-Terbuthylazine	0.69	1.57	2.05

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 77. *Triazine Pesticides concentrations in open sea samples*

Lab. Code:	TRIAZ-EMB-19-158	TRIAZ-EMB-19-160	TRIAZ-EMB-19-162-B
Sample name:	**JBSS-GE-UA-1-1 REP**	**JBSS-GE-UA-1-2 REP**	**JBSS-GE-UA-2**
Type of sample:	**MB Black Sea water**	**MB Black Sea water**	**MB Black Sea water**
Volume sampled (L):	**18.30**	**18.05**	**18.89**
Sampling period:	**28/07/2019**	**28/07/2019**	**30/07/2019**
Analysis date:	**11/11/2019**	**11/11/2019**	**11/12/2019**
Concentration	ng/L	ng/L	ng/L
Simazine	8.28	8.85	15
Atrazine	47	47	52
Terbuthylazine	9.49	9.86	1.70
Desethyl-Simazine	0.50	0.66	0.85
Desethyl-Atrazine	1.08	1.08	1.98
Desethyl-Terbuthylazine	1.88	2.35	1.26

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 78. *Triazine Pesticides concentrations in open sea samples*

Lab. Code:	TRIAZ-EMB-19-163	TRIAZ-EMB-19-164	TRIAZ-EMB-19-165
Sample name:	JBSS-GE-UA-3	JBSS-GE-UA-4	JBSS-GE-UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.89	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	31/07/2019
Analysis date:	11/11/2019	11/11/2019	11/11/2019
Concentration	ng/L	ng/L	ng/L
Simazine	9.10	14	7.68
Atrazine	38	53	76
Terbuthylazine	1.90	1.56	1.53
Desethyl-Simazine	0.21	0.17	0.47
Desethyl-Atrazine	0.77	0.88	1.07
Desethyl-Terbuthylazine	0.49	0.42	0.44

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 79. *Triazine Pesticides concentrations in open sea samples*

Lab. Code:	TRIAZ-EMB-19-166	TRIAZ-EMB-19-167	TRIAZ-EMB-19-168
Sample name:	JBSS-GE-UA-6	JBSS-GE-UA-7	JBSS-GE-UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.80	19.15	19.15
Sampling period:	31/07/2019	01/08/2020	01/08/2020
Analysis date:	11/11/2019	11/11/2019	11/11/2019
Concentration	ng/L	ng/L	ng/L
Simazine	11	9.48	12
Atrazine	42	51	51
Terbuthylazine	1.66	1.30	1.58
Desethyl-Simazine	0.25	0.12	0.06
Desethyl-Atrazine	1.07	0.58	0.48
Desethyl-Terbuthylazine	0.47	0.24	0.34

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

Table 80. *Triazine Pesticides concentrations in open sea samples*

Lab. Code:	TRIAZ-EMB-19-169	TRIAZ-EMB-19-170	TRIAZ-EMB-19-171-B	TRIAZ-EMB-19-172-B
Sample name:	**JBSS-GE-UA-9**	**JBSS-GE-UA-10**	**JBSS-GE-UA-11**	**JBSS-GE-UA-12**
Type of sample:	**MB Black Sea water**	**MB Black Sea water**	**MB Black Sea water**	**MB Black Sea water**
Volume sampled (L):	**19.45**	**18.90**	**19.85**	**19.22**
Sampling period:	**02/08/2020**	**03/08/2020**	**03/08/2020**	**03/08/2020**
Analysis date:	**11/11/2019**	**11/12/2019**	**11/12/2019**	**11/12/2019**
Concentration	ng/L	ng/L	ng/L	ng/L
Simazine	11	11	15	17
Atrazine	31	39	36	23
Terbuthylazine	1.29	1.45	1.55	1.46
Desethyl-Simazine	0.26	0.10	0.69	0.48
Desethyl-Atrazine	0.47	0.88	2.33	1.17
Desethyl-Terbuthylazine	0.32	0.56	1.31	0.65

The triazines have been analysed both with HRGC-HRMS and UHPLC-MS. Here the data reported were obtained by HRGC-HRMS, only the results of JBSS_GE-2 sample were obtained by UHPLC-MS/MS due to a lot of interference in HRGC-HRMS.

6.1.2.4. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT*

In Tables 81-88 the results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT obtained with 20L spot samples are reported.

Table 81. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in blank samples*

Lab. Code:	PAH-LBLK-240919	PAH-LBLK-250919	PAH-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	FIELD BLANK
Type of sample:	Field Blank	Field Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2020	25/09/2020	28/07-08/08/2019
Analysis date:	11/21/2019	11/21/2019	11/21/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	59	96	4279
Anthracene	20	19	562
Fluoranthene	30	8	1599
Pyrene	**<LOD**	10	1294
Benz(a)anthracene	4	2	64
Chrysene	5	4	192
Sum Benzo(b,j,k)fluoranthene	5	9	219
Benzo(e)pyrene	**<LOD**	**<LOD**	136
Benzo(a)pyrene	**<LOD**	7	114
Perylene	**<LOD**	8	93
Indeno(123-cd)pyrene	**<LOD**	8	95
Benzo(ghi)perylene	**<LOD**	10	242
Dibenz(ah)anthracene	3	**<LOD**	75
Coronene	**<LOD**	**<LOD**	306
BHT	260	340	39957
EHMC	3431	63175	9674

Table 82. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in samples from Ukraine coast and from outside the Danube delta (JBSS_GE_UA-1A)*

Lab. Code:	PAH-EMB-19-155	PAH-EMB-19-156	PAH-EMB-19-157
Sample name:	JBSS-GE-UA-1A	JBSS-GE-UA-2A	JBSS-GE-UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Analysis date:	11/21/2019	11/21/2019	11/21/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1777	8135	2068
Anthracene	612	1916	659
Fluoranthene	1329	3324	1604
Pyrene	597	1875	824
Benz(a)anthracene	115	62	76
Chrysene	180	226	240
Sum Benzo(b,j,k)fluoranthene	328	211	259
Benzo(e)pyrene	171	96	157
Benzo(a)pyrene	167	272	103
Perylene	145	122	58
Indeno(123-cd)pyrene	177	37	83
Benzo(ghi)perylene	291	162	428
Dibenz(ah)anthracene	228	35	63
Coronene	186	98	319
BHT	16926	46803	7305
EHMC	26840	90879	25714

Table 83. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in samples from the coast of Georgia, provided by Georgian partners

Lab. Code:	PAH-EMB-19-176-B	PAH-EMB-19-177-B	PAH-EMB-19-178
Sample name:	JBSS-GE-1	JBSS-GE-2	JBSS-GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	04/08/2019	07/08/2019	07/08/2019
Analysis date:	11/22/2019	11/22/2019	11/22/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	8165	18548	3368
Anthracene	1264	5369	789
Fluoranthene	49490	10776	2009
Pyrene	12753	6611	1805
Benz(a)anthracene	1275	688	215
Chrysene	2311	1788	614
Sum Benzo(b,j,k)fluoranthene	3263	1754	1203
Benzo(e)pyrene	2249	1060	500
Benzo(a)pyrene	1958	927	567
Perylene	2102	1005	1703
Indeno(123-cd)pyrene	1985	768	388
Benzo(ghi)perylene	7476	1844	882
Dibenz(ah)anthracene	1630	704	394
Coronene	7297	1471	705
BHT	88499	42603	15797
EHMC	435740	1727207	338567

Table 84. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in samples from the coast of Ukraine, provided by Ukrainian partners

Lab. Code:	PAH-EMB-19-173	PAH-EMB-19-174	PAH-EMB-19-175
Sample name:	JBSS-UA-10	JBSS-UA-11	JBSS-UA-15
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	19.30	19.55
Sampling period:	04/08/2019	04/08/2019	04/08/2019
Analysis date:	11/22/2019	11/22/2019	11/22/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1975	3027	8427
Anthracene	359	660	1587
Fluoranthene	811	2917	5249
Pyrene	885	1938	2316
Benz(a)anthracene	111	291	102
Chrysene	325	602	255
Sum Benzo(b,j,k)fluoranthene	658	1169	594
Benzo(e)pyrene	169	550	233
Benzo(a)pyrene	202	671	251
Perylene	114	1276	173
Indeno(123-cd)pyrene	182	488	229
Benzo(ghi)perylene	401	1419	575
Dibenz(ah)anthracene	141	278	152
Coronene	**<LOD**	832	196
BHT	13188	40429	43940
EHMC	394300	248893	1408233

Table 85. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-EMB-19-158	PAH-EMB-19-160	PAH-EMB-19-162
Sample name:	JBSS-GE-UA-1-1	JBSS-GE-UA-1-2	JBSS-GE-UA-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.30	18.05	18.89
Sampling period:	29/07/2019	29/07/2019	30/07/2019
Analysis date:	11/21/2019	11/21/2019	11/21/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	2241	2745	2155
Anthracene	476	417	443
Fluoranthene	1050	685	804
Pyrene	636	467	668
Benz(a)anthracene	33	30	32
Chrysene	194	149	178
Sum Benzo(b,j,k)fluoranthene	93	126	67
Benzo(e)pyrene	53	42	39
Benzo(a)pyrene	69	39	16
Perylene	64	23	19
Indeno(123-cd)pyrene	23	27	22
Benzo(ghi)perylene	77	64	49
Dibenz(ah)anthracene	38	29	39
Coronene	58	50	**<LOD**
BHT	6222	7183	7255
EHMC	10095	10839	43144

Table 86. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-EMB-19-163	PAH-EMB-19-164	PAH-EMB-19-165
Sample name:	JBSS-GE-UA-3	JBSS-GE-UA-4	JBSS-GE-UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.35	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	31/07/2019
Analysis date:	11/21/2019	11/21/2019	11/21/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1457	2301	1898
Anthracene	239	356	316
Fluoranthene	980	1046	668
Pyrene	655	801	640
Benz(a)anthracene	38	19	30
Chrysene	161	213	138
Sum Benzo(b,j,k)fluoranthene	124	177	113
Benzo(e)pyrene	61	62	29
Benzo(a)pyrene	63	29	20
Perylene	13	12	13
Indeno(123-cd)pyrene	41	32	32
Benzo(ghi)perylene	217	132	80
Dibenz(ah)anthracene	35	42	**<LOD**
Coronene	306	506	**<LOD**
BHT	4292	5984	4851
EHMC	22960	47405	41392

Table 87. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-EMB-19-166	PAH-EMB-19-167-B	PAH-EMB-19-168
Sample name:	JBSS-GE-UA-6	JBSS-GE-UA-7	JBSS-GE-UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.80	19.15	19.15
Sampling period:	31/07/2019	01/08/2019	01/08/2019
Analysis date:	11/22/2019	11/22/2019	11/22/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1580	Interferences	1200
Anthracene	189	Interferences	169
Fluoranthene	1321	Interferences	1163
Pyrene	745	Interferences	837
Benz(a)anthracene	23	202	21
Chrysene	110	601	105
Sum Benzo(b,j,k)fluoranthene	150	700	106
Benzo(e)pyrene	82	359	126
Benzo(a)pyrene	47	527	46
Perylene	22	155	107
Indeno(123-cd)pyrene	48	302	39
Benzo(ghi)perylene	215	680	437
Dibenz(ah)anthracene	99	484	22
Coronene	327	1116	309
BHT	6044	439331	4843
EHMC	52367	200739	15976

Table 88. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-EMB-19-169	PAH-EMB-19-170	PAH-EMB-19-171-B	PAH-EMB-19-172
Sample name:	JBSS-GE-UA-9	JBSS-GE-UA-10	JBSS-GE-UA-11	JBSS-GE-UA-12
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.45	18.90	19.85	19.22
Sampling period:	02/08/2019	03/08/2019	03/08/2019	03/08/2019
Analysis date:	11/22/2019	11/22/2019	11/22/2019	11/22/2019
Concentration	pg/L	pg/L	pg/L	pg/L
Phenanthrene	1143	1125	1097	1014
Anthracene	191	172	391	180
Fluoranthene	612	686	549	505
Pyrene	506	457	418	424
Benz(a)anthracene	80	9	12	27
Chrysene	280	78	119	125
Sum Benzo(b,j,k)fluoranthene	165	75	60	105
Benzo(e)pyrene	43	38	29	28
Benzo(a)pyrene	40	17	15	16
Perylene	**<LOD**	5	6	13
Indeno(123-cd)pyrene	61	39	21	21
Benzo(ghi)perylene	113	109	62	55
Dibenz(ah)anthracene	**<LOD**	20	27	18
Coronene	**<LOD**	119	60	**<LOD**
BHT	3446	3810	2841	2617
EHMC	19887	183171	90011	92000

6.1.2.5. *Polychlorinated Biphenyls (EC-7 PCBs)*

In Tables 89-96 the results of EC-7 Polychlorinated Biphenyls obtained with 20L spot samples are reported.

Table 89. *EC-7 Polychlorinated Biphenyls concentrations in blank samples*

Lab. Code:	P-LBLK-240919	P-LBLK-250919	P-FBLK-19-179
Sample name:	Laboratory Blank	Laboratory Blank	FIELD BLANK
Type of sample:	Laboratory Blank	Laboratory Blank	Field Blank
Volume sampled (L):	20.00	20.00	19.28
Sampling period:	24/09/2020	25/09/2020	28/07-08/08/2019
Analysis date:	1/9/2020	1/9/2020	1/9/2020
Concentration	**pg/L**	**pg/L**	**pg/L**
EC-7			
PCB 28	**<LOD**	**<LOD**	8.89
PCB 52	**<LOD**	**<LOD**	2.82
PCB 101	**<LOD**	**<LOD**	2.60
PCB 118	**<LOD**	**<LOD**	2.03
PCB 138	**<LOD**	**<LOD**	3.68
PCB 153	**<LOD**	**<LOD**	2.39
PCB 180	**<LOD**	**<LOD**	1.34
Sum EC-7 PCBs	---	---	24

Table 90. *EC-7 Polychlorinated Biphenyls concentrations in samples from the coast of Ukraine and from outside the Danube delta (JBSS_GE_UA-1A)*

Lab. Code:	P-EMB-19-155	P-EMB-19-156	P-EMB-19-157
Sample name:	JBSS_GE_UA-1A	JBSS-GE-UA-2A	JBSS-GE-UA-3A
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.65	18.34	18.55
Sampling period:	28/07/2019	28/07/2019	28/07/2019
Analysis date:	1/9/2020	1/9/2020	1/9/2020
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	8.71	14	10
PCB 52	6.55	10	6.11
PCB 101	12	20	7.67
PCB 118	8.66	16	5.55
PCB 138	29	32	21
PCB 153	12	21	7.94
PCB 180	6.02	3.00	4.68
Sum EC-7 PCBs	84	116	63

Table 91. EC-7 Polychlorinated Biphenyls concentrations in samples from the coast of Georgia, provided by Georgian partners

Lab. Code:	P-EMB-19-176	P-EMB-19-177	P-EMB-19-178
Sample name:	JBSS-GE-1	JBSS-GE-2	JBSS-GE-4
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	20.05	19.47
Sampling period:	07/08/2019	07/08/2019	07/08/2019
Analysis date:	1/13/2020	1/13/2020	1/13/2020
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	30	115	27
PCB 52	23	24	14
PCB 101	45	28	15
PCB 118	31	28	19
PCB 138	92	59	28
PCB 153	101	23	17
PCB 180	54	9.18	4.90
Sum EC-7 PCBs	376	287	125

Table 92. EC-7 Polychlorinated Biphenyls concentrations in samples from the coast of Ukraine, provided by Ukrainian partners

Lab. Code:	P-EMB-19-173	P-EMB-19-174	P-EMB-19-175
Sample name:	JBSS-UA-10	JBSS-UA-11	JBSS-UA-15
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	19.30	19.55
Sampling period:	04/08/2019	04/08/2019	04/08/2019
Analysis date:	1/13/2020	1/13/2020	1/13/2020
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	7.85	14	32
PCB 52	7.33	7.54	16
PCB 101	10	10	24
PCB 118	16	11	22
PCB 138	18	18	65
PCB 153	11	9.87	21
PCB 180	4.87	4.01	8.37
Sum EC-7 PCBs	75	75	189

Table 93. *EC-7 Polychlorinated Biphenyls concentrations concentrations in open sea samples*

Lab. Code:	P-EMB-19-158	P-EMB-19-160	P-EMB-19-162
Sample name:	JBSS-GE-UA-1-1	JBSS-GE-UA-1-2	JBSS-GE-UA-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.30	18.05	18.89
Sampling period:	29/07/2019	29/07/2019	30/07/2019
Analysis date:	1/10/2020	1/10/2020	1/10/2020
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	8.64	7.35	7.53
PCB 52	5.75	3.01	5.23
PCB 101	8.26	3.80	7.18
PCB 118	3.96	3.16	4.89
PCB 138	15	10	21
PCB 153	6.39	5.17	9.80
PCB 180	1.95	2.95	3.27
Sum EC-7 PCBs	50	36	59

Table 94. *EC-7 Polychlorinated Biphenyls concentrations in open sea samples*

Lab. Code:	P-EMB-19-163	P-EMB-19-164	P-EMB-19-165
Sample name:	JBSS-GE-UA-3	JBSS-GE-UA-4	JBSS-GE-UA-5
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.35	18.15	18.55
Sampling period:	30/07/2019	30/07/2019	30/07/2019
Analysis date:	1/10/2020	1/10/2020	1/10/2020
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	5.48	6.11	6.59
PCB 52	3.67	4.12	<LOD
PCB 101	8.14	6.14	6.76
PCB 118	4.02	2.81	<LOD
PCB 138	20	9.12	15
PCB 153	17	4.35	<LOD
PCB 180	9.31	<LOD	<LOD
Sum EC-7 PCBs	68	33	28

Table 95. *EC-7 Polychlorinated Biphenyls concentrations in open sea samples*

Lab. Code:	P-EMB-19-166	P-EMB-19-167	P-EMB-19-168
Sample name:	JBSS-GE-UA-6	JBSS-GE-UA-7	JBSS-GE-UA-8
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	18.55	19.15	19.15
Sampling period:	31/07/2019	01/08/2019	01/08/2019
Analysis date:	1/10/2020	1/10/2020	1/10/2020
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	5.26	22	3.04
PCB 52	3.06	11	1.82
PCB 101	6.78	19	3.10
PCB 118	3.35	9.65	1.72
PCB 138	14	45	7.78
PCB 153	5.65	13	3.77
PCB 180	2.51	3.30	<LOD
Sum EC-7 PCBs	40	124	21

Table 96. *EC-7 Polychlorinated Biphenyls concentrations in open sea samples*

Lab. Code:	P-EMB-19-169	P-EMB-19-170	P-EMB-19-171	P-EMB-19-172
Sample name:	JBSS-GE-UA-9	JBSS-GE-UA-10	JBSS-GE-UA-11	JBSS-GE-UA-12
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.45	18.90	19.85	19.22
Sampling period:	02/08/2019	03/08/2019	03/08/2019	03/08/2019
Analysis date:	1/13/2020	1/13/2020	1/13/2020	1/13/2020
Concentration	**pg/L**	**pg/L**	**pg/L**	**pg/L**
EC-7				
PCB 28	7.51	4.13	3.56	5.12
PCB 52	2.22	2.43	1.81	1.59
PCB 101	**<LOD**	3.67	3.47	3.37
PCB 118	4.45	2.19	2.58	**<LOD**
PCB 138	9.32	8.95	7.68	11
PCB 153	4.91	4.01	2.71	4.78
PCB 180	**<LOD**	**<LOD**	**<LOD**	**<LOD**
Sum EC-7 PCBs	28	25	22	26

6.2. Individual results for Large Volume Transect Samples

6.2.1. Organophosphate Compounds (OPCs)

In the Tables 97-101 the results of Organophosphate Compounds on filters, primary cells and secondary cells (breakthrough control) obtained by Large Volume Transect sampling are reported.

Table 97. *Organophosphate Compounds in Field Blank samples*

Lab. Code:	OPC-EMB-LV-19-185	OPC-EMB-17-066-FB
Sample name:	Field Filter Blank	Field Blank Cell
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	300	300
Sampling period:	28/07-08/08/2020	28/07-08/08/2020
Analysis date:	11/4/2019	11/4/2019
Concentration	ng/L	ng/L
TEP	0.01	0.01
TNPP	<LOD	<LOD
TIBP	0.27	0.02
TNBP	0.03	0.004
TCEP	0.01	0.001
TCPP	0.38	0.03
TDCPP	0.05	0.01
TBOEP	<LOD	<LOD
TPhP	0.002	0.002
EHDP	0.003	0.004
TEHP	0.000	0.000
TMPP	0.0007	0.0002
TIPPP	0.0001	<LOD
T35DMPP	0.0004	<LOD

Table 98. *Results of Organophosphate Compounds in Transect 1 (JBSS_XL_LVE-1)*

Lab. Code:	OPC-EMB-19-181	OPC-EMB-19-196-B	OPC-EMB-19-197-B
Sample name:	JBSS_XL_LVE-Filter 1	JBSS_XL_LVE-1 Cell 1	JBSS_XL_LVE-1 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.27	0.17
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	2.02	1.08
TNBP	<LOD	0.34	0.11
TCEP	<LOD	0.20	0.05
TCPP	<LOD	5.82	1.16
TDCPP	<LOD	0.68	0.11
TBOEP	<LOD	0.05	0.03
TPhP	<LOD	0.01	0.01
EHDP	<LOD	0.02	0.01
TEHP	<LOD	0.0013	0.0008
TMPP	<LOD	0.0044	<LOD
TIPPP	<LOD	0.0004	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 99. *Results of Organophosphate Compounds in Transect 2 (JBSS_XL_LVE-2)*

Lab. Code:	OPC-EMB-19-182	OPC-EMB-19-198-B	OPC-EMB-19-199-B
Sample name:	JBSS_XL_LVE-Filter 2	JBSS_XL_LVE-2 Cell 1	JBSS_XL_LVE-2 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.22	0.04
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	1.95	0.58
TNBP	<LOD	0.22	0.05
TCEP	<LOD	0.13	0.03
TCPP	<LOD	2.61	1.04
TDCPP	<LOD	0.36	0.10
TBOEP	<LOD	0.06	<LOD
TPhP	<LOD	0.007	0.005
EHDP	<LOD	0.017	0.013
TEHP	<LOD	0.0014	0.0009
TMPP	<LOD	0.0063	<LOD
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 100. *Results of Organophosphate Compounds in Transect 3 (JBSS_XL_LVE-3)*

Lab. Code:	OPC-EMB-19-183	OPC-EMB-19-200	OPC-EMB-19-201-B
Sample name:	JBSS_XL_LVE-Filter 3	JBSS_XL_LVE-3 Cell 1	JBSS_XL_LVE-3 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Analysis date:	11/4/2019	6/16/2020	11/5/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
TEP	**<LOD**	0.20	0.14
TNPP	**<LOD**	**<LOD**	**<LOD**
TIBP	**<LOD**	1.21	0.64
TNBP	**<LOD**	0.18	0.06
TCEP	**<LOD**	0.20	0.06
TCPP	**<LOD**	2.33	1.11
TDCPP	**<LOD**	0.45	0.12
TBOEP	**<LOD**	**<LOD**	**<LOD**
TPhP	**<LOD**	0.01	0.01
EHDP	**<LOD**	0.02	0.01
TEHP	**<LOD**	0.001	0.001
TMPP	**<LOD**	0.0043	0.0008
TIPPP	**<LOD**	**<LOD**	**<LOD**
T35DMPP	**<LOD**	**<LOD**	**<LOD**

Table 101. *Results of Organophosphate Compounds in Transect 4 (JBSS_XL_LVE-4)*

Lab. Code:	OPC-EMB-19-184	OPC-EMB-19-202-D	OPC-EMB-19-203-B
Sample name:	JBSS_XL_LVE-Filter 4	JBSS_XL_LVE-4 Cell 1	JBSS_XL_LVE-4 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Analysis date:	11/4/2019	11/6/2019	11/5/2019
Concentration	**ng/L**	**ng/L**	**ng/L**
TEP	**<LOD**	0.18	0.16
TNPP	**<LOD**	**<LOD**	**<LOD**
TIBP	**<LOD**	1.89	0.32
TNBP	**<LOD**	0.31	0.05
TCEP	**<LOD**	0.20	0.03
TCPP	**<LOD**	7.66	0.92
TDCPP	**<LOD**	0.94	0.09
TBOEP	**<LOD**	0.047	**<LOD**
TPhP	**<LOD**	0.024	0.007
EHDP	**<LOD**	0.029	0.016
TEHP	**<LOD**	0.002	0.001
TMPP	0.0011	0.0077	**<LOD**
TIPPP	**<LOD**	**<LOD**	**<LOD**
T35DMPP	0.0014	**<LOD**	**<LOD**

6.2.2. Chlorinated Pesticides

In Tables 102-106 the results of Chlorinated Pesticides on filters, primary cells and secondary cells (breakthrough control) obtained with LV Transect Sampling are reported.

Table 102. *Chlorinated Pesticides in Field blank samples*

Lab. Code:	OCP-EMB-LV-19-185	OCP-EMB-17-066-FB
Sample name:	Field Filter Blank	Field Blank Cell 1
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	300	300
Sampling period:	28/07-08/08/2020	28/07-08/08/2020
Analysis date:	11/28/2019	11/28/2019
Concentration	**pg/L**	**pg/L**
PeCBz	0.06	0.27
HCB	0.07	0.44
a-HCH	**<LOD**	0.03
b-HCH	**<LOD**	0.11
g-HCH	**<LOD**	0.07
d-HCH	**<LOD**	**<LOD**
e-HCH	**<LOD**	**<LOD**
Sum-HCHs	---	0.21
Heptachlor	0.01	0.01
Heptachlor-exo-epoxide	**<LOD**	**<LOD**
Heptachlor-endo-epoxide	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	---	---
Aldrin	**<LOD**	**<LOD**
Dieldrin	**<LOD**	**<LOD**
Endrin	**<LOD**	**<LOD**
Isodrin	**<LOD**	**<LOD**
Sum-Drins	---	---
trans-chlordane	0.005	0.005
cis-chlordane	0.004	0.006
Sum-Chlordane	0.010	0.011
Oxychlordane	**<LOD**	**<LOD**

Lab. Code:	OCP-EMB-LV-19-185	OCP-EMB-17-066-FB
Sample name:	Field Filter Blank	Field Blank Cell 1
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	300	300
Sampling period:	28/07-08/08/2020	28/07-08/08/2020
Analysis date:	11/28/2019	11/28/2019
Concentration	pg/L	pg/L
trans-nonachlor	<LOD	0.01
cis-nonachlor	<LOD	<LOD
Sum-nonachlor	---	0.01
Endosulfane-alpha	<LOD	<LOD
Endosulfane-beta	<LOD	<LOD
Sum-Endosulfanes	---	---
Endosulfane-sulphate	<LOD	<LOD
op-DDE	<LOD	<LOD
pp-DDE	<LOD	0.03
op-DDD	<LOD	<LOD
pp-DDD	<LOD	<LOD
op-DDT	<LOD	<LOD
pp-DDT	<LOD	<LOD
Sum-DDTtotal	---	0.03
Methoxychlor	<LOD	<LOD
Mirex	0.01	0.01
Others:		
HCBD	1.38	2.31
Dichlorvos	<LOD	<LOD
Trifluralin	<LOD	<LOD
Triallate	<LOD	<LOD
Chlorpyriphos	<LOD	<LOD
Chlorfenvinphos	<LOD	<LOD
Dicofol	<LOD	<LOD
Cypermethrins	<LOD	<LOD
Chlorothalonil	<LOD	0.06

Table 103. *Results of Chlorinated Pesticides in Transect 1 (JBSS_XL_LVE-1)*

Lab. Code:	OCP-EMB-19-181	OCP-EMB-19-196	OCP-EMB-19-197
Sample name:	JBSS_XL_LVE-Filter 1	JBSS_XL_LVE-1 Cell 1	JBSS_XL_LVE-1 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Analysis date:	11/28/2019	11/29/2019	11/28/2019
Concentration	pg/L	pg/L	pg/L
PeCBz	<LOD	1.68	<LOD
HCB	<LOD	3.91	<LOD
a-HCH	<LOD	94	8.93
b-HCH	1.18	2236	226
g-HCH	<LOD	40	5.15
d-HCH	<LOD	5.39	0.23
e-HCH	<LOD	2.21	0.17
Sum-HCHs	1.18	2378	241
Heptachlor	<LOD	0.04	<LOD
Heptachlor-exo-epoxide	<LOD	2.37	0.27
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Heptachlorepoxides	---	2.37	0.27
Aldrin	<LOD	<LOD	<LOD
Dieldrin	0.09	5.64	0.78
Endrin	<LOD	0.15	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	0.09	5.78	0.78
trans-chlordane	0.018	0.069	<LOD
cis-chlordane	0.020	0.038	0.020
Sum-Chlordane	0.04	0.11	0.02
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.07	<LOD
cis-nonachlor	<LOD	0.08	0.03
Sum-nonachlor	---	0.15	0.03

Lab. Code:	OCP-EMB-19-181	OCP-EMB-19-196	OCP-EMB-19-197
Sample name:	JBSS_XL_LVE-Filter 1	JBSS_XL_LVE-1 Cell 1	JBSS_XL_LVE-1 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Analysis date:	11/28/2019	11/29/2019	11/28/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.13	0.11
Sum-Endosulfanes	---	1.13	0.11
Endosulfane-sulphate	<LOD	0.75	0.08
op-DDE	<LOD	0.22	<LOD
pp-DDE	0.18	2.87	0.13
op-DDD	<LOD	3.47	<LOD
pp-DDD	0.10	13	0.31
op-DDT	<LOD	0.88	<LOD
pp-DDT	0.26	3.87	0.35
Sum-DDTtotal	0.54	24	0.79
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	0.016	0.018
Others:			
HCBD	<LOD	7.47	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	0.06	<LOD
Triallate	<LOD	2.04	0.21
Chlorpyriphos	0.62	194	6.06
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	0.68	<LOD

Table 104. *Results of Chlorinated Pesticides in Transect 2 (JBSS_XL_LVE-2)*

Lab. Code:	OCP-EMB-19-182	OCP-EMB-19-198	OCP-EMB-19-199
Sample name:	JBSS_XL_LVE-Filter 2	JBSS_XL_LVE-2 Cell 1	JBSS_XL_LVE-2 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Analysis date:	11/28/2019	11/29/2019	11/29/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	**<LOD**	0.96	**<LOD**
HCB	**<LOD**	2.88	**<LOD**
a-HCH	**<LOD**	64	9.52
b-HCH	0.91	1902	291
g-HCH	**<LOD**	46	3.81
d-HCH	**<LOD**	1.38	0.21
e-HCH	**<LOD**	0.63	0.12
Sum-HCHs	0.91	2015	305
Heptachlor	**<LOD**	0.04	**<LOD**
Heptachlor-exo-epoxide	**<LOD**	1.87	0.39
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	---	1.87	0.39
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	0.13	4.27	0.88
Endrin	**<LOD**	0.14	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	0.13	4.41	0.88
trans-chlordane	**<LOD**	0.16	**<LOD**
cis-chlordane	**<LOD**	0.13	**<LOD**
Sum-Chlordane	---	0.28	---
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	**<LOD**	0.05	**<LOD**
cis-nonachlor	**<LOD**	0.08	0.026
Sum-nonachlor	---	0.14	0.026

Lab. Code:	OCP-EMB-19-182	OCP-EMB-19-198	OCP-EMB-19-199
Sample name:	JBSS_XL_LVE-Filter 2	JBSS_XL_LVE-2 Cell 1	JBSS_XL_LVE-2 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Analysis date:	11/28/2019	11/29/2019	11/29/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	0.80	0.27
Sum-Endosulfanes	---	0.80	0.27
Endosulfane-sulphate	<LOD	0.28	0.05
op-DDE	<LOD	0.12	<LOD
pp-DDE	0.13	1.23	<LOD
op-DDD	<LOD	0.78	<LOD
pp-DDD	0.07	3.22	<LOD
op-DDT	<LOD	0.68	0.04
pp-DDT	<LOD	2.21	<LOD
Sum-DDTtotal	0.20	8.23	0.04
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
Others:			
HCBD	<LOD	5.93	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	2.04	<LOD
Chlorpyriphos	0.20	51	1.15
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	0.65	<LOD	<LOD
Chlorothalonil	<LOD	0.28	<LOD

Table 105. *Results of Chlorinated Pesticides in Transect 3 (JBSS_XL_LVE-3)*

Lab. Code:	OCP-EMB-19-183	OCP-EMB-19-200	OCP-EMB-19-201
Sample name:	JBSS_XL_LVE-Filter 3	JBSS_XL_LVE-3 Cell 1	JBSS_XL_LVE-3 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Analysis date:	11/28/2019	11/29/2019	11/29/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
PeCBz	**<LOD**	1.06	**<LOD**
HCB	**<LOD**	3.19	1.02
a-HCH	**<LOD**	63	14
b-HCH	**<LOD**	2254	487
g-HCH	**<LOD**	25	7.38
d-HCH	**<LOD**	1.57	0.28
e-HCH	**<LOD**	0.78	0.15
Sum-HCHs	---	2345	509
Heptachlor	**<LOD**	0.03	**<LOD**
Heptachlor-exo-epoxide	**<LOD**	2.06	0.48
Heptachlor-endo-epoxide	**<LOD**	**<LOD**	**<LOD**
Sum-Heptachlorepoxides	---	2.06	0.48
Aldrin	**<LOD**	**<LOD**	**<LOD**
Dieldrin	0.07	4.47	1.05
Endrin	**<LOD**	0.12	**<LOD**
Isodrin	**<LOD**	**<LOD**	**<LOD**
Sum-Drins	0.07	4.59	1.05
trans-chlordane	**<LOD**	0.04	0.02
cis-chlordane	**<LOD**	0.14	0.03
Sum-Chlordane	---	0.18	0.05
Oxychlordane	**<LOD**	**<LOD**	**<LOD**
trans-nonachlor	**<LOD**	0.04	0.02
cis-nonachlor	**<LOD**	0.09	**<LOD**
Sum-nonachlor	---	0.14	0.02

Lab. Code:	OCP-EMB-19-183	OCP-EMB-19-200	OCP-EMB-19-201
Sample name:	JBSS_XL_LVE-Filter 3	JBSS_XL_LVE-3 Cell 1	JBSS_XL_LVE-3 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Analysis date:	11/28/2019	11/29/2019	11/29/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.83	<LOD
Sum-Endosulfanes	---	1.83	---
Endosulfane-sulphate	<LOD	0.37	0.10
op-DDE	<LOD	0.06	<LOD
pp-DDE	<LOD	0.76	0.18
op-DDD	<LOD	0.78	<LOD
pp-DDD	0.03	3.50	0.36
op-DDT	<LOD	0.19	<LOD
pp-DDT	<LOD	2.69	<LOD
Sum-DDTtotal	0.03	7.98	0.55
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
Others:			
HCBD	<LOD	4.94	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	2.35	0.37
Chlorpyriphos	<LOD	32	5.99
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	0.46	1.77	<LOD
Chlorothalonil	<LOD	0.16	<LOD

Table 106. *Results of Chlorinated Pesticides in Transect 4 (JBSS_XL_LVE-4)*

Lab. Code:	OCP-EMB-19-184	OCP-EMB-19-202	OCP-EMB-19-203
Sample name:	JBSS_XL_LVE-Filter 4	JBSS_XL_LVE-4 Cell 1	JBSS_XL_LVE-4 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Analysis date:	11/28/2019	11/29/2019	11/29/2019
Concentration	pg/L	pg/L	pg/L
PeCBz	<LOD	2.05	1.02
HCB	<LOD	3.74	<LOD
a-HCH	<LOD	78	3.85
b-HCH	<LOD	2062	102
g-HCH	<LOD	38	1.97
d-HCH	<LOD	4.09	0.15
e-HCH	<LOD	2.06	<LOD
Sum-HCHs	---	2183	108
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	2.20	0.20
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Heptachlorepoxides	---	2.20	0.20
Aldrin	<LOD	<LOD	<LOD
Dieldrin	0.13	5.99	0.52
Endrin	<LOD	0.27	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	0.13	6.27	0.52
trans-chlordane	<LOD	0.11	0.028
cis-chlordane	<LOD	0.13	<LOD
Sum-Chlordane	---	0.24	0.03
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.08	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.08	---

Lab. Code:	OCP-EMB-19-184	OCP-EMB-19-202	OCP-EMB-19-203
Sample name:	JBSS_XL_LVE-Filter 4	JBSS_XL_LVE-4 Cell 1	JBSS_XL_LVE-4 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Analysis date:	11/28/2019	11/29/2019	11/29/2019
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	2.13	0.21
Sum-Endosulfanes	---	2.13	0.21
Endosulfane-sulphate	<LOD	0.77	<LOD
op-DDE	<LOD	0.31	0.15
pp-DDE	0.68	4.39	0.29
op-DDD	<LOD	3.79	0.04
pp-DDD	0.41	12	0.15
op-DDT	<LOD	0.98	<LOD
pp-DDT	0.27	4.87	0.26
Sum-DDTtotal	1.36	27	0.89
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	0.03	0.03
Others:			
HCBD	<LOD	7.86	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	0.30	0.12
Triallate	<LOD	2.26	<LOD
Chlorpyriphos	0.19	121	1.67
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	0.34	<LOD

6.2.3. Triazines

In Tables 107-110 the results of Triazine Pesticides on filters, primary cells and secondary cells (breakthrough control) obtained with LV Transect Sampling are reported.

Table 107. *Triazines in Field Blank samples*

Lab. Code:	OPC-EMB-LV-19-185	OPC-EMB-17-066-FB
Sample name:	Field Filter Blank	Field Blank Cell
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	300	300
Sampling period:	28/07-08/08/2020	28/07-08/08/2020
Analysis date:	11/4/2019	11/4/2019
Concentration	pg/L	pg/L
Simazine	<LOD	<LOD
Atrazine	<LOD	<LOD
Terbuthylazine	<LOD	<LOD
Desethyl-Simazine	<LOD	<LOD
Desethyl-Atrazine	<LOD	<LOD
Desethyl-Terbuthylazine	<LOD	<LOD

Table 108. *Results of Triazines in Transect 1 (JBSS_XL_LVE-1)*

Lab. Code:	OPC-EMB-19-181	OPC-EMB-19-196-B	OPC-EMB-19-197-B
Sample name:	JBSS_XL_LVE-Filter 1	JBSS_XL_LVE-1 Cell 1	JBSS_XL_LVE-1 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	1.48	0.61
Atrazine	<LOD	18	5.80
Terbuthylazine	<LOD	4.73	0.83
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.01	0.01
Desethyl-Terbuthylazine	<LOD	0.08	0.04

Table 109. *Results of Triazines in Transect 2 (JBSS_XL_LVE-2)*

Lab. Code:	OPC-EMB-19-182	OPC-EMB-19-198-B	OPC-EMB-19-199-B
Sample name:	JBSS_XL_LVE-Filter 2	JBSS_XL_LVE-2 Cell 1	JBSS_XL_LVE-2 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	1.49	0.20
Atrazine	<LOD	16	3.40
Terbuthylazine	<LOD	1.61	0.45
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.008	0.002
Desethyl-Terbuthylazine	<LOD	0.02	0.003

Table 110. *Results of Triazines in Transect 3 (JBSS_XL_LVE-3)*

Lab. Code:	OPC-EMB-19-183	OPC-EMB-19-200	OPC-EMB-19-201-B
Sample name:	JBSS_XL_LVE-Filter 3	JBSS_XL_LVE-3 Cell 1	JBSS_XL_LVE-3 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Analysis date:	11/4/2019	6/16/2020	11/5/2019
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	0.85	0.76
Atrazine	<LOD	12	7.86
Terbuthylazine	<LOD	1.32	0.54
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.005	0.007
Desethyl-Terbuthylazine	<LOD	0.03	0.02

Table 111. *Results of Triazines in Transect 4 (JBSS_XL_LVE-4)*

Lab. Code:	OPC-EMB-19-184	OPC-EMB-19-202-D	OPC-EMB-19-203-B
Sample name:	JBSS_XL_LVE-Filter 4	JBSS_XL_LVE-4 Cell 1	JBSS_XL_LVE-4 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Analysis date:	11/4/2019	11/6/2019	11/5/2019
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	1.53	0.49
Atrazine	<LOD	17	3.96
Terbuthylazine	<LOD	4.78	0.71
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.01	0.01
Desethyl-Terbuthylazine	<LOD	0.07	0.02

6.2.4. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

In the Tables 112-116 the results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT on filters, primary cells and secondary cells (breakthrough control) obtained with LV Transect Sampling are reported.

Table 112. *Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Field Blank samples*

Lab. Code:	OPC-EMB-LV-19-185	OPC-EMB-17-066-FB
Sample name:	Field Filter Blank	Field Blank Cell
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	300	300
Sampling period:	28/07-08/08/2020	28/07-08/08/2020
Analysis date:	11/4/2019	11/4/2019
Concentration	pg/L	pg/L
Phenanthrene	11	7.88
Anthracene	0.95	0.28
Fluoranthene	2.78	1.54
Pyrene	2.21	3.23
Benz(a)anthracene	0.33	0.10
Chrysene	0.69	0.82
Sum Benzo(b,j,k)fluoranthene	0.65	0.40
Benzo(e)pyrene	0.11	0.01
Benzo(a)pyrene	0.04	0.01
Perylene	0.08	0.02
Indeno(123-cd)pyrene	0.05	0.02
Benzo(ghi)perylene	0.15	0.03
Dibenz(ah)anthracene	0.05	0.03
Coronene	0.26	**<LOD**
BHT	355	34
EHMC	581	8.86

Table 113. *Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 1 JBSS_XL_LVE-1)*

Lab. Code:	OPC-EMB-19-181	OPC-EMB-19-196-B	OPC-EMB-19-197-B
Sample name:	JBSS_XL_LVE-Filter 1	JBSS_XL_LVE-1 Cell 1	JBSS_XL_LVE-1 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	15	316	52
Anthracene	**<LOD**	25	**<LOD**
Fluoranthene	10	43	15
Pyrene	**<LOD**	230	30
Benz(a)anthracene	1.72	1.58	0.38
Chrysene	4.09	34	2.05
Sum Benzo(b,j,k)fluoranthene	5.35	8.46	1.13
Benzo(e)pyrene	1.98	1.68	0.23
Benzo(a)pyrene	2.12	0.43	**<LOD**
Perylene	1.05	0.71	0.21
Indeno(123-cd)pyrene	2.48	1.03	0.23
Benzo(ghi)perylene	3.94	0.83	0.32
Dibenz(ah)anthracene	1.54	0.75	0.30
Coronene	3.33	**<LOD**	**<LOD**
BHT	**<LOD**	**<LOD**	**<LOD**
EHMC	**<LOD**	309	135

Table 114. *Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 2 (JBSS_XL_LVE-2)*

Lab. Code:	OPC-EMB-19-182	OPC-EMB-19-198-B	OPC-EMB-19-199-B
Sample name:	JBSS_XL_LVE-Filter 2	JBSS_XL_LVE-2 Cell 1	JBSS_XL_LVE-2 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	**<LOD**	312	17
Anthracene	**<LOD**	11	**<LOD**
Fluoranthene	10.30	23	3.35
Pyrene	16.47	151	10.79
Benz(a)anthracene	2.18	0.48	**<LOD**
Chrysene	5.30	25	**<LOD**
Sum Benzo(b,j,k)fluoranthene	7.85	6.30	1.22
Benzo(e)pyrene	2.88	1.00	**<LOD**
Benzo(a)pyrene	3.47	0.23	0.15
Perylene	1.16	0.36	**<LOD**
Indeno(123-cd)pyrene	4.16	0.78	0.40
Benzo(ghi)perylene	6.54	0.89	0.40
Dibenz(ah)anthracene	2.22	0.67	0.21
Coronene	5.56	0.93	0.67
BHT	**<LOD**	**<LOD**	**<LOD**
EHMC	**<LOD**	888	43

Table 115. *Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 3 (JBSS_XL_LVE-3)*

Lab. Code:	OPC-EMB-19-183	OPC-EMB-19-200	OPC-EMB-19-201-B
Sample name:	JBSS_XL_LVE-Filter 3	JBSS_XL_LVE-3 Cell 1	JBSS_XL_LVE-3 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Analysis date:	11/4/2019	6/16/2020	11/5/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	**<LOD**	271	36
Anthracene	**<LOD**	10	**<LOD**
Fluoranthene	4.69	10	3.96
Pyrene	**<LOD**	150	21
Benz(a)anthracene	1.13	**<LOD**	**<LOD**
Chrysene	3.03	32	3.20
Sum Benzo(b,j,k)fluoranthene	4.69	8.44	1.21
Benzo(e)pyrene	1.88	1.55	0.19
Benzo(a)pyrene	1.78	0.40	**<LOD**
Perylene	0.77	0.26	0.19
Indeno(123-cd)pyrene	2.61	1.42	0.38
Benzo(ghi)perylene	4.39	0.75	0.33
Dibenz(ah)anthracene	1.39	0.48	**<LOD**
Coronene	3.73	0.93	0.58
BHT	**<LOD**	**<LOD**	**<LOD**
EHMC	**<LOD**	709	80

Table 116. *Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 4 (JBSS_XL_LVE-4)*

Lab. Code:	OPC-EMB-19-184	OPC-EMB-19-202-D	OPC-EMB-19-203-B
Sample name:	JBSS_XL_LVE-Filter 4	JBSS_XL_LVE-4 Cell 1	JBSS_XL_LVE-4 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Analysis date:	11/4/2019	11/6/2019	11/5/2019
Concentration	pg/L	pg/L	pg/L
Phenanthrene	22	462	26
Anthracene	**<LOD**	23	**<LOD**
Fluoranthene	7.11	84	17
Pyrene	9.54	290	26
Benz(a)anthracene	1.04	1.96	**<LOD**
Chrysene	2.95	32	**<LOD**
Sum Benzo(b,j,k)fluoranthene	4.73	11	1.61
Benzo(e)pyrene	1.61	2.91	0.26
Benzo(a)pyrene	1.78	0.71	**<LOD**
Perylene	1.86	1.14	0.27
Indeno(123-cd)pyrene	2.80	1.44	0.43
Benzo(ghi)perylene	4.44	1.58	0.49
Dibenz(ah)anthracene	1.61	0.62	0.24
Coronene	5.23	1.05	**<LOD**
BHT	**<LOD**	200	**<LOD**
EHMC	**<LOD**	1082	24

6.2.5. Polychlorinated Biphenyls

In the Tables 117-121 the results of EC-7 Polychlorinated Biphenyls on filters, primary cells and secondary cells (breakthrough control) obtained with LV Transect Sampling are reported.

Table 117. *EC-7 Polychlorinated Biphenyls in Field blank samples*

Lab. Code:	OPC-EMB-LV-19-185	OPC-EMB-17-066-FB
Sample name:	Field Filter Blank	Field Blank Cell
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	300	300
Sampling period:	28/07-08/08/2020	28/07-08/08/2020
Analysis date:	11/4/2019	11/4/2019
Concentration	pg/L	pg/L
EC-7		
PCB 28	<LOD	<LOD
PCB 52	<LOD	<LOD
PCB 101	<LOD	<LOD
PCB 118	<LOD	<LOD
PCB 138	<LOD	<LOD
PCB 153	<LOD	<LOD
PCB 180	<LOD	<LOD
Sum EC-7 PCBs	---	---

Table 118. *EC-7 Polychlorinated Biphenyls in Transect 1 (JBSS_XL_LVE-1)*

Lab. Code:	OPC-EMB-19-181	OPC-EMB-19-196-B	OPC-EMB-19-197-B
Sample name:	JBSS_XL_LVE-Filter 1	JBSS_XL_LVE-1 Cell 1	JBSS_XL_LVE-1 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
EC-7			
PCB 28	0.17	4.53	0.10
PCB 52	0.26	1.20	0.10
PCB 101	0.26	0.68	0.09
PCB 118	0.13	0.37	**<LOD**
PCB 138	**<LOD**	0.39	**<LOD**
PCB 153	**<LOD**	0.48	**<LOD**
PCB 180	**<LOD**	0.14	**<LOD**
Sum EC-7 PCBs	0.83	7.80	0.28

Table 119. *EC-7 Polychlorinated Biphenyls in Transect 2 ((JBSS_XL_LVE-2)*

Lab. Code:	OPC-EMB-19-182	OPC-EMB-19-198-B	OPC-EMB-19-199-B
Sample name:	JBSS_XL_LVE-Filter 2	JBSS_XL_LVE-2 Cell 1	JBSS_XL_LVE-2 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Analysis date:	11/4/2019	11/5/2019	11/5/2019
Concentration	**pg/L**	**pg/L**	**pg/L**
EC-7			
PCB 28	**<LOD**	1.66	0.14
PCB 52	**<LOD**	0.42	0.18
PCB 101	**<LOD**	0.31	0.14
PCB 118	**<LOD**	0.17	**<LOD**
PCB 138	**<LOD**	0.18	**<LOD**
PCB 153	**<LOD**	0.25	**<LOD**
PCB 180	**<LOD**	**<LOD**	**<LOD**
Sum EC-7 PCBs	---	2.99	0.46

Table 120. *EC-7 Polychlorinated Biphenyls in Transect 3 ((JBSS_XL_LVE-3)*

Lab. Code:	OPC-EMB-19-183	OPC-EMB-19-200	OPC-EMB-19-201-B
Sample name:	JBSS_XL_LVE-Filter 3	JBSS_XL_LVE-3 Cell 1	JBSS_XL_LVE-3 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Analysis date:	11/4/2019	6/16/2020	11/5/2019
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	<LOD	1.78	0.20
PCB 52	<LOD	0.38	0.21
PCB 101	<LOD	0.25	0.15
PCB 118	<LOD	0.13	<LOD
PCB 138	<LOD	0.18	<LOD
PCB 153	<LOD	0.19	<LOD
PCB 180	<LOD	<LOD	<LOD
Sum EC-7 PCBs	---	2.91	0.55

Table 121. *EC-7 Polychlorinated Biphenyls in Transect 4 ((JBSS_XL_LVE-4)*

Lab. Code:	OPC-EMB-19-184	OPC-EMB-19-202-D	OPC-EMB-19-203-B
Sample name:	JBSS_XL_LVE-Filter 4	JBSS_XL_LVE-4 Cell 1	JBSS_XL_LVE-4 Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Analysis date:	11/4/2019	11/6/2019	11/5/2019
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	0.12	2.56	0.60
PCB 52	<LOD	1.29	0.62
PCB 101	<LOD	1.03	1.04
PCB 118	<LOD	0.52	0.93
PCB 138	<LOD	0.83	0.88
PCB 153	<LOD	0.65	1.06
PCB 180	<LOD	<LOD	0.25
Sum EC-7 PCBs	0.12	6.88	5.38

6.3. Final results for Large Volume Transect Samples

In the following tables the final concentrations of apolar and semi-polar compounds detected in the different water compartments are reported: in particulate matter (filters), dissolved in water (sum of Cell 1 and Cell 2) and in whole water (sum of particulate matter and dissolved fractions).

6.3.1. Organophosphate Compounds

Table 122. *Final concentration of Organophosphate Compounds in Transect 1 (JBSS_XL_LVE-1)*

Transect:	JBSS_XL_LVE-1		
Sample name:	Filter 1	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.44	0.44
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	3.10	3.10
TNBP	<LOD	0.45	0.45
TCEP	<LOD	0.26	0.26
TCPP	<LOD	6.98	6.98
TDCPP	<LOD	0.78	0.78
TBOEP	<LOD	0.076	0.076
TPhP	<LOD	0.018	0.018
EHDP	<LOD	0.028	0.028
TEHP	<LOD	0.002	0.002
TMPP	<LOD	0.004	0.004
TIPPP	<LOD	0.0004	0.0004
T35DMPP	<LOD	<LOD	<LOD

Table 123. *Final concentration of Organophosphate Compounds in Transect 2 (JBSS_XL_LVE-2)*

Transect:	JBSS_XL_LVE-2		
Sample name:	Filter 2	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.26	0.26
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	2.53	2.53
TNBP	<LOD	0.27	0.27
TCEP	<LOD	0.16	0.16
TCPP	<LOD	3.65	3.65
TDCPP	<LOD	0.46	0.46
TBOEP	<LOD	0.055	0.055
TPhP	<LOD	0.012	0.012
EHDP	<LOD	0.030	0.030
TEHP	<LOD	0.002	0.002
TMPP	<LOD	0.006	0.006
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 124. *Final concentration of Organophosphate Compounds in Transect 3 (JBSS_XL_LVE-3)*

Transect:	JBSS_XL_LVE-3		
Sample name:	Filter 3	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.34	0.34
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	1.85	1.85
TNBP	<LOD	0.25	0.25
TCEP	<LOD	0.26	0.26
TCPP	<LOD	3.43	3.43
TDCPP	<LOD	0.57	0.57
TBOEP	<LOD	<LOD	<LOD
TPhP	<LOD	0.012	0.012
EHDP	<LOD	0.031	0.031
TEHP	<LOD	0.002	0.002
TMPP	<LOD	0.005	0.005
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 125. *Final concentration of Organophosphate Compounds in Transect 4 (JBSS_XL_LVE-4)*

Transect:	JBSS_XL_LVE-4		
Sample name:	Filter 4	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.34	0.34
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	2.21	2.21
TNBP	<LOD	0.36	0.36
TCEP	<LOD	0.23	0.23
TCPP	<LOD	8.58	8.58
TDCPP	<LOD	1.04	1.04
TBOEP	<LOD	0.047	0.047
TPhP	<LOD	0.031	0.031
EHDP	<LOD	0.044	0.044
TEHP	<LOD	0.004	0.004
TMPP	0.0011	0.009	0.010
TIPPP	<LOD	<LOD	<LOD
T35DMPP	0.0014	0.0014	0.003

6.3.2. Chlorinated Pesticides

Table 126. *Final concentration of Chlorinated Pesticides in Transect 1 (JBSS_XL_LVE-1)*

Transect:	JBSS_XL_LVE-1		
Sample name:	Filter 1	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
PeCBz	<LOD	1.68	1.68
HCB	<LOD	3.91	3.91
a-HCH	<LOD	103	103
b-HCH	1.18	2463	2464
g-HCH	<LOD	45	45
d-HCH	<LOD	5.62	5.62
e-HCH	<LOD	2.38	2.38
Sum-HCHs	1.18	2619	2621
Heptachlor	<LOD	0.04	0.04
Heptachlor-exo-epoxide	<LOD	2.63	2.63
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Heptachlorepoxides	---	2.63	2.63
Aldrin	<LOD	<LOD	<LOD
Dieldrin	0.09	6.42	6.51
Endrin	<LOD	0.15	0.15
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	0.09	6.57	6.66
trans-chlordane	0.018	0.069	0.09
cis-chlordane	0.020	0.058	0.08
Sum-Chlordane	0.04	0.13	0.16
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.07	0.07

Transect:	JBSS_XL_LVE-1		
Sample name:	Filter 1	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
cis-nonachlor	<LOD	0.11	0.11
Sum-nonachlor	---	0.17	0.17
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.25	1.25
Sum-Endosulfanes	---	1.25	1.25
Endosulfane-sulphate	<LOD	0.83	0.83
op-DDE	<LOD	0.22	0.22
pp-DDE	0.18	3.01	3.19
op-DDD	<LOD	3.47	3.47
pp-DDD	0.10	13	13
op-DDT	<LOD	0.88	0.88
pp-DDT	0.26	4.22	4.47
Sum-DDTtotal	0.54	25	25
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	0.034	0.03
Others;			
HCBD	<LOD	7.47	7.47
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	0.061	0.06
Triallate	<LOD	2.25	2.25
Chlorpyriphos	0.62	200	201
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	0.68	0.68

Table 127. *Final concentration of Chlorinated Pesticides in Transect 2 (JBSS_XL_LVE-2)*

Transect:	JBSS_XL_LVE-2		
Sample name:	Filter 2	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
PeCBz	<LOD	0.96	0.96
HCB	<LOD	2.88	2.88
a-HCH	<LOD	74	74
b-HCH	0.91	2193	2194
g-HCH	<LOD	50	50
d-HCH	<LOD	1.58	1.58
e-HCH	<LOD	0.75	0.75
Sum-HCHs	0.91	2320	2321
Heptachlor	<LOD	0.04	0.04
Heptachlor-exo-epoxide	<LOD	2.26	2.26
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Heptachlorepoxides	---	2.26	2.26
Aldrin	<LOD	<LOD	<LOD
Dieldrin	0.13	5.16	5.29
Endrin	<LOD	0.14	0.14
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	0.13	5.29	5.43
trans-chlordane	<LOD	0.16	0.16
cis-chlordane	<LOD	0.13	0.13
Sum-Chlordane	---	0.28	0.28
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.05	0.05
cis-nonachlor	<LOD	0.11	0.11
Sum-nonachlor	---	0.16	0.16

Transect:	JBSS_XL_LVE-2		
Sample name:	Filter 2	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.07	1.07
Sum-Endosulfanes	---	1.07	1.07
Endosulfane-sulphate	<LOD	0.33	0.33
op-DDE	<LOD	0.12	0.12
pp-DDE	0.13	1.23	1.36
op-DDD	<LOD	0.78	0.78
pp-DDD	0.07	3.22	3.28
op-DDT	<LOD	0.71	0.71
pp-DDT	<LOD	2.21	2.21
Sum-DDTtotal	0.20	8.27	8.47
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
Others;			
HCBD	<LOD	5.93	5.93
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	2.04	2.04
Chlorpyriphos	0.20	52	52
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	0.65	<LOD	0.65
Chlorothalonil	<LOD	0.28	0.28

Table 128. *Final concentration of Chlorinated Pesticides in Transect 3 (JBSS_XL_LVE-3)*

Transect:	JBSS_XL_LVE-3		
Sample name:	Filter 3	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
PeCBz	<LOD	1.06	1.06
HCB	<LOD	4.21	4.21
a-HCH	<LOD	78	78
b-HCH	<LOD	2741	2741
g-HCH	<LOD	33	33
d-HCH	<LOD	1.85	1.85
e-HCH	<LOD	0.93	0.93
Sum-HCHs	---	2854	2854
Heptachlor	<LOD	0.03	0.03
Heptachlor-exo-epoxide	<LOD	2.55	2.55
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Heptachlorepoxides	---	2.55	2.55
Aldrin	<LOD	<LOD	<LOD
Dieldrin	0.07	5.52	5.60
Endrin	<LOD	0.12	0.12
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	0.07	5.64	5.71
trans-chlordane	<LOD	0.06	0.06
cis-chlordane	<LOD	0.17	0.17
Sum-Chlordane	---	0.23	0.23
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.06	0.06
cis-nonachlor	<LOD	0.09	0.09
Sum-nonachlor	---	0.16	0.16

Transect:	JBSS_XL_LVE-3		
Sample name:	Filter 3	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.83	1.83
Sum-Endosulfanes	---	1.83	1.83
Endosulfane-sulphate	<LOD	0.46	0.46
op-DDE	<LOD	0.06	0.06
pp-DDE	<LOD	0.94	0.94
op-DDD	<LOD	0.78	0.78
pp-DDD	0.03	3.86	3.89
op-DDT	<LOD	0.19	0.19
pp-DDT	<LOD	2.69	2.69
Sum-DDTtotal	0.03	8.53	8.56
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
Others;			
HCBD	<LOD	4.94	4.94
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	2.73	2.73
Chlorpyriphos	<LOD	38	38
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	0.46	1.77	2.23
Chlorothalonil	<LOD	0.16	0.16

Table 129. *Final concentration of Chlorinated Pesticides in Transect 4 (JBSS_XL_LVE-4)*

Transect:	JBSS_XL_LVE-4		
Sample name:	Filter 4	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
PeCBz	<LOD	3.07	3.07
HCB	<LOD	3.74	3.74
a-HCH	<LOD	81	81
b-HCH	<LOD	2164	2164
g-HCH	<LOD	40	40
d-HCH	<LOD	4.24	4.24
e-HCH	<LOD	2.06	2.06
Sum-HCHs	---	2291	2291
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	2.40	2.40
Heptachlor-endo-epoxide	<LOD	<LOD	0.00
Sum-Heptachlorepoxides	---	2.40	2.40
Aldrin	<LOD	<LOD	<LOD
Dieldrin	0.13	6.51	6.63
Endrin	<LOD	0.27	0.27
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	0.13	6.78	6.91
trans-chlordane	<LOD	0.14	0.14
cis-chlordane	<LOD	0.13	0.13
Sum-Chlordane	---	0.27	0.27
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.08	0.08
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.08	0.08

Transect:	JBSS_XL_LVE-4		
Sample name:	Filter 4	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	2.35	2.35
Sum-Endosulfanes	---	2.35	2.35
Endosulfane-sulphate	<LOD	0.77	0.77
op-DDE	<LOD	0.45	0.45
pp-DDE	0.68	4.68	5.37
op-DDD	<LOD	3.83	3.83
pp-DDD	0.41	13	13
op-DDT	<LOD	0.98	0.98
pp-DDT	0.27	5.13	5.40
Sum-DDTtotal	1.36	28	29
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	0.07	0.07
Others;			
HCBD	<LOD	7.86	7.86
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	0.42	0.42
Triallate	<LOD	2.26	2.26
Chlorpyriphos	0.19	123	123
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
Chlorothalonil	<LOD	0.34	0.34

6.3.3. Triazines

Table 130. *Final concentration of Triazines in Transect 1 (JBSS_XL_LVE-1)*

Transect:	JBSS_XL_LVE-1		
Sample name:	Filter 1	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	2.09	2.09
Atrazine	<LOD	23	23
Terbuthylazine	<LOD	5.56	5.56
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.02	0.02
Desethyl-Terbuthylazine	<LOD	0.11	0.11

Table 131. *Final concentration of Triazines detected in Transect 2 JBSS_XL_LVE-2)*

Transect:	JBSS_XL_LVE-2		
Sample name:	Filter 2	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	1.69	1.69
Atrazine	<LOD	20	20
Terbuthylazine	<LOD	2.06	2.06
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.01	0.01
Desethyl-Terbuthylazine	<LOD	0.02	0.02

Table 132. *Final concentration of Triazines in Transect 3 (JBSS_XL_LVE-3)*

Transect:	JBSS_XL_LVE-3		
Sample name:	Filter 3	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	1.61	1.61
Atrazine	<LOD	20	20
Terbuthylazine	<LOD	1.86	1.86
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.01	0.01
Desethyl-Terbuthylazine	<LOD	0.04	0.04

Table 133. *Final concentration of Triazines in Transect 4 (JBSS_XL_LVE-4)*

Transect:	JBSS_XL_LVE-4		
Sample name:	Filter 4	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	2.02	2.02
Atrazine	<LOD	20.84	20.84
Terbuthylazine	<LOD	5.49	5.49
Desethyl-Simazine	<LOD	<LOD	<LOD
Desethyl-Atrazine	<LOD	0.02	0.02
Desethyl-Terbuthylazine	<LOD	0.09	0.09

6.3.4. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

Table 134. *Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 1 (JBSS_XL_LVE-1)*

Transect:	JBSS_XL_LVE-1		
Sample name:	Filter 1	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Phenanthrene	15	368	383
Anthracene	**<LOD**	25	25
Fluoranthene	10	58	68
Pyrene	**<LOD**	260	260
Benz(a)anthracene	1.72	1.96	3.68
Chrysene	4.09	36	40
Sum Benzo(b,j,k)fluoranthene	5.35	10	15
Benzo(e)pyrene	1.98	1.91	3.89
Benzo(a)pyrene	2.12	0.43	2.55
Perylene	1.05	0.92	1.96
Indeno(123-cd)pyrene	2.48	1.26	3.74
Benzo(ghi)perylene	3.94	1.15	5.09
Dibenz(ah)anthracene	1.54	1.05	2.59
Coronene	3.33	**<LOD**	3.33
BHT	**<LOD**	**<LOD**	**<LOD**
EHMC	**<LOD**	443	443

Table 135. *Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 2 (JBSS_XL_LVE-2)*

Transect:	**JBSS_XL_LVE-2**		
Sample name:	**Filter 2**	**Σ Cell 1 and Cell 2**	**Σ Filter-cell1 - Cell 2**
Type of sample:	**LV-TS Black Sea Water**	**LV-TS Black Sea Water**	**LV-TS Black Sea Water**
Volume sampled (L):	**266**	**266**	**266**
Sampling period:	**30/07/2020**	**30/07/2020**	**30/07/2020**
Matrix / Phase:	**Suspended Particulate Matter**	**Dissolved**	**Whole Water**
Concentration	pg/L	pg/L	pg/L
Phenanthrene	**<LOD**	329	329
Anthracene	**<LOD**	11	11
Fluoranthene	10	27	37
Pyrene	16	161	178
Benz(a)anthracene	2.18	0.48	2.66
Chrysene	5.30	25	30
Sum Benzo(b,j,k)fluoranthene	7.85	7.52	15
Benzo(e)pyrene	2.88	1.00	3.88
Benzo(a)pyrene	3.47	0.37	3.84
Perylene	1.16	0.36	1.52
Indeno(123-cd)pyrene	4.16	1.18	5.34
Benzo(ghi)perylene	6.54	1.29	7.83
Dibenz(ah)anthracene	2.22	0.88	3.10
Coronene	5.56	1.59	7.15
BHT	**<LOD**	**<LOD**	**<LOD**
EHMC	**<LOD**	931	931

Table 136. *Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 3 (JBSS_XL_LVE-3)*

Transect:	**JBSS_XL_LVE-3**		
Sample name:	**Filter 3**	**Σ Cell 1 and Cell 2**	**Σ Filter-cell1 - Cell 2**
Type of sample:	**LV-TS Black Sea Water**	**LV-TS Black Sea Water**	**LV-TS Black Sea Water**
Volume sampled (L):	**408**	**408**	**408**
Sampling period:	**01/08/2020**	**01/08/2020**	**01/08/2020**
Matrix / Phase:	**Suspended Particulate Matter**	**Dissolved**	**Whole Water**
Concentration	pg/L	pg/L	pg/L
Phenanthrene	**<LOD**	307	307
Anthracene	**<LOD**	10	10
Fluoranthene	4.69	14	19
Pyrene	**<LOD**	171	171
Benz(a)anthracene	1.13	**<LOD**	1.13
Chrysene	3.03	35	38
Sum Benzo(b,j,k)fluoranthene	4.69	10	14
Benzo(e)pyrene	1.88	1.74	3.62
Benzo(a)pyrene	1.78	0.40	2.18
Perylene	0.77	0.45	1.22
Indeno(123-cd)pyrene	2.61	1.80	4.41
Benzo(ghi)perylene	4.39	1.08	5.47
Dibenz(ah)anthracene	1.39	0.48	1.87
Coronene	3.73	1.52	5.25
BHT	**<LOD**	**<LOD**	**<LOD**
EHMC	**<LOD**	789	789

Table 137. *Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 4 (JBSS_XL_LVE-4)*

Transect:	**JBSS_XL_LVE-4**		
Sample name:	**Filter 4**	**Σ Cell 1 and Cell 2**	**Σ Filter-cell1 - Cell 2**
Type of sample:	**LV-TS Black Sea Water**	**LV-TS Black Sea Water**	**LV-TS Black Sea Water**
Volume sampled (L):	**180**	**180**	**180**
Sampling period:	**08/08/2020**	**08/08/2020**	**08/08/2020**
Matrix / Phase:	**Suspended Particulate Matter**	**Dissolved**	**Whole Water**
Concentration	pg/L	pg/L	pg/L
Phenanthrene	22	488	510
Anthracene	**<LOD**	23	23
Fluoranthene	7.11	101	108
Pyrene	9.54	316	326
Benz(a)anthracene	1.04	1.96	3.00
Chrysene	2.95	32	35
Sum Benzo(b,j,k)fluoranthene	4.73	12	17
Benzo(e)pyrene	1.61	3.17	4.77
Benzo(a)pyrene	1.78	0.71	2.48
Perylene	1.86	1.41	3.27
Indeno(123-cd)pyrene	2.80	1.87	4.67
Benzo(ghi)perylene	4.44	2.07	6.50
Dibenz(ah)anthracene	1.61	0.85	2.46
Coronene	5.23	1.05	6.28
BHT	**<LOD**	200	200
EHMC	**<LOD**	1106	1106

6.3.5. Polychlorinated Biphenyls (EC-7 PCBs)

Table 138. *Final concentration of EC-7 Polychlorinated Biphenyls in Transect 1 (JBSS_XL_LVE-1)*

Transect:	JBSS_XL_LVE-1		
Sample name:	Filter 1	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	307	307	307
Sampling period:	29/07/2020	29/07/2020	29/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	0.17	4.63	4.79
PCB 52	0.26	1.29	1.56
PCB 101	0.26	0.77	1.03
PCB 118	0.13	0.37	0.51
PCB 138	<LOD	0.39	0.39
PCB 153	<LOD	0.48	0.48
PCB 180	<LOD	0.14	0.14
Sum EC-7 PCBs	0.83	8.08	8.91

Table 139. *Final concentration of EC-7 Polychlorinated Biphenyls in Transect 2 (JBSS_XL_LVE-2)*

Transect:	JBSS_XL_LVE-2		
Sample name:	Filter 2	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	266	266	266
Sampling period:	30/07/2020	30/07/2020	30/07/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L		pg/L
EC-7			
PCB 28	<LOD	1.80	1.80
PCB 52	<LOD	0.60	0.60
PCB 101	<LOD	0.45	0.45
PCB 118	<LOD	0.17	0.17
PCB 138	<LOD	0.18	0.18
PCB 153	<LOD	0.25	0.25
PCB 180	<LOD	<LOD	<LOD
Sum EC-7 PCBs	---	3.46	3.46

Table 140. *Final concentration of EC-7 Polychlorinated Biphenyls in Transect 3 (JBSS_XL_LVE-3)*

Transect:	JBSS_XL_LVE-3		
Sample name:	Filter 3	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	408	408	408
Sampling period:	01/08/2020	01/08/2020	01/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L		pg/L
EC-7			
PCB 28	<LOD	1.98	1.98
PCB 52	<LOD	0.58	0.58
PCB 101	<LOD	0.40	0.40
PCB 118	<LOD	0.13	0.13
PCB 138	<LOD	0.18	0.18
PCB 153	<LOD	0.19	0.19
PCB 180	<LOD	<LOD	<LOD
Sum EC-7 PCBs	---	3.47	3.47

Table 141. *Final concentration of EC-7 Polychlorinated Biphenyls in Transect 4 (JBSS_XL_LVE-4)*

Transect:	JBSS_XL_LVE-4		
Sample name:	Filter 4	Σ Cell 1 and Cell 2	Σ Filter-cell1 - Cell 2
Type of sample:	LV-TS Black Sea Water	LV-TS Black Sea Water	LV-TS Black Sea Water
Volume sampled (L):	180	180	180
Sampling period:	08/08/2020	08/08/2020	08/08/2020
Matrix / Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L		pg/L
EC-7			
PCB 28	0.12	3.16	3.28
PCB 52	<LOD	1.91	1.91
PCB 101	<LOD	2.07	2.07
PCB 118	<LOD	1.45	1.45
PCB 138	<LOD	1.72	1.72
PCB 153	<LOD	1.70	1.70
PCB 180	<LOD	0.25	0.25
Sum EC-7 PCBs	0.12	12	12

7. Conclusions

The analysis of organic contaminants at ultra trace level (low pg/L) in large marine water samples is challenging with respect to sampling procedures, sample preparation and the analytical measurements. It requires a high level of analytical quality control, clean working techniques, experience in sample handling and the use of specific analytical instrumentation.
Both techniques applied during the 2019 EMBLAS-Plus Joint Open Sea Campaign, spot sampling and transect sampling, provide specific results and have specific applications. Spot samples of 20 L volume can easily be acquired and transported, they provide a single result by extraction of filter and adsorbent in one step. After sample extraction in a field laboratory by a dedicated manifold, the combined filtration/adsorption cartridge can easily be transported to the analytical laboratory. This methodology can target a wide range of polar and non-polar substances and achieves very low detection limits.
Large scale transect sampling provides basin scale representative results on contaminants concentrations in the dissolved and particulate phase of the water masses, needed to target large scale pollution of the water column, including atmospheric input and the future budgeting of contaminants in different marine environmental compartments. Due to the large sample volume, the detection capability of the method is very high and ultra-low concentrations of non-polar persistent substances can be analysed.
The transect approach, performing sampling during cruising, does not require specific ship time and can potentially be applied on ships of opportunity. The extracts from large sample volumes collected during transect sampling allow also the application of different analytical techniques, including suspect screening and non-target analysis due to the availability of more analyte material.

The two different sampling methodologies were successfully applied during the EMBLAS-Plus 2019 cruise for a total of 39 samples collected, 112 substances analysed and about 4088 final individual results.

Please note that the interpretation of the analytical results with regards to their spatial distribution or potential sources is not within the scope of this report.

Some technical considerations which might be of use for the interpretation of the results are reported hereafter:

- While some absolute recoveries of standards in samples collected by stainless steel tanks were low, the use of internal isotope labelled standards allowed the control of the analytical procedure. Very good reproducibility for all detectable compounds was obtained with the 20L sampling device using stainless steel sampling containers (Mariani Box).

- The scarce quality of distilled water used for the 20L spot field blank sample made the blank unusable. It is preferable not to extract water in the field blank because it might induce contamination from handling and transport.

- A very good agreement was obtained comparing the results of Triazine pesticides analysed both by HRGC-HRMS and by UHPLC-MS/MS.

- Very good sampling efficiency for all detectable compounds was obtained with extralarge sampling device even sampling up to 408L (Large Volume Transect Sampling).

- Large Volume Transect Sampling allowed to reach the most stringent and challenging EQS for Heptachlor with the LOQ of 0.12 pg/L 0.07 pg/L and 0.05 pg/L respectively for the sampled volumes of 180L, 300L and 408L (EQS: 0.2 pg/L).

- For the Heptachlor-exo-epoxide the most stringent and challenging EQS of 0.2 pg/L was possible to reach only in the 408L sample with the LOQ of 0.16 pg/L. In the other samples the EQS was achieved as LOD of 0.14 pg/L and 0.09 pg/L respectively for the sampled volumes of 180L and 300L. For the congener Heptachlor-endo-epoxide the LOD of 408L sample was very close to EQS but not achieved.

- High concentrations of Triazines, in particular Atrazine, were found (tens nanograms per liter) in the 20L spot samples. These compounds were analysed also by LV transect sampling, confirming the detected high levels of contamination.

- Low molecular weight PAHs (e.g.: Naphthalene, Acenaphthylene, Acenapthene and Fluorene) have been analysed but not reported, as the methodology resulted to be not suitable for their quantification.

- Low molecular weight of HCBD and its high volatility makes the methodology not suitable for its analysis. The concentration of HCBD must be considered as indicative only.

- For the interpretation of elevated concentrations of EHMC, a sunscreen agent, including its occurrence in blanks, possible sources of contamination should be taken into consideration, both on-site due to personal application and during sample handling and preparation.

- The interpretation of the results including the mapping of the concentrations and the identification of eventual gradients and concentration distributions, will provide further quality control of the analytical results through probability considerations and source attribution analysis.

The analytical results, together with samples of sediment and biota, can provide a holistic approach for marine contaminant assessments, supporting also modelling of chemical's pathways for source attribution. This is needed in order to plan and facilitate the implementation of pollution reduction measures.

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List of abbreviations and definitions

Chemical elements are identified by their respective symbols as defined by the International Union of Pure and Applied Chemistry (IUPAC).

Throughout this report, the following abbreviations and symbols are used:

BHT	2.6-Di-tert-butyl-4-methylphenol
CAD	Collision Gas
CUR	Curtain Gas
CRM	Certified reference material
CXP	Collision Cell Exit Potential
DG	Directorate-General
E1	Estrone
E2	17β-estradiol
EE2	17α-ethinyl estradiol
EC	European Commission
EHMC	2-Ethylhexyl-methoxycinnamate
EI	Electron Impact
EP	Entrance Potential
EU	European Union
GC	Gas chromatography
GS1	Ion Source gas 1
GS2	Ion Source gas 2
HDPE	High Density Polyethylene
HLB	Hydrophilic-lipophilic balanced
IPs	Identification points
IS	Internal standard/Ion Transfer voltage
JRC	Joint Research Centre
LOD	Limit of detection
LOQ	Limit of quantification
LV-TS	Large Volume Transect Sampling
MRM	Multiple reaction monitoring
MS	Mass spectrometry
MSFD	Marine Strategy Framework Directive
OCPs	Organochloride pesticides
OPCs	Organophosphate compounds
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
PPG	Polypropylene glycol
PS	Priority substances
QC	Quality control sample
R^2	Coefficient of determination
RT	Retention time
SD	Standard deviation
S/N	Signal to Noise
SPE	Solid-phase extraction
TEM	Temperature
UHPLC	Ultra-high-pressure liquid chromatography
WFD	Water Framework Directive

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