



Environmental Monitoring  
in the Black Sea



*Empowered lives.  
Resilient nations.*

National Pilot Monitoring Studies and  
Joint Open Sea Surveys in Georgia,  
Russian Federation and Ukraine, 2017

Final Scientific Report -  
ANNEXES

**NOVEMBER** 2018

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# Annex 1: Phytoplankton intercomparison

## REPORT

### III intercomparison results of the phytoplankton samples processing

April 2018, Odessa

#### Responsible person of the Phytoplankton Intercomparison:

Andrii Zotov - Institute of Marine Biology, NAS Ukraine (IMB)

#### List of Participants:

	Name	Country	Affiliation	Contacts
1.	Gvarishvili Tsuri	Georgia	National Environmental Agency, Batumi (NEA)	ciuri-gvarishvili@rambler.ru
2.	Larisa Pautova	The Russian Federation	P.P. Shirshov Institute of Oceanology RAS, Moscow (SIO)	larisapautova@yahoo.com
3.	Olga Yasakova	The Russian Federation	Southern Scientific Center RAS, Rostov-on-Don (SSC)	yasak71@mail.ru
4.	Natalya Derezyuk	Ukraine	Odessa National University, Odessa (ONU)	n.derezyuk@onu.edu.ua
5.	Andrii Zotov	Ukraine	Institute of Marine Biology, Odessa (IMB)	zotovab@ukr.net
6.	Galyna Terenko	Ukraine	Ukrainian Scientific Center of Ecology Sea, Odessa (UkrSCES)	adlafia@mail.ru

#### Sampling Date:

Joss-UA-GE, St. 3, 27 m – 29.08.2017

Joss-UA-GE, St. 4, 30 m – 30.08.2017

Joss-UA-GE, St. 5, 30 m - 30.08.2017

#### Sampling Location:

Station	Lat, °N	Long, °E	Bottom depth, m
Joss-UA-GE, St. 3	44° 51'	31° 20'	62
Joss-UA-GE, St. 4	44° 06'	31° 34'	1165
Joss-UA-GE, St. 5	43° 24'	31° 50'	1919

**Number of samples passed to experts:**

	Expert Name	Country	Affiliation	Number of samples
1.	Gvarishvili Tsuri	Georgia	NEA	3
2.	Larisa Pautova	The Russian Federation	SIO	3
3.	Olga Yasakova	The Russian Federation	SSC	3
4.	Natalya Derezyuk	Ukraine	ONU	3
5.	Andrii Zotov	Ukraine	IMB	3
6.	Galyna Terenko	Ukraine	UkrSCES	3

**Features of phytoplankton samples processing:**

Laboratory	Sample concentration	Microscope type	Volume of condensed sample	Volume of subsample	Magnification
GE	Back filtration (to 80-120 ml) + sedimentation method (to 10-25 ml)	KRUSS, (inverted)	10–25 ml	0.05 ml 1.0 ml	200, 400
RF	Back filtration (to 80-120 ml) + sedimentation method (to 10-25 ml)	LOMO, Ergoval, (upright)	10–25 ml	0.05 ml, 1.0 ml	160, 400
UA	Back filtration (to 80-120 ml) + sedimentation method (to 10-25 ml)	LOMO, Ceiss (upright)	10–25 ml	0.05 ml, 0.1 ml	200, 400, 600

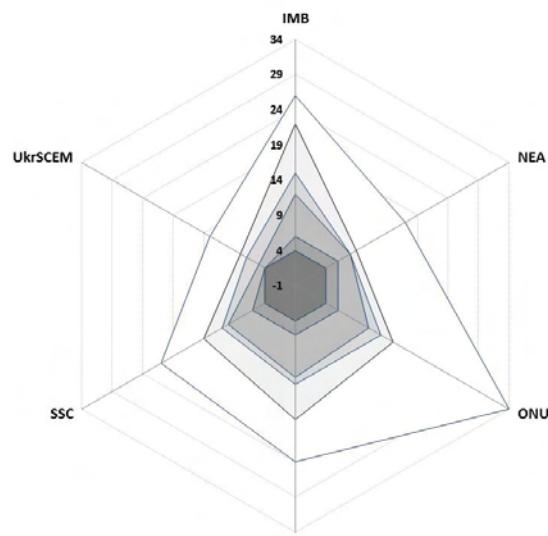
**Results of Intercomparison.**

After processing of the phytoplankton samples in the laboratories of national institutions, the experts provide the results prepared according to “Format Protocol Station” (Template) which has been approved during the practical study of Odessa Workshop in February 2016. Detailed data of processing are presented in: Annex 1 – Phytoplankton taxonomic comparison; Annex 2 – Phytoplankton sample analysis\_Joss\_St\_3; Annex 3 – Phytoplankton sample analysis\_Joss\_St\_4; Annex 4 – Phytoplankton sample analysis\_Joss\_St\_5.

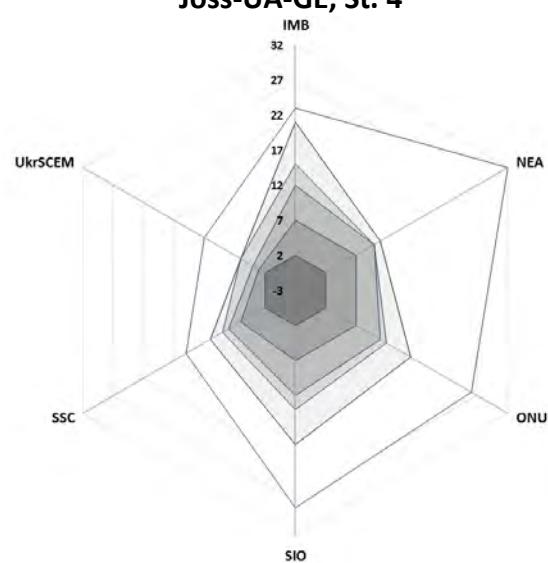
Comparison of the species lists identified by the experts that participated in intercorparision procedure is presented in Annex 1.

Fig. 1 shows the number of species that for each of the experts coincides with the taxonomic identification results of 2, 3, 4, 5 or all other participants. Each expert identified from 2 (St. 4, 5) to 4 (St. 3) of dominant phytoplankton species (Fig. 1). The number of mass species determined by at least four intercomparison participants was 12 (St. 3, 4) and 8 (St. 5) (Fig. 1). The total number of species identified by different experts for each of the samples differed by of 2.5 times an average. So, for the St. 3 the number of species varied from 13 (UkrSCES) to 34 (ONU); for the St. 4—from 12 (UkrSCES) to 32 (NEA) and for the St. 5 – from 14 (UkrSCES) to 32 (NEA) (Fig. 1). Thus, the results of the taxonomic composition identification are not characterized by significant differences in the number of coincidences in species lists of different participants.

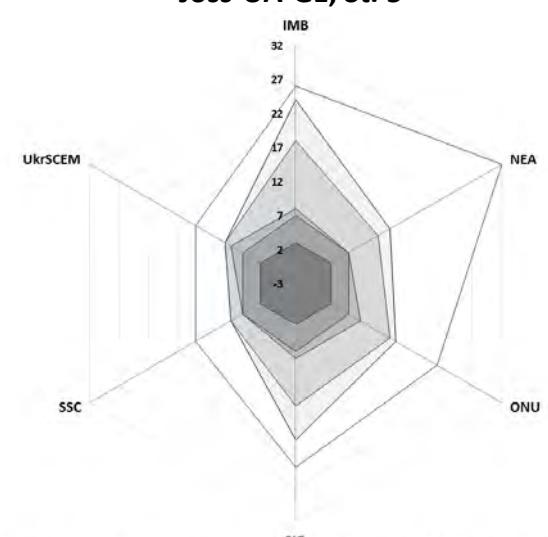
### Joss-UA-GE, St. 3



### Joss-UA-GE, St. 4



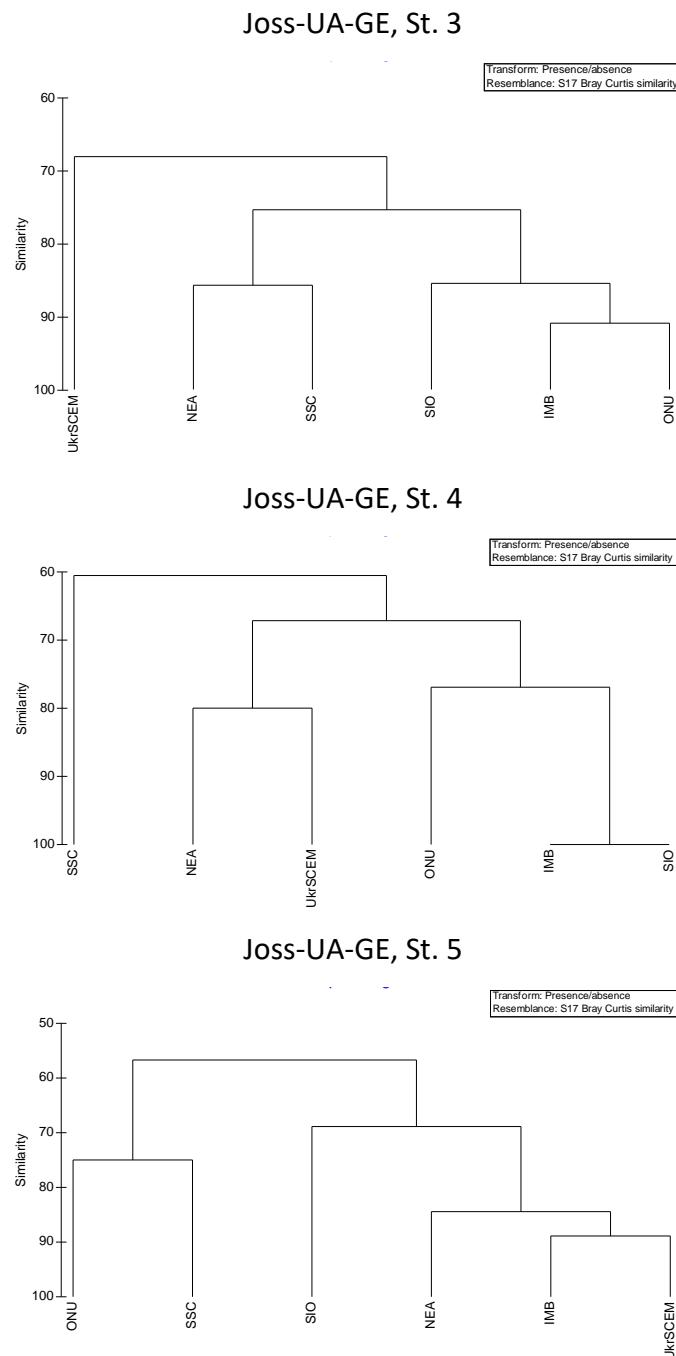
### Joss-UA-GE, St. 5



□ No of species    □ congruence, no less 2    □ no less 3    □ no less 4    □ no less 5    □ for 6

**Fig. 1. Concurrence of the taxonomic composition of phytoplankton for intercomparison samples by EMBLAS's partners.**

Analysis of the Bray-Curtis similarity for the taxonomic composition at the Class level showed higher results. A feature of the distribution of this Bray-Curtis similarity values for all three stations was the presence of a group of 5 (St. 3, 4) or 4 (St. 5) experts with a similarity level above 70% (Fig. 2). For other experts, this indicator varied from 57% (St. 5) to 67% (St. 3) (Fig. 2).

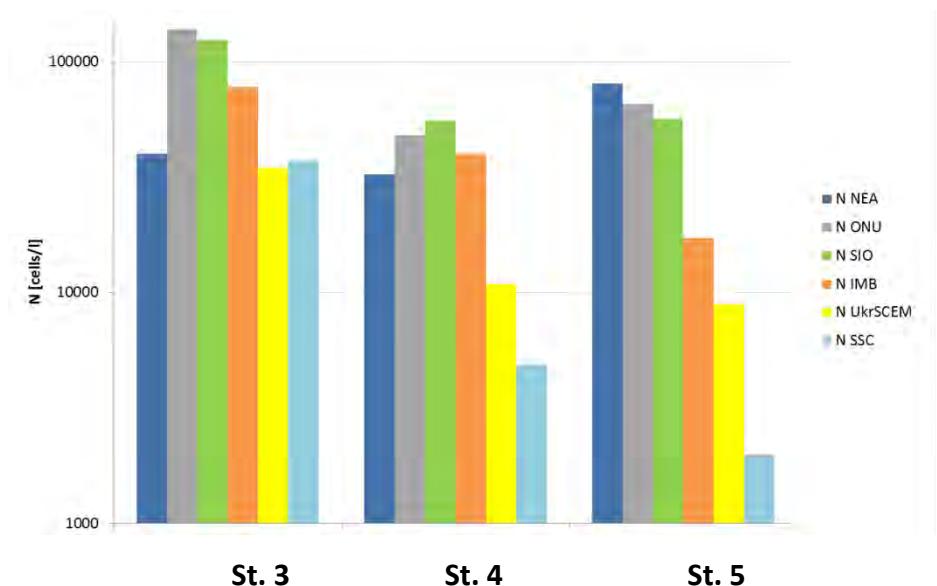


**Fig. 2. The Bray-Curtis similarity for the phytoplankton taxonomic composition at the Class level for intercomparison samples.**

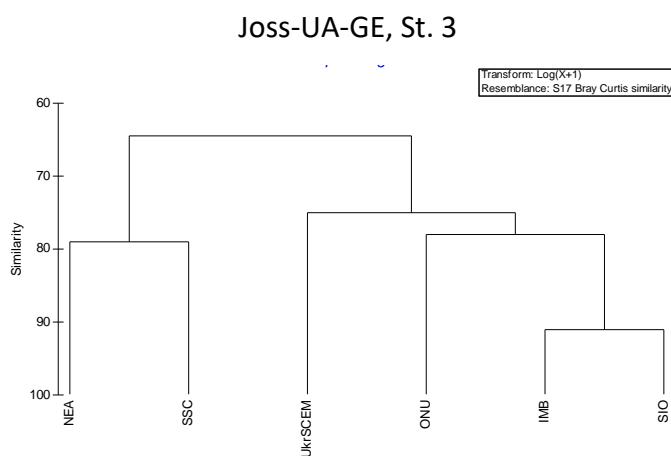
The total values of phytoplankton abundance, identified by experts, varied significantly for each of the stations. The maximum and minimum values of abundance differed 3.9 (St. 3), 11.4 (St. 4) and 40.4 (St. 5) times (Fig. 3). The spread of biomass values of phytoplankton at each of the three

stations was also high. The maximum and minimum value of biomass for each of the stations differ respectively 10.2, 15.8 and 15.3 times (Fig. 5).

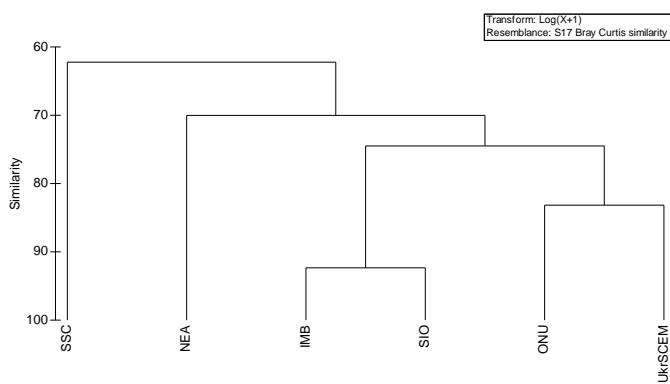
The analysis of the Bray-Curtis similarity for the abundance and biomass of taxonomic classes revealed a higher similarity (Fig. 4. 6). For both abundance and biomass, the Bray-Curtis similarity distribution for most participants was characterized by a similarity level above 70%.



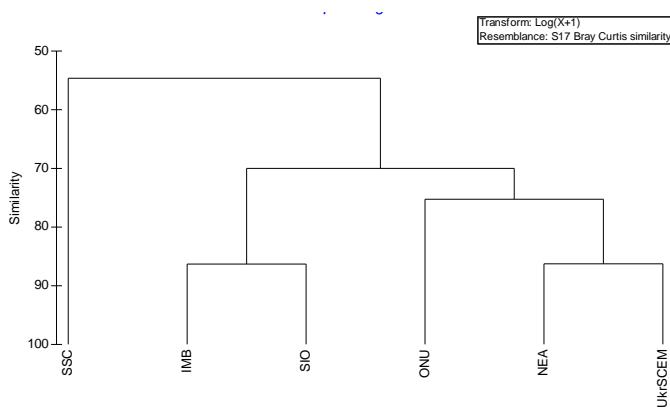
**Fig. 3. The phytoplankton abundance in the intercomparison samples for EMBLAS partners.**



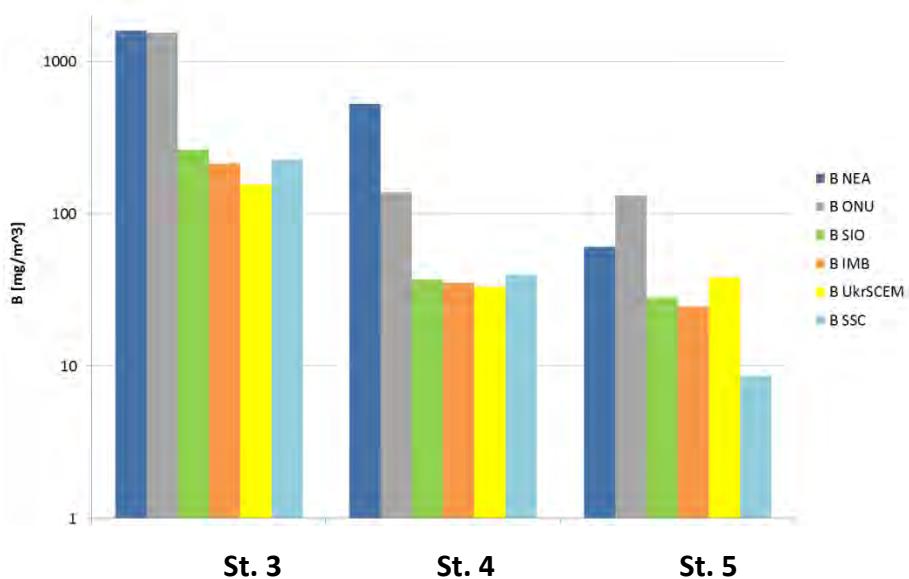
### Joss-UA-GE, St. 4



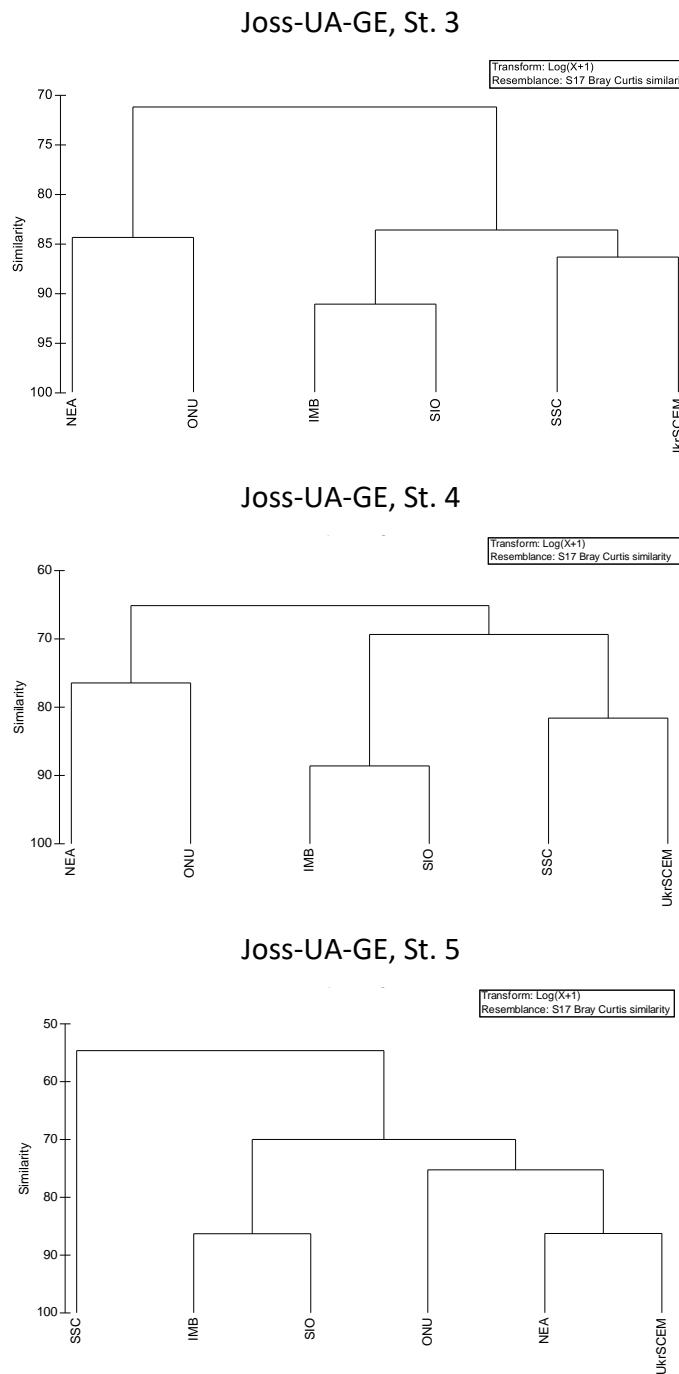
### Joss-UA-GE, St. 5



**Fig. 4. The Bray-Curtis similarity for the phytoplankton abundance at the Class level for intercomparison samples.**



**Fig. 5. The phytoplankton biomass in the intercomparison samples for EMLAS partners.**



**Fig. 6. The Bray-Curtis similarity for the phytoplankton biomass at the Class level for intercomparison samples.**

The minimum value of the Bray-Curtis similarity for both abundance and biomass of taxonomic classes was 55% (St. 5) (Fig. 6).

The differences in the values of biomass determined the discrepancies of the assessment of ecological status based on this index (Table 1). The status for St. 3, 4 и 5 for most of EMBLAS's partners was corresponded the «High» category. The exception was St. 3, where two out of six experts defined the ecological status as "Moderate" (Table 1).

**Table 1. Result of comparative analysis of the biomass for determination of the ESC correspond to intercomparison phytoplankton samples by EMBLAS's partners.**

EMBLAS's partner	Biomass, mg.m <sup>-3</sup>		
	St. 3	St. 4	St. 5
NEA (Ge)	Moderate	High	High
ONU (Uk)	Moderate	High	High
SIO (RF)	High	High	High
IMB (Uk)	High	High	High
UkrSCES (Uk)	High	High	High
SSC (RF)	High	High	High

Significant discrepancies in the quantitative results of sample processing required the use of an objective criterion for estimating these differences. In this regard, the experts results were compared not with the control result, but based deviation from the average value of the indicators ( $X_x$ ), calculated from the results of each expert ( $X_i$ ). To assessment of the correctness of the calculation of the indicator for each expert, the deviation ( $\Delta$ ) from the mean values abundance and biomass of phytoplankton was calculated. Deviation was calculated by formula:

$$\Delta = \frac{(X_i - X_x) * 100\%}{X_x}$$

This approach allows estimating the percentage of deviation in the quantitative indicators for each expert. Eligibility criteria were accepted for the values of deviation less than 100%. For all stations this criterion was consistent with the results of the 4 from the 6 experts participating in III Intercomparison of the phytoplankton samples processing (Table 2).

**Table 2. Evaluation of deviation in determining the values of quantitative indicators of III Intercomparison of phytoplankton samples by EMBLAS's partners.**

EMBLAS's partner	B [mg/m3]			N [cells/l]		
	St. 3	St. 4	St. 5	St. 3	St. 4	St. 5
NEA (Ge)	140	290	25	-47	2	109
ONU (Uk)	131	2	170	83	50	70
SIO (RF)	-61	-72	-42	65	74	47
IMB (Uk)	-68	-74	-49	3	25	-55
UKRSCIES (Uk)	-66	-71	-82	-51	-85	-95
SSC (RF)	-77	-75	-22	-54	-66	-77

### Conclusions:

Exercises of the III Intercomparison of phytoplankton samples consisted of two components:

*Identification of the taxonomic composition of phytoplankton samples.* Analysis the results of the taxonomic composition identification are not revealed significant differences in the number of coincidences in species lists of different participants.

*Calculation of phytoplankton indicators for the determination of Ecological Status Class.* To assess this component, the criterion of less than 100% of the deviation from the average value obtained by all experts was used. The inconsistency with this criterion in the estimation of quantitative indices for no less than two stations was considered as a negative result.

Estimation of the results obtained by experts during the two stages of the III Intercomparison of the phytoplankton showed the following:

EMBLAS's partner	B
NEA (Ge)	-
ONU (Uk)	-
SIO (RF)	+
IMB (Uk)	+
UkrSCES (Uk)	+
SSC (RF)	+

Thus, the following experts have obtained the positive results of the III Intercomparisen of phytoplankton exercises: Galyna Terenko (UkrSCES, Ukraine), Andrii Zotov (IMB, Ukraine), Larisa Pautova (SIO, The Russian Federation) Olga Yasakova (The Russian Federation)

Responsible person  
of the phytoplankton Intercomparison:

Andrii Zotov

25-04-2018

Odessa

## Annex 2: Zooplankton intercomparison

Boris Alexandrov - Institute of Marine Biology, National Academy of Sciences of Ukraine

The main objective of this exercise was to collect samples for biological parameters by the partners of the EMBLAS, following their routine methodology of sampling and analysis for assessment of the comparability of data collected during the expedition to the R/V "Mare Nigrum" (27 August - 30 September 2017). The aim of the exercise was to compare the qualitative and quantitative composition of the mesozooplankton as the result of processing the samples, as well as assessing the quality of the marine environment on biological indicators. These exercises are expected to produce valuable results for making recommendations for further improvement and harmonization of research (monitoring) methodology in the Black Sea. In intercomparison exercise III on mesozooplankton were participated all scientific organizations that took part in EMBLAS-II Project (Table 1) in contrast to intercomparison exercise I and II (were organized in 2016) in which the representatives of the Russian Federation did not participate.

**Table 1. List of Participants of intercomparison exercise III on mesozooplankton**

	Name	Country	Affiliation	Contacts
1	Marina Mgelandze	Georgia	National Environmental Agency, Batumi (NEA)	mari.mgelandze@gmail.com
2	Aleksandr Korshenko	Russian Federation	State Oceanographic Institute, Moscow (SOI)	korshenko58@mail.ru
3	Tamara Shiganova	Russian Federation	P.P. Shirshov Institute of Oceanology RAS (IO)	shiganov@ocean.ru
4	Mikhail Nabokin	Ukraine	Ukrainian Scientific Center of Ecology of Sea, Odesa (USC)	miobiusa@lenta.ru
5	Pavel Lumkis	Ukraine	I.I. Mechnikov Odesa National University (ONU)	pasha.lumkis@gmail.com
6	Vasiliy Dyadichko	Ukraine	Institute of Marine Biology, Odesa (IMB)	wasilij_d@mail.ru

To perform the exercise, each participant was given a set of three samples fixed by neutralized formaldehyde. Each initial sample was divided into 8 equal parts using the Motoda Plankton Sample Divider. All samples were taken during. Characteristics of samples see in Table 2.

**Table 2. Characteristics of mesozooplankton samples participating in Intercomparison exercise III**

No Station	Data,	Coordinates		*Haul horizon, m	Volume of fil- tering water, m <sup>3</sup>
		Day/Month	Latitude	Longitude	
2	29/08	43° 13' N	31° 14' E	0-17	3.461
3	29/08	44° 51' N	31° 20' E	0-20	4.071
6	31/08	43° 25' N	32° 52' E	0-14	2.850

\*Two vertical hauls were carried out using the Judy net (mouth diameter 36 cm, mesh size 150 µm) at each station.

## Results of intercomparison

Analysis of the data showed that sample processing in 2017 had more good results, than in previous intercomporison exercise II in 2016. The main differences between intercomparison exercises that were conducted in 2016 and in 2017 were the increase in the amplitude of the spread of the values of the qualitative composition of the plankton, but significantly less deviation of quantitative characteristics as total abundance and biomass. Even though the biological diversity of the samples collected in 2017 was higher than in 2016 (30-33 taxa and 17-26 respectively), and the average number of registered species was also higher (13 and 19 respectively), the variation of values at the processing of samples significantly increased with an increase in the number of specialists from 4 to 6. If the average percentage of dominant species founded by experts from different organizations in 2016 was 3-7, then in 2017 the variation increased to 1-53%. Increasing the number of experts who analysed similar samples did not significantly reduce the percentage of species found by all of them. In intercomparison exercises III and II, the percentage of total species was  $29.7 \pm 2.2$  and  $32.7 \pm 8.0\%$  respectively (Annex. Results of intercomparison exercises for biota: Zooplankton, Table 3 in “Mare Nigrum” Report on 2016 and 2017).

**Table 3. Results of intercomparison exercises III by identification of species composition of zooplankton at all investigated samples**

NN	Taxon (Species)	NEA	SOI	IO	USC	IMB	ONU
Sample 2							
1	<i>Noctiluca scintillans</i> Kofoid & Swezy, 1921				+		+
2	<i>Ceratium tripos</i> (O.F.Müller) Nitzsch				+		
3	<i>Aurelia aurita</i> (Linnaeus, 1758)	+					
4	<i>Beroe ovata</i> , Bruguière, 1789			+	+		
5	Ctenophora, larvae	+					
6	Hydrozoa sp.		+	+			
7	Rotatoria gen.sp.		+				+
8	Copepod g.sp., ova			+			
9	Copepod g.sp., nauplii	+	+	+	+	+	+
10	<i>Acartia clausi</i> Giesbrecht, 1889	+	+	+	+	+	+
11	<i>Acartia</i> sp. (clausi+tonsa)	+			+	+	+
12	<i>Centropages ponticus</i> Karavaev, 1894	+	+	+	+	+	+
13	<i>Paracalanus parvus</i> (Claus, 1863)	+	+	+	+	+	+
14	<i>Pseudocalanus elongatus</i> (Boeck, 1865)			+			
15	<i>Oithona davisi</i> (Ferrari F.D. and Orsi, 1984)	+	+	+	+	+	+
16	<i>Oithona similis</i> , Claus, 1866	+		+			+
17	<i>Evadne spinifera</i> P.E. Muller, 1867	+					
18	<i>Penilia avirostris</i> Dana, 1849	+	+	+	+	+	+
19	<i>Pleopis polyphaemoides</i> (Leucart, 1859)	+	+	+	+		
20	<i>Podonevadne trigona</i> (G.O. Sars, 1897)					+	
21	<i>Pseudoevadne tergestina</i> Claus, 1864	+	+	+	+		+

NN	Taxon (Species)	NEA	SOI	IO	USC	IMB	ONU
22	<i>Parasagitta setosa</i> (Müller, 1847), ova			+			
23	<i>Parasagitta setosa</i> (Müller, 1847), adultis	+	+	+	+	+	+
24	<i>Oikopleura (Vexillaria) dioica</i> Fol, 1872	+	+	+	+	+	+
25	Bivalvia g. sp., larvae	+	+	+	+	+	+
26	Cirripedia, nauplii			+	+		
27	Gastropoda g. sp., larvae			+	+		
28	Polychaeta g. sp., larvae	+	+	+	+	+	+
29	Pisces sp., ova	+	+	+			
30	Pisces sp., larvae			+			
31	Nematoda sp.					+	
32	Ostracoda sp.		+				
33	Doliolidae ???		+				
	TOTALLY	18	17	22	19	13	15

## Sample 3

1	<i>Noctiluca scintillans</i> Kofoid & Swezy, 1921	+	+	+	+		+
2	<i>Ceratium tripos</i> (O.F.Müller) Nitzsch				+		
3	<i>Aurelia aurita</i> (Linnaeus, 1758)	+					
4	<i>Beroe ovata</i> , Bruguière, 1789, ova			+			
5	<i>Beroe ovata</i> , Bruguière, 1789, larvae	+		+	+		
6	<i>Pleurobrachia pileus</i> (O. F. Müller, 1776)	+					
7	Hydrozoa sp.			+	+		
8	Rotatoria gen.sp.		+				+
9	Copepod g.sp., ova			+			
10	Copepod g.sp., nauplii	+	+	+	+		+
11	<i>Acartia clausi</i> Giesbrecht, 1889	+	+	+	+	+	+
12	<i>Acartia</i> sp. (clausi+tonsa)	+			+	+	+
13	<i>Centropages ponticus</i> Karavaev, 1894	+	+	+	+	+	+
14	<i>Paracalanus parvus</i> (Claus, 1863)	+	+	+	+	+	+
15	<i>Pseudocalanus elongatus</i> (Boeck, 1865)			+			
16	<i>Oithona davisae</i> (Ferrari F.D. and Orsi, 1984)	+	+	+	+	+	+
17	<i>Oithona similis</i> , Claus, 1866	+		+			+
18	<i>Evadne spinifera</i> P.E. Muller, 1867	+		+			+
19	<i>Penilia avirostris</i> Dana, 1849	+	+	+	+	+	+
20	<i>Pleopis polyphaemoides</i> (Leucart, 1859)	+	+	+	+		
21	<i>Podonevadne trigona</i> (G.O. Sars, 1897)					+	
22	<i>Pseudoevadne tergestina</i> Claus, 1864	+	+	+	+		+
23	<i>Parasagitta setosa</i> (Müller, 1847), ova			+			
24	<i>Parasagitta setosa</i> (Müller, 1847), adultis	+	+	+	+	+	+
25	<i>Oikopleura (Vexillaria) dioica</i> Fol, 1872	+	+	+	+	+	+
26	Bivalvia g. sp., larvae	+	+	+	+		+
27	Cirripedia, nauplii (cyparis)			+	+	+	
28	Gastropoda g. sp., larvae		+	+	+		
29	Polychaeta g. sp., larvae	+	+	+	+	+	+

NN	Taxon (Species)	NEA	SOI	IO	USC	IMB	ONU
30	Pisces sp., ova	+					
31	Pisces sp., larvae			+			
	TOTALLY	20	15	24	19	11	16
Sample 6							
1	<i>Noctiluca scintillans</i> Kofoid & Swezy, 1921	+	+	+	+	+	+
2	<i>Ceratium tripos</i> (O.F.Müller) Nitzsch				+		
3	<i>Beroe ovata</i> , Bruguière, 1789, ova			+			
4	<i>Beroe ovata</i> , Bruguière, 1789, larvae			+	+		
5	<i>Pleurobrachia pileus</i> (O. F. Müller, 1776)	+					
6	Hydrozoa sp.				+		
7	Rotatoria gen.sp.		+				+
8	Copepoda g. sp., ova			+			
9	Copepoda g. sp., nauplii	+	+	+	+	+	+
10	<i>Acartia clausi</i> Giesbrecht, 1889	+	+	+	+	+	+
11	<i>Acartia</i> sp. (clausi+tonsa)	+	+		+	+	+
12	<i>Calanus euxinus</i> Hulsemann, 1991	+	+	+	+	+	
13	<i>Centropages ponticus</i> Karavaev, 1894	+	+	+	+	+	+
14	<i>Paracalanus parvus</i> (Claus, 1863)	+	+	+	+	+	+
15	<i>Pseudocalanus elongatus</i> (Boeck, 1865)			+			
16	Cyclopoida sp.					+	
17	<i>Oithona davisiæ</i> (Ferrari F.D. and Orsi, 1984)	+	+	+	+	+	+
18	<i>Oithona similis</i> , Claus, 1866	+		+			+
19	<i>Evadne spinifera</i> P.E. Muller, 1867						+
20	<i>Penilia avirostris</i> Dana, 1849			+	+		+
21	<i>Pleopis polyphaemoides</i> (Leucart, 1859)	+		+	+	+	
22	<i>Pseudoevadne tergestina</i> Claus, 1864	+	+	+	+		+
23	<i>Parasagitta setosa</i> (Müller, 1847), ova			+			
24	<i>Parasagitta setosa</i> (Müller, 1847), adultis	+	+	+	+	+	+
25	<i>Oikopleura dioca</i> Fol, 1872	+	+	+	+	+	+
26	Bivalvia g. sp., larvae	+		+	+	+	+
27	Cirripedia, nauplii				+		
28	Gastropoda g. sp., larvae				+		
29	Polychaeta g. sp., larvae	+	+	+	+	+	+
30	Pisces sp., ova		+				
	TOTALLY	16	14	20	20	14	16

Dominant species marked in grey (it was assumed that the number of mass species is  $\geq 2\%$  of the total number of zooplankton)

For convenience in assessing the quality of the marine environment at the stations under analysis, the threshold values are given in Table 4.

**Table 4. Normative value of zooplankton metrics (characteristics) for different kind of marine water quality in shelf area**

Characteristics (mesozooplankton metrics)	Numerical value	Water quality
MSFD variant (two-point evaluation system)		
Total mesozooplankton biomass (mg·m <sup>-3</sup> )	280-550	GES
	< 280	LES
Copepods biomass (% to total mesozooplankton)	> 42	GES
	≤ 42	LES
<i>Noctiluca</i> biomass (% to total mesozooplankton)	< 30	GES
	≥ 30	LES
Shannon-Weaver index (bit·ind <sup>-1</sup> )	≥ 3 (2,5)*	GES
	< 3 (2,5)*	LES
WFD variant (five-point rating system)		
Total mesozooplankton biomass, mg·m <sup>-3</sup> **		
Spring	> 300	High
	300-150	Good
	150-70	Medium
	70-10	Poor
	< 10	Bad
Summer	> 600	High
	600-350	Good
	350-200	Medium
	200-40	Poor
	< 40	Bad
Autumn	> 250	High
	250-150	Good
	150-70	Medium
	70-10	Poor
	< 10	Bad

Note:

GES – Good Environment Status

LES – Low Environment Status

\* Significance of index for open sea areas in brackets

\*\* According Moncheva, Boicenco, 2014 and Stefanova et al., 2015.

Despite the error made by the majority of experts because of inattention (not taking into account the division of the original sample into 8 equal parts), it can not practically repeat itself when performing self-monitoring. Seawater quality assessment of 6 experts was close and only at station 6 its assessment differed within two points in the WFD system (Table 5). The scatter in the values of the quantitative characteristics of mesozooplankton was insignificant (Table 6), and in comparison with the results of the exercise of 2016 it turned out to be almost two times lower (Table 7). In order to compare the obtained results, as well as to analyze the inaccuracies

detected (highlighted in red), the Excel file is presented with the results of the processing of samples of all the experts who participated in the exercise.

**Table 5. Comparative characteristics of water quality on the base of zooplankton indicators**

Indicators of water quality	NEA	SOI	IO	USC	IMB	ONU
Station 2						
Zooplankton abundance, ind·m <sup>-3</sup>	24157 3020	16090	15360 1920	11357	16392 2049	14832 1854
Zooplankton total biomass, mg·m <sup>-3</sup>	786,9 98,4	475,4	172,4 21,5	168,9	644,8 80,6	342,4 42,8
Copepoda biomass, %	20,6	27,5	5,3	40,3	20,8	38,7
Noctiluca biomass, %	0,0	0,0	0,0	0,0	0,0	1,7
Biodiversity by Shannon indices, bit·ind <sup>-1</sup>	2,678	1,738	2,693	1,931	2,790	2,890
Water quality status (MSFD)	LES	LES	LES	LES	LES	LES
Water quality status (WFD)	High Medium	High Good	Good Good	Good Good	High Poor	High Poor
Station 3						
Zooplankton abundance, ind·m <sup>-3</sup>	24580 3072	25730	30978 3872	16598	23736 2967	15192 1899
Zooplankton total biomass, mg·m <sup>-3</sup>	693,9 86,7	393,9	252,3 31,5	254,1	762,4 95,3	278,4 34,8
Copepoda biomass, %	20,7	49,4	63,8	33,2	25,1	47,8
Noctiluca biomass, %	0,1	0,1	0,5	0,0	0,0	2,0
Biodiversity by Shannon indices, bit·ind <sup>-1</sup>	2,794	1,670	2,603	1,995	3,538	3,059
Water quality status (MSFD)	LES	LES	LES	LES	LES	LES
Water quality status (WFD)	High Medium	High Good	Good Good	Good Good	High Poor	Good Bad
Station 6						
Zooplankton abundance, ind·m <sup>-3</sup>	19856 2482	18692	22318 2790	14564	26112 3264	10160 1270
Zooplankton total biomass, mg·m <sup>-3</sup>	278,6 34,8	167,5	170,4 21,3	129,7	877,6 109,7	85,6 10,7
Copepoda biomass, %	49,6	51,1	72,7	85,2	96,4	65,0
Noctiluca biomass, %	2,5	3,0	17,3	1,6	1,6	5,9
Biodiversity by Shannon indices, bit·ind <sup>-1</sup>	2,522	1,762	2,830	1,849	3,612	2,608
Water quality status (MSFD)	LES	LES	LES	LES	GES	LES
Water quality status (WFD)	Good Poor	Good	Good	Medium	High Poor	Medium Bad

Note. **NEA** - National Environmental Agency of Fishery and Black Sea monitoring (Georgia); **IO** – P.P.Shirshov Institute of Oceanology RAS, **SOI** – State Oceanographic Institute (Russia); **USC** – Ukrainian Scientific Center of Ecology of the Sea, **IMB** – Institute of Marine Biology NASU, **ONU** – Odessa national university I.I. Mechnikov (Ukraine).

Underlined values calculated incorrectly. Main part of participants of intercomparison exercise forgot that each of the three general (initial) samples were divided into 8 equal parts with the help of the Motoda divisor. Incorrectly calculated values including Water Quality are marked in red.

**Table 6. The average values of some mesozooplankton characteristics in the control stations (calculated according data in Table 5)**

№ station	Abundance		Biomass	
	N, ind·m <sup>-3</sup>	Average error, %	B, mg·m <sup>-3</sup>	Average error, %
2	16365 ± 1725	10,5	431,8 ± 102,9	23,8
3	22802 ± 2422	10,6	439,2 ± 94,3	21,5
6	18617 ± 2305	12,4	284,9 ± 121,4	42,6
Average	11,2 ± 0,6			29,3 ± 6,7

**Table 7. Average deviation of the results of total mesozooplankton abundance and biomass determination by all participants in control samples of the intercomparison exercise II (2016) and III (2017)**

Intercomparison exercise	Abundance, %	Biomass, %
II (2016)	42,1 ± 1,8	39,6 ± 5,4
III (2017)	11,2 ± 0,6	29,3 ± 6,7

### Remarks

Most participants in the exercise (4 of 6) did not take into account that the original sample was divided into 8 equal parts. Therefore, the total biomass of mesozooplankton has been underestimated by eight times, which has led to a significant underestimation of the quality of sea water (Table 5). Practically all the participants of the exercise did not pay attention to the coordinates of the stations under study and made a mistake in assessing the water quality according to the Shannon index, since station number 6 was in the open sea and required a different threshold value. Finally, the last omission of participants in the exercise was also associated with inattention. One of the key indicators in determining the quality of sea water is the total biomass of mesozooplankton, and according to the WFD criteria it is assessed on a five-point scale. Unlike in 2016, the R / V "Mare Nigrum" flight did not pass in spring, but in summer, so it was correct to use the scale of values for the summer period of the year (Moncheva, Boicenco, 2014).

NEA (Marina Mgelandze) - Used an inaccurate formula to find the Shannon index, the values of which were found correctly, but had a negative value.

SOI (Aleksandr Korshenko) - Values of water quality assessment for total biomass for all three samples are underestimated by one point from the five-point WFD assessment system (see Table 4). Representatives of Doliolidae in the Black Sea have never been registered.

IO (Tamara Shiganova) - Unfortunately, after processing all three samples of zooplankton, the author did not present the results of the water quality assessment, which was made by B. Alexandrov in the result of the processing the mesozooplankton samples. During determination the species composition of the samples, only this expert identified the presence of copepoda *Pseudocalanus elongatus*, and as the dominant species. In addition, no one other than this author has registered a high number of the *Parasagitta setosa* eggs in the samples.

USC (Mikhail Nabokin) - It is not accepted to register the *Ceratium tripos* in zooplankton samples, since this species belongs to peredinium algae and the overall result of determining the number and biomass of zooplankton can be distorted. The only exception to this rule is the consideration of the colorless flagellate *Noctiluca scintillans*, which also refers to peridinia algae.

ONU (Pavel Lumkis) - Only this expert from the other one used the template, which was not developed by the USC specialists, but its own system of counting the results («калькулятор Евгения Газетова»). But this system was based on the same calculation formulas and produced good results. The results of the quantitative processing of the samples are very close to the results of other participants in the exercise, both in determining the abundance and biomass of mesozooplankton.

IMB (Vasiliy Dyadichko) - used an inaccurate formula to find the Shannon index, the values of which were found correctly, but had a negative value.

Despite the comments made on the whole, the results obtained showed little variability in assessing the species composition, abundance and biomass of mesozooplankton, as well as high similarity in assessing the quality of marine water.

## Conclusions and recommendations

1. All experts that took part in intercomparison exercise III have good results and they have good qualification in biological monitoring of marine water on the base of mesozooplankton investigations.
2. When quantitatively taking into account mesozooplankton, it is recommended not to take into account bottom invertebrates, which happened to be in a plankton sample, for example, Nematoda, Ostracoda, Harpacticoida, Amphipoda, Isopoda. Do not take into account the large species of phytoplankton, except *Noctiluca scintillans*.
3. For a more accurate assessment of the quality of sea water, develop national scales with threshold values for different seasons of the year, for open, coastal and transit waters. In addition, combine the various characteristics of zooplankton in the integral index.
4. In connection with the complexities of some dominant mesozooplankton species identification that belong to native and non-indigenous species (for example *Acartia clausa*, *A. tonsa*, *Oithona similis*, *O. davisae*) it is not recommended to use the percentage of exotic species from the total number of native species as an indicator of the quality of marine waters. More perspective to use Biopollution level index (Olenin et al., 2007) as indicator of water quality. This indicator combine all non-indigenous species that registered in pelagic and benthic communities.
5. The processing of mesozooplankton samples and the creation of a database on this biological indicator using the template, which was developed by the USC, showed good results. Bad side of this template, which can be recommended for monitoring the marine ecosystems of the Black Sea, is the existence of two parallel methods for determining the biomass of zooplankton (a key indicator of water quality): the determination of biomass based on standard weights and on measuring the size of aquatic invertebrates. The differences in the total biomass of zooplankton, calculated by these methods, averages  $52\pm7\%$  (see Excel annexes of A. Korshenko and M. Nabokin), which may cause differences in the assessment of water quality. To eliminate this defect, remove from the template in the calculation of biomass by standard weights, as an old and not precise method.

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## Annex 3: Macrophytobentos Intercomparison

### Results of intercomparing exercises for biota: Macrophytes

Responsible person: Galina Minicheva - Institute Marine Biology, NAS Ukraine (IMB)

#### **III Intercomparison results of the macrophytobenthos samples processing**

*April 2018, Odessa*

#### **List of Participants:**

	Name	Country	Affiliation	Contacts
1.	Miriam Tsetskhladze	Georgia	National Environmental Agency, Batumi (NEA)	m-marie@mail.ru
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3.	Irina Tretyak	Ukraine	Ukrainian Scientific Centre of Ecology of the Sea, Odessa (UkrSCES)	<a href="mailto:iratretiak9@gmail.com">iratretiak9@gmail.com</a> <a href="mailto:iratretiak9@ukr.net">iratretiak9@ukr.net</a>
4.	Fedor Tkachenko	Ukraine	Odessa National University, Odessa (ONU)	<a href="mailto:tvf@ukr.net">tvf@ukr.net</a>
5.	Anna Marinets	Ukraine	Institute of Marine Biology, Odessa (IMB)	<a href="mailto:samoylenko_anna@ukr.net">samoylenko_anna@ukr.net</a>

#### Sampling Data:

5 September 2017

#### Sampling Location:

Georgia coast, Adjara region, “Green Cape” polygon

#### Sampling Conditions:

Water temperature – 21,8 C°. Salinity – 17‰. Substrate -nature rocks and stones. Depth – 5,5 m. Cover of substrate by macrophytes communities – 10-20 %. Size of frame – (10x10) cm.

#### Value of samples passed to experts:

	Expert Name	Country	Affiliation	Number of samples
1.	Miriyam Tsetskhladze	Georgia	NEA	3
2.	Dmitry Afanasyev	The Russian Federation	SFU_DB	3
3.	Irina Tretyak	Ukraine	UkrSCES	3
4.	Fedor Tkachenko	Ukraine	ONU	3
5.	Anna Marinets	Ukraine	IMB	3

## Results of Intercomparison

After processing of the macrophyte samples in the laboratories of national institutions, the experts provide of the results correspond to “Format Protocol Station” (Template) which is approved on the practical study of the Odessa Workshop in February 2016 and with use Data Set Template which is approved in May 2017. Data of processing presented: Annex 1 – (Miriyam Tsetskhadze, NEA); Annex 2 – (Dmitry Afanasyev, SFU\_DB); Annex 3 – (Irina Tretyak, UkrSCES); Annex 4 – (Fedor Tkachenko, ONU); Annex 5 – (Anna Marinets, IMB).

The results of the determination of the floristic composition for sampels macrophytobenthos of III Intercomparison exercises by the EMBLAS's partners are presented in Tab. 1. Each of the partner participants identified from 5 to 8 species which are related to macrophytes and fouling microalgae.

**Table 1. Result to determination of the floristic composition of III Intercomparison macrophytobenthos samples (Batumi coast, “Green Cap”, 05.10.2011) by the EMBLAS's partners.**

Species	EMBLAS's partner					Number of identifications
	IMB (UA)	ONU (UA)	UkrSCES (UA)	SFU_DB (RF)	NEA (GE)	
<i>Chlorophyta</i>						
<i>Ulva intestinalis</i> (Linnaeus) Nees = <i>Enteromorpha intestinalis</i> (L.) Link.	-	+	-	-	-	1
<i>Ulva compressa</i> L.	-	+	-	-	-	1
<i>Cladophora albida</i> (Huds.) Kutz.	-	+	-	-	-	1
<i>C. laetevirens</i> (Dillw.) Kütz.	+	+	+	+	+	5
<i>Rhodophyta</i>						
<i>Gelidium crinale</i> (Hare ex Turner) Gaillon	+	-	+	+	+	4
<i>Stylonema alsidii</i> (Zanard.) K.M. Drew	+	+	+	+	+	5
<i>Acrochaetium hallanicum</i> f. <i>parvulum</i> (Kylin) Rosenvinge	-	+	-	-	-	1
<i>Acrochaetium secundatum</i> (Lyngb.) Nägeli	+	-	+	+	+	5
<i>Ceramium virgatum</i> Roth	+	+	+	+	+	5
<i>Ceramium diaphanum</i> var. <i>elegans</i> (Roth) Roth = <i>Ceramium elegans</i> Ducl.	-	-	+	-	-	1
<i>Ochrophyta</i>						
<i>Stilophora tenella</i> (Esper) P.C. Silva	-	+	-	-	-	1
Total	5	8	6	5	5	

 Mass species

The Frequency of occurrence (P, %) of algae species in the samples is a characteristic its role in the structure of the phytocenoses. Species with 100% occurrence are the dominant elements of the communities and has highly probability to be present in all samples. If the participant has determined a species that is not found in the samples of other experts and this species has 100% occurrence, existent high probability that this species is not determined correctly. If species was determinate only in one sample and has a low occurrence (33%), this may be result from the heterogeneous distribution of algae in the biotope. An example of a possible incorrect definition can be the following species: *Stilophora tenella* (Esper) P.C. Silva – 1 identification, P - 100% (ONU's expert); *Acrochaetium hallanicum f. parvulum* (Kylin) Rosenvinge – 1 identification, P - 66,6 % (ONU's expert) (Tab.2.).

**Table 2. Result to determination of the Frequency of occurrence (P, %) for floristic composition of intercomparison macrophytobenthos samples (Batumi coast, “Green Cap”, 05.10.201) by the EMBLAS’s partners.**

Species	EMBLAS's partner				
	IMB (UA)	ONU (UA)	UkrSCES (UA)	SFU_DB (RF)	NEA (Ge)
<i>Ulva intestinalis</i> (Linnaeus) Nees = <i>Enteromorpha intestinalis</i> (L.) Link.	-	33,3	-	-	-
<i>Ulva compressa</i> L.	-	33,3	-	-	-
<i>Cladophora albida</i> (Huds.) Kutz.	-	33,3	-	-	-
<i>C. laetevirens</i> (Dillw.) Kütz.	33,3	33,3	100	66	100
<i>Gelidium crinale</i> (Hare ex Turner) Gaillon	100	-	100	100	100
<i>Stilonema alsidii</i> (Zanard.) K.M. Drew	100	66,6	100	66	100
<i>Acrochaetium hallanicum f. parvulum</i> (Kylin) Rosenvinge	-	66,6	-	-	-
<i>Acrochaetium secundatum</i> (Lyngb.) Nägeli	100	-	100	100	100
<i>Ceramium virgatum</i> Roth	100	100	100	66	100
<i>Ceramium diaphanum</i> var. <i>elegans</i> (Roth) Roth = <i>Ceramium elegans</i> Ducl.	-	-	33,3	-	-
<i>Stilophora tenella</i> (Esper) P.C. Silva	-	100	-	-	-
Total	5	8	6	5	5

For evaluation the stage of determining the floristics composition of macrophytobenthos samples of the III Intercomparisen exercise, the main criterion was the correct determination of three dominant species. When determining the floristics composition, this indicator is very important, because the values of the specific surface of the three dominants it is necessary to calculate the indicator  $S/W_{3D}$  – on basis it should determinate the Ecological Status Class (ESC). According to criterion - the correct identification of the three dominants of the community, the 4 experts from the 5 experts participating in III Intercomparisen exercise were well consulted (Tab. 3).

**Table 3. Evaluation of the correctness of the identification of floristics composition of III Intercomparison macrophytes samples by EMBLAS's partners.**

EMBLAS's partner	Total number species identified by expert	Common species identified by all experts	Single species identified only by one expert	Identification of dominant species			Evaluation results
				<i>Gelidium crinale</i>	<i>Cladophora laetevirens</i>	<i>Ceramium virgatum</i>	
NEA (GE)	5	5	-	+	+	+	Good
SFU_DB (RF)	5	5	-	+	+	+	Good
UkrSCES (UA)	6	5	1	+	+	+	Good
ONU (UA)	8	3	5	-	+	+	Bad
IMB (UA)	5	5	-	+	+	+	Good

Summaries comparative analysis of the results of calculating the values of the main macrophyte indicators and determining the categories of the Ecological Status Class obtained by experts (see Annex1-5) on their basis is presented in Table 4.

**Table 4. Result of comparative analysis of the main indicators for determining of the ESC of III Intercomparison macrophytes samples (Batumi coast, “Green Cap”, 05.10.2011) by EMBLAS's partners.**

EMBLAS's partner	Biomass of over growth, kg.m <sup>-2</sup>	Ecological Evaluation Index					
		S/W <sub>3DP</sub> , m <sup>2</sup> .kg <sup>-1</sup>	ESC	S/W <sub>x</sub> , m <sup>2</sup> .kg <sup>-1</sup>	ESC	Slph, units	ESC
NEA (GE)	0,182	30,8	Moderate	146,0	Poor	3,53	High
SFU_DB (RF)	0,202	42,6	Moderate	135,8	Poor	3,99	High
UkrSCES (UA)	0,207	34,4	Moderate	137,6	Poor	3,62	High
ONU (UA)	0,273	44,2	Moderate	106,3	Moderate	8,73	High
IMB (UA)	0,149	31,98	Moderate	139,4	Poor	3,28	High

Since each of the experts processed the individual samples which selected on the monosensuous habitat, the results could be consist the natural spatial different quality of macrophytobenthos distribution. For this reason, the results were compared, not with the control samples, but with the average value of the indicators ( $X_x$ ), calculated from the results of each expert ( $X_i$ ). To assess the correctness of the calculation of the indicator for each expert, the deviation ( $\Delta$ ) from the mean values characteristic of macrophytes monocenosis was calculated. Deviation was calculated by formula:

$$\Delta = \frac{(X_i - X_x) * 100\%}{X_x}$$

The adopted approach allows estimating the percentage of deviation in the calculated indicators for each expert. Criterion of qualification used the criterion value of deviation less than 30%. According to this criterion the 4 experts from the 5 experts participating in III Intercomparisen Macrophytobenthos exercise were well consulted (Tab. 5).

**Table 5. Evaluation of the correctness of determining the values of ESC-indicators of III Intercomparison macrophytes samples by EMBLAS's partners.**

EMBLAS's partner	Deviation from average value of indicators, %				Average deviation for all indicators, %	Evaluation results
	Biomass	S/W <sub>3Dp</sub>	S/Wx	SI <sub>ph</sub>		
NEA (GE)	- 10,4	-16,1	+ 9,7	-16,7	13,2	Good
SFU_DB (RF)	- 0,5	+ 16,0	+2,1	- 13,2	8,0	Good
UkrSCES (UA)	- 1,9	- 6,3	- 3,0	- 21,3	8,2	Good
ONU (UA)	+ 34,5	+20,4	+ 25,0	+ 89,7	42,4	Bad
IMB (UA)	- 27,1	- 12,6	+ 19,6	- 28,6	21,9	Good

### Conclusions:

1. Exercises of the III Intercomparisen of Macrophytobenthos consisted the two stages:
  - I. Identification of the floristic composition of the macrophytobenthos samples. For evaluation this stage, the criterion of correct identification of the first three dominants of community was used.
  - II. Calculation of indicators for the determination of Ecological Status Class. For evaluation this stage, the criterion of less than 30% of the deviation from the average value obtained by all experts was used.
2. Estimation of the results obtained by experts during the two stages of the III Intercomparisen of Macrophytobenthos showed the following:

EMBLAS's partner	Evaluation result	
	Identification of the floristic composition	Calculation of indicators
NEA (GE)	Good	Good
SFU_DB (RF)	Good	Good
UkrSCES (UA)	Good	Good
ONU (UA)	Bad	Bad
IMB (UA)	Good	Good

Thus, the following experts have obtained positive results of the III Intercomparison of Macrophytobenthos exercises: **Miriam Tsetskhadze**, National Environmental Agency (Georgia); **Dmitry Afanasyev** Southern Federal University, Department of Botany (The Russian Federation); **Irina Tretyak** Ukrainian Scientific Center of Ecology Sea (Ukraine); **Anna Marinets**, Institute of Marine Biology NASU (Ukraine).

Galina Minicheva

18-04-2018

Odessa

## Annex 4: List of phytoplankton species

Species 2017	CW UA	OW GE	OW UA	Shelf UA
Dinophyceae				
Ahradina pulchra Lohmann, 1903				+
Akashiwo sanguinea (K.Hirasaka) G.Hansen & Ø.Moestrup, 2000	+		+	+
Alexandrium sp.				+
Alexandrium tamarensse (Lebour, 1925) Balech, 1995				+
Amphidinium klebsii Kofoid & Swezy, 1921				+
Amphidinium operculatum Claparède & Lachmann, 1859				+
Ceratium fusus (Ehrenberg) Dujardin, 1841	+		+	+
Ceratium fusus var. seta (Ehrenberg) Sournia				+
Ceratium hirundinella f. silesiacum Schröder, 1918			+	
Ceratium tripos (O.F.Müller) Nitzsch, 1817	+	+	+	+
Cochlodinium helicoides Lebour, 1925	+			+
Dinophyceae gen. sp.	+	+	+	+
Dinophysis acuminata Claparède & Lachmann, 1859	+		+	+
Dinophysis caudata Saville-Kent, 1881			+	+
Dinophysis norvegica Claparède & Lachmann, 1859				+
Dinophysis ovata Claparéde & Lachmann, 1859			+	
Dinophysis ovum (F.Schütt) T.H.Abé	+		+	+
Dinophysis sacculus Stein, 1883	+			+
Diplopsalis lenticula Bergh, 1881	+	+	+	+
Durinskia agilis (Kofoid & Swezy) Saburova, Chomérat & Hoppenrath, 2012	+			
Durinskia dybowskii (Wołoszynska) S.Carty, 2014	+			
Ensicalifera carinata Matsuoka, Kobayashi & Gains, 1990	+			
Glenodinium paululum Lindernann	+	+	+	+
Glenodinium pilula (Ostenfeld) Schiller, 1935	+		+	+
Glenodinium sp.		+		+
Gonyaulax cochlea Meunier, 1919	+			
Gonyaulax minima Matzenauer, 1933				+
Gonyaulax polygramma Stein, 1883				+
Gonyaulax spinifera (Claparède & Lachmann) Diesing, 1866	+			+
Gymnodinium agiliforme Schiller, 1928	+			
Gymnodinium arcticum Wulff, 1919		+		
Gymnodinium cneocoides T.M.Harris, 1940	+			
Gymnodinium najadeum J.Schiller, 1928	+			+
Gymnodinium simplex (Lohmann) Kofoid & Swezy, 1921				+
Gymnodinium sp.	+	+	+	+
Gymnodinium uberrimum (G.J.Allman) Kofoid & Swezy, 1921	+			
Gymnodinium wulffii J.Schiller, 1933	+	+	+	+
Gyrodinium cornutum (Pouchet) Kofoid & Swezy, 1921	+	+	+	+
Gyrodinium fusiforme Kofoid & Swezy, 1921	+		+	+
Gyrodinium fusus (Meunier) Akselman, 1985			+	
Gyrodinium lachryma (Meunier) Kofoid & Swezy, 1921	+		+	+

Species 2017	CW UA	OW GE	OW UA	Shelf UA
Gyrodinium nasutum (Wulff) Schiller, 1933	+			
Gyrodinium pingue (Schütt) Kofoid & Swezy, 1921	+	+	+	+
Gyrodinium sp.		+	+	+
Gyrodinium spirale (Bergh) Kofoid & Swezy, 1921				+
Gyrodinium varians (Wulff) Schiller, 1933				+
Heterocapsa triquetra (Ehrenberg) Stein, 1883	+			+
Katodinium fungiforme (Anissimova) Fott, 1957		+		
Katodinium sp.			+	+
Lessardia elongata Saldarriaga & F.J.R.Taylor, 2003	+			
Levanderina fissa (Levander) Ø.Moestrup, P.Hakanen, G.Hansen, N.Daugbjerg & M.Ellegaard, 2014			+	+
Lingulodinium polyedrum (F.Stein) J.D.Dodge, 1989	+		+	+
Margalefidinium citron (Kofoid & Swezy, 1921) F.Gómez, Richlen & D.M.Anderson, 2017	+			
Oblea rotunda (Lebour) Balech ex Sournia, 1973	+	+		
Palatinus apiculatus (Ehrenberg) S.C.Craveiro, A.J.Calado, N.Daugbjerg & Ø.Moestrup, 2009				+
Peridiniella danica (Paulsen) Y.B.Okolodkov & J.D.Dodge, 1995		+	+	
Peridiniopsis penardii (Lemmermann) Bourrelly, 1968	+		+	+
Peridinium aciculiferum Lemmermann, 1900	+			
Phalacroma ovatum (Claparède & Lachmann) Jorgensen, 1923				+
Phalacroma rotundatum (Claparéde & Lachmann) Kofoid & Michener, 1911	+	+		+
Polykrikos schwarzii Bütschli, 1873				+
Prorocentrum cordatum (Ostenfeld) J.D.Dodge, 1975	+	+	+	+
Prorocentrum cordatum var. aralensis (Kisselev) Krachmalny, 1993		+		
Prorocentrum micans Ehrenberg, 1834	+	+	+	+
Prorocentrum oblongum (Schiller) Ab-				+
Prorocentrum ponticus Krachmalny & Terenko, 2002				+
Prorocentrum scutellum Schröder, 1900	+			
Protoceratium reticulatum (Claparède & Lachmann) Bütschli, 1885	+			
Protoperdinium bipes (Paulsen, 1904) Balech, 1974			+	+
Protoperdinium brevipes (Paulsen, 1908) Balech, 1974		+		+
Protoperdinium conicum (Gran, 1900) Balech, 1974				+
Protoperdinium crassipes (Kofoid, 1907) Balech, 1974				+
Protoperdinium divergens (Ehrenberg, 1840) Balech, 1974	+		+	+
Protoperdinium granii (Ostenfeld) Balech, 1974	+		+	+
Protoperdinium leonis (Pavillard, 1916) Balech, 1974	+			
Protoperdinium oblongum (Aurivillius) Parke & Dodge, 1976	+			
Protoperdinium pallidum (Ostenfeld, 1899) Balech, 1973	+			
Protoperdinium pellucidum Bergh, 1881	+		+	+
Protoperdinium solidicorne (Mangin, 1926) Balech, 1974	+			
Protoperdinium sp.	+			+
Protoperdinium steinii (Jørgensen, 1899) Balech, 1974	+	+	+	+
Protoperdinium subinerme (Paulsen) Loeblich III, 1969	+			
Scrippsiella trochoidea (Stein) Loeblich III, 1976	+	+	+	+
Spatulodinium pseudonociluca (Pouchet) J.Cachon & M.Cachon, 1968	+			
Torodinium robustum Kofoid & Swezy, 1921	+			
Tovellia coronata (Woloszynska) Moestrup, Lindberg & Daugbjerg, 2005	+			

Species 2017	CW UA	OW GE	OW UA	Shelf UA
<i>Tripos furca</i> (Ehrenberg) F.Gómez, 2013	+	+	+	+
<i>Woloszynska neglecta</i> (Schilling) R.H.Thompson, 1951	+			
<i>Woloszynska pascheri</i> (Suchlandt) Stosch, 1973	+			
Bacillariophyceae				
<i>Achnanthes longipes</i> C.Agardh, 1824	+			
<i>Amphipleura</i> sp.	+			
<i>Amphora crassa</i> Gregory, 1857				+
<i>Amphora hyalina</i> Kützing, 1844	+			+
<i>Amphora</i> sp.	+			+
<i>Asterionella formosa</i> Hassall, 1850	+			
<i>Attheya decora</i> T.West, 1860	+			
<i>Attheya septentrionalis</i> (Østrup) R.M.Crawford, 1994	+			
<i>Aulacoseira granulata</i> (Ehrenberg) Simonsen, 1979	+			
Bacillariophyceae gen. sp.				+
<i>Cerataulina pelagica</i> (Cleve) Hendey, 1937	+	+	+	+
<i>Ceratoneis closterium</i> Ehrenberg, 1839	+	+		+
<i>Chaetoceros affinis</i> Lauder, 1864	+			
<i>Chaetoceros compressus</i> Lauder, 1864	+			
<i>Chaetoceros curvisetus</i> Cleve, 1889	+			
<i>Chaetoceros danicus</i> Cleve, 1889			+	
<i>Chaetoceros insignis</i> Proschkina-Lavrenko, 1955				+
<i>Chaetoceros laciniosus</i> F.Schütt, 1895				+
<i>Chaetoceros lorenzianus</i> Grunow, 1863	+	+		
<i>Chaetoceros muelleri</i> Lemmermann, 1898	+			
<i>Chaetoceros similis</i> Cleve, 1896				+
<i>Chaetoceros similis</i> f. <i>solitarius</i> Proschkina-Lavrenko, 1955	+		+	+
<i>Chaetoceros simplex</i> Ostenfeld, 1902	+			+
<i>Chaetoceros socialis</i> H.S.Lauder, 1864	+			+
<i>Chaetoceros subtilis</i> Cleve, 1896	+			
<i>Chaetoceros tenuissimus</i> Meunier, 1913	+			
<i>Chaetoceros thronsenii</i> (Marino, Montresor & Zingone) Marino, Montresor & Zingone, 1991		+		
<i>Cocconeis pediculus</i> Ehrenberg, 1838	+		+	+
<i>Cocconeis scutellum</i> Ehrenberg, 1838	+			+
<i>Cocconeis</i> sp.	+			+
<i>Coscinodiscopsis jonesiana</i> (Greville) E.A.Sar & I.Sunesen, 2008				+
<i>Coscinodiscus janischii</i> A.Schmidt, 1878	+			
<i>Coscinodiscus oculus-iridis</i> (Ehrenberg) Ehrenberg, 1840	+			
<i>Cyclotella chaetoceras</i> Lemmermann, 1900				+
<i>Cyclotella choctawhatcheeana</i> Prasad, 1990	+		+	+
<i>Cyclotella</i> sp.		+		+
<i>Cylindrotheca closterium</i> (Ehrenberg) Reimann & J.C.Lewin, 1964	+			
<i>Cymatopleura solea</i> (Brébisson) W.Smith, 1851	+			
<i>Cymbella</i> sp.	+			
<i>Cymbopleura</i> sp.				+
<i>Dactyliosolen fragilissimus</i> (Bergon) Hasle, 1996	+		+	

Species 2017	CW UA	OW GE	OW UA	Shelf UA
Diatoma tenuis C.Agardh, 1812	+			+
Diatoma vulgare Bory de Saint-Vincent, 1824	+			
Diploneis bombus (Ehrenberg) Ehrenberg, 1853	+		+	
Ditylum brightwellii (T.West) Grunow, 1885	+			+
Entomoneis paludosa (W.Smith) Reimer, 1975	+			+
Fragilaria sp.	+			
Grammatophora marina (Lyngbye) Kützing, 1844	+			
Gyrosigma acuminatum (Kützing) Rabenhorst, 1853	+			
Gyrosigma fasciola (Ehrenberg) J.W.Griffith & Henfrey, 1856	+			
Halamphora costata (W.Smith) Levkov, 2009			+	
Halamphora sp.				+
Hemiaulus hauckii Grunow ex Van Heurck, 1882				+
Hemiaulus sp.				+
Leptocylindrus danicus Cleve, 1889	+			+
Licmophora ehrenbergii (Kützing) Grunow, 1867	+			
Licmophora gracilis (Ehrenberg) Grunow, 1867	+		+	+
Licmophora sp.				+
Lyrella lyra (Ehrenberg) Karajeva, 1978	+			
Lyrella sp.	+			
Melosira moniliformis (O.F.Müller) C.Agardh, 1824	+			
Melosira nummuloides C.Agardh, 1824	+			
Melosira varians C.Agardh, 1827	+			
Navicula cancellata Donkin, 1872	+			
Navicula cryptocephala Kützing, 1844	+		+	
Navicula lanceolata Ehrenberg, 1838	+			+
Navicula pennata A.Schmidt, 1876	+			+
Navicula radiosha Kützing, 1844	+			
Navicula salinarum Grunow, 1880	+			
Navicula sp.	+		+	+
Nitzschia holsatica Hustedt, 1930	+			+
Nitzschia hybrida Grunow	+			
Nitzschia longissima (Brébisson) Ralfs, 1861			+	+
Nitzschia sigma (Kützing) W.Smith, 1853	+			
Nitzschia sigmoidea (Nitzsch) W.Smith, 1853	+			
Nitzschia sp.	+			+
Nitzschia tenuirostris Mer.	+		+	+
Paralia sulcata (Ehrenberg) Cleve, 1873	+			+
Pinnularia sp.				+
Plagiotropis lepidoptera (Gregory) Kuntze, 1898	+			
Pleurosigma angulatum (Queckett) W.Smith, 1852	+			+
Pleurosigma elongatum W.Smith, 1852	+		+	
Proboscia alata (Brightwell) Sundström, 1986	+		+	+
Proboscia alata f. alata (Brightwell) Sündstrom, 1986			+	+
Pseudo-nitzschia delicatissima (Cleve) Heiden, 1928	+	+	+	+
Pseudo-nitzschia pseudodelicatissima (Hasle) Hasle, 1993				+

Species 2017	CW UA	OW GE	OW UA	Shelf UA
<i>Pseudo-nitzschia pungens</i> (Grunow ex Cleve) G.R.Hasle, 1993	+			+
<i>Pseudo-nitzschia seriata</i> (Cleve) H.Peragallo, 1899	+		+	
<i>Pseudosolenia calcar-avis</i> (Schultze) B.G.Sundström, 1986	+	+	+	+
<i>Rhoicosphenia abbreviata</i> (C.Agardh) Lange-Bertalot, 1980	+			
<i>Skeletonema costatum</i> (Greville) Cleve, 1873	+			+
<i>Stephanodiscus hantzschii</i> Grunow, 1880	+			+
<i>Striatella unipunctata</i> (Lyngbye) C.Agardh, 1832	+			
<i>Synedra baculus</i> Gregory, 1857				+
<i>Synedra</i> sp.	+		+	+
<i>Tabularia fasciculata</i> (C.Agardh) D.M.Williams & Round, 1986	+			+
<i>Thalassionema nitzschiooides</i> (Grunow) Mereschkowsky, 1902	+	+	+	+
<i>Thalassiosira baltica</i> (Grunow) Ostenfeld, 1901	+	+		+
<i>Thalassiosira nordenskioeldii</i> Cleve, 1873	+			
<i>Thalassiosira parva</i> Proschkina-Lavrenko, 1955	+			+
<i>Thalassiosira punctigera</i> (Castracane) Hasle, 1983	+			+
<i>Thalassiosira</i> sp.			+	+
<i>Tryblionella compressa</i> (J.W.Bailey) M.Poulin, 1990		+	+	+
<b>Cyanophyceae</b>				
<i>Aphanizomenon flosaqueae</i> Ralfs ex Bornet & Flahault, 1886	+			+
<i>Cyanophyceae</i> Schaffner, 1909	+			
<i>Dolichospermum flosaqueae</i> (Brébisson ex Bornet & Flahault) P.Wacklin, L.Hoffmann & J.Komárek, 2009	+			
<i>Dolichospermum spiroides</i> (Klebhan) Wacklin, L.Hoffmann & Komárek, 2009	+			
<i>Glaucospira laxissima</i> (G.S.West) Simic, Komárek & Dordevic, 2014	+			+
<i>Jaginema kisselevii</i> (Anissimova) Anagnostidis & Komárek, 1988	+			+
<i>Limnothrix planctonica</i> (Woloszynska) Meffert, 1988	+			
<i>Merismopedia minima</i> G.Beck, 1897	+			
<i>Merismopedia tenuissima</i> Lemmermann, 1898	+			
<i>Nodularia spumigena</i> Mertens ex Bornet & Flahault, 1886	+			
<i>Planktolyngbya limnetica</i> (Lemmermann) Komárová-Legnerová & Cronberg, 1992	+			
<b>Chlorophyceae</b>				
<i>Acutodesmus acuminatus</i> (Lagerheim) Tsarenko, 2001	+			
<i>Acutodesmus obliquus</i> (Turpin) Hegewald & Hanagata, 2000	+			
<i>Chlorogonium elongatum</i> (P.A.Dangeard) Francé, 1897	+			
<i>Desmodesmus communis</i> (E.Hegewald) E.Hegewald, 2000	+			
<i>Hyaloraphidium contortum</i> Pascher & Korshikov, 1931	+			+
<i>Kirchneriella lunaris</i> (Kirchner) Möbius, 1894	+	+	+	+
<i>Lacunastrum gracillimum</i> (West & G.S. West) H.McManus, 2011	+			
<i>Monactinus simplex</i> (Meyen) Corda, 1839	+			
<i>Monoraphidium arcuatum</i> (Korshikov) Hindák, 1970	+			
<i>Monoraphidium contortum</i> (Thuret) Komárová-Legnerová, 1969	+			+
<i>Monoraphidium komarkovae</i> Nygaard, 1979	+			+
<i>Mychonastes jurisii</i> (Hindák) Krienitz, C.Bock, Dadheeck & Proschold, 2011	+			
<i>Neocystis ovalis</i> (Korshikov) Hindák, 1988	+			
<i>Pediastrum duplex</i> Meyen, 1829	+			
<i>Raphidocelis danubiana</i> (Hindák) Marvan, Komárek & Comas, 1984	+			+

Species 2017	CW UA	OW GE	OW UA	Shelf UA
Scenedesmus sp.				+
Tetraedron triangulare Korshikov, 1953	+			
Prymnesiophyceae				
Acanthoica quattrospina Lohmann, 1903	+		+	+
Anacanthoica acanthos (Schiller) Deflandre, 1952	+			
Anacanthoica lithostratos (Schiller) Norris, 1984	+			
Coccolithus sp.				+
Emiliania huxleyi (Lohmann) W.W.Hay & H.P.Mohler, 1967	+	+	+	+
Pontosphaera haekelii Lohmann	+			
Pontosphaera nigra Schiller				+
Pontosphaera sp.	+			+
Prymnesiophyceae gen. sp.			+	+
Prymnesium parvum N.Carter, 1937	+			+
Syracosphaera dentata Lohmann				+
Syracosphaera sp.			+	+
Euglenoidea				
Euglena viridis (O.F.Müller) Ehrenberg, 1830	+	+		+
Eutreptia lanowii Steuer, 1904	+			+
Eutreptia viridis Perty, 1852		+		
Phacus sp.				+
Chrysophyceae				
Chrysamoeba radians Klebs, 1892	+			
Dinobryon balticum (Schütt) Lemmermann, 1900	+	+		+
Dinobryon faculiferum (Willén) Willén, 1992		+		+
Dinobryon sociale (Ehrenberg) Ehrenberg, 1834	+			
Ochromonas oblonga N.Carter, 1937	+			
Ollicola vangoorii (W.Conrad) Vørs, 1992	+	+	+	+
Cryptophyceae				
Hillea fusiformis (J.Schiller) J.Schiller, 1925	+	+	+	+
Hillea marina Butcher, 1952				+
Plagioselmis nannoplantica (H.Skuja) G.Novarino, I.A.N.Lucas & S.Morrall, 1994	+			
Plagioselmis prolonga Butcher ex G.Novarino, I.A.N.Lucas & S.Morrall, 1994		+	+	+
Plagioselmis sp.				+
Trebouxiophyceae				
Actinastrum aciculare Playfair, 1917	+			
Actinastrum sp.	+			
Crucigenia fenestrata (Schmidle) Schmidle, 1900	+			
Crucigenia sp.		+		+
Oocystis borgei J.W.Snow, 1903	+			+
Oocystis lacustris Chodat, 1897	+			
Oocystis sp.				+
Dictyochophyceae				
Apedinella radians (Lohmann) P.H.Campbell, 1973	+	+		+
Dictyocha speculum Ehrenberg, 1839		+	+	+
Pseudopedinella pyriformis N.Carter, 1937	+			

Species 2017	CW UA	OW GE	OW UA	Shelf UA
Ulvophyceae				
<i>Binuclearia lauterbornii</i> (Schmidle) Proschkina-Lavrenko, 1966	+			
Chlorodendrophyceae				
<i>Tetraselmis inconspicua</i> Butcher, 1959	+	+		+
<i>Tetraselmis</i> sp.				+
Chlorophyta incertae sedis				
<i>Poropila dubia</i> J.Schiller, 1925	+			
Choanoflagellatea				
<i>Bicosta minor</i> (Reynolds) Leadbeater, 1978	+			
<i>Diaphanoeca grandis</i> Ellis, 1930				+
Cryptophyta incertae sedis				
<i>Leucocryptos marina</i> (Braarud) Butcher, 1967	+			
Imbricatea				
<i>Paulinella ovalis</i> (A.Wulff) P.W.Johnson, P.E.Hargraves & J.M.Sieburth, 1988	+		+	+
Conjugatophyceae				
<i>Closterium lineatum</i> Ehrenberg ex Ralfs, 1848	+			
Prasinophyceae				
<i>Pterosperma jorgensenii</i> J.Schiller, 1925		+		+

## Annex 5: List of zooplankton species

### List of microzooplankton species 2017 – IMB

#### Species composition of microzooplankton (ciliates and metazoa)

Taxa	FPZ	NPMS-UA	JOSS
Amphileptus sp.	+	-	-
Askenasia regina Earlander et Mont., 2002	+	-	-
Askenasia stellaris (Eichw., 1852)	+	+	+
Balanion comatum (Wulff, 1919)	+	-	-
Cardiostomatella sp.	+	-	-
Chlamydodontida fam. gen. sp.	+	-	-
Choreotrichida fam. gen. sp.	-	-	+
Cyclotrichium sphaericum F.-Fr., 1924	+	-	-
Dartintinnus alderae Smith, Song, Gavrilova, Kurilov, Liu, McManus and Santoferrara, 2017	+	+	+
Eutintinnus lususundae (Entz, 1884)	+	+	+
Foissneridium constrictum (Meunier, 1910) Agatha, 2011 (= S. constrictum (Meun., 1910))	+	-	-
Helicostoma oblongum Cohn, 1866	+	+	-
Hypotrichia fam. gen. sp.	+	-	-
Laboea strobila (Lohm., 1908)	+	-	-
Lohmanniella oviformis (Leeg., 1915)	+	+	+
Lynnella semiglobulosa Liu, Yi, Lin & Al-Rasheid, 2011	+	-	-
Mesodinium pulex (Clap. et Lachm., 1858)	-	-	+
Mesodinium rubrum (=Myrionecta rubra) (Lohm., 1908)	+	+	+
Mesodinium sp.	+	-	+
Metacylis mediterranea (Mereschk., 1881)	+	+	+
Monodinium balbianii nanum (Fab.-Dom., 1888)	+	-	+
Pelagostrobilidium conicum (Kahl, 1932) Liu, Yi, Lin, Warren & Song, 2012	+	-	-
Pelagostrobilidium epacrum (Lynn & Montagnes, 1988) Agatha, Str?der-Kypke, Beran & Lynn, 2005	+	+	-
Pelagostrobilidium spirale (Leeg., 1915)	+	+	+
Prorodon spp.	-	+	-
Stenosemella nivalis (Meun., 1910)	-	-	+
Strobilidium sp.	+	-	-
<b>Strobilidium sp. (очень мелкий)</b>	-	-	+
Strobilidium sp. undinum?	+	-	-
Strombidinopsis sp.	-	+	+
Strombidium acutum (Leeg., 1915)	+	+	+
Strombidium capitatum Kahl, 1932	+	+	+
Strombidium conicum (Lohm., 1908)	+	+	+
Strombidium dalum Lynn et al., 1988	-	-	+
Strombidium emergens (Leeg., 1915)	+	+	+
Strombidium epidemum Lynn et al., 1988	+	+	+
Strombidium filificum Kahl, 1932	-	+	-
Strombidium lagenula F.-Fr., 1924	+	+	-
Strombidium rhynchum Lynn et al., 1988	+	-	-
Strombidium spp.	+	+	-
Strombidium tintinnodes Entz, 1884	+	+	+

Taxa	FPZ	NPMS-UA	JOSS
<i>Strombidium vestitum</i> (Leeg., 1915)	+	+	+
<i>Tiarina fusus</i> (Clap. et Lachm., 1857)	+	-	+
<i>Tintinnida proter fam. gen. sp. 1</i>	-	-	+
<i>Tintinnida proter fam. gen. sp. 2</i>	-	-	+
<i>Tintinnidium mucicola</i> (Clap. et Lachm., 1858)	-	+	-
<i>Tintinnopsis beroidea</i> Entz, 1884 (= <i>T. strigosa</i> Meunier, 1919)	+	-	-
<i>Tintinnopsis nana</i> Lohmann, 1908	-	+	-
<i>Tintinnopsis parvula</i> Jørgensen, 1912	+	-	-
<i>Tintinnopsis tubulosa</i> Levander, 1900	+	-	-
<i>Tontonia</i> sp. 1	+	+	+
<i>Tontonia</i> sp. 2	+	+	+
<i>Urotricha</i> sp. 1	+	-	+
<i>Urotricha</i> sp. 2	+	-	-
<i>Urotricha</i> sp. 3	+	+	+
<i>Vasicola ciliata</i> Tatem, 1869	-	+	-
<i>Centrohelida</i> fam. gen. sp.	+	-	+
<i>Lepadella</i> sp.	-	+	-
<i>Rotaria</i> sp.	-	+	-
<i>Synchaeta baltica</i> Ehrenberg, 1834	+	-	-
<i>Synchaeta</i> sp.	+	+	+
<i>Synchaeta</i> sp. 1	+	-	-
<i>Synchaeta</i> sp. 2	+	-	-
Acartia-like larvae	+	+	+
<i>Bivalvia</i> veliger sp.	-	+	+
<i>Bivalvia</i> veliger sp. 1	-	+	+
<i>Bivalvia</i> veliger sp. 2	-	+	+
Harpacticoida fam. gen. spp.	-	-	+
Larvae unidentified sp. 1	-	-	+
Larvae unidentified sp. 2	-	-	+
<i>Oikopleura</i> ( <i>Vexillaria</i> ) <i>dioica</i> larvae Fol, 1872	+	-	+
Ova unidentified	+	+	-
Polychaeta larvae fam. gen. sp.	-	+	-

## **Annex 6: List of macrophytobenthos species**

Detailed results of intercomparison are in five Excel files (DCT Macrophytobenthos) – one file per participants of the intercomparison.

## **Annex 7: Report on the Chemical Contaminant Measurements from Surveys 2017 (EC JRC)**



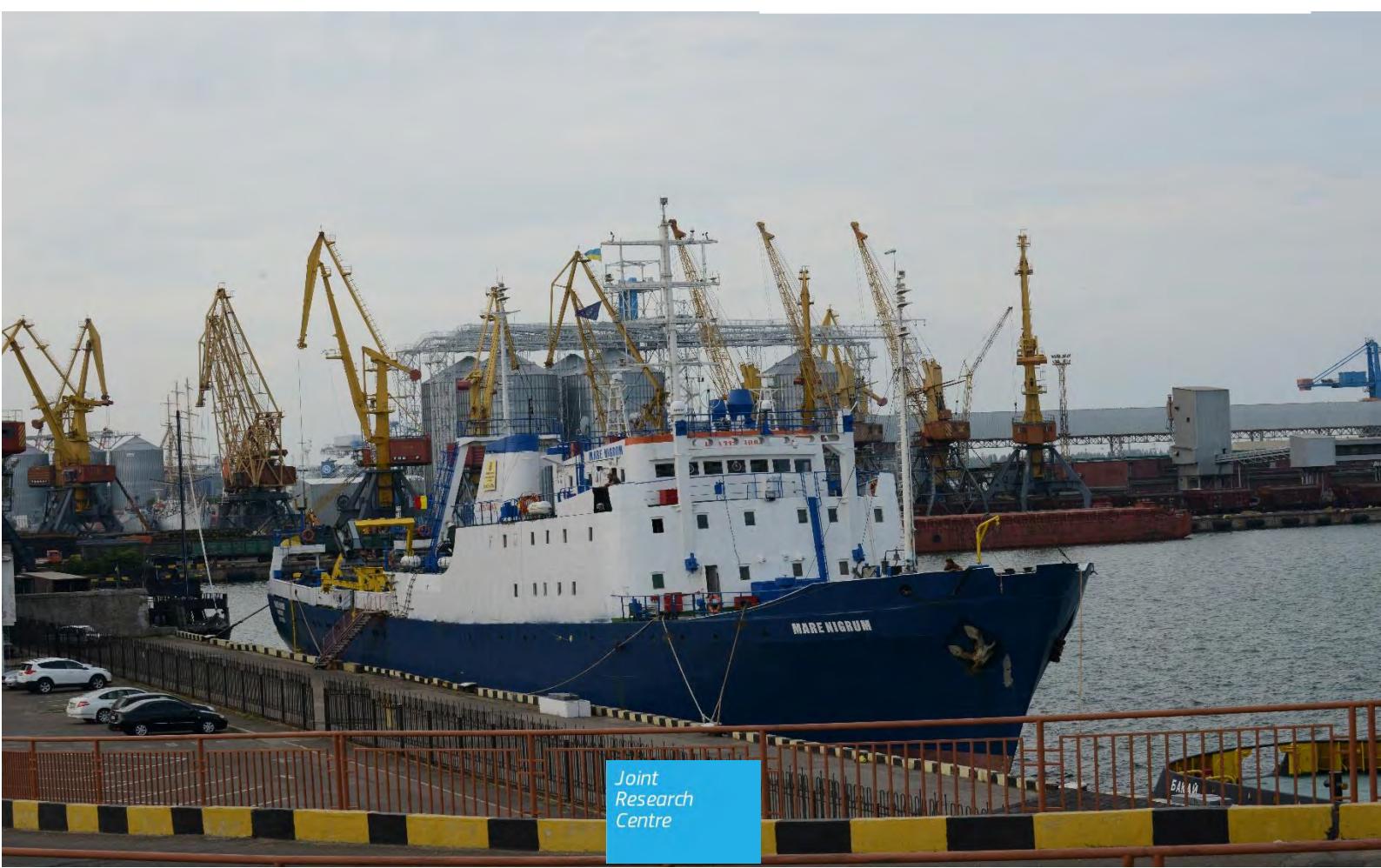
# JRC TECHNICAL REPORTS

EMBLAS II - Joint Black Sea Survey 2017  
JRC Chemical Contaminant Measurements

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

Giulio Mariani, Simona Tavazzi,  
Helle Skejo, Peter Oswald, Bernd  
Manfred Gawlik, Georg Hanke

2018



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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 EU/UNDP Project EMBLAS-II, aiming at improving the monitoring and the availability of analytical results 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 for a total of 49 samples collected, 104 substances analysed (35 of these are part of the WFD Priority Substance list) and about 4266 final individual results.

## **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 a Commission Decision (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 at 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, increasing 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 DG NEAR project EMBLAS-II (<http://emblasproject.org/>) aims at improving the protection of the Black Sea environment. 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 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-II in 2016 (EMBLAS-II 2017, Mariani 2017), JRC provided, besides continued collaboration for the monitoring of marine litter, extended support in 2017. In close collaboration with the EMBLAS-II Project coordination team and the Environmental Institute (Slovakia), sampling strategies and work planning have been agreed. The samples for organic trace contaminant analysis have been taken during the 2017 EMBLAS II Joint Black Sea Survey and during dedicated field campaigns in Ukraine and Georgia.

Peter Oswald, EMBLAS-II Chemistry Expert (Environmental Institute), visited JRC in order to prepare sampling material for the cruise, including the pre-extraction of filters and adsorbants. The material was shipped at Environmental Institute and then transported to the departure port, Costanta, Romania and the sampling/ extraction devices were installed on Mare Nigrum on 26.8.2017.

The sampling on board was performed by Peter Oswald (Georg Hanke, JRC , joined the cruise for the first 1 day leg from Constanta, Romania to Odessa, Ukraine) with support by Ksenia Toholukova (Ukrainian Scientific Centre of Ecology of the Sea) during the duration of the cruise.

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 Georgia, 3 in Ukraine, 1 inside and 1 outside the Danube delta area, 12 samples in open sea and 9 samples for QA/QC and as replicates.

Additional samples were collected for the University of Athen, to support non-target analysis. The 20 L samples have been filtered and extracted on-board of Mare Nigrum with a JRC developed manifold (Mariani 207, Mariani 2017a) 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 300-600 L volume) during transects on the moving ship for on-line filtration and extraction with two subsequent cartridges for later reference analysis at JRC.

The resulting filtration/extraction disks and cartridges have then been shipped to JRC in Ispra, Italy. After the cruise, the visiting scientist (Peter Oswald), in collaboration with Helle Skejo, prepared samples for instrumental analysis in the JRC Ispra laboratory facility. All the samples after extraction were divided in three fractions in order to support also other partner laboratories (University of Athens and Enviroment Institute of Slovak Repuplic) for non-target measurements.

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

The samples were analysed with multi-compound methodologies including 104 substances selected from the following categories: corrosion inhibitor, antioxidants, anti-epileptic drug and metabolite, hypolipidemic agent, nonsteroidal anti-inflammatory drugs (NSAID), sunscreen, fluorinated compounds, antibiotic, insecticide, neonicotinoid insecticide, herbicide, algicide, dielectric and coolant fluid, products by incomplete combustion of matter, plasticizers, chlorinated and phosphate flame retardants. Among them, 35 substances are enclosed in the priority list of WFD (EU 2000).

The sampling and analytical work provided 4266 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 zodiac or glass fiber boat, or in case of rough sea, through a CTD system with Niskin sampling bottles (Figure 1). Coastal surface water samples from Georgia and Ukraine were sampled in HDPE containers.



*Figure 1 Glass fiber boat and CTD system*

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 20 different spots were sampled: 3 in Georgia, 3 in Ukraine, 1 inside and 1 outside the Danube delta and 12 samples in the open see. In Table 1 samples and sampling conditions are reported.

Figures 2, 3 and 4 show the maps of the sampling points. Figure 5 shows the filters arrived in the laboratory after the cruise. Figures 6-11 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)	Boat type (Glass Fiber or Zodiac)	Coastal sample provenience	Type	Tank N.	Extraction Box number	Weight of full canister (kg)	Filtration volume (L)	Internal Standard mix add (yes or no)	Sample destination
1-A	45°08'19.3"	29°10'11.1"	26.8.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	1	20	23.75	20.05	YES	Breakthrough Test JRC,UoA
1'-A	45°08'19.3"	29°10'11.1"	26.8.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	2	18	23.65	20	YES	JRC for repeatability
1-JOSS	46°23'10.7"	31°01'14.7"	28.8.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	1	20	23.50	19.8	YES	JRC,UoA
2-JOSS	45°12'53.3"	31°15'56.4"	29.8.2017	Surface	0	CTD		20 Liter Sea Water samples in steel tank	1	20	24.00	20.3	YES	JRC,UoA
3-JOSS	44°51'07.8"	31°20'31.4"	29.8.2017	Surface	0	CTD		20 Liter Sea Water samples in steel tank	2	18	23.65	20	YES	JRC,UoA
4-JOSS	44°06'45.8"	31°34'08.3"	30.8.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	X	20	22.4	21.45	YES	JRC,UoA
5-JOSS	43°23'45.9"	31°48'43.7"	30.8.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	3	18	24.2	20.55	YES	JRC,UoA
6-JOSS	43°24'50.3"	32°52'02.8"	31.8.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	3	20	23.5	19.85	YES	JRC,UoA
7-JOSS	43°22'49.7"	34°46'01.9"	31.8.2017	Surface	0	CTD		20 Liter Sea Water samples in steel tank	2	20	23.5	19.85	YES	JRC,UoA
8-JOSS	43°31'13.0"	36°03'34.2"	1.9.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	3	18	23.5	19.85	YES	JRC,UoA
9-JOSS	42°14'21.0"	39°52'58.0"	2.9.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	3	20	23.8	20.15	YES	JRC,UoA
10-JOSS	42°06'12.0"	40°20'11.5"	2.9.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	2	20	22.65	19	YES	JRC,UoA
11-JOSS	41°56'15.3"	40°49'36.2"	2.9.2017	Surface	0	CTD		20 Liter Sea Water samples in steel tank	1	20	23.5	19.8	YES	JRC,UoA
12-JOSS	41°47'03.3"	41°13'16.3"	3.9.2017	Surface	0	Z		20 Liter Sea Water samples in steel tank	2	18	23.25	19.6	YES	JRC,UoA
1-GE	41°39'23.69"	41°37'59.73"	3.9.2017	Surface	0	HDPE	GE-delivery Batumi port	20 Liter Sea Water samples in steel tank	P6	20	21.4	20.65	YES	Breakthrough Test JRC,UoA
2-GE	41°39'0.05"	41°38'42.17"	3.9.2017	Surface	0	HDPE	GE-delivery Batumi port	20 Liter Sea Water samples in steel tank	P7	18	21.35	20.6	YES	JRC,UoA
3-GE	41°41'30.37"	41°42'13.14"	3.9.2017	Surface	0	HDPE	GE-delivery Green Cape	20 Liter Sea Water samples in steel tank	P8	18	21.4	20.65	YES	JRC,UoA
FB-1				Surface	0	HDPE		20 Liter Sea Water samples in steel tank	P1	18	22.3	21.34	YES	JRC,UoA
FB-2				Surface	0	HDPE		20 Liter Sea Water samples in steel tank	P2	20	22.25	21.29	YES	JRC,UoA
CW-7	46°33.207'	30°46.154'	6.9.2017	Surface	0	HDPE	UA-delivery	20 Liter Sea Water samples in steel tank	P3	18	23.15	22.25	YES	Breakthrough Test JRC,UoA
CW-8	46°04.136'	31°10.283'	6.9.2017	Surface	0	HDPE	UA-delivery	20 Liter Sea Water samples in steel tank	P4	20	23.75	22.85	YES	JRC,UoA
CW-5	46°04.136'	30°27.830'	5.9.2017	Surface	0	HDPE	UA-delivery	20 Liter Sea Water samples in steel tank	P5	20	24.30	23.4	YES	JRC,UoA

Sample Code	Latit.	Long.	Date	Water sampling Depth	Site Depth (meters)	Boat type (Glass Fiber or Zodiac)	Coastal sample provenience	Type	Tank N.	Extraction Box number	Weight of full canister (kg)	Filtration volume (L)	Internal Standard mix add (yes or no)	Sample destination
1-UoA			As JOSS_4	Surface	0	Z	UoA calibration	20 Liter Sea Water samples in steel tank	1	18	23.4	19.7	YES	UoA
2-UoA			As JOSS_4	Surface	0	Z	UoA calibration	20 Liter Sea Water samples in steel tank	2	20	23.5	19.85	YES	UoA
3-UoA			As JOSS_4	Surface	0	Z	UoA calibration	20 Liter Sea Water samples in steel tank	3	18	23.4	19.75	YES	UoA
4-UoA			As JOSS_4	Surface	0	Z	UoA calibration	20 Liter Sea Water samples in steel tank	4	20	23.0	19.35	YES	UoA
1-C	45°20'42.0"	29°51'22.0"	7.9.2017	Surface	0	CTD		20 Liter Sea Water samples in steel tank	4	20	24.45	20.8	YES	JRC,UoA

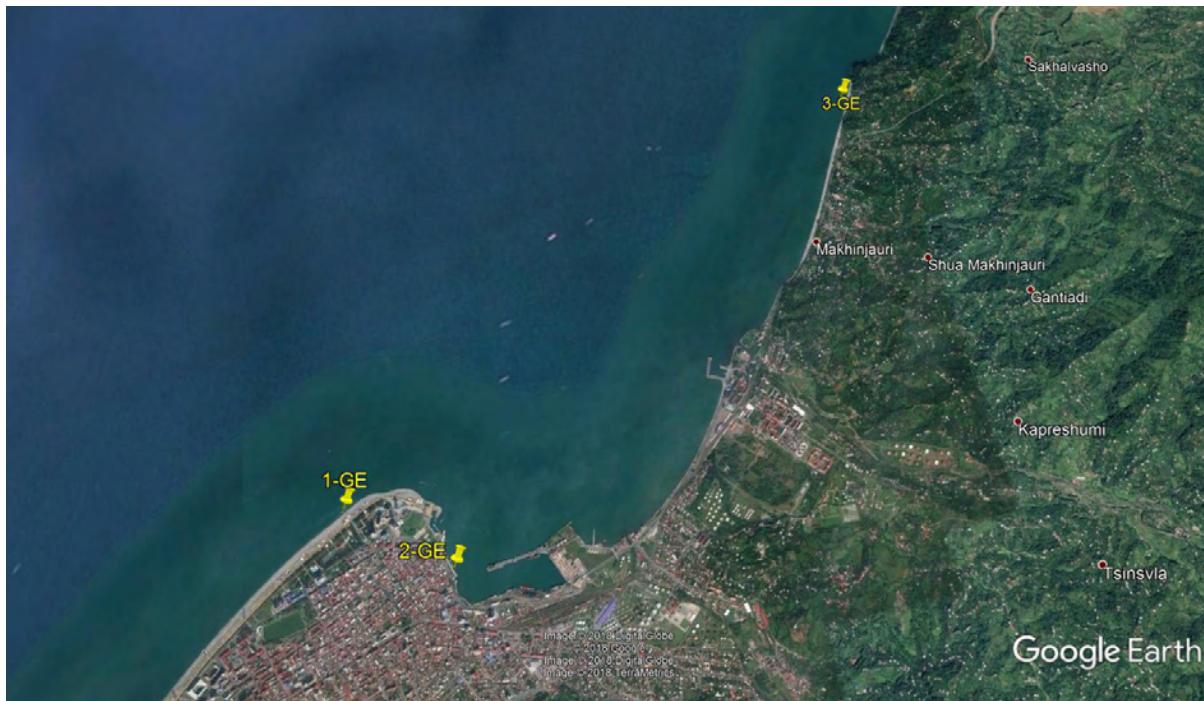


Figure 2: Sampling points on coast of Georgia

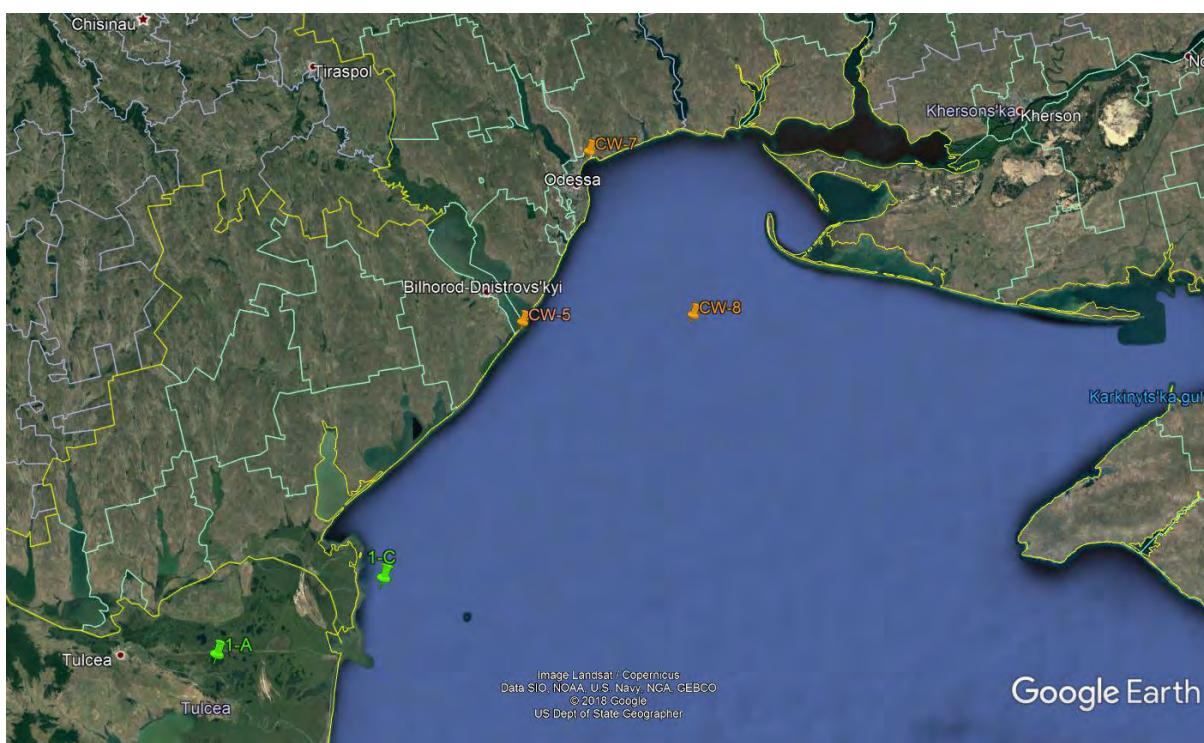


Figure 3: Sampling points nn coast of Ukraine and in Danube delta zone

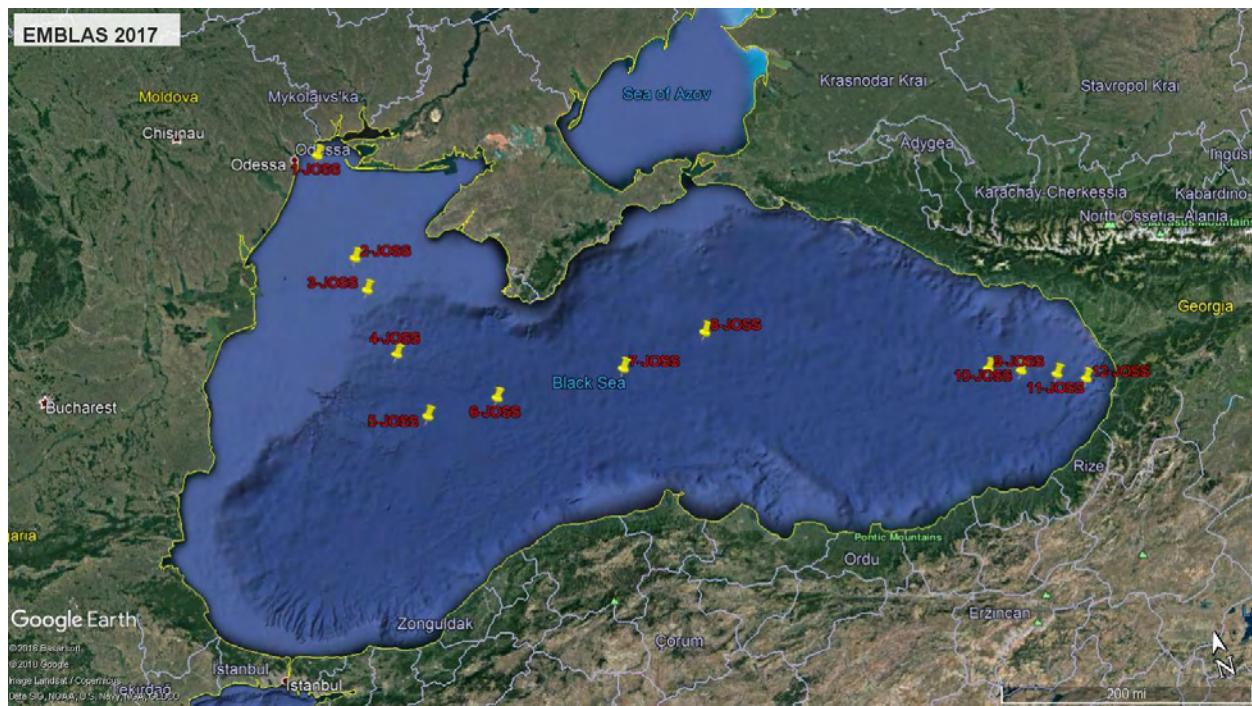


Figure 4: 12 Sampling points in open sea



Figure 5: Filtration/adsorption cartridge samples

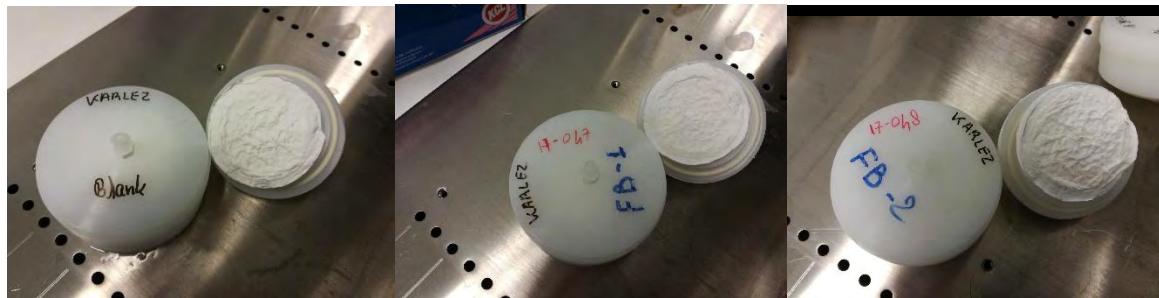


Figure 6: HLB disks used for samples: Field blank



Figure 7: HLB disks used in Danube River area for samples: 1A, 1A' for reproducibility test, 1A-BT for breakthrough evaluation and 1-C



Figure 8: HLB disks used in Georgia for samples: 1 GE, 1GE-BT for breakthrough evaluation, 2 GE and 3 GE



Figure 9: HLB disks used in Ukraina for samples: CW-5, CW-7, CW-7 BT for breakthrough evaluation and CW-8



Figure 10: HLB disks used in open sea for samples 1-JOSS, 2-JOSS, 3-JOSS, 4-JOSS, 5-JOSS, 6-JOSS, 7-JOSS and 8-JOSS



*Figure 11: HLB disks used in open sea for samples 9-JOSS, 10-JOSS, 11-JOSS and 12-JOSS*

### 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 12.

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 labelled internal standards and filtered/extracted on site at an average flow of 0.14 l/min.



*Figure 12: Sampling device used for sampling*

HLB disk activation, drying and elution were performed using an automatic extractor (J2 Scientific, Figure 13).

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 <sub>2</sub> for 30 min at 20 ml/min	
Labelled Syringe Standard spiking		
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 13: 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 reconstituting solution, divided in two portions one for JRC and one for UoA. The JRC portions were analysed by UHPLC-MS/MS.

### 3.3. Large Volume Transect Sampling

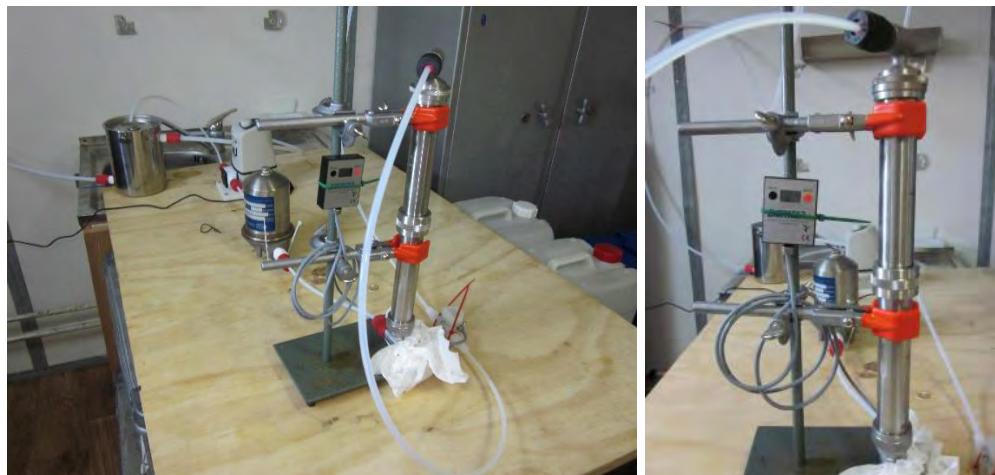
A total of 5 transects were sampled across the Black Sea within the 2017 EMBLAS Joint Open Sea Survey Georgia – Ukraine (JOSS GE-UA) with the Large Volume Transect Sampling (LV-TS) method.

A 10 mm o.d./8 mm i.d. teflon tube has been mounted inside a steel protection tube on the right side of the ships hull, secured with steel cables, in order to collect sea water during navigation (Figure 14). The open sampling tube inlet was directed towards the navigation direction at ca. 1 m 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 14: Sampling metal probe mounted on hull of the ship*

From the overflow container a fraction 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 and then to 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 15) was kept at 220 ml/min, controlled with a digital flowmeter. The first cell was a primary extraction cartridge and 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 15: 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 analysed for several contaminants before the use, in order to evaluate and reaching background contamination values appropriated at the purpose.

The transect coordinates and the sampled volume of each transect are the following:

- Filter 1 (JOSS 1 - Cell 1; JOSS 1 - Cell 2)
- Starting date: 28/08/2017 – End date: 03/09/2017):
  - o stretch - GPS data – **start point: N: 46°29'53.2"**, E: 30°46'30.0" and end point: **N: 43°01'21.3", E: 37°33'51.3"**
  - o loaded volume: 600 L
- Filter 2 (JOSS 2 - Cell 1; JOSS 2 - Cell 2)
- Starting date: 03/09/2017 – End date: 05/09/2017):
  - o stretch - GPS data – **start point: N: 41°40'56.6"**, E: 41°37'38.3" and end point: **N: 42°55'49.2", E: 37°01'46.3"**
  - o loaded volume: 303 L
- Filter 3 (JOSS 3 - Cell 1; JOSS 3 - Cell 2)
- Starting date: 05/09/2017 – End date: 06/09/2017)
  - o stretch - GPS data – **start point: N: 42°55'49.2"**, E: 37°01'46.3" and end point: **N: 43°24'53.1", E: 32°52'00.1"**
  - o loaded volume: 293 L
- Filter 4 (JOSS 4 - Cell 1; JOSS 4 - Cell 2)
- Starting date: 06/09/2017 – End date: 07/09/2017):
  - o stretch - GPS data – **start point: N: 43°24'53.1"**, E: 32°52'00.1" and end point: **N: 46°29'57.6", E: 30°46'06.3"**
  - o loaded volume: 300 L
- Filter 5 (JOSS 5 - Cell 1; JOSS 5 - Cell 2)
- Starting date: 07/09/2017 – End date: 08/09/2017):
  - o stretch - GPS data – **start point: N: 46°29'57.6"**, E: 30°46'06.3" and end point: **N: 44°06'50.8", E: 28°43'40.4"**
  - o loaded volume: 295 L

The graphical view of transects is shown in Fig.16. Figure 17 shows the overlapping between LV Transect Sampling and the 12 spot samples in open sea.

Figure 18 shows the LV filters and cells arrived in the laboratory after the cruise and show filters as they appeared after extraction.



Figure 16: LV sampling transects in open sea

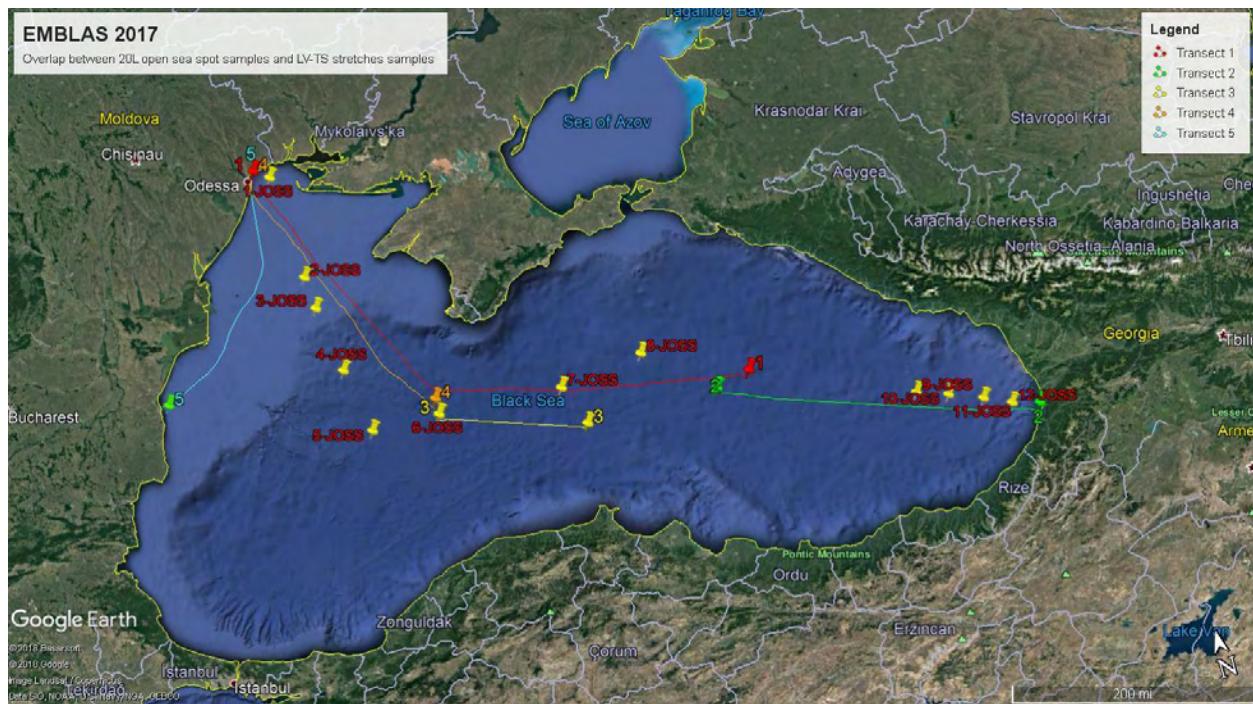


Figure 17: Overlapping between LV transects and the 12 spot samples in open sea



*Figure 18: LV filters and cells arrived in the laboratory after the cruise and the filter cartridges after extraction*

### 3.4. Large Volume Transect Sampling extraction method

Each transect sample consist 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. 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 3.

*Table 3: 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 <sub>2</sub> evaporation	2	Final volume 100 µl in Toluene
	Submitted to HRGC-HRMS analysis for background		

Extraction cells	Method/Activity	Cycles	Solvent
	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 19). 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 methanolic phase was carried out by liquid-liquid extraction (LLE) using Hexane. The Hexane was evaporated to 10 ml and divided in 3 fractions: one for JRC analysis, one for University of Athens and the last one for SK Environmental Institute.

Working conditions are summarized in Table 4.



Figure 19: Cells extraction on ASE system and filter extraction on USE system

Table 4: 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	LLE	3	Hexane 100%

Extraction cells	Method/Activity	Cycles	Solvent
Extracts evaporation after LLE	N <sub>2</sub> evaporation	1	Final volume 10 ml in Hexane
Each extract	Subdivided into 3 parts		JRC (3 ml), UoA (3.5 ml), EI (3.5 ml)
<b>JRC's part of extract</b>			
Extracts	Spiking with both apolar internal and syringe mixture of standards		
Extracts evaporation	N <sub>2</sub> evaporation		Volume 100 µl in toluene
	Submitted to HRGC-HRMS analysis		

Filter cartridges (Figure 19) including those used for the field blank evaluation were extracted using an ultra-sonic extraction method (USE). As for the cells, the filter were cleaned, extracted and analysed before sampling. Filters pre-cleaning and conditioning method is summarized in Table 5.

*Table 5: 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		

Filters	Method/Activity	Cycles	Solvent
	mixture of standards		
Extracts evaporation	N <sub>2</sub> 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%
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 USE 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 to 10 ml and divided in 3 fractions: one for JRC analysis, one for University of Athens and one for SK Environmental Institute.

Filters were processed separately, all working conditions are summarized in Table 6.

*Table 6: 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	LLE	3	Hexane 100%
Extracts evaporation after LLE	N <sub>2</sub> evaporation	1	Final volume 10 ml in hexane

Filters	Method/Activity	Cycles	Solvent
Each extract	Subdivided into 3 parts		JRC (3 ml), UoA (3.5 ml), EI (3.5 ml)
<b>JRC's part of extract</b>			
Extracts	Spiking with both apolar and syringe mixture of standards	1	
Extracts evaporation	N <sub>2</sub> evaporation	1	Volume 100 µl in Toluene

## 4. Analytical methods

### 4.1. QA/QC

Quantification of selected analytes was performed using isotopic dilution method, implying the use of 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 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 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 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 where just one ion was recorded).

Levels of analytical and field blanks were controlled during all process (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

Multi-residual UHPLC-MS/MS using isotope dilution method was developed and included the following analytes (Table 7):

*Table 7: Polar compound analysed by UHPLC-MS/MS*

Analyte ID	Use/Application
Benzotriazole	Corrosion inhibitor
2,4-D	
Atrazine	
Simazine	
MCPA	Herbicide
Terbutryn	
Terbutylazine	
Aclonifen	

Analyte ID	Use/Application
Irgarol	Algicide
Carbamazepine	Anti-epileptic drug
10,11-dihydro-10,11-dihydroxy-carbamazepine	Metabolite of carbamazepine
Bezafibrate	
Gemfibrozil	Fibrate (hypolipidemic agent)
Ibuprofen	
Naproxen	NSAID
Diclofenac	
PFBS	
PFHpA	
PFHxA	
PFHxS	Fluorinated compounds
PFNA	
PFOA	
PFOS	
Sulfamethazine	
Sulfamethoxazole	Antibiotic
Acetamiprid	Neonicotinoid insecticide

#### 4.2.1. UHPLC conditions

Experimental conditions for polar compounds UHPLC-MSMS analysis are reported in Table 8.

*Table 8: UHPLC experimental conditions for polar compounds chromatographic separation*

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:	600 µL/min
Injection volume:	10 µL
Analytical column:	Hypersil GOLD, 2.1x100 mm, 1.9 µm, Thermo Scientific

Mobile phase:	A: CH <sub>3</sub> CO <sub>2</sub> NH <sub>4</sub> 5 mM B: Acetonitrile: Methanol, 9:1 (% , v/v)
Reconstituting solution	A: B, 90:10, (% v/v)

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

Table 9: UHPLC gradient scheme

Time (min)	Mobile phase (A%)	Mobile Phase B (%)
0	90	10
1	90	10
10	10	90
10.5	10	90
11	90	10
15	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 preserved their molecular identity. The data were collected using the software program Analyst 1.6.1 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: Polarity Switching 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):  $\pm$  4500.00

Ion Source Gas 1 (GS1) 55

Ion Source Gas 2 (GS2) 45

Target Scan Time 0.1 sec

MRM detection window 10 sec

Table 10: QTRAP MS/MS parameters

Analyte ID	Internal standard	Q1	Q3	DP	EP	CE	CXP
1-H-Benzotriazole	Benzotriazole d <sub>4</sub>	120	65	209	10	29	13
1-HBenzotriazole 1	Benzotriazole d <sub>4</sub>	120	92	209	10	24	13
Benzotriazole d <sub>4</sub>		124	69	56	10	35	13
2,4-D	2,4-D <sup>13</sup> C <sub>6</sub>	219	161	-130	-10	-24	-11
2,4-D 1	2,4-D <sup>13</sup> C <sub>6</sub>	219	125	-130	-10	-38	-11
2,4-D <sup>13</sup> C <sub>6</sub>		225	167	-68	-10	-19	-11
Bezafibrate	Bezafibrate D <sub>4</sub>	360	274	-100	-10	-24	-11
Bezafibrate 1	Bezafibrate D <sub>4</sub>	360	154	-100	-10	-39	-11
Bezafibrate D <sub>4</sub>		364	278	-165	-10	-24	-11
Gemfibrozil	Gemfibrozil d <sub>6</sub>	249	121	-100	-10	-30	-11
Gemfibrozil 1	Gemfibrozil d <sub>6</sub>	249	106	-100	-10	-60	-11
Gemfibrozil d <sub>6</sub>		255	121	-100	-10	-20	-11
Diclofenac	Diclofenac <sup>13</sup> C <sub>6</sub>	294	250	-42	-10	-16	-11
Diclofenac 1	Diclofenac <sup>13</sup> C <sub>6</sub>	294	214	-42	-10	-29	-11
Diclofenac <sup>13</sup> C <sub>6</sub>		300	256	-173	-10	-15	-11
Ibuprofen	Iburpofen <sup>13</sup> C <sub>3</sub>	205	161	-132	-10	-10	-11
Ibuprofen 1	Iburpofen <sup>13</sup> C <sub>3</sub>	205	159	-132	-10	-10	-11
Iburpofen <sup>13</sup> C <sub>3</sub>		208	163	-81	-10	-11	-11
Naproxen	Naproxen <sup>13</sup> C <sub>3</sub>	229	169	-100	-10	-47	-11
Naproxen 1	Naproxen <sup>13</sup> C <sub>3</sub>	229	185	-100	-10	-10	-11
Naproxen <sup>13</sup> C <sub>3</sub>		233	169	-42	-10	-46	-11

Analyte ID	Internal standard	Q1	Q3	DP	EP	CE	CXP
Atrazine	Atrazine $^{13}\text{C}_3$	216	174	258	10	25	13
Atrazine	Atrazine $^{13}\text{C}_3$	216	104	258	10	40	13
Atrazine $^{13}\text{C}_3$		219	177	100	10	25	13
Carbamazepine	Carbamazepine $d_{10}$	237	194	250	10	28	13
Carbamazepine	Carbamazepine $d_{10}$	237	165	250	10	60	13
Carbamazepine $d_{10}$		247	204	234	10	31	13
10,11-dihydro 10,11-dihydroxy carbamazepine	Carbamazepine $d_{10}$	271	180	80	10	47	13
10,11-dihydro 10,11-dihydroxy carbamazepine 1	Carbamazepine $d_{10}$	271	210	80	10	19	13
10,11-dihydro 10,11-dihydroxy carbamazepine 2	Carbamazepine $d_{10}$	271	253	80	10	10	13
Simazine	Simazine $^{13}\text{C}_3$	202	104	253	10	34	13
Simazine	Simazine $^{13}\text{C}_3$	202	132	253	10	26	13
Simazine $^{13}\text{C}_3$		205	70	218	10	45	13
Simazine $^{13}\text{C}_3$		205	106	218	10	35	13
Simazine $^{13}\text{C}_3$		205	134	218	10	27	13
Sulfamethoxazole	Sulfamethoxazole $^{13}\text{C}_6$	254	156	150	10	22	13
Sulfamethoxazole 1	Sulfamethoxazole $^{13}\text{C}_6$	254	92	150	10	38	13
Sulfamethoxazole $^{13}\text{C}_6$		260	98	70	10	36	13
Aclonifen	Aclonifen $d_5$	265.3	248	120	10	21	13
Aclonifen 1	Aclonifen $d_5$	265.3	194	120	10	25	13

Analyte ID	Internal standard	Q1	Q3	DP	EP	CE	CXP
Aclonifen d <sub>5</sub>		270	253	120	10	22	13
Terbutryn	Terbutryn d <sub>5</sub>	242	186	255	10	25	13
Terbutryn 1	Terbutryn d <sub>5</sub>	242	91	255	10	36	13
Terbutryn d <sub>5</sub>		247	191	228	10	27	13
Terbutylazine	Atrazine <sup>13</sup> C <sub>3</sub>	230	174	219	10	26	13
Terbutylazine	Atrazine <sup>13</sup> C <sub>3</sub>	230	132	219	10	35	13
Acetamiprid	Acetamiprid d <sub>3</sub>	223	126	80	10	29	13
Acetamiprid	Acetamiprid d <sub>3</sub>	223	73	80	10	76	13
Acetamiprid d <sub>3</sub>		226	126	80	10	27	13
Cybutrine	Cybutryune d <sub>9</sub>	254	198	261	10	26	13
Cybutrine 1	Cybutryune d <sub>9</sub>	263	74	269	10	61	13
Cybutryune d <sub>9</sub>		263	199	269	10	27	13
PFBS	PFOS <sup>13</sup> C <sub>4</sub>	299	80	-260	-10	-66	-11
PFBS 1	PFOS <sup>13</sup> C <sub>4</sub>	299	99	-260	-10	-39	-11
PFHpA	PFOA <sup>13</sup> C <sub>4</sub>	363	319	-116	-10	-14	-11
PFHpA 1	PFOA <sup>13</sup> C <sub>4</sub>	363	169	-116	-10	-24	-11
PFHxA	PFHxA <sup>13</sup> C <sub>2</sub>	313	269	-107	-10	-12	-11
PFHxA 1	PFHxA <sup>13</sup> C <sub>2</sub>	313	119	-107	-10	-28	-11
PFHxA <sup>13</sup> C <sub>2</sub>		315	270	-60	-10	-13	-11
PFHxS	PFOS <sup>13</sup> C <sub>4</sub>	399	80	-260	-10	-93	-11
PFHxS 1	PFOS <sup>13</sup> C <sub>4</sub>	399	99	-260	-10	-66	-11
PFNA	PFNA <sup>13</sup> C <sub>5</sub>	463	419	-122	-10	-19	-11
PFNA 1	PFNA <sup>13</sup> C <sub>5</sub>	463	219	-122	-10	-25	-11

Analyte ID	Internal standard	Q1	Q3	DP	EP	CE	CXP
PFNA $^{13}\text{C}_5$		468	423	-57	-10	-16	-11
PFOA	PFOA $^{13}\text{C}_4$	413	369	-122	-10	-16	-11
PFOA 1	PFOA $^{13}\text{C}_4$	413	169	-122	-10	-26	-11
PFOA $^{13}\text{C}_4$		417	372	-119	-10	-15	-11
PFOS	PFOS $^{13}\text{C}_4$	499	80	-260	-10	-97	-11
PFOS 1	PFOS $^{13}\text{C}_4$	499	99	-260	-10	-83	-11
PFOS $^{13}\text{C}_4$		503	80	-276	-10	-104	-11

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

#### 4.3. GC-MS for semi-polar and apolar compound analysis

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

EC-7 PCBs, Pesticides, Atrazine, 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) two ions of the isotopic cluster were selected coming from the fragmentation and chosen on the basis of close elution of different OCPs and the dynamic mass range of the HRMS. For not chlorinated pesticides two most abundant ions were selected coming from the fragmentation 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 molecular ion and -15 m/z ion were recorded. For EHMC the two most abundant ions were recorded.

For OPCs two most abundant ions were selected coming from the fragmentation and 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 with 0.25 mm i.d. (inner diameter) and 0.25  $\mu\text{m}$  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  $\text{min}^{-1}$  of He, GC-MS interface at 300 °C and a

GC program rate: 80 °C for 1 min., 10 °C min<sup>-1</sup> to 250 °C for 5 min., then 5 °C min<sup>-1</sup> to 300 °C for a final isotherm of 1 min.

In Table 11 the exact recorded masses and retention time in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

*Table 11: 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	
	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	TNBP-d27	
	139.0155	155.0468	12.25	TNBP		
	124.9998	155.0468	13.84	TIBP		
4	261.0598	263.0568	15.09	TCEP-d12	TCEP-d12	p-terphenyl-d14
	248.9845	250.9786		TCEP		
			15.65	TCPP-1		
	277.0158	279.0128	15.78	TCPP-2		
			15.89	TCPP-3		
	244.1969		19.87	p-terphenyl-d14		
5	393.9775	395.9746	21.38	TDCPP-d15	TDCPP-d15	
	380.8939	382.9746	21.62	TDCPP		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
6	299.1618	300.1652	22.42	TBOEP- <sup>13</sup> C <sub>6</sub>		
	303.1752	304.1785	22.43	TBOEP	TBOEP- <sup>13</sup> C <sub>6</sub>	
	343.1228	344.1306	22.69	TPhP- <sup>13</sup> C <sub>18</sub>		
	339.1503	341.1644	22.56	TPhP-d15		
	325.0624	326.0702	22.70	TPhP		
7	250.0389	251.0468	23.03	EHDP	TPhP- <sup>13</sup> C <sub>18</sub>	
8	98.9842	113.1325	23.44	TEHP		
9	367.1094	368.1172	403.1893	419.2206	30.27	T35DMPP-d9
					26.85	TMPP-1
					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. Pesticides and Chlorinated Flame Retardants

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 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<sup>-1</sup> of He, GC-MS interface at 250 °C and a GC program rate: 100 °C for 1 min., 10 °C min<sup>-1</sup> to 270 °C for 5 min., then 30 °C min<sup>-1</sup> to 300 °C for a final isotherm of 9 min.

In Table 12 exact recorded mass and retention time in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

*Table 12: HRGC-HRMS experimental conditions for 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		
	184.9765	186.9735	8.44	Dichlorvos	Dichlorvos-d6	
	230.8512	232.8483	8.26	HCBD <sup>13</sup> C <sub>4</sub>		
	222.8408	224.8408	8.26	HCBD	HCBD <sup>13</sup> C <sub>4</sub>	
2	255.8693	257.8663	12.15	PeCBz <sup>13</sup> C <sub>6</sub>		
	249.8491	251.8462	12.15	PeCBz	PeCBz <sup>13</sup> C <sub>6</sub>	
	264.0227	306.0696	13.54	Trifluralin	γ-HCH- <sup>13</sup> C <sub>6</sub>	
3	222.9341	224.9312	14.21	α-HCH- <sup>13</sup> C <sub>6</sub>		
	216.9140	218.9110	14.21	α-HCH	α-HCH- <sup>13</sup> C <sub>6</sub>	
	222.9341	224.9312	14.75	β-HCH- <sup>13</sup> C <sub>6</sub>		
	222.9341	224.9312	14.92	γ-HCH- <sup>13</sup> C <sub>6</sub>		
	216.9140	218.9110	14.75	β-HCH	γ-HCH- <sup>13</sup> C <sub>6</sub>	
	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
3	268.0324	270.0295	15.41	Triallate		
	216.9140	218.9110	15.63	$\epsilon$ -HCH		
	289.8297	291.8268	14.42	HCB- <sup>13</sup> C <sub>6</sub>		
	283.8096	285.8067	14.42	HCB		HCB- <sup>13</sup> C <sub>6</sub>
	276.8264	278.8234	16.43	Heptachlor- <sup>13</sup> C <sub>10</sub>		
	271.8096	273.8067	16.44	Heptachlor		Heptachlor- <sup>13</sup> C <sub>10</sub>
	324.0196	326.0167	16.98	Chlorpyriphos-d10		
	313.9539	315.9539	17.07	Chlorpyriphos		Chlorpyriphos-d10
	269.8799	271.8769	17.12	Chloran 542		Aldrin- <sup>13</sup> C <sub>12</sub>
4	269.8799	271.8769	17.18	Aldrin- <sup>13</sup> C <sub>12</sub>		
	262.8564	264.8535	17.19	Aldrin		Aldrin- <sup>13</sup> C <sub>12</sub>
	333.0629	335.0599	17.77	Chlorfenvinphos-d10		
	323.0001	324.9972	17.85	Chlorfenvinphos		Chlorfenvinphos-d10
	375.9125	377.9095	17.79	Isodrin- <sup>13</sup> C <sub>12</sub>		
	363.8722	365.8693	17.80	Isodrin		Isodrin- <sup>13</sup> C <sub>12</sub>
	362.8772	364.8743	17.95	Cis-Heptachlor-epoxide- <sup>13</sup> C <sub>10</sub>		$p,p'$ -DDD-d8
	352.8437	354.8407	17.95	Cis-Heptachlor-epoxide	Cis-Heptachlor-epoxide- <sup>13</sup> C <sub>10</sub>	
	352.8437	354.8407	18.04	Trans-Heptachlor-epoxide		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
6	396.8382	398.8353	17.97	Oxychlordane- <sup>13</sup> C <sub>10</sub>		
	386.8047	388.8017	17.97	Oxychlordane	Oxychlordane- <sup>13</sup> C <sub>10</sub>	
	327.9777	329.9748	18.40	o,p-DDE- <sup>13</sup> C <sub>12</sub>		
	315.9375	3179345	18.40	o,p-DDE	o,p-DDE- <sup>13</sup> C <sub>12</sub>	
	347.9027	349.8997	18.73	<b>a</b> -Endosulfan- <sup>13</sup> C <sub>9</sub>		
	338.8725	340.8695	18.73	<b>a</b> -Endosulfan	<b>a</b> -Endosulfan- <sup>13</sup> C <sub>9</sub>	
	382.8590	384.8560	18.42	Trans-Chlordan- <sup>13</sup> C <sub>10</sub>		
	372.8254	374.8225	18.42	Trans-Chlordan	Trans-Chlordan- <sup>13</sup> C <sub>10</sub>	
	372.8254	374.8225	18.74	Cis-Chlordan		
	416.8200	418.8170	18.83	Trans-Nonachlor- <sup>13</sup> C <sub>10</sub>		
	406.7864	408.7835	18.83	Trans-Nonachlor	Trans-Nonachlor- <sup>13</sup> C <sub>10</sub>	
7	327.9777	329.9748	19.00	<b>p,p'</b> -DDE- <sup>13</sup> C <sub>12</sub>		
	315.9375	3179345	19.01	<b>p,p'</b> -DDE	<b>p,p'</b> -DDE- <sup>13</sup> C <sub>12</sub>	
	247.0481	249.0449	19.23	o,p-DDD- <sup>13</sup> C <sub>12</sub>		
	235.0076	237.0046	19.24	o,p-DDD	o,p-DDD- <sup>13</sup> C <sub>12</sub>	
	269.8799	271.8769	19.28	Dieldrin- <sup>13</sup> C <sub>12</sub>		
	262.8564	264.8535	19.28	Dieldrin	Dieldrin - <sup>13</sup> C <sub>12</sub>	
8	269.8799	271.8769	19.79	Endrin- <sup>13</sup> C <sub>12</sub>		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
8	262.8564	264.8535	19.81	Endrin	Endrin- <sup>13</sup> C <sub>12</sub>	
	243.0578	245.0550	19.91	p,p'-DDD-d8		
	235.0076	237.0046	19.96	p,p'-DDD	o,p-DDD- <sup>13</sup> C <sub>12</sub>	
	239.8605	341.8575	19.99	β-Endosulfan- <sup>13</sup> C <sub>9</sub>		
	234.8437	236.8408	20.00	β-Endosulfan	β-Endosulfan- <sup>13</sup> C <sub>9</sub>	
	247.0481	249.0449	20.09	o,p-DDT- <sup>13</sup> C <sub>12</sub>		
	235.0076	237.0046	20.10	o,p-DDT	o,p-DDT- <sup>13</sup> C <sub>12</sub>	
	239.8605	341.8575	20.24	Cis-Nonachlor- <sup>13</sup> C <sub>10</sub>		
	234.8437	236.8408	20.25	Cis-Nonachlor	Cis-Nonachlor- <sup>13</sup> C <sub>10</sub>	
9	247.0481	249.0449	20.09	p,p'-DDT- <sup>13</sup> C <sub>12</sub>		
	235.0076	237.0046	20.10	p,p'-DDT	p,p'-DDT- <sup>13</sup> C <sub>12</sub>	
	276.8264	278.8234	21.02	Endosulfan-sulfate- <sup>13</sup> C <sub>9</sub>		
	271.8096	273.8067	21.03	Endosulfan-sulfate	Endosulfan-sulfate- <sup>13</sup> C <sub>9</sub>	
10	239.1475	240.1508	22.44	Methoxychlor- <sup>13</sup> C <sub>12</sub>		
	227.1067	228.1106	22.45	Methoxychlor		
	258.0527	261.0498	22.44	Dicofol-d8	Methoxychlor- <sup>13</sup> C <sub>12</sub>	
	251.0025	252.9995	22.68	Dicofol		
11	276.8264	278.8234	24.14	Mirex- <sup>13</sup> C <sub>10</sub>		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	271.8096	273.8067	24.15	Mirex	Mirex- <sup>13</sup> C <sub>10</sub>	
	169.0452	171.0423	25.93 26.08	Trans-Cypermehtrin-d6 (2 isomers)		
12	163.0081	165.0052	25.88 25.98 26.09	Mix solution of Trans and Cis-Cypermehtrin (4 isomers)	Trans-Cypermehtrin-d6	
13	271.8096	273.8067	35.57	Syn-Dechlorane Plus	Mirex- <sup>13</sup> C <sub>10</sub>	
	271.8096	273.8067	36.73	Syn-Dechlorane Plus		

#### 4.3.3. Triazines

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<sup>-1</sup> of He, GC-MS interface at 280 °C and a GC program rate: 100 °C for 1 min., 7 °C min<sup>-1</sup> to 160 °C for 6 min., then 30 °C min<sup>-1</sup> to 320 °C for a final isotherm of 4 min.

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

HRGC-HRMS was used for triazines analysis only for the LV transect samples. The other samples were analysed using UPLC-MS/MS.

Table 13: HRGC-HRMS experimental conditions for triazine analysis

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
1	201.0781	203.0752	18.91	Simazine	Atrazine- <sup>13</sup> C <sub>3</sub>	p,p'-DDD-d8

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	200.0703	202.0673	19.01	Atrazine $^{13}\text{C}_3$		
	203.0804	205.0774	19.01	Atrazine	Atrazine $^{13}\text{C}_3$	
	214.0854	216.0824	19.28		Atrazine $^{13}\text{C}_3$	
	243.0578	245.0550	24.15	p,p'-DDD-d8		

#### 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  $\mu\text{m}$  film (Agilent J&W, USA).

Gas chromatographic conditions were:

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

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

Table 14: 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		
	128.0621		7.87	Naphthalene	Naphthalene-d8	
2	160.1123		10.94	Acenaphthylene-d8		
	152.0621		10.97	Acenaphthylene	Acenaphthylene-d8	Biphenyl-d10
	164.1405		9.91	Biphenyl-d10		
	164.1405		11.31	Acenaphthene-d10		
	154.0777		11.39	Acenaphthene	Acenaphthene-d10	

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	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		
	178.0777		14.87	Phenanthrene	Phenanthrene-d10	
	188.1405		14.93	Anthracene-d10		
	178.0777		14.98	Anthracene	Anthracene-d10	
	212.1405		17.72	Fluoranthene-d10		
4	202.0777		17.77	Fluoranthene	Fluoranthene-d10	p-terphenyl-d14
	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		
	240.1687		21.16	Benzo(a)anthracene-d12		
5	228.0934		21.21	Benzo(a)anthracene	Chrysene-d12	Benzo(a)anthracene-d12
	240.1687		21.25	Chrysene-d12		
	228.0934		21.31	Chrysene	Chrysene-d12	

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
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	
	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 HT-8 capillary columns, 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<sup>-1</sup> of He, GC-MS interface at 280 °C and a GC program rate: Starting from 120 °C with 20 °C min<sup>-1</sup> to 180 °C, 2 °C min<sup>-1</sup> to 260 °C, and 5 °C min<sup>-1</sup> to 300 °C isotherm for 4 min.

In Table 15 exact recorded mass and retention time in HRGC-HRMS for native compounds, internal and syringe labelled standards are reported.

*Table 15: 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 <sup>13</sup> C <sub>12</sub>		PCB-31 <sup>13</sup> C <sub>12</sub>
	268.0016	269.9986	23.58	PCB-28 <sup>13</sup> C <sub>12</sub>		
	255.9613	257.9584	23.61	PCB-28	PCB-28 <sup>13</sup> C <sub>12</sub>	
	301.9626	303.9597	25.76	PCB-52 <sup>13</sup> C <sub>12</sub>		
	289.9224	291.9194	25.78	PCB-52	PCB-52 <sup>13</sup> C <sub>12</sub>	
2	337.9207	339.9178	33.61	PCB-101 <sup>13</sup> C <sub>12</sub>		PCB-111 <sup>13</sup> C <sub>12</sub>
	325.8804	327.8775	33.63	PCB-101 <sup>13</sup> C <sub>12</sub>	PCB-101 <sup>13</sup> C <sub>12</sub>	
	337.9207	339.9178	36.40	PCB-111 <sup>13</sup> C <sub>12</sub>		
	337.9207	339.9178	40.34	PCB-118 <sup>13</sup> C <sub>12</sub>		
	325.8804	327.8775	40.37	PCB-118	PCB-118 <sup>13</sup> C <sub>12</sub>	
	371.8817	373.8788	41.76	PCB-153 <sup>13</sup> C <sub>12</sub>		
	359.8415	361.8385	41.79	PCB-153	PCB-153 <sup>13</sup> C <sub>12</sub>	
	371.8817	373.8788	44.46	PCB-138 <sup>13</sup> C <sub>12</sub>		

Group number	m/z 1	m/z 2	RT	Analyte	Internal Standard	Recovery Standard
	359.8415	361.8385	44.49	PCB-138	PCB-138 $^{13}\text{C}_{12}$	
3	405.8428	407.8398		PCB-180 $^{13}\text{C}_{12}$		
	393.8025	395.7995		PCB-180	PCB-180 $^{13}\text{C}_{12}$	PCB-170 $^{13}\text{C}_{12}$
	405.8428	407.8398		PCB-170 $^{13}\text{C}_{12}$		

## 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 (Mariani Box).

#### 5.1.1. Polar compounds

Table 16 reports average recovery of internal standards and relative coefficients of variation calculated in real samples and sampling efficiency evaluated in real samples 1-GE and 1-A, samples for which the break-through tests were available. The break-through tests for CW-7 is not reported because recovery of internal standards was not appreciable in the sample CW-7 BT.

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

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

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

Table 17 report **methods' LODs** (calculated as signal to noise 3:1) and LOQs (calculated as signal to noise 10:1).

The real samples 1-A and 1-A' were sampled for the reproducibility test. In table 18 the concentrations of detected compounds, their relative coefficients of variation calculated, below the table their graphs are reported.

*Table 16: Recovery, Sampling efficiency of Polar Compounds*

	Steel tank container		HDPE container GE samples		HDPE container CW samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %	Average Recovery (%)	CV %	1-GE	1-A
Benzotriazole d <sub>4</sub>	0.4	98.6	1.5	86.5	1.2	23.6	55.2	55.0
2,4-D <sup>13</sup> C <sub>6</sub>	15.9	99.4	84.0	55.9	31.9	60.2	59.6	50.0
Atrazine 13C <sub>3</sub>	25.0	23.4	36.2	31.1	11.5	57.0	50.5	50.0
Bezafibrate d <sub>4</sub>	24.9	19.7	48.8	37.6	14.4	52.6	62.9	41.2
Carbamazepine d <sub>10</sub>	24.3	24.3	37.4	33.4	10.6	56.2	55.2	46.2
Diclofenac <sup>13</sup> C <sub>6</sub>	9.9	12.6	15.3	33.3	10.2	50.0	50.0	50.0
Gemfibrozil d <sub>6</sub>	37.7	27.3	50.9	34.7	24.8	54.8	46.4	43.9

Ibuprofen $^{13}\text{C}_3$	82.6	22.7	132.7	36.1	43.1	38.3	50.0	40.8
Cybutryny d <sub>9</sub>	13.0	27.1	9.6	46.8	7.9	60.9	37.1	41.3
MCPA d <sub>3</sub>	20.9	87.9	112.6	57.1	50.0	54.0	57.0	49.5
Naproxen $^{13}\text{C}_3$	36.3	31.7	74.8	31.5	23.8	49.5	66.7	42.9
PFHxA $^{13}\text{C}_2$	40.1	53.9	131.3	42.3	33.8	50.6	60.5	49.3
PFOA $^{13}\text{C}_4$	127.5	25.8	138.9	10.5	46.8	37.1	47.3	42.6
PFOS $^{13}\text{C}_4$	74.8	31.5	90.2	15.6	27.7	82.8	57.5	37.1
PFNA $^{13}\text{C}_5$	209.1	25.2	293.8	26.2	129.8	53.2	55.1	46.3
Sulfamethazine $^{13}\text{C}_6$	1.1	66.2	2.0	70.7	1.8	26.8	47.2	60.2
Sulfamethoxazole $^{13}\text{C}_6$	0.7	196.4	0.5	72.4	0.9	40.1	53.9	62.8
Terbutryny d <sub>5</sub>	18.7	25.4	16.6	34.8	11.7	61.0	42.4	43.9
Acetamiprid d <sub>3</sub>	9.8	19.1	16.7	95.2	4.6	48.1	60.0	42.2
Aclonifen d <sub>5</sub>	16.9	41.7	12.9	57.8	14.0	109.1	39.8	32.6

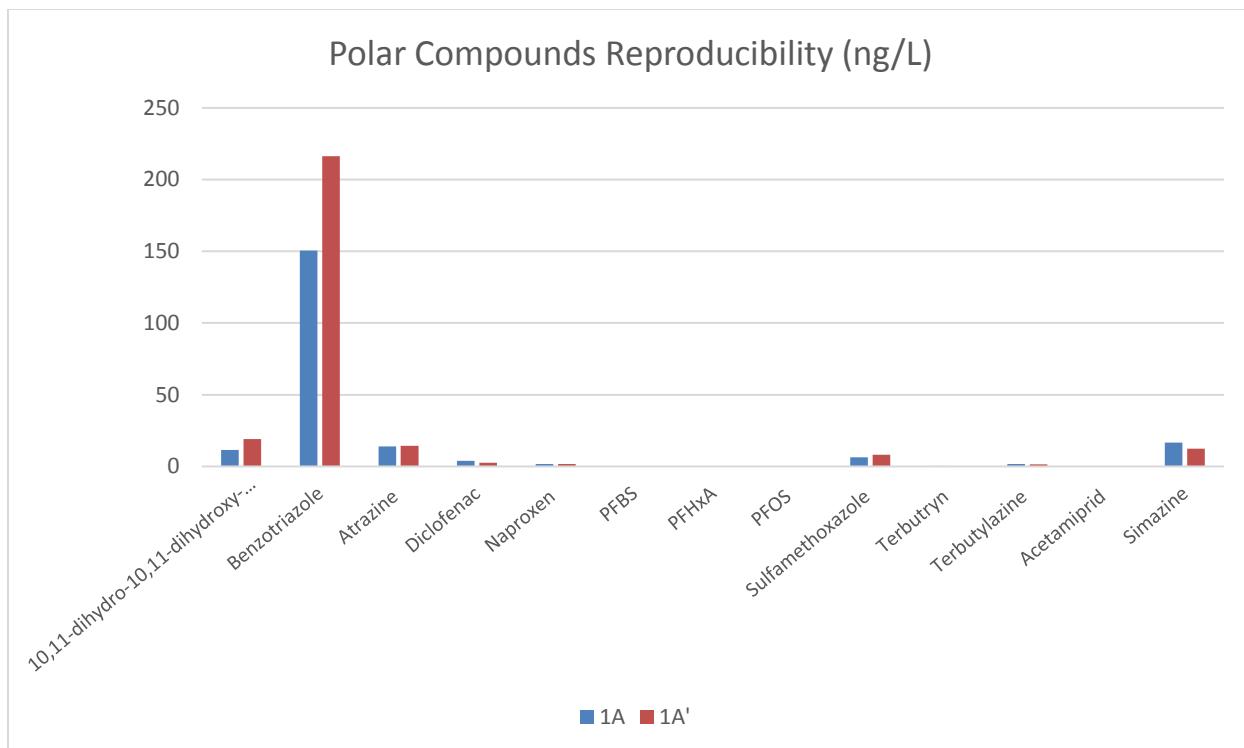
Table 17: LOD/LOQ of Polar Compounds

	LOD 3/1	LOQ 10/1
	ng/l	ng/l
10,11-dihydro 10,11-dihydroxy carbamazepine	0.001	0.004
Benzotriazole	0.597	1.97
2,4-D	0.017	0.06
Atrazine	0.006	0.02
Bezafibrate	0.006	0.02
Carbamazepine	0.006	0.02
Diclofenac	0.182	0.6
Gemfibrozil	0.003	0.01
Ibuprofen	1.227	4.05
Irgarol	0.006	0.02
MCPA	0.01	0.03
Naproxen	0.042	0.14
PFBS	0.001	0.004
PFHpA	0.007	0.02
PFHxA	0.006	0.02
PFHxS	0.002	0.01
PFNA	0.032	0.11
PFOA	0.003	0.01
PFOS	0.005	0.02
Sulfamethazine	0.001	0
Sulamethoxazole	0.021	0.07
Terbutryny	0.003	0.01
Terbutylazine	0.091	0.3
Acetamiprid	0.015	0.05

	LOD 3/1	LOQ 10/1
	ng/l	ng/l
Aclonifen	0.008	0.03
Simazine	0.23	0.76

Table 18: Reproducibility data and graph of Polar Compounds

Lab. Code:	17-020	17-021		
Sampling Code:	1A	1A'		
Concentration	ng/L	ng/L	Average	Cv %
10,11-dihydro-10,11-dihydroxy-carbamazepine	11.427	19.077	15.25	35.47
Benzotriazole	150.617	216.309	183	25.32
Atrazine	14.082	14.520	14.30	2.16
Diclofenac	3.838	2.550	3.19	28.52
Naproxen	1.632	1.667	1.65	1.52
PFBS	0.006	0.020	0.01	80.15
PFHxA	BLOQ	0.120	0.12	---
PFOS	0.079	0.097	0.09	14.21
Sulfamethoxazole	6.335	8.138	7.24	17.62
Terbutryn	0.328	0.251	0.29	18.95
Terbutylazine	1.659	1.563	1.61	4.21
Acetamiprid	0.135	0.119	0.13	8.88
Simazine	16.713	12.358	14.54	21.19



### **5.1.2.** Semi-polar and apolar compounds

Tables 19, 22, 24 and 27 report: average recovery of internal standards and relative coefficients of variation calculated in real samples, sampling efficiency evaluated in real samples 1-GE and 1-A for which the break-through tests were available. The break-through tests for CW-7 is not reported because no recovery of internal standards was appreciable in the sample CW-7 BT.

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

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

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

The recovery of the samples collected in steel tank and HDPE (GE and CW) containers are reported separately because substantial difference in recovery was found between them. Very low recovery was detected in samples collected in HDPE containers.

Tables 20, 23, 25 and 28 report method LODs (calculated as signal to noise 3:1) and LOQs (calculated as signal to noise 10:1) respectively for OPCs, pesticides, PAHs and PCBs.

The real samples 1-A and 1-A' were sampled for the reproducibility test. In the tables 21, 26 and 29 are reported the concentrations of compounds detected, their relative coefficients of variation calculated and graph respectively for OPCs, PAHs and PCBs. Reproducibility test results for pesticides are not reported because a signal suppression occurred in the sample 1-A' during the HRGC-HRMS analysis.

### 5.1.2.1. Organophosphate Compounds OPCs

*Table 19: Recovery and Sampling efficiency of Organophosphate Compounds OPCs*

	Steel tank container		HDPE container GE samples		HDPE container CW samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %	Average Recovery	CV %	1-GE	1-A
TEP-D9	7.83	47.49	17.04	36.61	6.76	51.88	45.72	49.45
TNPP-D21	64.12	28.28	61.17	38.39	16.99	60.09	58.21	56.45
TNBP-D27	87.96	27.28	42.45	50.30	16.75	88.88	52.84	56.64
TCEP-D12	68.87	31.49	49.47	42.92	14.73	88.97	57.70	55.65
TDCPP-D15	74.17	32.87	24.75	64.48	12.67	109.18	46.16	57.08
TBOEP-13C6	110.75	30.43	56.25	61.50	24.18	66.94	55.03	58.74
TPhP-13C18	57.13	30.07	19.73	61.49	10.49	99.12	46.72	57.62
T35DMPP-D9	35.84	35.70	5.14	27.86	6.63	93.46	57.74	46.64

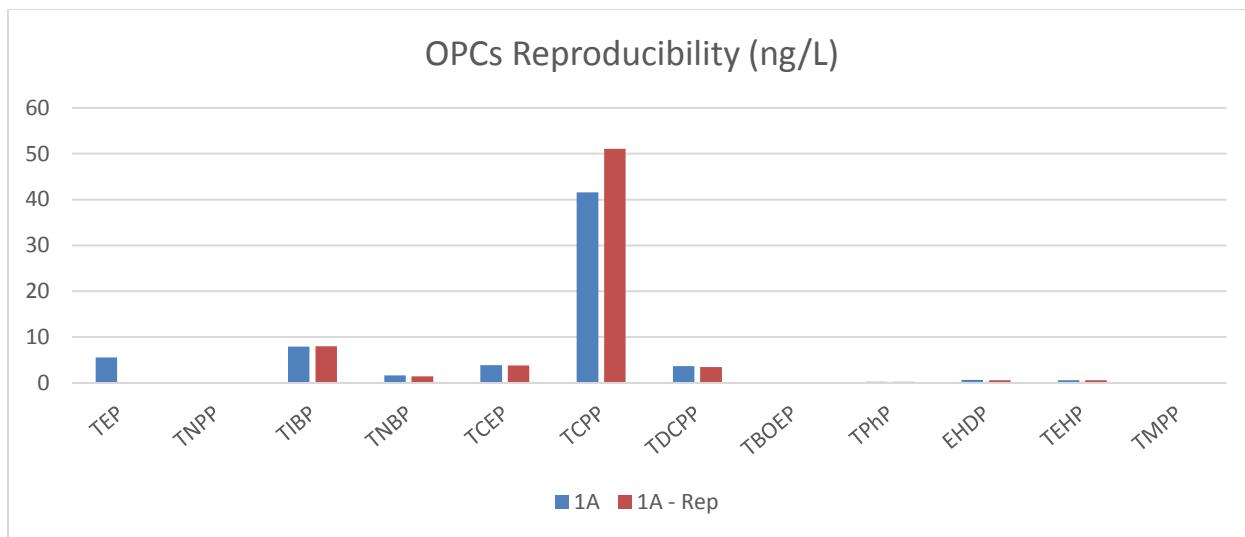
*Table 20: LOD/LOQ of Organophosphate Compounds OPCs*

	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
TNBP	0.02	0.067
TCEP	0.03	0.1
TCPP	0.015	0.05
TDCPP	0.005	0.017

	LOD 3/1	LOQ 10/1
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 21: Reproducibility data and graph of Organophosphate Compounds (OPCs)

Lab. Code:	OPC-17-020	OPC-17-021		
Sampling Code:	1A	1A - Rep		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	20.05	20.00		
Sampling period:	2017	2017		
Analysis date:	11/30/2017	11/30/2017		
Concentration	ng/L	ng/L	Average	Cv %
TEP	5.58	n.d.	5.58	---
TNPP	Below LOD	Below LOD		
TIBP	7.95	7.99	7.97	0.41
TNBP	1.66	1.48	1.57	8.19
TCEP	3.89	3.84	3.87	0.88
TCPP	41.54	51.05	46.29	14.51
TDCPP	3.69	3.44	3.56	4.92
TBOEP	Below LOD	Below LOD	---	---
TPhP	0.28	0.27	0.27	2.76
EHDP	0.70	0.62	0.66	7.85
TEHP	0.59	0.62	0.60	3.01
TMPP	0.15	Below LOD	0.15	---
TIPPP	Below LOD	Below LOD	---	---
T35DMPP	Below LOD	Below LOD	---	---
n.d.: not detected for interference				



### 5.1.2.2. Pesticides and Chlorinated Flame Retardants

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.

Low molecular weight of HCBD and his high volatility makes the methodology not suitable for this substance. The concentration of HCBD must be considered as only indicative.

Table 22: Recovery and Sampling efficiency of Pesticides

	Steel tank container		HDPE container GE samples		HDPE container CW samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	Average Recovery	CV %	Average Recovery (%)	CV %	1-GE	1-A
C13 HCBD	4.63	126.0	5.88	45.70	1.06	109.24	64.15	50.76
D6 Dichlorvos	17.09	61.2	21.85	51.30	6.30	80.08	56.44	77.14
C13_PeCBz	15.73	66.7	12.92	55.58	2.98	80.34	50.07	54.72
D14 Trifluralin								
13C HCB	16.07	46.6	7.32	47.32	6.09	100.72	41.68	48.19
13C a-HCH	40.33	20.0	21.14	54.80	7.83	88.42	55.01	55.09
13C_g-HCH	44.94	19.7	20.56	49.17	8.38	98.44	51.48	56.47
13C_Heptachlor	37.93	30.2	7.94	80.66	3.58	125.23	67.69	54.18
D10 Chlorpyriphos	50.10	22.7	13.68	70.12	11.10	110.44	59.89	53.19
C13_Aldrin	25.22	33.2	4.79	64.93	2.20	117.78	60.18	51.23
D10 Chlорfenvinphos	98.80	20.9	0.004	77.82	18.98	90.15	37.15	57.40
C13 Isodrin	25.72	27.1	0.98	26.80	5.68	145.71	66.95	50.46

	Steel tank container		HDPE container GE samples		HDPE container CW samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	Average Recovery	CV %	Average Recovery (%)	CV %	1-GE	1-A
C13_Oxychlordane	32.55	28.4	0.23	40.47	4.07	147.36	54.02	56.27
C13_Endosulfane-alpha	50.33	24.2	12.66	71.76	5.77	164.55	62.44	53.86
C13_Heptachlor-exo-epoxide	44.68	23.2	0.07	43.13	5.71	121.99	37.12	58.67
C13_trans-chlordanne	29.65	29.1	5.16	96.27	4.03	110.10	66.69	53.36
C13_trans-nonachlor	29.12	28.2	5.55	74.21	5.34	115.09	58.07	50.86
C13_op-DDE	25.69	39.3	4.06	98.59	3.90	103.70	83.01	70.31
C13_pp-DDE	28.28	29.5	4.70	56.67	4.04	100.29	55.44	52.15
C13_op-DDD	43.40	28.2	8.49	82.89	5.82	108.67	65.98	55.82
C13_op-DDT	35.58	32.1	6.25	58.75	4.16	97.68	52.01	43.47
C13_pp-DDT	36.74	34.3	6.03	57.36	4.17	67.61	53.86	50.97
C13_Dieldrin	44.97	24.6	9.82	83.75	5.30	132.31	68.30	25.11
C13_Endrin	25.18	79.1	15.99	77.91	8.60	127.15	63.75	55.12
C13_Endosulfane-beta	47.34	22.1	13.25	79.75	6.12	143.49	64.51	55.84
C13_cis-nonachlor	31.03	29.6	5.84	77.57	3.57	134.96	59.93	50.41
C13_Endosulfane-sulphate	47.64	22.5	10.94	69.85	6.98	120.91	62.50	56.30
C13_Methoxychlor	57.68	19.6	48.51	34.09	14.02	101.26	68.48	54.63
C13_Mirex	25.13	20.6	4.30	23.12	4.94	110.31	77.93	59.60
D6_Cypermethrin	37.78	27.3	5.12	35.37	4.55	105.52	57.82	52.54

Table 23: LOD and LOQ of Pesticides and Chlorinated Flame Retardants

	LOD 3/1	LOQ 10/1
<i>Chlorinated Pesticides:</i>	pg/L	pg/L
PeCBz	0.5	1.67
HCB	0.5	1.67
a-HCH	3	10
b-HCH	3	10
g-HCH	3	10
d-HCH	3	10
e-HCH	3	10
Heptachlor	0.8	2.67
Heptachlor-exo-epoxide	1.5	5

	LOD 3/1	LOQ 10/1
Heptachlor-endo-epoxide	10	33.3
Aldrin	3.5	11.7
Dieldrin	2.5	8.33
Endrin	2	6.67
Isodrin	15	50
trans-chlordane	5	16.7
cis-chlordane	5	16.7
Oxychlordane	3	10
trans-nonachlor	0.5	1.67
cis-nonachlor	3	10
Endosulfane-alpha	15	50
Endosulfane-beta	2	6.67
Endosulfane-sulphate	0.5	1.67
op-DDE	2	6.67
pp-DDE	2	6.67
op-DDD	1.5	5
pp-DDD	1.5	5
op-DDT	3	10
pp-DDT	3	10
Methoxychlor	12	40
Mirex	0.5	1.67
<i>Others Pesticides:</i>		
HCBD	2	6.67
Dichlorvos	15	50
Trifluralin	1	3.33
Triallate	7	23.3
Chlorpyriphos	3.5	11.7

	LOD 3/1	LOQ 10/1
Chlorfenvinphos	25	83.3
Dicofol	180	600
Cypermethrins	35	117
<i>Chlorinated Flame Retardants</i>		
Chloran 542	313	855
syn-Decchlorane Plus	3.2	8
anti-Decchlorane Plus	2.1	5.3

### 5.1.2.3. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC, BHT

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

BHT and EHMC are not reported in the graph. EHMC is a sunscreen product, thus the interpretation of elevated concentrations should take eventual contamination on-site or during sample handling and preparation into account.

Table 24: Recovery and Sampling efficiency of PAHs, EHMC and BHT

	Steel tank container		HDPE container GE samples		HDPE container CW samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %	Average Recovery (%)	CV %	1-GE	1-A
Phenanthrene-d10	30.38	32.02	14.56	54.41	5.78	100.9	51.77	55.62
Anthracene-d10	25.71	32.26	12.01	58.22	5.11	99.9	50.96	59.17
Fluoranthene-d10	43.22	31.61	13.16	59.82	7.63	98.94	51.54	54.44
Pyrene-d10	43.14	31.39	13.79	60.62	7.64	101.4	52.63	55.06
Chrysene-d12	45.07	34.90	13.14	58.34	8.66	83.55	51.10	51.45
Benzo(b)fluoranthene-d12	34.09	35.71	10.03	47.35	7.25	77.84	57.02	56.75
Perylene-d12	30.17	37.46	6.54	49.59	5.40	97.53	60.41	51.86
Benzo(a)pyrene-d12	29.07	37.28	6.73	45.04	5.00	98.47	59.75	53.39
Benzo(e)pyrene-d12	37.12	35.15	8.80	50.25	6.59	100.0	55.72	52.41
Indeno(123-cd)pyrene-d12	32.32	32.13	5.73	36.80	6.09	97.91	69.25	56.29
Benzo(ghi)perylene-d12	31.84	34.49	6.24	29.04	5.90	97.91	70.44	57.83
Dibenz(ah)anthracene-d12	31.01	32.76	5.92	35.44	5.87	98.72	67.79	56.60
Coronene-d12	21.89	44.71	2.94	18.66	2.36	112.3	<b>76.47</b>	<b>60.38</b>
BHT-21	23.35	118.6	7.45	77.78	2.91	100.7	83.56	<b>77.74</b>

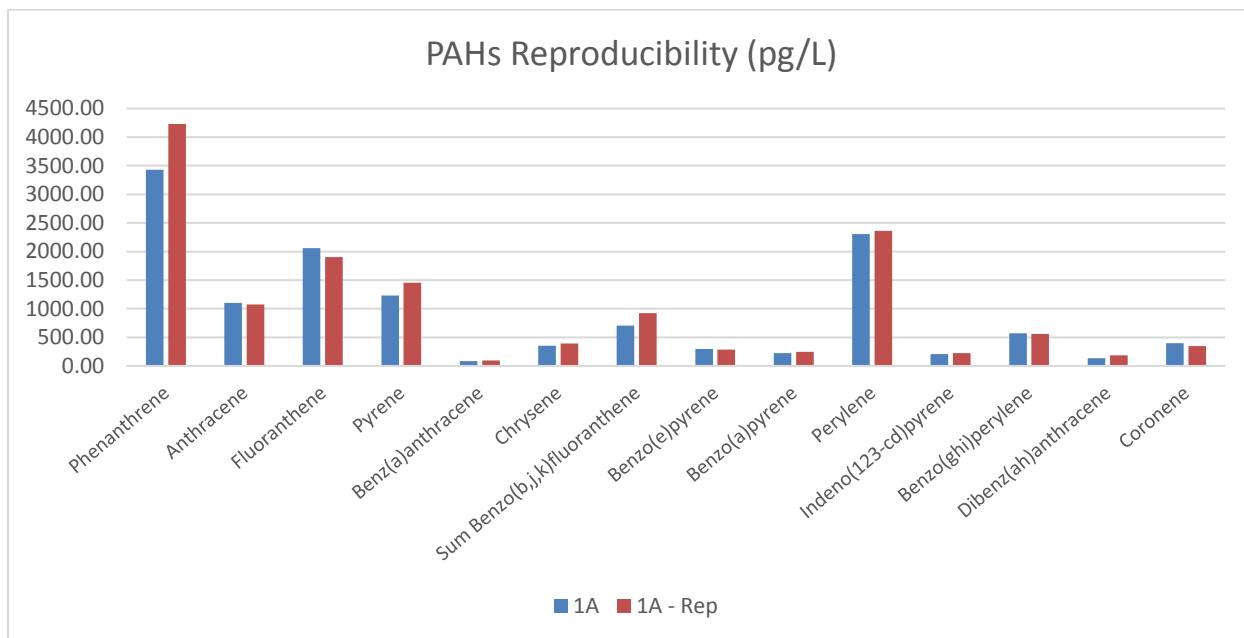
Table 25: LOD and LOQ of PAHs, EHMC and BHT

	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
Dibenz(ah)anthracene	2	6.7
Coronene	4	13.3
BHT	60	200
EHMC	60	200

Table 26: Reproducibility data and graph of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT

Lab. Code:	PAH-17-020	PAH-17-021		
Sample name:	1A	1A - Rep		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	20.05	20.00		
Sampling period:	2017	2017		
Analysis date:	12/7/2017	12/7/2017		
Concentration	pg/L	pg/L	Average	Cv %
Phenanthrene	3432	4229	3830	14.72
Anthracene	1100	1075	1087	1.63
Fluoranthene	2058	1902	1980	5.57

Lab. Code:	PAH-17-020	PAH-17-021		
Sample name:	1A	1A - Rep		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	20.05	20.00		
Sampling period:	2017	2017		
Analysis date:	12/7/2017	12/7/2017		
Concentration	pg/L	pg/L	Average	Cv %
Pyrene	1233	1456	1344	11.71
Benz(a)anthracene	82	97	89	11.43
Chrysene	351	389	370	7.35
Sum Benzo(b,j,k)fluoranthene	704	924	814	19.09
Benzo(e)pyrene	296	285	290	2.69
Benzo(a)pyrene	225	244	234	5.65
Perylene	2304	2364	2334	1.79
Indeno(123-cd)pyrene	207	226	217	6.40
Benzo(ghi)perylene	571	559	565	1.47
Dibenz(ah)anthracene	133	183	158	22.51
Coronene	397	349	373	9.22
BHT	74467	64846	69657	9.77
EHMC	4972	93561	49266	127.15



#### 5.1.2.4. Polychlorinated Biphenyls (PCBs)

Table 27: Recovery and sampling efficiency of PCBs

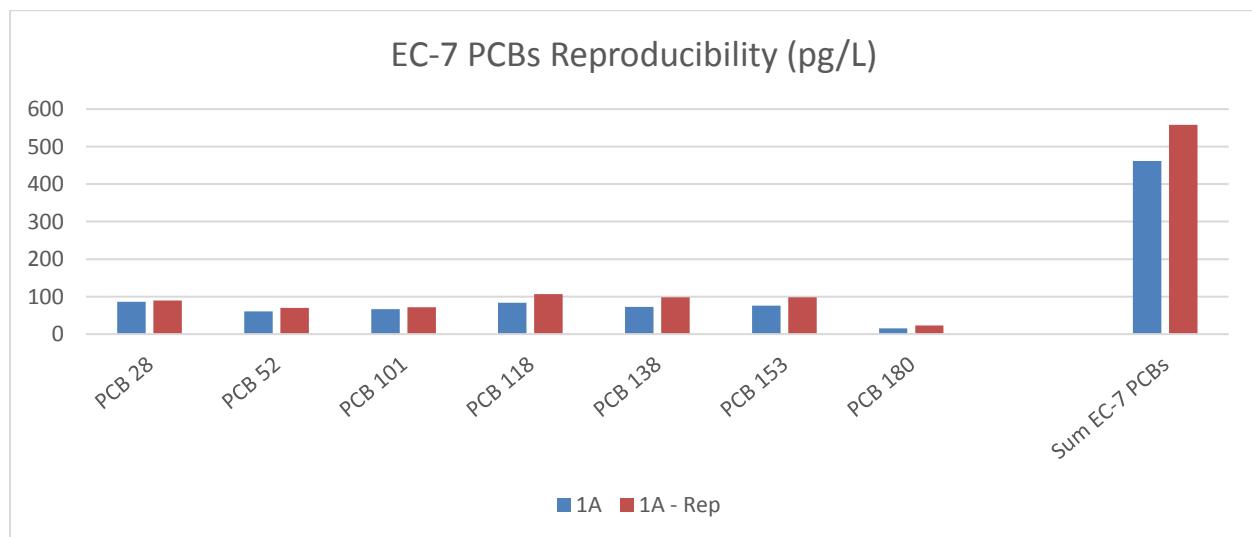
	Steel tank container		HDPE container GE samples		HDPE container CW samples		Sampling efficiency (%)	
	Average Recovery (%)	CV %	Average Recovery (%)	CV %	Average Recovery (%)	CV %	1-GE	1-A
13C12 PCB 28	44.81	39.60	10.06	56.01	7.19	85.90	47.34	45.90
13C12 PCB 52	32.64	40.82	7.10	64.85	4.61	99.01	40.48	46.53
13C12 PCB 101	29.23	41.94	5.57	61.86	3.89	99.87	47.47	48.05
13C12 PCB 118	32.67	40.58	6.12	56.17	4.98	102.7	50.65	52.53
13C12 PCB 138	31.70	36.63	6.10	35.24	5.45	92.86	60.28	53.91
13C12 PCB 153	30.10	36.53	5.79	43.28	5.47	92.41	58.30	51.09
13C12 PCB 180	31.65	30.87	5.39	29.58	6.28	102.8	66.20	55.54

Table 28: 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 29: Reproducibility data and graph of Polychlorinated Biphenyls (PCBs)

Lab. Code:	PCB-17-020	PCB-17-021		
Sample name:	1A	1A - Rep		
Type of sample:	MB Black Sea water	MB Black Sea water		
Volume sampled (L):	20.05	20.00		
Sampling period:	2017	2017		
Analysis date:	12/13/2017	12/13/2017		
Concentration	pg/L	pg/L	Average	Cv %
EC-7				
PCB 28	86.17	89.89	88.03	2.99
PCB 52	60.40	69.61	65.00	10.02
PCB 101	66.33	71.92	69.13	5.72
PCB 118	83.93	107	95.47	17.09
PCB 138	72.53	98.32	85.43	21.34
PCB 153	76.31	98.15	87.23	17.70
PCB 180	15.60	22.96	19.28	26.97
Sum EC-7 PCBs	461	558	510	13.40



## 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. Results are shown in Tables 30, 32, 34 and 36: reporting average of analytical recovery of internal standards and their relative coefficients of variation obtained in filter and cells samples, and method LOD (calculated as blank value plus 3 standard deviation) and LOQ (calculated as blank value plus 10 standard deviation) respectively for OPCs, pesticides, PAHs and PCBs.

Tables 31, 33, 35 and 37 report respectively for OPCs, pesticides, PAHs and PCBs the sampling efficiency obtained in LV transect samples during the cruise. The sampling efficiency was possible to evaluate 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:

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

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

### 5.2.1. Organophosphate Compounds OPCs

*Table 30: Filters and cells analytical recovery and LOD/LOQ of Organophosphate Compounds OPCs*

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %			
						ng/L	ng/L
<b>TEP-D9</b>	76.72	11.37	20.40	66.17	<b>TEP</b>	0.09	0.24
<b>TNPP-D21</b>	89.81	16.77	94.24	45.56	<b>TNPP</b>	0.10	0.24
<b>TNBP-D27</b>	105.38	21.10	100.60	41.79	<b>TIBP</b>	0.068	0.177
<b>TCEP-D12</b>	111.14	17.00	76.88	33.78	<b>TNBP</b>	0.014	0.027
<b>TDCPP-D15</b>	103.66	18.03	88.82	36.37	<b>TCEP</b>	0.003	0.008
<b>TBOEP-13C6</b>	95.48	41.64	105.72	40.85	<b>TCPP</b>	0.012	0.019
<b>TPhP-13C18</b>	84.77	12.63	58.28	35.74	<b>TDCPP</b>	0.005	0.012
<b>T35DMPP-D9</b>	86.20	16.03	68.21	35.55	<b>TBOEP</b>	0.01	0.03

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
					<b>TPhP</b>	0.004	0.006
					<b>EHDP</b>	0.006	0.009
					<b>TEHP</b>	0.001	0.002
					<b>TMPP</b>	0.0003	0.0005
					<b>TIPPP</b>	0.001	0.002
					<b>T35DMPP</b>	<i>0.0006</i>	0.0015

Table 31: Sampling efficiency in 5 transect samplings of Organophosphate Compounds OPCs

Transects					
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5
	Sampling Efficiency (%)				
<b>TEP</b>	34.90	41.78	37.77	24.27	29.47
<b>TNPP</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>TIBP</b>	80.55	88.04	78.14	72.45	82.31
<b>TNBP</b>	75.90	87.82	77.59	78.07	81.59
<b>TCEP</b>	70.14	82.71	77.25	57.56	75.85
<b>TCPP</b>	80.54	92.74	84.21	76.08	86.07
<b>TDCPP</b>	82.82	92.97	84.51	79.41	86.67
<b>TBOEP</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>TPhP</b>	>99	>99	67.30	60.89	59.82
<b>EHDP</b>	n.e.	79.00	62.11	61.26	63.74
<b>TEHP</b>	>99	48.08	58.50	64.12	59.82
<b>TMPP</b>	94.42	85.98	27.19	95.44	n.e.
<b>TIPPP</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>T35DMPP</b>	n.e.	n.e.	n.e.	n.e.	n.e.
n.e.: Not Evaluable					

## 5.2.2. Pesticides and Chlorinated Flame Retardants

Dicofol has been analysed but not reported in the recovery table and in the results because of compound degradation, see paragraph 4.1.2.2.

*Table 32: Filters and cells analytical recovery and LOD/LOQ of Pesticides and Chlorinated Flame Retardants*

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
					<b><i>Chlorinated Pesticides</i></b>	<b>pg/L</b>	<b>pg/L</b>
<b>C13 HCBD</b>	32.82	49.99	43.24	16.52	<b>PeCBz</b>	0.34	0.63
<b>D6 Dichlorvos</b>	66.58	25.69	95.49	13.37	<b>HCB</b>	0.53	0.99
<b>C13_PeCBz</b>	52.04	21.40	66.57	10.40			
<b>13C HCB</b>	38.85	44.14	38.12	25.29	<b>a-HCH</b>	0.38	1.04
<b>13C a-HCH</b>	71.60	15.58	83.85	9.11	<b>b-HCH</b>	11	31
<b>13C_g-HCH</b>	75.43	13.94	85.08	8.07	<b>g-HCH</b>	0.39	0.86
<b>13C_Heptachlor</b>	108.00	17.26	127.54	11.43	<b>d-HCH</b>	0.15	0.37
<b>D10 Chlorpyriphos</b>	124.17	20.71	146.29	12.91	<b>e-HCH</b>	0.24	0.60
<b>C13_Aldrin</b>	74.09	14.78	74.12	11.37	<b>Sum-HCHs</b>		
<b>D10 Chlорfenvinphos</b>	226.32	22.70	298.67	15.45			
<b>C13_Isodrin</b>	68.85	16.67	70.32	8.88	<b>Heptachlor</b>	0.10	0.24
<b>C13_Oxychlordane</b>	44.26	33.15	44.55	16.14			
<b>C13_Endosulfane-alpha</b>	107.22	10.92	127.96	8.77	<b>Heptachlor-exo-epoxide</b>	0.29	0.74
<b>C13_Heptachlor-exo-epoxide</b>	55.87	20.77	58.22	11.36	<b>Heptachlor-endo-epoxide</b>	1.65	4.49
<b>C13_trans-chlordane</b>	40.05	44.66	40.52	15.66	<b>Sum-Hetachlorepoxydes</b>		
<b>C13_trans-nonachlor</b>	36.33	40.58	35.89	16.29			
<b>C13_op-DDE</b>	73.75	27.90	76.49	18.34	<b>Aldrin</b>	0.39	0.99
<b>C13_pp-DDE</b>	77.55	18.97	77.21	17.08	<b>Dieldrin</b>	0.27	0.63
<b>C13_op-DDD</b>	72.50	17.24	80.79	15.37	<b>Endrin</b>	0.32	0.65
<b>C13_op-DDT</b>	90.03	13.96	99.50	6.76	<b>Isodrin</b>	1.98	4.88
<b>C13_pp-DDT</b>	94.80	19.73	107.06	7.00	<b>Sum-Drins</b>		
<b>C13_Dieldrin</b>	62.20	20.49	64.61	7.97			
<b>C13_Endrin</b>	94.39	9.70	128.03	11.81	<b>trans-chlordane</b>	0.06	0.13
<b>C13_Endosulfane-beta</b>	72.47	17.29	67.13	32.33	<b>cis-chlordane</b>	0.06	0.14
<b>C13_cis-nonachlor</b>	56.77	18.77	57.52	9.05	<b>Sum-Chlordan</b>		
<b>C13_Endosulfane-sulphate</b>	55.27	29.10	50.24	16.57			

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
					<b><i>Chlorinated Pesticides</i></b>		<b>pg/L</b>
<b>C13_Methoxychlor</b>	107.33	17.07	147.53	18.03	<b>Oxychlordane</b>		0.21
<b>C13_Mirex</b>	45.16	19.21	46.77	14.63			
<b>D6 Cypermethrin</b>	141.83	19.26	176.01	15.49	<b>trans-nonachlor</b>		0.08
					<b>cis-nonachlor</b>		0.09
<b>C13_Atrazine</b>	75.88	18.61	74.42	18.26	<b>Sum-nonachlor</b>		
					<b>Endosulfane-alpha</b>		1.25
					<b>Endosulfane-beta</b>		0.19
					<b>Sum-Endosulfanes</b>		
					<b>Endosulfane-sulphate</b>		0.10
							0.23
					<b>op-DDE</b>		0.24
					<b>pp-DDE</b>		0.34
					<b>op-DDD</b>		0.42
					<b>pp-DDD</b>		0.11
					<b>op-DDT</b>		0.33
					<b>pp-DDT</b>		0.35
					<b>Sum-DDTtotal</b>		
					<b>Methoxychlor</b>		1.78
					<b>Mirex</b>		0.04
							0.08
					<b><i>Others Pesticides</i></b>		<b>pg/L</b>
					<b>HCBD</b>		12
					<b>Dichlorvos</b>		4.54
					<b>Trifluralin</b>		0.09
					<b>Triallate</b>		0.22
					<b>Chlorpyriphos</b>		0.84
					<b>Chlorfenvinphos</b>		2.08
					<b>Dicofol</b>		8.60
					<b>Cypermethrins</b>		---
							---
					<b><i>Chlorinated Flame Retardants</i></b>		<b>pg/L</b>

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
					<b><i>Chlorinated Pesticides</i></b>	<b>pg/L</b>	<b>pg/L</b>
					<b>Chloran 542</b>	187	486
					<b>syn-Decchlorane Plus</b>	0.20	0.49
					<b>anti-Decchlorane Plus</b>	0.13	0.31
					<b><i>Triazine</i></b>	<b>ng/L</b>	<b>ng/L</b>
					<b>Simazine</b>	0.010	0.018
					<b>Atrazine</b>	0.004	0.006
					<b>Terbutylazine</b>	0.001	0.002

Table 33: Sampling efficiency in 5 transect samples of Pesticides and Chlorinated Flame Retardants

	Transects				
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5
	Sampling Efficiency (%)				
<b>PeCBz</b>	>99	>99	>99	90.02	76.62
<b>HCB</b>	>99	>99	>99	84.03	91.04
<b>a-HCH</b>	89.60	96.72	92.52	87.35	93.98
<b>b-HCH</b>	89.41	96.37	91.97	83.60	93.03
<b>g-HCH</b>	89.54	95.19	91.42	86.54	93.28
<b>d-HCH</b>	91.83	>99	>99	85.89	92.76
<b>e-HCH</b>	>99	n.e.	>99	84.42	92.72
<b>Sum-HCHs</b>					
<b>Heptachlor</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Heptachlor-exo-epoxide</b>	85.70	>99	89.28	90.56	93.15
<b>Heptachlor-endo-epoxide</b>	n.e.	n.e.	n.e.	72.38	n.e.
<b>Sum-Hetachlorepoxydes</b>					

Transects					
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5
	Sampling Efficiency (%)				
<b>Aldrin</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Dieldrin</b>	86.91	93.28	87.77	90.36	92.45
<b>Endrin</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Isodrin</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Sum-Drins</b>					
<b>trans-chlordane</b>	n.e.	n.e.	n.e.	>99	>99
<b>cis-chlordane</b>	n.e.	n.e.	n.e.	>99	>99
<b>Sum-Chlordanes</b>					
<b>Oxychlordanes</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>trans-nonachlor</b>	>99	n.e.	n.e.	>99	>99
<b>cis-nonachlor</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Sum-nonachlor</b>					
<b>Endosulfane-alpha</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Endosulfane-beta</b>	85.55	>99	>99	76.80	>99
<b>Sum-Endosulfanes</b>					
<b>Endosulfane-sulphate</b>	84.03	>99	>99	84.72	92.21
<b>op-DDE</b>	n.e.	n.e.	n.e.	n.e.	>99
<b>pp-DDE</b>	>99	>99	>99	>99	96.30
<b>op-DDD</b>	>99	>99	>99	>99	98.42
<b>pp-DDD</b>	98.15	98.19	>99	>99	98.89
<b>op-DDT</b>	>99	>99	>99	>99	>99
<b>pp-DDT</b>	>99	>99	>99	>99	>99
<b>Sum-DDTtotal</b>					
<b>Methoxychlor</b>	n.e.	n.e.	74.53	n.e.	n.e.
<b>Mirex</b>	n.e.	n.e.	51.35	n.e.	n.e.

Transects					
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5
	Sampling Efficiency (%)				
<b>Others;</b>					
<b>HCBD</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Dichlorvos</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Trifluralin</b>	>99	n.e.	n.e.	n.e.	>99
<b>Triallate</b>	>99	n.e.	>99	>99	93.07
<b>Chlorpyriphos</b>	95.90	>99	96.91	98.89	98.69
<b>Chlorfenvinphos</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Dicofol</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Cypermethrins</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>Simazine</b>	66.28	73.35	69.20	46.24	71.26
<b>Atrazine</b>	71.05	82.40	74.40	54.34	77.20
<b>Terbutylazine</b>	80.69	90.57	81.55	68.77	85.78
<b>Chloran 542</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>syn-Dechlorane Plus</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>anti-Dechlorane Plus</b>	n.e.	n.e.	n.e.	n.e.	n.e.
<b>n.e.: Not Evaluable</b>					

### 5.2.3. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

Table 34: Filter and extraction cells analytical recovery and LOD/LOQ of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
						pg/L	pg/L
<b>Phenanthrene-d10</b>	58.56	25.34	55.20	71.50	<b>Phenanthrene</b>	40.6	86.4
<b>Anthracene-d10</b>	54.91	24.06	49.35	52.62	<b>Anthracene</b>	11.1	23.7

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
<b>Fluoranthene-d10</b>	68.52	21.20	54.60	31.41	<b>Fluoranthene</b>	7.94	16.0
<b>Pyrene-d10</b>	67.17	19.79	54.03	31.53	<b>Pyrene</b>	24.7	61.0
<b>Chrysene-d12</b>	98.21	38.10	90.72	8.89	<b>Benz(a)anthracene</b>	2.22	6.23
<b>Benzo(b)fluoranthene-d12</b>	89.74	23.02	91.91	11.96	<b>Chrysene</b>	4.92	12.9
<b>Perylene-d12</b>	81.46	11.09	89.34	14.78	<b>Sum Benzo(b,j,k)fluoranthene</b>	1.84	3.74
<b>Benzo(a)pyrene-d12</b>	82.98	11.36	89.33	9.57	<b>Benzo(e)pyrene</b>	0.39	0.94
<b>Benzo(e)pyrene-d12</b>	85.73	12.30	92.44	6.76	<b>Benzo(a)pyrene</b>	0.31	0.75
<b>Indeno(123-cd)pyrene-d12</b>	85.79	11.83	102.28	31.36	<b>Perylene</b>	0.42	0.88
<b>Benzo(ghi)perylene-d12</b>	87.31	9.60	110.24	30.24	<b>Indeno(123-cd)pyrene</b>	0.38	0.97
<b>Dibenz(ah)anthracene-d12</b>	84.89	11.90	103.04	27.64	<b>Benzo(ghi)perylene</b>	0.60	1.35
<b>Coronene-12</b>	90.21	11.27	126.95	60.60	<b>Dibenz(ah)anthracene</b>	0.51	1.29
<b>BHT</b>	27.51	17.44	78.33	58.82	<b>Coronene</b>	1.34	3.48
					<b>BHT</b>	390	1016
					<b>EHMC</b>	71	113

Table 35: Sampling efficiency in 5 transect samples of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

	Transects				
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5
	Sampling Efficiency (%)				
<b>Phenanthrene</b>	n.e.	>99	>99	>99	>99
<b>Anthracene</b>	n.e.	>99	>99	>99	>99
<b>Fluoranthene</b>	n.e.	>99	50.3	>99	>99
<b>Pyrene</b>	n.e.	>99	>99	>99	>99
<b>Benz(a)anthracene</b>	n.e.	>99	n.e.	n.e.	>99
<b>Chrysene</b>	n.e.	>99	>99	>99	>99

		Transects				
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5	
	Sampling Efficiency (%)					
<b>Sum Benzo(b,j,k)fluoranthene</b>	n.e.	>99	87.0	>99	>99	
<b>Benzo(e)pyrene</b>	n.e.	>99	79.1	>99	>99	
<b>Benzo(a)pyrene</b>	n.e.	>99	54.9	n.e.	>99	
<b>Perylene</b>	n.e.	>99	>99	>99		93.3
<b>Indeno(123-cd)pyrene</b>	n.e.	85.6	68.4	>99		
<b>Benzo(ghi)perylene</b>	n.e.	>99	25.0	59.7		77.4
<b>Dibenz(ah)anthracene</b>	n.e.	52.7	>99	>99		
<b>Coronene</b>	n.e.	n.e.	n.e.	n.e.		
<b>BHT</b>	n.e.	n.e.	n.e.	n.e.		
<b>EHMC</b>	n.e.	>99	58.6	>99		51.4
<b>n.e.: Not Evaluable</b>						

#### 5.2.4. Polychlorinated biphenyls (PCBs)

Table 36: Filter and extraction cells analytical recovery and LOD/LOQ of Polychlorinated biphenyls (PCBs)

	LV-TS Filter		LV-TS Cells			LOD	LOQ
	Average Recovery (%)	CV %	Average Recovery (%)	CV %		Blank +3sd	Blank +10sd
						pg/L	pg/L
<b>13C12 PCB 28</b>	78.06	10.68	89.57	7.45	PCB 28	0.16	0.24
<b>13C12 PCB 52</b>	79.00	13.48	118.94	16.38	PCB 52	0.23	0.42
<b>13C12 PCB 101</b>	81.80	10.55	86.45	7.05	PCB 101	0.21	0.28
<b>13C12 PCB 118</b>	85.74	11.56	90.79	8.03	PCB 118	0.33	0.72
<b>13C12 PCB 138</b>	77.16	9.04	92.42	11.99	PCB 138	0.58	1.04
<b>13C12 PCB 153</b>	77.47	8.77	95.19	17.89	PCB 153	0.63	0.98
<b>13C12 PCB 180</b>	87.54	14.64	89.18	8.75	PCB 180	0.32	0.49

Table 37: Sampling efficiency in 5 transect samples of Polychlorinated biphenyls (PCBs)

	Transects				
	JOSS 1	JOSS 2	JOSS 3	JOSS 4	JOSS 5
	Sampling Efficiency (%)				
<b>EC-6</b>					
<b>PCB 28</b>	>99	>99	88.01	84.13	97.71
<b>PCB 52</b>	>99	>99	>99	80.38	>99
<b>PCB 101</b>	>99	>99	>99	>99	95.05
<b>PCB 118</b>	>99	>99	n.e.	>99	>99
<b>PCB 138</b>	>99	n.e.	n.e.	>99	>99
<b>PCB 153</b>	>99	n.e.	n.e.	>99	80.77
<b>PCB 180</b>	>99	n.e.	n.e.	>99	>99
<b>n.e.: Not Evaluable</b>					

## 6. Analytical results

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

### 6.1. Mariani Box 20L spot samples

#### 6.1.1. Polar compounds

In Table 38 to 44 the results of Polar Compounds obtained with 20L spot samples are reported.

*Table 38: Polar Compounds concentrations in Blank samples*

	FBLK-FILTER	17-047	17-048
Analyte ID/ Sample ID	Field Blank Filter	FB-1	FB-2
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	0.025	6.93	5.93
Benzotriazole	7.20	< LOD	< LOD
2,4-D	< LOD	< LOD	< LOD
Carbamazepine	< LOD	< LOD	< LOD
Bezafibrate	< LOD	< LOD	< LOD
Atrazine	< LOD	< LOD	< LOD
Diclofenac	< LOD	< LOD	< LOD
Gemfibrozil	< LOD	< LOD	< LOD
Ibuprofen	< LOD	< LOD	< LOD
Irgarol	< LOD	< LOD	< LOD
MCPA	< LOD	< LOD	< LOD
Naproxen	< LOD	< LOD	< LOD
PFBS	< LOD	< LOD	< LOD
PFHpA	0.11	< LOD	< LOD
PFHxA	< LOD	< LOD	< LOD
PFHxS	0.031	< LOD	< LOD
PFNA	0.29	< LOD	< LOD
PFOA	0.27	< LOD	< LOD
PFOS	< LOD	< LOD	0.097
Sulfamethazine	< LOD	< LOD	< LOD
Sulfamethoxazole	< LOD	< LOD	0.008

	FBLK-FILTER	17-047	17-048
Analyte ID/ Sample ID	Field Blank Filter	FB-1	FB-2
Concentration:	ng/L	ng/L	ng/L
Terbutryny	<LOD	<LOD	<LOD
Terbutylazine	<LOD	<LOD	<LOD
Acetamiprid	<LOD	<LOD	<LOD
Aclonifen	<LOD	<LOD	<LOD
Simazine	<LOD	<LOD	<LOD

Table 39: Polar Compounds concentrations in samples from inside (1A and 1A') and 1 outside (1C) Danube delta

	17-020	17-021	17-046
Analyte ID/ Sample ID	1A	1A'	1-C
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	11.43	19.08	5.06
Benzotriazole	151	216	<LOD
2,4-D	<LOD	<LOD	0.997
Carbamazepine	<LOD	12.51	2.80
Bezafibrate	<LOD	<LOD	<LOD
Atrazine	14.08	14.52	18.42
Diclofenac	3.84	2.55	<LOD
Gemfibrozil	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	0.37
Naproxen	1.63	1.67	0.72
PFBS	0.006	0.020	0.007
PFHpA	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD

	17-020	17-021	17-046
Analyte ID/ Sample ID	1A	1A'	1-C
Concentration:	ng/L	ng/L	ng/L
PFOA	<LOD	<LOD	<LOD
PFOS	0.079	0.097	0.044
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	6.33	8.14	5.22
Terbutryn	0.33	0.25	0.11
Terbutylazine	1.66	1.56	0.93
Acetamiprid	0.13	0.12	0.11
Aclonifen	<LOD	<LOD	<LOD
Simazine	16.71	12.36	n.a.

Table 40: Polar Compounds concentrations in samples from Georgia Coast

	17-035	17-037	17-038
Analyte ID/ Sample ID	1-GE	2-GE	3-GE
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	3.03	2.44	2.14
Benzotriazole	15.51	29.02	16.80
2,4-D	1.29	0.21	0.25
Carbamazepine	<LOD	1.70	0.76
Bezafibrate	<LOD	0.013	0.039
Atrazine	<LOD	18.98	19.83
Diclofenac	<LOD	2.82	1.86
Gemfibrozil	<LOD	0.021	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD

	17-035	17-037	17-038
Analyte ID/ Sample ID	1-GE	2-GE	3-GE
Concentration:	ng/L	ng/L	ng/L
PFHxA	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD
PFOS	0.022	0.024	0.020
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	<LOD	1.49	<LOD
Terbutryn	<LOD	<LOD	<LOD
Terbutylazine	<LOD	<LOD	0.36
Acetamiprid	0.063	<LOD	<LOD
Aclonifen	<LOD	<LOD	<LOD
Simazine	17.67	20.78	23.06

Table 41: Polar Compounds concentrations in samples from Ukraine Coast

	17-039	17-040	17-041
Analyte ID/ Sample ID	CW-5	CW-7	CW-8
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	6.88	4.56	4.20
Benzotriazole	39.77	17.93	21.74
2,4-D	<LOD	<LOD	<LOD
Carbamazepine	5.51	<LOD	<LOD
Bezafibrate	0.072	0.021	0.090
Atrazine	17.63	18.27	18.62
Diclofenac	<LOD	1.41	2.63
Gemfibrozil	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	<LOD
Naproxen	<LOD	1.66	1.13

	17-039	17-040	17-041
Analyte ID/ Sample ID	CW-5	CW-7	CW-8
Concentration:	ng/L	ng/L	ng/L
PFBS	<LOD	<LOD	0.005
PFHpA	0.021	0.040	0.061
PFHxA	0.091	0.075	0.045
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	0.040	<LOD
PFOA	<LOD	<LOD	<LOD
PFOS	0.023	0.054	0.025
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	2.14	1.75	1.61
Terbutryn	0.24	<LOD	<LOD
Terbutylazine	1.74	1.20	0.56
Acetamiprid	<LOD	<LOD	0.071
Aclonifen	<LOD	<LOD	<LOD
Simazine	7.11	27.49	17.23

Table 42: Polar Compounds concentrations in open sea samples

	17-022	17-033	17-023
Analyte ID/ Sample ID	1-JOSS	2-JOSS	3-JOSS
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	3.217	3.563	6.199
Benzotriazole	<LOD	<LOD	<LOD
2,4-D	0.545	<LOD	<LOD
Carbamazepine	<LOD	<LOD	<LOD
Bezafibrate	0.09	0.11	0.22
Atrazine	16.97	16.67	15.71
Diclofenac	<LOD	<LOD	<LOD
Gemfibrozil	<LOD	0.015	<LOD
Ibuprofen	<LOD	<LOD	<LOD

	17-022	17-033	17-023
Analyte ID/ Sample ID	1-JOSS	2-JOSS	3-JOSS
Concentration:	ng/L	ng/L	ng/L
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD
PFOS	0.22	<LOD	0.25
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	<LOD	1.99	2.31
Terbutryn	<LOD	<LOD	<LOD
Terbutylazine	0.51	0.65	0.88
Acetamiprid	0.096	0.13	0.094
Aclonifen	<LOD	<LOD	<LOD
Simazine	6.79	20.34	5.06

Table 43: Polar Compounds concentrations in open sea samples

	17-024	17-025	17-026
Analyte ID/ Sample ID	4-JOSS	5-JOSS	6-JOSS
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	3.392	4.339	2.828
Benzotriazole	<LOD	<LOD	<LOD
2,4-D	<LOD	<LOD	<LOD
Carbamazepine	<LOD	<LOD	<LOD
Bezafibrate	<LOD	<LOD	<LOD
Atrazine	16.97	20.96	18.77
Diclofenac	<LOD	<LOD	<LOD
Gemfibrozil	<LOD	<LOD	<LOD

	17-024	17-025	17-026
Analyte ID/ Sample ID	4-JOSS	5-JOSS	6-JOSS
Concentration:	ng/L	ng/L	ng/L
Ibuprofen	<LOD	<LOD	<LOD
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD
PFOS	0.23	0.22	0.23
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	0.81	0.81	0.50
Terbutryn	<LOD	<LOD	<LOD
Terbutylazine	<LOD	<LOD	<LOD
Acetamiprid	0.067	0.075	0.054
Aclonifen	<LOD	<LOD	<LOD
Simazine	7.97	9.17	6.33

Table 44: Polar Compounds concentrations in open sea samples

	17-027	17-028	17-29
Analyte ID/ Sample ID	7-JOSS	8-JOSS	9-JOSS
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	2.345	2.763	1.773
Benzotriazole	<LOD	<LOD	<LOD
2,4-D	<LOD	<LOD	<LOD
Carbamazepine	<LOD	<LOD	<LOD
Bezafibrate	<LOD	<LOD	<LOD
Atrazine	21.85	16.79	17.58
Diclofenac	<LOD	<LOD	<LOD

	17-027	17-028	17-29
Analyte ID/ Sample ID	7-JOSS	8-JOSS	9-JOSS
Concentration:	ng/L	ng/L	ng/L
Gemfibrozil	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD
PFOS	0.18	0.25	0.019
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	<LOD	0.95	0.40
Terbutryny	<LOD	<LOD	<LOD
Terbutylazine	<LOD	0.30	0.30
Acetamiprid	0.057	0.074	0.081
Aclonifen	<LOD	<LOD	<LOD
Simazine	6.06	7.31	17.20

Table 44: Polar Compounds concentrations in open sea samples

	17-030	17-031	17-032
Analyte ID/ Sample ID	10-JOSS	11-JOSS	12-JOSS
Concentration:	ng/L	ng/L	ng/L
10,11-dihydro-10,11-dihydroxy-carbamazepine	2.158	2.165	1.962
Benzotriazole	<LOD	<LOD	<LOD
2,4-D	<LOD	<LOD	<LOD
Carbamazepine	<LOD	<LOD	<LOD
Bezafibrate	<LOD	<LOD	<LOD

	17-030	17-031	17-032
Analyte ID/ Sample ID	10-JOSS	11-JOSS	12-JOSS
Concentration:	ng/L	ng/L	ng/L
Atrazine	19.10	17.61	16.94
Diclofenac	<LOD	<LOD	<LOD
Gemfibrozil	<LOD	<LOD	<LOD
Ibuprofen	<LOD	<LOD	<LOD
Irgarol	<LOD	<LOD	<LOD
MCPA	<LOD	<LOD	<LOD
Naproxen	<LOD	<LOD	<LOD
PFBS	<LOD	<LOD	<LOD
PFHpA	<LOD	<LOD	<LOD
PFHxA	<LOD	<LOD	<LOD
PFHxS	<LOD	<LOD	<LOD
PFNA	<LOD	<LOD	<LOD
PFOA	<LOD	<LOD	<LOD
PFOS	0.022	0.025	0.025
Sulfamethazine	<LOD	<LOD	<LOD
Sulfamethoxazole	<LOD	<LOD	1.46
Terbutryny	<LOD	<LOD	<LOD
Terbutylazine	0.36	0.34	0.38
Acetamiprid	0.13	0.11	0.12
Aclonifen	<LOD	0.54	<LOD
Simazine	20.48	25.64	15.14

## 6.1.2. Semi-polar and Apolar Compounds

### 6.1.2.1. Organophosphate Compounds (OPCs)

In Table 45 to 52 are reported the results of Organophosphate Compounds obtained with 20L spot samples are reported.

Table 45: Organophosphate Compounds (OPCs) concentrations in blank samples

Lab. Code:	OPC-FBLK-FILTER	OPC-17-047	OPC-17-048
Sample name:	Field Blank Filter	FB-1	FB-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	21.29	21.34	21.29
Sampling period:	2017	2017	2017
Analysis date:	11/29/2017	11/29/2017	11/29/2017
Concentration	ng/L	ng/L	ng/L
TEP	0.30	12.45	14.90
TNPP	<LOD	<LOD	<LOD
TIBP	0.04	<LOD	0.10
TNBP	0.05	1.05	1.41
TCEP	0.10	0.94	1.66
TCPP	0.17	23.38	25.33
TDCPP	0.02	0.11	0.15
TBOEP	<LOD	2.10	2.64
TPhP	<LOD	0.33	0.35
EHDP	0.04	0.16	0.25
TEHP	0.004	0.04	0.05
TMPP	<LOD	0.16	0.10
TIPPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

*Table 46: Organophosphate Compounds (OPCs) Compounds concentrations in samples from inside (1A and 1A') and 1 outside (1C) Danube delta*

Lab. Code:	OPC-17-020	OPC-17-021	OPC-17-046
Sample name:	1A	1A - Rep	1-C
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.05	20.00	20.80
Sampling period:	2017	2017	2017
Analysis date:	11/30/2017	11/30/2017	11/30/2017
Concentration	ng/L	ng/L	ng/L
TEP	5.58	n.d.	29.36
TNPP	< LOD	< LOD	< LOD
TIBP	7.95	7.99	4.52
TNBP	1.66	1.48	0.80
TCEP	3.89	3.84	3.80
TCPP	41.54	51.05	18.82
TDCPP	3.69	3.44	1.38
TBOEP	< LOD	< LOD	0.99
TPhP	0.28	0.27	0.11
EHDP	0.70	< LOD	0.20
TEHP	0.59	0.62	0.18
TMPP	0.15	< LOD	0.08
TIPPP	< LOD	< LOD	< LOD
T35DMPP	< LOD	< LOD	< LOD
n.d.: not detected because of interference			

Table 47: Organophosphate Compounds (OPCs) concentrations in samples from Georgia Coast

Lab. Code:	OPC-17-035	OPC-17-037	OPC-17-038
Sample name:	1-GE	2-GE	3-GE
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.65	20.60	20.65
Sampling period:	2017	2017	2017
Analysis date:	11/30/2017	11/30/2017	11/30/2017
Concentration	ng/L	ng/L	ng/L
TEP	2.88	1.35	0.96
TNPP	<LOD	<LOD	<LOD
TIBP	9.23	5.33	6.31
TNBP	1.95	1.47	0.87
TCEP	15.91	4.60	6.29
TCPP	21.58	7.36	9.07
TDCPP	1.43	0.88	0.78
TBOEP	1.76	<LOD	<LOD
TPhP	8.24	3.24	3.53
EHDP	1.63	0.99	0.52
TEHP	0.42	0.84	0.16
TMPP	4.00	0.25	1.43
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

Table 48: Organophosphate Compounds (OPCs) concentrations in samples from Ukraine Coast

Lab. Code:	OPC-17-039	OPC-17-041	OPC-17-040
Sample name:	CW-5	CW-8	CW-7
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	23.40	22.85	22.25
Sampling period:	2017	2017	2017
Analysis date:	11/30/2017	11/30/2017	11/30/2017
Concentration	ng/L	ng/L	ng/L
TEP	1.28	1.62	1.38
TNPP	<LOD	<LOD	<LOD
TIBP	6.44	4.12	3.62
TNBP	2.96	1.01	1.25
TCEP	6.07	4.73	5.23
TCPP	22.77	10.31	12.73
TDCPP	2.57	0.69	0.81
TBOEP	<LOD	<LOD	<LOD
TPhP	1.16	0.16	0.52
EHDP	1.78	0.22	0.41
TEHP	0.61	0.06	0.24
TMPP	0.88	0.07	0.74
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

*Table 49: Organophosphate Compounds (OPCs) concentrations in open sea samples*

Lab. Code:	OPC-17-22	OPC-17-33	OPC-17-23
Sample name:	1-JOSS	2-JOSS	3-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.80	20.30	20.00
Sampling period:	2017	2017	2017
Analysis date:	11/29/2017	11/30/2017	11/29/2017
Concentration	ng/L	ng/L	ng/L
TEP	1.69	6.38	5.05
TNPP	<LOD	<LOD	<LOD
TIBP	2.64	3.78	4.05
TNBP	0.56	0.94	0.79
TCEP	4.86	4.16	5.75
TCPP	10.43	15.55	16.49
TDCPP	0.45	0.70	0.84
TBOEP	<LOD	<LOD	<LOD
TPhP	0.07	0.10	0.12
EHDP	0.18	0.33	0.33
TEHP	0.04	0.06	0.05
TMPP	0.05	0.06	0.07
TI PPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

Table 50: Organophosphate Compounds (OPCs) concentrations in open sea samples

Lab. Code:	OPC-17-24	OPC-17-25	OPC-17-26
Sample name:	4-JOSS	5-JOSS	6-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	21.45	20.55	19.85
Sampling period:	2017	2017	2017
Analysis date:	11/29/2017	11/29/2017	11/30/2017
Concentration	ng/L	ng/L	ng/L
TEP	1.22	2.07	2.10
TNPP	<LOD	<LOD	<LOD
TIBP	0.71	2.05	2.22
TNBP	0.51	0.54	0.56
TCEP	4.03	4.05	3.83
TCPP	6.55	7.24	7.44
TDCPP	0.32	0.44	0.53
TBOEP	<LOD	<LOD	<LOD
TPhP	0.12	0.16	0.13
EHDP	0.22	0.48	0.16
TEHP	0.06	0.07	0.05
TMPP	0.18	0.06	0.07
TI PPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

Table 51: Organophosphate Compounds (OPCs) concentrations in open sea samples

Lab. Code:	OPC-17-27	OPC-17-28	OPC-17-29
Sample name:	7-JOSS	8-JOSS	9-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	19.85	20.15
Sampling period:	2017	2017	2017
Analysis date:	11/30/2017	11/30/2017	11/30/2017
Concentration	ng/L	ng/L	ng/L
TEP	12.31	7.65	7.03
TNPP	<LOD	<LOD	<LOD
TIBP	2.02	2.23	1.88
TNBP	0.52	0.70	0.46
TCEP	4.12	3.86	3.73
TCPP	7.79	10.37	9.93
TDCPP	0.33	0.44	0.40
TBOEP	<LOD	0.53	<LOD
TPhP	0.09	0.14	0.14
EHDP	0.37	0.25	0.82
TEHP	0.03	0.05	0.04
TMPP	0.06	0.08	0.07
TI PPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

Table 52: Organophosphate Compounds (OPCs) concentrations in open sea samples

Lab. Code:	OPC-17-30	OPC-17-31	OPC-17-32
Sample name:	10-JOSS	11-JOSS	12-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.00	19.80	19.60
Sampling period:	2017	2017	2017
Analysis date:	11/30/2017	11/30/2017	11/30/2017
Concentration	ng/L	ng/L	ng/L
TEP	6.26	7.59	7.33
TNPP	<LOD	<LOD	<LOD
TIBP	2.60	2.43	2.26
TNBP	0.59	0.73	0.73
TCEP	4.70	4.14	4.53
TCPP	9.93	10.20	11.45
TDCPP	0.40	0.41	0.46
TBOEP	<LOD	<LOD	<LOD
TPhP	0.15	0.10	0.25
EHDP	0.16	0.27	0.30
TEHP	0.04	0.03	0.05
TMPP	0.06	0.10	0.07
TI PPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD
n.d.: not detected because of interference			

### 6.1.2.2. Pesticides and Chlorinated Flame Retardants

In the Table 53 to 60 the results of Pesticides and Chlorinated Flame Retardants obtained with 20L spot samples are reported.

*Table 53: Pesticides and Chlorinated Flame Retardants concentrations in blank samples*

Lab. Code:	OCP-FBLK-FILTER	OCP-17-047	OCP-17-048
Sample name:	Field Blank Filter	FB-1	FB-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	21.34	21.29
Sampling period:	2017	2017	2017
Analysis date:	11/20/2017	11/20/2017	11/20/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	2.06	49.58	72.46
HCB	2.48	25.80	32.06
a-HCH	<LOD	3.20	<LOD
b-HCH	2.01	3.88	3.74
g-HCH	<LOD	8.70	9.59
d-HCH	<LOD	<LOD	<LOD
e-HCH	<LOD	<LOD	<LOD
Sum-HCHs	2.01	15.77	15.75
Heptachlor		<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	<LOD	<LOD
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	---	---
Aldrin	<LOD	<LOD	<LOD
Dieldrin	<LOD	<LOD	<LOD
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	---	---
trans-chlordane	<LOD	<LOD	<LOD
cis-chlordane	<LOD	<LOD	<LOD

Lab. Code:	OCP-FBLK-FILTER	OCP-17-047	OCP-17-048
Sample name:	Field Blank Filter	FB-1	FB-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	21.34	21.29
Sampling period:	2017	2017	2017
Analysis date:	11/20/2017	11/20/2017	11/20/2017
Concentration	pg/L	pg/L	pg/L
Sum-Chlordane	---	---	---
Oxychlordane	< LOD	< LOD	< LOD
trans-nonachlor	< LOD	0.61	< LOD
cis-nonachlor	< LOD	< LOD	< LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	< LOD	< LOD	< LOD
Endosulfane-beta	< LOD	2.58	< LOD
Sum-Endosulfanes	---	2.58	---
Endosulfane-sulphate	< LOD	< LOD	< LOD
op-DDE	< LOD	< LOD	< LOD
pp-DDE	< LOD	< LOD	< LOD
op-DDD	< LOD	< LOD	< LOD
pp-DDD	< LOD	< LOD	< LOD
op-DDT	< LOD	< LOD	< LOD
pp-DDT	< LOD	< LOD	< LOD
Sum-DDTtotal	---	---	---
Methoxychlor	< LOD	< LOD	< LOD
Mirex	< LOD	< LOD	< LOD
<i>Other pesticides:</i>			
HCBD	7.01	331.47	358.06
Dichlorvos	< LOD	< LOD	< LOD
Trifluralin	< LOD	< LOD	< LOD
Triallate	< LOD	< LOD	< LOD

Lab. Code:	OCP-FBLK-FILTER	OCP-17-047	OCP-17-048
Sample name:	Field Blank Filter	FB-1	FB-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	21.34	21.29
Sampling period:	2017	2017	2017
Analysis date:	11/20/2017	11/20/2017	11/20/2017
Concentration	pg/L	pg/L	pg/L
Chlorpyriphos	< LOD	< LOD	< LOD
Chlorfenvinphos	< LOD	< LOD	< LOD
Dicofol	No stable	No stable	No stable
Cypermethrins	< LOD	< LOD	< LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	< LOD	< LOD	< LOD
syn-Dechlorane Plus	< LOD	< LOD	< LOD
anti-Dechlorane Plus	< LOD	< LOD	< LOD

Table 54: Pesticides and Chlorinated Flame Retardants concentrations in samples from inside (1A and 1A') and 1 outside (1C) Danube delta

Lab. Code:	OCP-17-020	OPC-17-021	ATR-DDT-17-046
Sample name:	1A	1A - Rep	1-C
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.05	20.00	20.80
Sampling period:	2017	2017	2017
Analysis date:	11/22/2017	11/30/2017	11/29/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	29.90	---	---
HCB	34.86	---	---
a-HCH	156.81	<i>Sample not analysed due to instrument signal suppression</i>	<i>Sample not analysed due to instrument signal suppression. Results only for DDTs</i>
b-HCH	1682.45		
g-HCH	97.17		
d-HCH	38.54		
e-HCH	20.99		
Sum-HCHs	1995.96		
Heptachlor	< LOD	---	---
Heptachlor-exo-epoxide	< LOD	---	---
Heptachlor-endo-epoxide	< LOD	---	---
Sum-Hetachlorepoxydes	---	---	---
Aldrin	< LOD	---	---
Dieldrin	19.32	---	---
Endrin	< LOD	---	---
Isodrin	< LOD	---	---
Sum-Drins	19.32	---	---
trans-chlordane	< LOD	---	---
cis-chlordane	< LOD	---	---
Sum-Chlordanes	---	---	---
Oxychlordane	< LOD	---	---
trans-nonachlor	< LOD	---	---
cis-nonachlor	< LOD	---	---
Sum-nonachlor	---	---	---
Endosulfane-alpha	< LOD	---	---
Endosulfane-beta	< LOD	---	---
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	7.77	---	---

Lab. Code:	OCP-17-020	OPC-17-021	ATR-DDT-17-046
Sample name:	1A	1A - Rep	1-C
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.05	20.00	20.80
Sampling period:	2017	2017	2017
Analysis date:	11/22/2017	11/30/2017	11/29/2017
Concentration	pg/L	pg/L	pg/L
op-DDE	10.15	---	<LOD
pp-DDE	77.18	---	57.97
op-DDD	118.90	---	54.29
pp-DDD	383.81	---	177.33
op-DDT	<LOD	---	<LOD
pp-DDT	31.56	---	<LOD
Sum-DDTtotal	621.59	---	289.58
Methoxychlor	<LOD	---	---
Mirex	<LOD	---	---
<i>Other pesticides:</i>			
HCBD	79.37	---	---
Dichlorvos	<LOD	---	---
Trifluralin	<LOD	---	---
Triallate	33.26	---	---
Chlorpyriphos	50.97	---	---
Chlorfenvinphos	<LOD	---	---
Dicofol	No stable	---	---
Cypermethrins	<LOD	---	---
<i>Chlorinated Flame Retardants:</i>			
Chloran 542	<LOD	---	---
syn-Decchlorane Plus	<LOD	---	---
anti-Decchlorane Plus	<LOD	---	---

Table 55: Pesticides and Chlorinated Flame Retardants concentrations in samples from Georgia Coast

Lab. Code:	OCP-17-035	OCP-17-037	OCP-17-038
Sample name:	1-GE	2-GE	3-GE
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.65	20.60	20.65
Sampling period:	2017	2017	2017
Analysis date:	11/22/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	25.77	27.79	19.72
HCB	41.34	54.69	32.92
a-HCH	172.02	102.71	129.80
b-HCH	4955.16	3348.99	5019.29
g-HCH	88.30	44.70	55.30
d-HCH	6.69	<LOD	3.77
e-HCH	3.93	<LOD	<LOD
Sum-HCHs	5226.09	3496.39	5208.15
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	no Rec	no Rec	no Rec
Heptachlor-endo-epoxide	no Rec	no Rec	no Rec
Sum-Hetachlorepoxydes	---	---	---
Aldrin	<LOD	<LOD	<LOD
Dieldrin	45.74	<LOD	22.11
Endrin	<LOD	<LOD	<LOD
Isodrin	no Rec	no Rec	no Rec
Sum-Drins	45.74	---	22.11
trans-chlordane	no Rec	<LOD	<LOD
cis-chlordane	no Rec	<LOD	<LOD
Sum-Chlordane	---	---	---
Oxychlordane	no Rec	no Rec	no Rec
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	25.72	<LOD	<LOD
Sum-Endosulfanes	25.72	---	---
Endosulfane-sulphate	<LOD	<LOD	2.49

Lab. Code:	OCP-17-035	OCP-17-037	OCP-17-038
Sample name:	1-GE	2-GE	3-GE
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.65	20.60	20.65
Sampling period:	2017	2017	2017
Analysis date:	11/22/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
op-DDE	<LOD	<LOD	<LOD
pp-DDE	41.63	176.11	40.46
op-DDD	<LOD	104.81	23.63
pp-DDD	70.74	304.14	82.51
op-DDT	<LOD	<LOD	<LOD
pp-DDT	91.82	86.75	46.53
Sum-DDTtotal	204.19	671.80	193.13
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	54.07	96.58	46.32
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	172.18	168.74	97.56
Chlorfenvinphos	<LOD	no Rec	no Rec
Dicofol	No stable	No stable	No stable
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Chloran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	29.3
anti-Decchlorane Plus	<LOD	<LOD	28.8

*Table 56: Pesticides and Chlorinated Flame Retardants concentrations in samples from Ukraine Coast*

Lab. Code:	OCP-17-039	OCP-17-041	OCP-17-040
Sample name:	CW-5	CW-8	CW-7
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	23.40	22.85	22.25
Sampling period:	2017	2017	2017
Analysis date:	11/21/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	128.08	32.93	13.68
HCB	150.57	17.66	<LOD
a-HCH	118.89	115.85	71.27
b-HCH	1690.65	3092.27	2619.71
g-HCH	57.85	50.71	10.52
d-HCH	<LOD	<LOD	<LOD
e-HCH	<LOD	<LOD	<LOD
Sum-HCHs	1867.39	3258.82	2701.51
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	<LOD	<LOD
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	---	---
Aldrin	<LOD	<LOD	<LOD
Dieldrin	<LOD	<LOD	<LOD
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	---	---
trans-chlordane	<LOD	<LOD	<LOD
cis-chlordane	<LOD	<LOD	<LOD
Sum-Chlordanes	---	---	---
Oxychlordane	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	<LOD	<LOD
Sum-Endosulfanes	---	---	---
Endosulfane-sulphate	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-039	OCP-17-041	OCP-17-040
Sample name:	CW-5	CW-8	CW-7
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	23.40	22.85	22.25
Sampling period:	2017	2017	2017
Analysis date:	11/21/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
op-DDE	<LOD	<LOD	<LOD
pp-DDE	266.19	<LOD	<LOD
op-DDD	<LOD	<LOD	20.74
pp-DDD	244.45	<LOD	71.59
op-DDT	<LOD	<LOD	<LOD
pp-DDT	<LOD	<LOD	<LOD
Sum-DDTtotal	510.64	---	92.32
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	526.37	202.08	0.00
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	112.45	31.20	<LOD
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	No stable	No stable	No stable
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Chloran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 57: Pesticides and Chlorinated Flame Retardants concentrations in open sea samples

Lab. Code:	OCP-17-22	OCP-17-33	OCP-17-23
Sample name:	1-JOSS	2-JOSS	3-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.80	20.30	20.00
Sampling period:	2017	2017	2017
Analysis date:	11/22/2017	11/21/2017	11/20/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	6.76	11.05	8.22
HCB	8.11	9.36	11.01
a-HCH	496.50	115.97	125.44
b-HCH	3377.20	3337.56	3344.42
g-HCH	53.73	78.23	74.35
d-HCH	4.68	4.95	6.03
e-HCH	3.34	3.33	3.77
Sum-HCHs	3935.46	3540.05	3554.01
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	7.31	2.23	3.41
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	7.31	2.23	3.41
Aldrin	<LOD	<LOD	<LOD
Dieldrin	8.50	10.70	8.92
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	8.50	10.70	8.92
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	3.41	<LOD
Sum-Endosulfanes	---	3.41	---
Endosulfane-sulphate	1.79	1.41	1.13
op-DDE	2.28	<LOD	<LOD

Lab. Code:	OCP-17-22	OCP-17-33	OCP-17-23
Sample name:	1-JOSS	2-JOSS	3-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.80	20.30	20.00
Sampling period:	2017	2017	2017
Analysis date:	11/22/2017	11/21/2017	11/20/2017
Concentration	pg/L	pg/L	pg/L
pp-DDE	6.02	6.53	18.23
op-DDD	2.60	<LOD	16.47
pp-DDD	17.84	24.26	48.85
op-DDT	<LOD	<LOD	3.10
pp-DDT	7.69	7.08	11.43
Sum-DDTtotal	36.43	37.88	98.08
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	15.66	52.55	23.10
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	148.23	147.05	143.38
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	No stable	No stable	No stable
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 58: Pesticides and Chlorinated Flame Retardants concentrations in open sea samples

Lab. Code:	OCP-17-24	OCP-17-25	OCP-17-26
Sample name:	4-JOSS	5-JOSS	6-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	21.45	20.55	19.85
Sampling period:	2017	2017	2017
Analysis date:	11/20/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	33.08	6.76	5.46
HCB	23.28	12.30	11.18
a-HCH	99.91	98.44	99.65
b-HCH	3398.33	2690.34	2689.19
g-HCH	45.15	47.35	43.62
d-HCH	<LOD	<LOD	<LOD
e-HCH	<LOD	<LOD	<LOD
Sum-HCHs	3543.39	2836.13	2832.46
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	2.97	2.69	2.24
Heptachlor-endo-epoxide	2.23	2.16	1.98
Sum-Hetachlorepoxydes	2.97	2.69	2.24
Aldrin	<LOD	<LOD	<LOD
Dieldrin	10.30	9.28	11.11
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	10.30	9.28	11.11
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	77.84	39.50	<LOD
Endosulfane-beta	24.07	17.74	3.98
Sum-Endosulfanes	101.90	57.24	3.98
Endosulfane-sulphate	0.94	1.26	0.89
op-DDE	<LOD	2.49	<LOD

Lab. Code:	OCP-17-24	OCP-17-25	OCP-17-26
Sample name:	4-JOSS	5-JOSS	6-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	21.45	20.55	19.85
Sampling period:	2017	2017	2017
Analysis date:	11/20/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
pp-DDE	7.55	8.75	14.00
op-DDD	<LOD	<LOD	<LOD
pp-DDD	15.03	4.66	12.65
op-DDT	<LOD	<LOD	<LOD
pp-DDT	9.05	18.01	13.48
Sum-DDTtotal	31.63	33.92	40.13
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	161.30	82.94	42.96
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	140.23	63.51	127.72
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	No stable	No stable	No stable
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 59: Pesticides and Chlorinated Flame Retardants concentrations in open sea samples

Lab. Code:	OCP-17-27	OCP-17-28	OCP-17-29
Sample name:	7-JOSS	8-JOSS	9-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	19.85	20.15
Sampling period:	2017	2017	2017
Analysis date:	11/21/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	7.58	5.79	14.33
HCB	12.64	7.82	8.68
a-HCH	104.18	87.43	80.49
b-HCH	3438.43	3224.36	3773.17
g-HCH	58.64	48.50	46.35
d-HCH	<LOD	<LOD	<LOD
e-HCH	<LOD	<LOD	<LOD
Sum-HCHs	3601.26	3360.29	3900.00
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	2.39	2.96	3.17
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	2.39	2.96	3.17
Aldrin	<LOD	<LOD	<LOD
Dieldrin	9.15	10.95	11.01
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	9.15	10.95	11.01
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.63	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.63	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	5.21	4.80	3.98
Sum-Endosulfanes	5.21	4.80	3.98
Endosulfane-sulphate	0.71	0.69	1.20
op-DDE	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-27	OCP-17-28	OCP-17-29
Sample name:	7-JOSS	8-JOSS	9-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	19.85	20.15
Sampling period:	2017	2017	2017
Analysis date:	11/21/2017	11/21/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
pp-DDE	5.87	9.16	7.39
op-DDD	<LOD	<LOD	<LOD
pp-DDD	13.10	9.83	14.18
op-DDT	<LOD	<LOD	<LOD
pp-DDT	4.89	12.15	11.30
Sum-DDTtotal	23.86	31.15	32.87
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	46.02	86.44	538.58
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	43.90	32.98	18.27
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	No stable	No stable	No stable
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 60: Pesticides and Chlorinated Flame Retardants concentrations in open sea samples

Lab. Code:	OCP-17-30	OCP-17-31	OCP-17-32
Sample name:	10-JOSS	11-JOSS	12-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.00	19.80	19.60
Sampling period:	2017	2017	2017
Analysis date:	11/21/2017	11/22/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	11.07	15.48	12.05
HCB	8.30	7.68	8.07
a-HCH	88.58	107.14	79.18
b-HCH	4404.90	4517.30	3869.91
g-HCH	54.09	78.48	43.96
d-HCH	<LOD	3.89	<LOD
e-HCH	<LOD	<LOD	<LOD
Sum-HCHs	4547.57	4706.80	3993.05
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	3.62	3.81	3.15
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	3.62	3.81	3.15
Aldrin	<LOD	<LOD	<LOD
Dieldrin	12.81	n.d	10.45
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	12.81	---	10.45
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	9.23	3.76
Sum-Endosulfanes	---	9.23	3.76
Endosulfane-sulphate	1.53	1.81	1.60
op-DDE	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-30	OCP-17-31	OCP-17-32
Sample name:	10-JOSS	11-JOSS	12-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.00	19.80	19.60
Sampling period:	2017	2017	2017
Analysis date:	11/21/2017	11/22/2017	11/21/2017
Concentration	pg/L	pg/L	pg/L
pp-DDE	5.04	< LOD	4.91
op-DDD	< LOD	< LOD	< LOD
pp-DDD	11.69	10.68	11.06
op-DDT	< LOD	< LOD	< LOD
pp-DDT	7.26	10.50	7.75
Sum-DDTtotal	23.98	21.18	23.71
Methoxychlor	< LOD	< LOD	< LOD
Mirex	< LOD	< LOD	< LOD
<i>Other pesticides:</i>			
HCBD	138.79	46.46	221.66
Dichlorvos	< LOD	< LOD	< LOD
Trifluralin	< LOD	< LOD	< LOD
Triallate	< LOD	< LOD	< LOD
Chlorpyriphos	24.35	16.21	14.11
Chlorfenvinphos	< LOD	< LOD	< LOD
Dicofol	No stable	No stable	No stable
Cypermethrins	< LOD	< LOD	< LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	< LOD	< LOD	< LOD
syn-Decchlorane Plus	< LOD	< LOD	< LOD
anti-Decchlorane Plus	< LOD	< LOD	< LOD

### 6.1.2.3. Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT

In Tables 61 to 68 the results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT obtained with 20L spot samples are reported.

*Table 61: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in blank samples*

Lab. Code:	PAH-FBLK-FILTER	PAH-17-047	PAH-17-048
Sample name:	Field Blank Filter	FB-1	FB-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	21.34	21.29
Sampling period:	2017	2017	2017
Analysis date:	12/6/2017	12/6/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	399	9182	5576
Anthracene	56.54	836	871
Fluoranthene	94.22	529	1013
Pyrene	34.53	274	420
Benz(a)anthracene	<LOD	17.58	13.50
Chrysene	4.67	126	92.22
Sum Benzo(b,j,k)fluoranthene	9.52	91.14	73.06
Benzo(e)pyrene	<LOD	25.87	88.20
Benzo(a)pyrene	<LOD	16.91	43.76
Perylene	<LOD	21.32	15.59
Indeno(123-cd)pyrene	<LOD	15.44	39.24
Benzo(ghi)perylene	<LOD	74.20	376
Dibenz(ah)anthracene	<LOD	<LOD	<LOD
Coronene	55.35	31.42	360
BHT	<LOD	<LOD	<LOD
EHMC	495	3958	6531

*Table 62: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in samples from inside (1A and 1A') and 1 outside (1C) Danube delta*

Lab. Code:	PAH-17-020	PAH-17-021	PAH-17-046
Sample name:	1A	1A - Rep	1-C
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.05	20.00	20.80
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	3432	4229	2076
Anthracene	1100	1075	1109
Fluoranthene	2058	1902	795
Pyrene	1233	1456	522
Benz(a)anthracene	82.18	96.63	63.85
Chrysene	351	389	221
Sum Benzo(b,j,k)fluoranthene	704	924	459
Benzo(e)pyrene	296	285	189
Benzo(a)pyrene	225	244	192
Perylene	2304	2364	1260
Indeno(123-cd)pyrene	207	226	132
Benzo(ghi)perylene	571	559	380
Dibenz(ah)anthracene	133	183	125
Coronene	397	349	350
BHT	74467	64846	70563
EHMC	4972	93561	9700

*Table 63: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in samples from Georgia Coast*

Lab. Code:	PAH-17-035	PAH-17-037	PAH-17-038
Sample name:	1-GE	2-GE	3-GE
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.65	20.60	20.65
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	4089	8528	2865
Anthracene	975	2953	762
Fluoranthene	1605	7002	1325
Pyrene	1350	3994	866
Benz(a)anthracene	82.43	652	41.15
Chrysene	355	2273	344
Sum Benzo(b,j,k)fluoranthene	583	4794	581
Benzo(e)pyrene	274	2006	320
Benzo(a)pyrene	146	1411	172
Perylene	770	4991	641
Indeno(123-cd)pyrene	284	1702	289
Benzo(ghi)perylene	722	3549	734
Dibenz(ah)anthracene	198	1472	283
Coronene	729	3044	1018
BHT	89454	84273	51495
EHMC	3145	17348	6419

*Table 64: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in samples from Ukraine Coast*

Lab. Code:	PAH-17-039	PAH-17-041	PAH-17-040
Sample name:	CW-5	CW-8	CW-7
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	23.40	23.85	22.25
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	22188	2450	5584
Anthracene	6251	1149	1895
Fluoranthene	8116	904	5675
Pyrene	9039	551	4956
Benz(a)anthracene	919	59.90	2000
Chrysene	1982	173	3565
Sum Benzo(b,j,k)fluoranthene	4301	498	16097
Benzo(e)pyrene	3725	154	3684
Benzo(a)pyrene	5992	173	4714
Perylene	12105	94.72	2085
Indeno(123-cd)pyrene	3853	155	3494
Benzo(ghi)perylene	6262	266	6634
Dibenz(ah)anthracene	2549	102	2905
Coronene	1509	84.57	3497
BHT	267758	55477	184583
EHMC	35174	10550	95585

*Table 65: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-17-22	PAH-17-33	PAH-17-23
Sample name:	1-JOSS	2-JOSS	3-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.80	20.30	20.00
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1559	1620	1657
Anthracene	398	539	398
Fluoranthene	285	384	507
Pyrene	294	320	414
Benz(a)anthracene	6.47	10.27	8.23
Chrysene	65.31	73.11	67.91
Sum Benzo(b,j,k)fluoranthene	65.31	96.90	85.73
Benzo(e)pyrene	19.56	28.09	21.64
Benzo(a)pyrene	11.47	11.95	9.92
Perylene	9.82	7.96	11.57
Indeno(123-cd)pyrene	16.00	21.93	15.52
Benzo(ghi)perylene	43.15	71.59	35.48
Dibenz(ah)anthracene	16.48	21.05	< LOD
Coronene	56.16	71.73	25.46
BHT	17827	29998	20702
EHMC	2440	3337	4608

*Table 66: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-17-24	PAH-17-25	PAH-17-26
Sample name:	4-JOSS	5-JOSS	6-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	21.45	20.55	19.85
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	8270	1348	1521
Anthracene	766	255	404
Fluoranthene	292	541	258
Pyrene	344	350	321
Benz(a)anthracene	14.93	12.90	7.84
Chrysene	86.50	94.59	63.80
Sum Benzo(b,j,k)fluoranthene	178	141	105
Benzo(e)pyrene	43.65	57.08	21.13
Benzo(a)pyrene	59.68	42.55	8.57
Perylene	28.03	17.41	7.46
Indeno(123-cd)pyrene	42.14	39.39	11.77
Benzo(ghi)perylene	90.47	100	25.29
Dibenz(ah)anthracene	30.78	<LOD	<LOD
Coronene	59.30	63.78	<LOD
BHT	10905	23276	28944
EHMC	2138	6487	3939

*Table 67: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-17-27	PAH-17-28	PAH-17-29
Sample name:	7-JOSS	8-JOSS	9-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	19.85	20.15
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1391	1044	1273
Anthracene	167	359	611
Fluoranthene	557	325	452
Pyrene	441	231	302
Benz(a)anthracene	21.36	5.63	7.45
Chrysene	169	52.56	62.06
Sum Benzo(b,j,k)fluoranthene	392	94.32	130
Benzo(e)pyrene	86.12	35.50	28.92
Benzo(a)pyrene	61.67	14.41	16.16
Perylene	25.54	10.66	8.41
Indeno(123-cd)pyrene	86.68	22.30	34.84
Benzo(ghi)perylene	184	62.13	60.83
Dibenz(ah)anthracene	14.42	<LOD	<LOD
Coronene	111	33.75	37.55
BHT	16114	19344	28103
EHMC	2949	8298	2095

*Table 68: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT concentrations in open sea samples*

Lab. Code:	PAH-17-30	PAH-17-31	PAH-17-32
Sample name:	10-JOSS	11-JOSS	12-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.00	19.80	19.60
Sampling period:	2017	2017	2017
Analysis date:	12/7/2017	12/7/2017	12/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	1105	1648	1279
Anthracene	414	584	512
Fluoranthene	282	374	368
Pyrene	241	291	282
Benz(a)anthracene	14.04	14.56	11.98
Chrysene	52.28	72.67	101
Sum Benzo(b,j,k)fluoranthene	88.76	163	188
Benzo(e)pyrene	26.03	48.63	49.64
Benzo(a)pyrene	11.02	24.12	25.31
Perylene	8.56	13.73	13.76
Indeno(123-cd)pyrene	29.52	41.81	57.27
Benzo(ghi)perylene	80.12	87.94	86.45
Dibenz(ah)anthracene	<LOD	16.10	22.12
Coronene	63.02	66.02	82.10
BHT	22746	34448	30582
EHMC	3183	1302	1712

#### 6.1.2.4. Polychlorinated Biphenyls (EC-7 PCBs)

In Tables 69 to 76 the results of EC-7 Polychlorinated Biphenyls obtained with 20L spot samples are reported.

*Table 69: EC-7 Polychlorinated Biphenyls concentrations in blank samples*

Lab. Code:	PCB-FBLK-FILTER	PCB-17-047	PCB-17-048
Sample name:	Field Blank Filter	FB-1	FB-2
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.00	21.34	21.29
Sampling period:	2017	2017	2017
Analysis date:	12/11/2017	12/11/2017	12/11/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	0.74	7.22	9.09
PCB 52	< LOD	2.52	2.52
PCB 101	0.55	3.27	3.85
PCB 118	< LOD	1.80	1.36
PCB 138	2.11	8.38	8.05
PCB 153	1.04	8.38	9.53
PCB 180	< LOD	3.98	5.96
Sum EC-7 PCBs	4.44	35.55	40.35

*Table 70: EC-7 Polychlorinated Biphenyls concentrations in samples from inside (1A and 1A') and 1 outside (1C) Danube delta*

Lab. Code:	PCB-17-020	PCB-17-021	PCB-17-046
Sample name:	1A	1A - Rep	1-C
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.05	20.00	20.80
Sampling period:	2017	2017	2017
Analysis date:	12/13/2017	12/13/2017	12/13/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	86.17	89.89	29.07
PCB 52	60.40	69.61	27.22
PCB 101	66.33	71.92	30.96
PCB 118	83.93	107.00	31.34
PCB 138	72.53	98.32	45.95
PCB 153	76.31	98.15	44.27
PCB 180	15.60	22.96	13.01
Sum EC-7 PCBs	461	558	222

*Table 71: EC-7 Polychlorinated Biphenyls concentrations concentrations in samples from Georgia Coast*

Lab. Code:	PCB-17-035	PCB-17-037	PCB-17-038
Sample name:	1-GE	2-GE	3-GE
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	20.65	20.60	20.65
Sampling period:	2017	2017	2017
Analysis date:	12/12/2017	12/12/2017	12/12/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	24.89	78.93	22.24
PCB 52	24.48	64.77	18.59
PCB 101	30.18	91.25	24.17
PCB 118	18.56	42.47	14.49
PCB 138	62.16	108.19	51.17
PCB 153	33.86	101.18	32.17
PCB 180	<LOD	49.76	<LOD
Sum EC-7 PCBs	194	537	163

*Table 72: EC-7 Polychlorinated Biphenyls concentrations concentrations in samples from Ukraine Coast*

Lab. Code:	PCB-17-039	PCB-17-041	PCB-17-040
Sample name:	CW-5	CW-8	CW-7
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	23.40	22.85	22.50
Sampling period:	2017	2017	2017
Analysis date:	12/12/2017	12/12/2017	12/12/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	38.42	10.52	31.06
PCB 52	33.50	9.40	37.84
PCB 101	68.32	6.13	61.06
PCB 118	37.56	6.08	49.39
PCB 138	153.46	22.17	80.50
PCB 153	91.25	13.11	58.42
PCB 180	<LOD	4.72	<LOD
Sum EC-7 PCBs	423	72.12	318

*Table 73: EC-7 Polychlorinated Biphenyls concentrations concentrations in open sea samples*

Lab. Code:	PCB-17-022	PCB-17-033	PCB-17-023
Sample name:	1-JOSS	2-JOSS	3-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.80	20.30	20.00
Sampling period:	2017	2017	2017
Analysis date:	12/11/2017	12/12/2017	12/11/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	3.82	4.30	5.85
PCB 52	2.11	2.61	5.77
PCB 101	2.95	3.15	6.70
PCB 118	1.85	1.58	4.05
PCB 138	6.42	5.23	16.77
PCB 153	6.93	5.97	10.35
PCB 180	4.44	<LOD	3.93
Sum EC-7 PCBs	28.52	22.84	53.42

*Table 74: EC-7 Polychlorinated Biphenyls concentrations concentrations in open sea samples*

Lab. Code:	PCB-17-024	PCB-17-025	PCB-17-026
Sample name:	4-JOSS	5-JOSS	6-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	21.45	20.55	19.85
Sampling period:	2017	2017	2017
Analysis date:	12/11/2017	12/11/2017	12/12/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	6.60	5.06	4.58
PCB 52	3.44	4.70	2.14
PCB 101	5.09	7.27	5.78
PCB 118	1.98	4.25	2.19
PCB 138	11.54	17.97	14.28
PCB 153	10.33	14.36	9.40
PCB 180	3.17	4.96	4.74
Sum EC-7 PCBs	42.16	58.57	43.11

*Table 75: EC-7 Polychlorinated Biphenyls concentrations in open sea samples*

Lab. Code:	PCB-17-027	PCB-17-028	PCB-17-029
Sample name:	7-JOSS	8-JOSS	9-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.85	19.85	20.15
Sampling period:	2017	2017	2017
Analysis date:	12/12/2017	12/12/2017	12/12/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	4.32	3.17	3.90
PCB 52	2.79	2.06	2.60
PCB 101	3.95	3.28	4.55
PCB 118	2.00	2.26	1.73
PCB 138	8.58	8.64	10.84
PCB 153	6.69	6.43	8.92
PCB 180	2.83	2.18	3.51
Sum EC-7 PCBs	31.17	28.03	36.04

Table 76: EC-7 Polychlorinated Biphenyls concentrations in open sea samples

Lab. Code:	PCB-17-030	PCB-17-031-bis	PCB-17-032
Sample name:	10-JOSS	11-JOSS	12-JOSS
Type of sample:	MB Black Sea water	MB Black Sea water	MB Black Sea water
Volume sampled (L):	19.00	19.80	19.60
Sampling period:	2017	2017	2017
Analysis date:	12/12/2017	12/13/2017	12/12/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	3.14	3.41	3.68
PCB 52	2.32	1.87	2.28
PCB 101	3.46	2.22	3.03
PCB 118	1.86	1.99	2.66
PCB 138	8.63	6.54	6.06
PCB 153	7.67	6.06	4.71
PCB 180	2.73	2.43	2.43
Sum EC-7 PCBs	29.80	24.53	24.85

## 6.2. Individual results for Large Volume Transect Samples

### 6.2.1. Organophosphate Compounds (OPCs)

In the Tables 77 to 82 the results of Organophosphate Compounds, filters, primary cells and secondary cells (breakthrough control) obtained with the Large Volume Transect sampling are reported.

*Table 77: Organophosphate Compounds in Field blank samples*

Lab. Code:	OPC-17-055	OPC-17-067	OPC-17-069
Sample name:	Filter BLK	Field Blank Cell 1	Field Blank Cell 2
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Analysis date:	11/15/2017	11/17/2017	11/17/2017
Concentration	ng/L	ng/L	ng/L
TEP	0.0053	0.046	0.035
TNPP	0.058	0.023	0.021
TI BP	0.040	0.014	0.012
TNBP	0.008	0.010	0.007
TCEP	0.0006	0.002	0.001
TCPP	0.010	0.010	0.008
TDCPP	0.003	0.001	0.002
TBOEP	0.006	0.009	0.006
TPhP	0.003	0.002	0.002
EHDP	0.005	0.004	0.005
TEHP	0.0005	0.0007	0.0007
TMPP	0.0002	0.0003	0.0002
TI PPP	0.0002	0.0005	0.0005
T35DMPP	0.0001	0.0002	0.0003

Bold numbers are LOD calculated on signal to noise 3/1 in order to use the value obtained for the estimation of LOD blank +3std and LOQ blank +10std

Table 78 : Results of Organophosphate Compounds in Transect 1 (JOSS 1)

Lab. Code:	OPC-17-050	OPC-17-056	OPC-17-057	Sum Cell 1 and 2
Sample name:	Filter 1	JOSS 1 - Cell 1	JOSS 1 - Cell 2	JOSS 1
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	180.00	180.00	180.00	180.00
Sampling period:	2017	2017	2017	2017
Analysis date:	11/17/2017	11/15/2017	11/15/2017	
Concentration	ng/L	ng/L	ng/L	ng/L
TEP	<LOD	0.22	0.41	0.62
TNPP	<LOD	<LOD	<LOD	<LOD
TIBP	<LOD	2.12	0.51	2.63
TNBP	<LOD	0.32	0.10	0.42
TCEP	<LOD	0.17	0.073	0.24
TCPP	0.017	3.85	0.93	4.78
TDCPP	<LOD	0.34	0.069	0.40
TBOEP	<LOD	<LOD	<LOD	<LOD
TPhP	<LOD	0.021	0.003	0.023
EHDP	<LOD	<LOD	<LOD	<LOD
TEHP	0.002	0.001	<LOD	0.001
TMPP	<LOD	0.017	0.001	0.018
TI PPP	<LOD	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD	<LOD

Table 79: Results of Organophosphate Compounds in Transect 2 (JOSS 2)

Lab. Code:	OPC-17-051	OPC-17-058	OPC-17-059	Sum Cell 1 and 2
Sample name:	Filter 2	JOSS 2 - Cell 1	JOSS 2 - Cell 2	JOSS 2
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.90	90.90	90.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	11/17/2017	11/15/2017	11/15/2017	
Concentration	ng/L	ng/L	ng/L	ng/L
TEP	<LOD	0.37	0.52	0.90
TNPP	<LOD	<LOD	<LOD	<LOD
TIBP	<LOD	2.60	0.35	2.95
TNBP	<LOD	0.31	0.044	0.36
TCEP	<LOD	0.20	0.041	0.24
TCPP	0.044	4.29	0.34	4.63
TDCPP	<LOD	0.29	0.022	0.32
TBOEP	<LOD	<LOD	<LOD	<LOD
TPhP	<LOD	0.012	<LOD	0.012
EHDP	<LOD	0.027	0.007	0.035
TEHP	<LOD	0.002	0.002	0.003
TMPP	<LOD	0.007	0.001	0.008
TI PPP	<LOD	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD	<LOD

Table 80: Results of Organophosphate Compounds in Transect 3 (JOSS 3)

Lab. Code:	OPC-17-052	OPC-17-060	OPC-17-061	Sum Cell 1 and 2
Sample name:	Filter 3	JOSS 3 - Cell 1	JOSS 3 - Cell 2	JOSS 3
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	87.90	87.90	87.90	87.90
Sampling period:	2017	2017	2017	2017
Analysis date:	11/17/2017	11/16/2017	11/15/2017	
Concentration	ng/L	ng/L	ng/L	ng/L
TEP	<LOD	0.43	0.71	1.15
TNPP	<LOD	<LOD	<LOD	<LOD
TIBP	<LOD	2.20	0.61	2.81
TNBP	<LOD	0.34	0.10	0.44
TCEP	<LOD	0.22	0.06	0.28
TCPP	0.023	3.50	0.66	4.16
TDCPP	0.008	0.29	0.05	0.34
TBOEP	<LOD	<LOD	<LOD	<LOD
TPhP	0.004	0.008	0.004	0.011
EHDP	0.014	0.016	0.010	0.025
TEHP	0.002	0.002	0.001	0.003
TMPP	<LOD	0.010	0.026	0.035
TI PPP	<LOD	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD	<LOD

*Table 81: Results of Organophosphate Compounds in Transect 4 (JOSS 4)*

Lab. Code:	OPC-17-053	OPC-17-062	OPC-17-063	Sum Cell 1 and 2
Sample name:	Filter 4	JOSS 4 - Cell 1	JOSS 4 - Cell 2	JOSS 4
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.00	90.00	90.00	90.00
Sampling period:	2017	2017	2017	2017
Analysis date:	11/17/2017	11/16/2017	11/15/2017	
Concentration	ng/L	ng/L	ng/L	ng/L
TEP	<LOD	0.20	0.64	0.84
TNPP	<LOD	<LOD	<LOD	<LOD
TIBP	<LOD	1.99	0.76	2.75
TNBP	<LOD	0.38	0.11	0.49
TCEP	<LOD	0.12	0.089	0.21
TCPP	0.021	3.89	1.22	5.11
TDCPP	<LOD	0.37	0.096	0.47
TBOEP	<LOD	<LOD	<LOD	<LOD
TPhP	<LOD	0.008	0.005	0.013
EHDP	<LOD	0.017	0.011	0.028
TEHP	0.002	0.002	0.001	0.003
TMPP	<LOD	0.006	0.0003	0.006
TI PPP	<LOD	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD	<LOD

*Table 82: Results of Organophosphate Compounds in Transect 5 (JOSS 5)*

Lab. Code:	OPC-17-054	OPC-17-064	OPC-17-065	Sum Cell 1 and 2
Sample name:	Filter 5	JOSS 5 - Cell 1	JOSS 5 - Cell 2	JOSS 5
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	88.50	88.50	88.50	88.50
Sampling period:	2017	2017	2017	2017
Analysis date:	11/15/2017	11/16/2017	11/15/2017	
Concentration	ng/L	ng/L	ng/L	ng/L
TEP	<LOD	0.36	0.87	1.23
TNPP	<LOD	<LOD	<LOD	<LOD
TIBP	<LOD	3.54	0.76	4.31
TNBP	<LOD	0.53	0.12	0.65
TCEP	<LOD	0.14	0.04	0.18
TCPP	0.056	6.54	1.06	7.60
TDCPP	<LOD	0.77	0.12	0.89
TBOEP	<LOD	<LOD	<LOD	<LOD
TPhP	<LOD	0.015	0.010	0.025
EHDP	0.006	0.022	0.013	0.035
TEHP	0.006	0.005	0.004	0.009
TMPP	0.0005	0.008	<LOD	0.008
TI PPP	<LOD	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD	<LOD

## 6.2.2. Pesticides and Chlorinated Flame Retardants

In Tables 83 to 88 the results of Pesticides and Chlorinated Flame Retardants obtained with LV Transect Sampling are reported.

*Table 83: Pesticides and Chlorinated Flame Retardants in Field blank samples*

Lab. Code:	OCP-17-055	OCP-17-067	OCP-17-069-BIS
Sample name:	Filter BLK	Field Blank Cell 1	Field Blank Cell 2
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Analysis date:	10/23/2017	10/23/2017	10/24/2017
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	0.16	0.22	0.24
HCB	0.25	0.37	0.37
a-HCH	0.03	0.04	0.20
b-HCH	0.12	0.37	5.34
g-HCH	0.20	0.12	0.26
d-HCH	0.05	0.03	0.09
e-HCH	0.07	0.03	0.14
Sum-HCHs			
Heptachlor	0.06	0.04	0.02
Heptachlor-exo-epoxide	0.04	0.07	0.16
Heptachlor-endo-epoxide	0.18	0.21	0.90
Sum-Hetachlorepoxydes			
Aldrin	0.06	0.09	0.22
Dieldrin	0.07	0.12	0.17
Endrin	0.23	0.16	0.14
Isodrin	0.50	0.51	1.22
Sum-Drins			
trans-chlordane	0.02	0.02	0.04
cis-chlordane	0.03	0.01	0.03
Sum-Chlordanes			
Oxychlordane	0.04	0.04	0.12
trans-nonachlor	0.00	0.01	0.04
cis-nonachlor	0.04	0.05	0.06
Sum-nonachlor			
Endosulfane-alpha	1.01	0.92	0.78
Endosulfane-beta	0.09	0.13	0.13
Sum-Endosulfanes			
Endosulfane-sulphate	0.04	0.03	0.07
op-DDE	0.15	0.06	0.11
pp-DDE	0.22	0.13	0.20
op-DDD	0.13	0.03	0.23
pp-DDD	0.08	0.06	0.05
op-DDT	0.25	0.29	0.28
pp-DDT	0.06	0.14	0.20
Sum-DDTtotal			
Methoxychlor	0.37	1.05	0.35

Lab. Code:	OCP-17-055	OCP-17-067	OCP-17-069-BIS
Sample name:	Filter BLK	Field Blank Cell 1	Field Blank Cell 2
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Analysis date:	10/23/2017	10/23/2017	10/24/2017
Concentration	pg/L	pg/L	pg/L
Mirex	0.03	0.03	0.03
<i>Other pesticides:</i>			
HCBD	2.94	7.14	5.51
Dichlorvos	0.37	0.82	1.12
Trifluralin	0.02	0.02	0.06
Triallate	0.13	0.05	0.09
Chlorpyriphos	0.42	0.10	0.40
Chlorgenvinphos	1.78	4.31	4.87
Dicofol	0.28	0.23	0.19
Cypermethrins	0.32	0.49	0.78
<i>Chlorinated Flame Retardants:</i>			
Chloran 542	24.69	46.09	106.90
syn-Decchlorane Plus	0.057	0.036	0.117
anti-Decchlorane Plus	0.032	0.040	0.081

Bold numbers are LOD calculated on signal to noise 3/1 in order to use the value obtained for the estimation of LOD blank +3std and LOQ blank +10std

Table 84: Results of Pesticides and Chlorinated Flame Retardants in Transect 1 (JOSS 1)

Lab. Code:	OCP-17-050	OCP-17-056	OCP-17-057	Sum Cell 1 and 2
Sample name:	Filter 1	JOSS 1 - Cell 1	JOSS 1 - Cell 2	JOSS 1
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	180.00	180.00	180.00	180.00
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>				
PeCBz	< LOD	1.47	< LOD	1.47
HCB	< LOD	4.52	< LOD	4.52
a-HCH	< LOD	113	13.15	127
b-HCH	< LOD	3449	408	3858
g-HCH	< LOD	47.65	5.56	53.22
d-HCH	< LOD	2.74	0.24	2.99
e-HCH	< LOD	2.14	0.21	2.35
Sum-HCHs	---	3615	428	4043
Heptachlor	< LOD	< LOD	< LOD	< LOD
Heptachlor-exo-epoxide	< LOD	2.88	0.48	3.37
Heptachlor-endo-epoxide	< LOD	< LOD	< LOD	< LOD
Sum-Hetachlorepoxydes	---	2.88	0.48	3.37
Aldrin	< LOD	< LOD	< LOD	< LOD
Dieldrin	< LOD	6.91	1.04	7.95
Endrin	< LOD	< LOD	< LOD	< LOD
Isodrin	< LOD	< LOD	< LOD	< LOD
Sum-Drins	---	6.91	1.04	7.95
trans-chlordane	< LOD	< LOD	< LOD	< LOD
cis-chlordane	< LOD	< LOD	< LOD	< LOD
Sum-Chlordane	---	---	---	---
Oxychlordanane	< LOD	< LOD	< LOD	< LOD
trans-nonachlor	< LOD	0.12	< LOD	0.12
cis-nonachlor	< LOD	< LOD	< LOD	< LOD
Sum-nonachlor	---	0.12	---	0.12
Endosulfane-alpha	< LOD	< LOD	< LOD	< LOD
Endosulfane-beta	< LOD	1.30	0.22	1.52
Sum-Endosulfanes	---	1.30	0.22	1.52
Endosulfane-sulphate	< LOD	0.65	0.12	0.77
op-DDE	< LOD	< LOD	< LOD	< LOD
pp-DDE	< LOD	3.84	< LOD	3.84
op-DDD	< LOD	4.78	< LOD	4.78
pp-DDD	< LOD	12.16	0.23	12.39
op-DDT	< LOD	1.37	< LOD	1.37
pp-DDT	< LOD	5.29	< LOD	5.29
Sum-DDTtotal	---	27.44	0.23	27.67
Methoxychlor	< LOD	< LOD	< LOD	< LOD
Mirex	< LOD	< LOD	< LOD	< LOD
<i>Other pesticides:</i>				
HCBD	< LOD	< LOD	< LOD	< LOD
Dichlorvos	< LOD	< LOD	< LOD	< LOD

Lab. Code:	OCP-17-050	OCP-17-056	OCP-17-057	Sum Cell 1 and 2
Sample name:	Filter 1	JOSS 1 - Cell 1	JOSS 1 - Cell 2	JOSS 1
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	180.00	180.00	180.00	180.00
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Trifluralin	< LOD	0.70	< LOD	0.70
Triallate	< LOD	1.19	< LOD	1.19
Chlorpyriphos	< LOD	104	4.45	109
Chlorfenvinphos	< LOD	< LOD	< LOD	< LOD
Dicofol	< LOD	< LOD	< LOD	< LOD
Cypermethrins	< LOD	< LOD	< LOD	< LOD
<i>Chlorinated Flame Retardants:</i>				
Chloran 542	< LOD	< LOD	< LOD	< LOD
syn-Dechlorane Plus	< LOD	< LOD	< LOD	< LOD
anti-Dechlorane Plus	< LOD	< LOD	< LOD	< LOD

Table 85: Results of Pesticides and Chlorinated Flame Retardants in Transect 2 (JOSS 2)

Lab. Code:	OCP-17-051	OCP-17-058	OCP-17-059	Sum Cell 1 and 2
Sample name:	Filter 2	JOSS 2 - Cell 1	JOSS 2 - Cell 2	JOSS 2
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.90	90.90	90.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>				
PeCBz	<LOD	1.30	<LOD	1.30
HCB	<LOD	4.29	<LOD	4.29
a-HCH	<LOD	92.99	3.15	96.14
b-HCH	<LOD	3941	148	4089
g-HCH	<LOD	40.96	2.07	43.03
d-HCH	<LOD	1.71	<LOD	1.71
e-HCH	<LOD	<LOD	<LOD	<LOD
Sum-HCHs	---	4076	154	4230
Heptachlor	<LOD	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	3.84	0.25	4.09
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	3.84	0.25	4.09
Aldrin	<LOD	<LOD	<LOD	<LOD
Dieldrin	<LOD	8.91	0.64	9.55
Endrin	<LOD	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD	<LOD
Sum-Drins	---	8.91	0.64	9.55
trans-chlordanne	<LOD	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.99	<LOD	1.99
Sum-Endosulfanes	---	1.99		1.99
Endosulfane-sulphate	<LOD	0.96	<LOD	0.96
op-DDE	<LOD	<LOD	<LOD	<LOD
pp-DDE	<LOD	1.15	<LOD	1.15
op-DDD	<LOD	2.10	<LOD	2.10
pp-DDD	0.19	9.36	0.17	9.53
op-DDT	<LOD	0.59	<LOD	0.59
pp-DDT	<LOD	4.28	<LOD	4.28
Sum-DDTtotal	0.19	17.48	0.17	17.65
Methoxychlor	<LOD	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD	<LOD
<i>Other pesticides:</i>				
HCBD	<LOD	<LOD	<LOD	<LOD
Dichlorvos	<LOD	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-051	OCP-17-058	OCP-17-059	Sum Cell 1 and 2
Sample name:	Filter 2	JOSS 2 - Cell 1	JOSS 2 - Cell 2	JOSS 2
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.90	90.90	90.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Trifluralin	<LOD	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD	<LOD
Chlorpyriphos	<LOD	20.93	<LOD	20.93
Chlorfenvinphos	<LOD	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>				
Chloran 542	<LOD	<LOD	<LOD	<LOD
syn-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD
anti-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD

Table 86: Results of Pesticides and Chlorinated Flame Retardants in Transect 3 (JOSS 3)

Lab. Code:	OCP-17-052-BIS	OCP-17-060	OCP-17-061-BIS	Sum Cell 1 and 2
Sample name:	Filter 3	JOSS 3 - Cell 1	JOSS 3 - Cell 2	JOSS 3
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	87.90	87.90	87.90	87.90
Sampling period:	2017	2017	2017	2017
Analysis date:	10/24/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>				
PeCBz	<LOD	1.65	<LOD	1.65
HCB	<LOD	5.94	<LOD	5.94
a-HCH	<LOD	142	11.47	153
b-HCH	<LOD	4371	382	4753
g-HCH	<LOD	56.51	5.30	61.82
d-HCH	<LOD	2.54	<LOD	2.54
e-HCH	<LOD	2.37	<LOD	2.37
Sum-HCHs	---	4575	399	4973
Heptachlor	<LOD	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	4.18	0.50	4.68
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	4.18	0.50	4.68
Aldrin	<LOD	<LOD	<LOD	<LOD
Dieldrin	<LOD	9.01	1.26	10.27
Endrin	<LOD	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD	<LOD
Sum-Drins	---	9.01	1.26	10.27
trans-chlordanne	<LOD	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.88	<LOD	1.88
Sum-Endosulfanes	---	1.88	---	1.88
Endosulfane-sulphate	<LOD	0.68	<LOD	0.68
op-DDE	<LOD	<LOD	<LOD	<LOD
pp-DDE	<LOD	2.26	0.19	2.45
op-DDD	<LOD	2.90	<LOD	2.90
pp-DDD	0.28	12.56	<LOD	12.56
op-DDT	<LOD	1.02	<LOD	1.02
pp-DDT	<LOD	5.16	<LOD	5.16
Sum-DDTtotal	0.28	23.90	0.19	24.09
Methoxychlor	<LOD	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD	<LOD
<i>Other pesticides:</i>				
HCBD	<LOD	12.49	6.13	18.63
Dichlorvos	<LOD	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-052-BIS	OCP-17-060	OCP-17-061-BIS	Sum Cell 1 and 2
Sample name:	Filter 3	JOSS 3 - Cell 1	JOSS 3 - Cell 2	JOSS 3
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	87.90	87.90	87.90	87.90
Sampling period:	2017	2017	2017	2017
Analysis date:	10/24/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Trifluralin	<LOD	<LOD	<LOD	<LOD
Triallate	<LOD	1.00	<LOD	<LOD
Chlorpyriphos	1.21	62.50	2.00	64.49
Chlorfenvinphos	<LOD	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>				
Chloran 542	<LOD	<LOD	<LOD	<LOD
syn-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD
anti-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD

Table 87: Results of Pesticides and Chlorinated Flame Retardants in Transect 4 (JOSS 4)

Lab. Code:	OCP-17-053	OCP-17-062	OCP-17-063	Sum Cell 1 and 2
Sample name:	Filter 4	JOSS 4 - Cell 1	JOSS 4 - Cell 2	JOSS 4
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.00	90.00	90.00	90.00
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>				
PeCBz	<LOD	4.65	0.52	5.17
HCB	<LOD	5.13	0.97	6.11
a-HCH	<LOD	103	14.98	118
b-HCH	<LOD	2879	565	3444
g-HCH	<LOD	48.01	7.47	55.48
d-HCH	<LOD	2.98	0.49	3.47
e-HCH	<LOD	2.45	0.45	2.90
Sum-HCHs	---	3036	588	3625
Heptachlor	<LOD	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	3.13	0.33	3.46
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	3.13	0.33	3.46
Aldrin	<LOD	<LOD	<LOD	<LOD
Dieldrin	<LOD	6.74	0.72	7.46
Endrin	<LOD	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD	<LOD
Sum-Drins	---	6.74	0.72	7.46
trans-chlordanne	<LOD	0.10	<LOD	0.10
cis-chlordanne	<LOD	0.20	<LOD	0.20
Sum-Chlordanne	---	0.30	---	0.30
Oxychlordane	<LOD	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.11	<LOD	0.11
cis-nonachlor	<LOD	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.11	---	0.11
Endosulfane-alpha	<LOD	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.42	0.43	1.85
Sum-Endosulfanes	---	1.42	0.43	1.85
Endosulfane-sulphate	<LOD	0.70	0.13	0.82
op-DDE	<LOD	<LOD	<LOD	<LOD
pp-DDE	0.55	3.98	<LOD	3.98
op-DDD	0.45	6.35	<LOD	6.35
pp-DDD	1.12	25.41	<LOD	25.41
op-DDT	<LOD	0.88	<LOD	0.88
pp-DDT	0.24	4.05	<LOD	4.05
Sum-DDTtotal	2.36	40.66	---	40.66
Methoxychlor	<LOD	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD	<LOD
<i>Other pesticides:</i>				
HCBD	<LOD	<LOD	<LOD	<LOD
Dichlorvos	<LOD	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-053	OCP-17-062	OCP-17-063	Sum Cell 1 and 2
Sample name:	Filter 4	JOSS 4 - Cell 1	JOSS 4 - Cell 2	JOSS 4
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.00	90.00	90.00	90.00
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Trifluralin	<LOD	<LOD	<LOD	<LOD
Triallate	<LOD	1.27	<LOD	1.27
Chlorpyriphos	<LOD	106.66	1.20	107.86
Chlorfenvinphos	<LOD	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>				
Chloran 542	<LOD	<LOD	<LOD	<LOD
syn-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD
anti-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD

Table 88: Results of Pesticides and Chlorinated Flame Retardants in Transect 5 (JOSS 5)

Lab. Code:	OCP-17-054	OCP-17-064	OCP-17-065	Sum Cell 1 and 2
Sample name:	Filter 5	JOSS 5 - Cell 1	JOSS 5 - Cell 2	JOSS 5
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	88.50	88.50	88.50	88.50
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>				
PeCBz	<LOD	5.55	1.69	7.25
HCB	<LOD	5.88	0.58	6.46
a-HCH	<LOD	128	8.20	136
b-HCH	<LOD	2847	213	3060
g-HCH	<LOD	61.07	4.40	65.47
d-HCH	<LOD	7.04	0.55	7.59
e-HCH	<LOD	5.84	0.46	6.29
Sum-HCHs	---	3049	227	3276
Heptachlor	<LOD	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	3.88	0.29	4.16
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	3.88	0.29	4.16
Aldrin	<LOD	<LOD	<LOD	<LOD
Dieldrin	<LOD	9.97	0.81	10.79
Endrin	<LOD	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD	<LOD
Sum-Drins	---	9.97	0.81	10.79
trans-chlordanne	<LOD	0.24	<LOD	0.24
cis-chlordanne	<LOD	0.23	<LOD	0.23
Sum-Chlordanne	---	0.47	---	0.47
Oxychlordane	<LOD	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.12	<LOD	0.12
cis-nonachlor	<LOD	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.12	---	0.12
Endosulfane-alpha	<LOD	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.81	<LOD	1.81
Sum-Endosulfanes	---	1.81	---	1.81
Endosulfane-sulphate	<LOD	1.95	0.17	2.12
op-DDE	<LOD	1.16	<LOD	1.16
pp-DDE	2.76	14.97	0.58	15.55
op-DDD	1.56	34.12	0.55	34.67
pp-DDD	5.83	136	1.52	137
op-DDT	0.44	1.48	<LOD	1.48
pp-DDT	1.87	4.79	<LOD	4.79
Sum-DDTtotal	12.46	192	2.64	195
Methoxychlor	<LOD	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD	<LOD
<i>Other pesticides:</i>				
HCBD	<LOD	10.52	2.83	13.35
Dichlorvos	<LOD	<LOD	<LOD	<LOD

Lab. Code:	OCP-17-054	OCP-17-064	OCP-17-065	Sum Cell 1 and 2
Sample name:	Filter 5	JOSS 5 - Cell 1	JOSS 5 - Cell 2	JOSS 5
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	88.50	88.50	88.50	88.50
Sampling period:	2017	2017	2017	2017
Analysis date:	10/23/2017	10/24/2017	10/24/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Trifluralin	<LOD	0.28	<LOD	0.28
Triallate	<LOD	3.97	0.30	4.27
Chlorpyriphos	1.20	75.31	1.00	76.31
Chlorfenvinphos	<LOD	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>				
Chloran 542	<LOD	<LOD	<LOD	<LOD
syn-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD
anti-Dechlorane Plus	<LOD	<LOD	<LOD	<LOD

### 6.2.3. Triazines

In Tables 89 to 94 the results of Triazines obtained with the LV Transect Sampling are reported.

*Table 89: Triazines in Field blank samples*

Lab. Code:	TRIAZ-17-055	TRIAZ-17-067	TRIAZ-17-069
Sample name:	Filter BLK	Field Blank Cell 1	Field Blank Cell 2
<b>Type of sample:</b>	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Analysis date:	6/4/2018	6/4/2018	6/4/2018
Concentration	ng/L	ng/L	ng/L
Simazine	<b>0.005</b>	<b>0.005</b>	<b>0.007</b>
Atrazine	<b>0.003</b>	0.003	0.003
Terbutylazine	<b>0.001</b>	<b>0.0004</b>	<b>0.0004</b>
Sum triazines	---	---	---

**Bold numbers are LOD calculated on signal to noise 3/1 in order to use the value obtained for the estimation of LOD blank +3std and LOQ blank +10std**

*Table 90: Results of Triazines in Transect 1 (JOSS 1)*

Lab. Code:	TRIAZ-17-050	TRIAZ-17-056	TRIAZ-17-057	Sum Cell 1 and 2
Sample name:	Filter 1	JOSS 1 - Cell 1	JOSS 1 - Cell 2	JOSS 1
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	180.00	180.00	180.00	180.00
Sampling period:	2017	2017	2017	2017
Analysis date:	6/4/2018	6/5/2018	6/4/2018	
Concentration	ng/L	ng/L	ng/L	ng/L
Simazine	<LOD	2.45	1.25	3.70
Atrazine	0.009	20.88	8.51	29.39
Terbutylazine	<LOD	1.23	0.29	1.52
Sum triazines	0.009	24.57	10.05	34.61

*Table 91: Results of Triazines in Transect 2 (JOSS 2)*

Lab. Code:	TRIAZ-17-051	TRIAZ-17-058	TRIAZ-17-059	Sum Cell 1 and 2
Sample name:	Filter 2	JOSS 2 - Cell 1	JOSS 2 - Cell 2	JOSS 2
<b>Type of sample:</b>	LV-TS Black Sea water			
Volume sampled (L):	90.90	90.90	90.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	6/4/2018	6/5/2018	6/4/2018	
Concentration	ng/L	ng/L	ng/L	ng/L
Simazine	< LOD	3.22	1.17	4.39
Atrazine	0.022	28.92	6.18	35.10
Terbutylazine	< LOD	1.16	0.12	1.28
Sum triazines	0.022	33.31	7.47	40.78

*Table 92: Results of Triazines in Transect 3 (JOSS 3)*

Lab. Code:	TRIAZ-17-052	TRIAZ-17-060	TRIAZ-17-061	Sum Cell 1 and 2
Sample name:	Filter 3	JOSS 3 - Cell 1	JOSS 3 - Cell 2	JOSS 3
<b>Type of sample:</b>	LV-TS Black Sea water			
Volume sampled (L):	87.90	87.90	87.90	87.90
Sampling period:	2017	2017	2017	2017
Analysis date:	6/4/2018	6/5/2018	6/5/2018	
Concentration	ng/L	ng/L	ng/L	ng/L
Simazine	< LOD	3.46	1.54	5.00
Atrazine	0.034	28.16	9.69	37.85
Terbutylazine	< LOD	1.12	0.25	1.37
Sum triazines	0.034	32.74	11.48	44.22

*Table 93: Results of Triazines in Transect 4 (JOSS 4)*

Lab. Code:	TRIAZ-17-053	TRIAZ-17-062	TRIAZ-17-063	Sum Cell 1 and 2
Sample name:	Filter 4	JOSS 4 - Cell 1	JOSS 4 - Cell 2	JOSS 4
<b>Type of sample:</b>	LV-TS Black Sea water			
Volume sampled (L):	90.00	90.00	90.00	90.00
Sampling period:	2017	2017	2017	2017
Analysis date:	6/4/2018	6/5/2018	6/5/2018	
Concentration	ng/L	ng/L	ng/L	ng/L
Simazine	<LOD	1.60	1.86	3.46
Atrazine	0.019	15.30	12.86	28.16
Terbutylazine	<LOD	1.17	0.53	1.71
Sum triazines	0.019	18.08	15.25	33.33

*Table 94: Results of Triazines in Transect 5 (JOSS 5)*

Lab. Code:	TRIAZ-17-054	TRIAZ-17-064	TRIAZ-17-065	Sum Cell 1 and 2
Sample name:	Filter 5	JOSS 5 - Cell 1	JOSS 5 - Cell 2	JOSS 5
<b>Type of sample:</b>	LV-TS Black Sea water			
Volume sampled (L):	88.50	88.50	88.50	88.50
Sampling period:	2017	2017	2017	2017
Analysis date:	6/4/2018	6/5/2018	6/5/2018	
Concentration	ng/L	ng/L	ng/L	ng/L
Simazine	<LOD	2.11	0.85	2.97
Atrazine	0.0214	19.38	5.72	25.10
Terbutylazine	<LOD	1.78	0.29	2.07
Sum triazines	0.0214	23.27	6.87	30.14

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

In the Tables 95 to 100 the results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT obtained with LV Transect Sampling are reported.

*Table 95: Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Field blank samples*

Lab. Code:	PAHT-17-055	PAH-17-067	PAH-17-069
Sample name:	Filter BLK	Field Blank Cell 1	Field Blank Cell 2
<b>Type of sample:</b>	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Analysis date:	11/7/2017	11/7/2017	11/7/2017
Concentration	pg/L	pg/L	pg/L
Phenanthrene	13.73	26.38	22.92
Anthracene	7.79	4.39	5.07
Fluoranthene	3.77	5.81	3.85
Pyrene	3.99	14.38	8.83
Benz(a)anthracene	0.29	1.15	0.06
Chrysene	1.35	2.69	0.41
Sum Benzo(b,j,k)fluoranthene	1.34	0.89	0.86
Benzo(e)pyrene	0.24	0.15	0.08
Benzo(a)pyrene	0.16	0.16	0.06
Perylene	0.21	0.30	0.17
Indeno(123-cd)pyrene	0.22	0.07	0.08
Benzo(ghi)perylene	0.29	0.38	0.17
Dibenz(ah)anthracene	0.30	0.15	0.08
Coronene	0.76	0.31	0.18
BHT	32.39	211	121
EHMC	46.90	56.17	57.83

*Table 96: Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 1 (JOSS 1)*

Lab. Code:	PAH-17-050	PAH-17-056	PAH-17-057	Sum Cell 1 and 2
Sample name:	Filter 1	JOSS 1 - Cell 1	JOSS 1 - Cell 2	JOSS 1
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	180.00	180.00	180.00	180.00
Sampling period:	2017	2017	2017	2017
Analysis date:	11/7/2017	11/13/2017	11/13/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Phenanthrene	<LOD	543	n.d.	543
Anthracene	<LOD	33.72	n.d.	33.72
Fluoranthene	9.89	29.16	n.d.	29.16
Pyrene	<LOD	133	n.d.	133
Benz(a)anthracene	2.39	4.23	n.d.	4.23
Chrysene	6.61	49.43	n.d.	49.43
Sum Benzo(b,j,k)fluoranthene	9.91	17.16	n.d.	17.16
Benzo(e)pyrene	4.36	6.74	n.d.	6.74
Benzo(a)pyrene	4.72	1.50	n.d.	1.50
Perylene	2.30	1.03	n.d.	1.03
Indeno(123-cd)pyrene	4.27	1.68	n.d.	1.68
Benzo(ghi)perylene	7.69	1.94	n.d.	1.94
Dibenz(ah)anthracene	2.49	1.05	n.d.	1.05
Coronene	6.16	0.72	n.d.	0.72
BHT	<LOD	<LOD	n.d.	<LOD
EHMC	<LOD	<LOD	n.d.	<LOD
n.d.: not detected because of interferences				

*Table 97: Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 2 (JOSS 2)*

Lab. Code:	PAH-17-051	PAH-17-058	PAH-17-059	Sum Cell 1 and 2
Sample name:	Filter 2	JOSS 2 - Cell 1	JOSS 2 - Cell 2	JOSS 2
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.90	90.90	90.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	11/7/2017	11/13/2017	11/13/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Phenanthrene	<LOD	361	<LOD	361
Anthracene	<LOD	29.37	<LOD	29.37
Fluoranthene	10.29	30.66	<LOD	30.66
Pyrene	<LOD	120	<LOD	120
Benz(a)anthracene	<LOD	2.43	<LOD	2.43
Chrysene	<LOD	43.09	<LOD	43.09
Sum Benzo(b,j,k)fluoranthene	5.92	17.04	<LOD	17.04
Benzo(e)pyrene	1.86	4.92	<LOD	4.92
Benzo(a)pyrene	1.35	0.77	<LOD	0.77
Perylene	0.75	0.77	<LOD	0.77
Indeno(123-cd)pyrene	2.17	3.08	0.52	3.60
Benzo(ghi)perylene	3.46	1.86	<LOD	1.86
Dibenz(ah)anthracene	0.89	0.67	0.60	1.26
Coronene	3.77	<LOD	<LOD	<LOD
BHT	<LOD	<LOD	<LOD	<LOD
EHMC	77.17	93.25	<LOD	93.25

Table 98: Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 3 (JOSS 3)

Lab. Code:	PAH-17-052	PAH-17-060	PAH-17-061	Sum Cell 1 and 2
Sample name:	Filter 3	JOSS 3 - Cell 1	JOSS 3 - Cell 2	JOSS 3
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	87.90	87.90	87.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	11/7/2017	11/13/2017	11/13/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Phenanthrene	44.27	381	<LOD	381
Anthracene	13.87	30.16	<LOD	30.16
Fluoranthene	12.79	34.15	33.70	67.86
Pyrene	<LOD	127	<LOD	127.36
Benz(a)anthracene	2.28	<LOD	<LOD	<LOD
Chrysene	8.38	35.13	<LOD	35.13
Sum Benzo(b,j,k)fluoranthene	11.53	13.70	2.06	15.75
Benzo(e)pyrene	5.22	3.34	0.89	4.23
Benzo(a)pyrene	3.34	0.72	0.59	1.31
Perylene	1.19	0.43	<LOD	0.43
Indeno(123-cd)pyrene	4.90	1.47	0.68	2.15
Benzo(ghi)perylene	7.07	0.91	2.75	3.66
Dibenz(ah)anthracene	2.20	<LOD	<LOD	<LOD
Coronene	6.06	<LOD	1.64	1.64
BHT	<LOD	76.06	<LOD	76.06
EHMC	96.11	110	77.30	187

Table 99: Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 4 (JOSS 4)

Lab. Code:	PAH-17-053	PAH-17-062	PAH-17-063	Sum Cell 1 and 2
Sample name:	Filter 4	JOSS 4 - Cell 1	JOSS 4 - Cell 2	JOSS 4
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.00	90.00	90.00	90.00
Sampling period:	2017	2017	2017	2017
Analysis date:	11/7/2017	11/13/2017	11/13/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Phenanthrene	<LOD	463	<LOD	463
Anthracene	<LOD	26.55	<LOD	26.55
Fluoranthene	<LOD	16.81	<LOD	16.81
Pyrene	<LOD	116	<LOD	116.38
Benz(a)anthracene	<LOD	<LOD	<LOD	<LOD
Chrysene	<LOD	30.66	<LOD	30.66
Sum Benzo(b,j,k)fluoranthene	5.08	9.10	<LOD	9.10
Benzo(e)pyrene	1.98	1.78	<LOD	1.78
Benzo(a)pyrene	1.40	<LOD	<LOD	<LOD
Perylene	0.93	0.51	<LOD	0.51
Indeno(123-cd)pyrene	1.64	0.97	<LOD	0.97
Benzo(ghi)perylene	3.47	0.90	0.61	1.51
Dibenz(ah)anthracene	0.90	0.76	<LOD	0.76
Coronene	2.61	<LOD	<LOD	<LOD
BHT	<LOD	<LOD	<LOD	<LOD
EHMC	74.56	85.10	<LOD	85.10

Table 100: Results of Polycyclic Aromatic Hydrocarbons (PAHs), EHMC and BHT in Transect 5 (JOSS 5)

Lab. Code:	PAH-17-054	PAH-17-064	PAH-17-065	Sum Cell 1 and 2
Sample name:	Filter 5	JOSS 5 - Cell 1	JOSS 5 - Cell 2	JOSS 5
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	88.50	88.50	88.50	88.50
Sampling period:	2017	2017	2017	2017
Analysis date:	11/7/2017	11/14/2017	11/13/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
Phenanthrene	35.98	543	<LOD	543
Anthracene	<LOD	45.08	<LOD	45.08
Fluoranthene	19.08	52.50	<LOD	52.50
Pyrene	24.99	128	<LOD	128
Benz(a)anthracene	6.33	4.09	<LOD	4.09
Chrysene	13.92	42.90	<LOD	42.90
Sum Benzo(b,j,k)fluoranthene	18.86	10.67	<LOD	10.67
Benzo(e)pyrene	9.40	7.10	<LOD	7.10
Benzo(a)pyrene	8.54	1.42	<LOD	1.42
Perylene	119	57.22	4.09	61.30
Indeno(123-cd)pyrene	6.60	1.15	<LOD	1.15
Benzo(ghi)perylene	15.48	2.49	0.73	3.21
Dibenz(ah)anthracene	4.80	1.15	<LOD	1.15
Coronene	14.78	1.55	<LOD	1.55
BHT	<LOD	<LOD	<LOD	<LOD
EHMC	74.46	102	96.47	198

## 6.2.5. Polychlorinated Biphenyls

In the Tables 101 to 106 the results of EC-7 Polychlorinated Biphenyls obtained from the Large Volume Transect Sampling are reported.

*Table 101: EC-7 Polychlorinated Biphenyls in Field blank samples*

Lab. Code:	P-17-055	P-17-067	P-17-069
Sample name:	Filter BLK	Field Blank Cell 1	Field Blank Cell 2
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Analysis date:	10/24/2017	10/24/2017	10/24/2017
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	0.12	0.13	0.14
PCB 52	0.12	0.15	0.17
PCB 101	0.16	0.18	0.18
PCB 118	0.11	0.15	0.22
PCB 138	0.32	0.45	0.39
PCB 153	0.45	0.53	0.44
PCB 180	0.27	0.23	0.23
Sum EC-7 PCBs	1.56	1.83	1.78

*Table 102: EC-7 Polychlorinated Biphenyls in Transect 1 (JOSS 1)*

Lab. Code:	P-17-050	P-17-056	P-17-057	Sum Cell 1 and 2
Sample name:	Filter 1	JOSS 1 - Cell 1	JOSS 1 - Cell 2	JOSS 1
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	180.00	180.00	180.00	180.00
Sampling period:	2017	2017	2017	2017
Analysis date:	10/25/2017	10/25/2017	10/25/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
EC-7				
PCB 28	<LOD	1.51	<LOD	1.51
PCB 52	<LOD	0.80	<LOD	0.80
PCB 101	<LOD	0.67	<LOD	0.67
PCB 118	<LOD	0.51	<LOD	0.51
PCB 138	<LOD	0.62	<LOD	0.62
PCB 153	<LOD	0.88	<LOD	0.88
PCB 180	<LOD	0.34	<LOD	0.34
Sum EC-7 PCBs	---	5.34	---	5.34

Table 103: EC-7 Polychlorinated Biphenyls in Transect 2 (JOSS 2)

Lab. Code:	P-17-051	P-17-058	P-17-059	Sum Cell 1 and 2
Sample name:	Filter 2	JOSS 2 - Cell 1	JOSS 2 - Cell 2	JOSS 2
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.90	90.90	90.90	90.90
Sampling period:	2017	2017	2017	2017
Analysis date:	10/25/2017	10/25/2017	10/25/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
EC-7				
PCB 28	<LOD	0.83	<LOD	0.83
PCB 52	<LOD	0.46	<LOD	0.46
PCB 101	<LOD	0.38	<LOD	0.38
PCB 118	<LOD	0.25	<LOD	0.25
PCB 138	<LOD	<LOD	<LOD	<LOD
PCB 153	<LOD	<LOD	<LOD	<LOD
PCB 180	<LOD	<LOD	<LOD	<LOD
Sum EC-7 PCBs	---	1.93	---	1.93

Table 104: EC-7 Polychlorinated Biphenyls in Transect 3 (JOSS 3)

Lab. Code:	P-17-052	P-17-060	P-17-061	Sum Cell 1 and 2
Sample name:	Filter 3	JOSS 3 - Cell 1	JOSS 3 - Cell 2	JOSS 3
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	87.90	87.90	87.90	87.90
Sampling period:	2017	2017	2017	2017
Analysis date:	10/25/2017	10/25/2017	10/25/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
EC-7				
PCB 28	<LOD	1.17	0.16	1.33
PCB 52	<LOD	0.61	<LOD	0.61
PCB 101	<LOD	0.39	<LOD	0.39
PCB 118	<LOD	<LOD	<LOD	<LOD
PCB 138	<LOD	<LOD	<LOD	<LOD
PCB 153	<LOD	<LOD	<LOD	<LOD
PCB 180	<LOD	<LOD	<LOD	<LOD
Sum EC-7 PCBs	---	2.17	0.16	2.33

*Table 105: EC-7 Polychlorinated Biphenyls in Transect 4 (JOSS 4)*

Lab. Code:	P-17-053	P-17-062	P-17-063	Sum Cell 1 and 2
Sample name:	Filter 4	JOSS 4 - Cell 1	JOSS 4 - Cell 2	JOSS 4
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	90.00	90.00	90.00	90.00
Sampling period:	2017	2017	2017	2017
Analysis date:	10/25/2017	10/25/2017	10/25/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
EC-7				
PCB 28	< LOD	1.33	0.25	1.58
PCB 52	< LOD	1.02	0.25	1.27
PCB 101	< LOD	0.62	< LOD	0.62
PCB 118	< LOD	0.51	< LOD	< LOD
PCB 138	< LOD	0.61	< LOD	< LOD
PCB 153	< LOD	0.80	< LOD	< LOD
PCB 180	< LOD	0.35	< LOD	< LOD
Sum EC-7 PCBs	---	5.24	0.50	5.74

*Table 106: EC-7 Polychlorinated Biphenyls in Transect 5 (JOSS 5)*

Lab. Code:	P-17-054	P-17-064	P-17-065	Sum Cell 1 and 2
Sample name:	Filter 5	JOSS 5 - Cell 1	JOSS 5 - Cell 2	JOSS 5
Type of sample:	LV-TS Black Sea water			
Volume sampled (L):	88.50	88.50	88.50	88.50
Sampling period:	2017	2017	2017	2017
Analysis date:	10/25/2017	10/25/2017	10/25/2017	
Concentration	pg/L	pg/L	pg/L	pg/L
EC-7				
PCB 28	0.60	9.84	0.23	10.07
PCB 52	0.45	7.54	< LOD	7.54
PCB 101	0.71	4.74	0.25	4.98
PCB 118	0.83	4.39	< LOD	4.39
PCB 138	1.32	2.91	< LOD	2.91
PCB 153	1.43	3.02	0.72	3.74
PCB 180	0.48	0.61	< LOD	0.61
Sum EC-7 PCBs	5.81	33.05	1.20	34.25

### 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 107: Final concentration of Organophosphate Compounds in Transect 1 (JOSS 1)*

Transect:	JOSS 1		
Sample name:	Filter 1	Sum Cell 1 and 2	Sum Filter and Cells
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:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.62	0.62
TNPP	<LOD	<LOD	<LOD
TIBP	<LOD	2.63	2.63
TNBP	<LOD	0.42	0.42
TCEP	<LOD	0.24	0.24
TCPP	0.017	4.78	4.80
TDCPP	<LOD	0.40	0.40
TBOEP	<LOD	<LOD	<LOD
TPhP	<LOD	0.023	0.023
EHDP	<LOD	<LOD	<LOD
TEHP	0.002	0.001	0.003
TMPP	<LOD	0.018	0.018
TIPPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 108: Final concentration of Organophosphate Compounds in Transect 2 (JOSS 2)

Transect:	JOSS 2		
Sample name:	Filter 2	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.90	90.90	90.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	0.90	0.90
TNPP	<LOD	<LOD	<LOD
TI BP	<LOD	2.95	2.95
TNBP	<LOD	0.36	0.36
TCEP	<LOD	0.24	0.24
TCPP	0.044	4.63	4.67
TDCPP	<LOD	0.32	0.32
TBOEP	<LOD	<LOD	<LOD
TPhP	<LOD	0.012	0.012
EHDP	<LOD	0.035	0.035
TEHP	<LOD	0.003	0.003
TMPP	<LOD	0.008	0.008
TI PPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

Table 109: Final concentration of Organophosphate Compounds in Transect 3 (JOSS 3)

Transect:	JOSS 3		
Sample name:	Filter 3	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	87.90	87.90	87.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	< LOD	1.15	1.15
TNPP	< LOD	< LOD	< LOD
TIBP	< LOD	2.81	2.81
TNBP	< LOD	0.44	0.44
TCEP	< LOD	0.28	0.28
TCPP	0.023	4.16	4.18
TDCPP	0.008	0.34	0.35
TBOEP	< LOD	< LOD	< LOD
TPhP	0.004	0.011	0.016
EHDP	0.014	0.025	0.039
TEHP	0.002	0.003	0.005
TMPP	< LOD	0.035	0.035
TIPPP	< LOD	< LOD	< LOD
T35DMPP	< LOD	< LOD	< LOD

Table 110: Final concentration of Organophosphate Compounds in Transect 4 (JOSS 4)

Transect:	JOSS 4		
Sample name:	Filter 4	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	< LOD	0.84	0.84
TNPP	< LOD	< LOD	< LOD
TIBP	< LOD	2.75	2.75
TNBP	< LOD	0.49	0.49
TCEP	< LOD	0.21	0.21
TCPP	0.021	5.11	5.13
TDCPP	< LOD	0.47	0.47
TBOEP	< LOD	< LOD	< LOD
TPhP	< LOD	0.013	0.013
EHDP	< LOD	0.028	0.028
TEHP	0.002	0.003	0.005
TMPP	< LOD	0.006	0.006
TIPPP	< LOD	< LOD	< LOD
T35DMPP	< LOD	< LOD	< LOD

*Table 111: Final concentration of Organophosphate Compounds detected in Transect 5 (JOSS 5)*

Transect:	JOSS 5		
Sample name:	Filter 5	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	88.50	88.50	88.50
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
TEP	<LOD	1.23	1.23
TNPP	<LOD	<LOD	<LOD
TI BP	<LOD	4.31	4.31
TNBP	<LOD	0.65	0.65
TCEP	<LOD	0.18	0.18
TCPP	0.056	7.60	7.65
TDCPP	<LOD	0.89	0.89
TBOEP	<LOD	<LOD	<LOD
TPhP	<LOD	0.025	0.025
EHDP	0.006	0.035	0.041
TEHP	0.006	0.009	0.015
TMPP	0.0005	0.008	0.008
TI PPP	<LOD	<LOD	<LOD
T35DMPP	<LOD	<LOD	<LOD

### 6.3.2. Pesticides and Chlorinated Flame Retardants

Table 112: Final concentration of Pesticides and Chlorinated Flame Retardants in Transect 1 (JOSS 1)

Transect:	JOSS 1		
Sample name:	Filter 1	Sum Cell 1 and 2	Sum Filter and Cells
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:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	<LOD	1.47	1.47
HCB	<LOD	4.52	4.52
a-HCH	<LOD	127	127
b-HCH	<LOD	3858	3858
g-HCH	<LOD	53.22	53.22
d-HCH	<LOD	2.99	2.99
e-HCH	<LOD	2.35	2.35
Sum-HCHs	---	4043	4043
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	3.37	3.37
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	3.37	3.37
Aldrin	<LOD	<LOD	<LOD
Dieldrin	<LOD	7.95	7.95
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	7.95	7.95
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.12	0.12
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.12	0.12
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.52	1.52
Sum-Endosulfanes	---	1.52	1.52
Endosulfane-sulphate	<LOD	0.77	0.77
op-DDE	<LOD	<LOD	<LOD
pp-DDE	<LOD	3.84	3.84

Transect:	JOSS 1		
Sample name:	Filter 1	Sum Cell 1 and 2	Sum Filter and Cells
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:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
op-DDD	<LOD	4.78	4.78
pp-DDD	<LOD	12.39	12.39
op-DDT	<LOD	1.37	1.37
pp-DDT	<LOD	5.29	5.29
Sum-DDTtotal	---	27.67	27.67
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	<LOD	<LOD	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	0.70	0.70
Triallate	<LOD	1.19	1.19
Chlorpyriphos	<LOD	109	109
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Chloran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 113: Final concentration of Pesticides and Chlorinated Flame Retardants in Transect 2 (JOSS 2)

Transect:	JOSS 2		
Sample name:	Filter 2	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.90	90.90	90.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	<LOD	1.30	1.30
HCB	<LOD	4.29	4.29
a-HCH	<LOD	96.14	96.14
b-HCH	<LOD	4089	4089
g-HCH	<LOD	43.03	43.03
d-HCH	<LOD	1.71	1.71
e-HCH	<LOD	<LOD	<LOD
Sum-HCHs	---	4230	4230
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	4.09	4.09
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	4.09	4.09
Aldrin	<LOD	<LOD	<LOD
Dieldrin	<LOD	9.55	9.55
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	9.55	9.55
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.99	1.99
Sum-Endosulfanes	---	1.99	1.99
Endosulfane-sulphate	<LOD	0.96	0.96
op-DDE	<LOD	<LOD	
pp-DDE	<LOD	1.15	1.15
op-DDD	<LOD	2.10	2.10
pp-DDD	0.19	9.53	9.72
op-DDT	<LOD	0.59	0.59

Transect:	JOSS 2		
Sample name:	Filter 2	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.90	90.90	90.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
pp-DDT	<LOD	4.28	4.28
Sum-DDTtotal	0.19	17.65	17.85
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	<LOD	<LOD	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	<LOD	20.93	20.93
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 114: Final concentration of Pesticides and Chlorinated Flame Retardants in Transect 3 (JOSS 3)

Transect:	JOSS 3		
Sample name:	Filter 3	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	87.90	87.90	87.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	<LOD	1.65	1.65
HCB	<LOD	5.94	5.94
a-HCH	<LOD	153	153
b-HCH	<LOD	4753	4753
g-HCH	<LOD	61.82	61.82
d-HCH	<LOD	2.54	2.54
e-HCH	<LOD	2.37	2.37
Sum-HCHs	---	4973	4973
Heptachlor	<LOD	<LOD	
Heptachlor-exo-epoxide	<LOD	4.68	4.68
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	4.68	4.68
Aldrin	<LOD	<LOD	
Dieldrin	<LOD	10.27	10.27
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	10.27	10.27
trans-chlordanne	<LOD	<LOD	<LOD
cis-chlordanne	<LOD	<LOD	<LOD
Sum-Chlordanne	---	---	---
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	<LOD	<LOD
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	---	---
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.88	1.88
Sum-Endosulfanes	---	1.88	1.88
Endosulfane-sulphate	<LOD	0.68	0.68
op-DDE	<LOD	<LOD	<LOD
pp-DDE	<LOD	2.45	2.45
op-DDD	<LOD	2.90	2.90
pp-DDD	0.28	12.56	12.84
op-DDT	<LOD	1.02	1.02

Transect:	JOSS 3		
Sample name:	Filter 3	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	87.90	87.90	87.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
pp-DDT	<LOD	5.16	5.16
Sum-DDTtotal	0.28	24.09	24.37
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	<LOD	18.63	18.63
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	<LOD	<LOD
Chlorpyriphos	1.21	64.49	65.70
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 115: Final concentration of Pesticides and Chlorinated Flame Retardants in Transect 4 (JOSS 4)

Transect:	JOSS 4		
Sample name:	Filter 4	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	<LOD	5.17	5.17
HCB	<LOD	6.11	6.11
a-HCH	<LOD	118	118
b-HCH	<LOD	3444	3444
g-HCH	<LOD	55.48	55.48
d-HCH	<LOD	3.47	3.47
e-HCH	<LOD	2.90	2.90
Sum-HCHs	---	3625	3625
Heptachlor	<LOD	<LOD	<LOD
Heptachlor-exo-epoxide	<LOD	3.46	3.46
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	3.46	3.46
Aldrin	<LOD	<LOD	<LOD
Dieldrin	<LOD	7.46	7.46
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	7.46	7.46
trans-chlordanne	<LOD	0.10	0.10
cis-chlordanne	<LOD	0.20	0.20
Sum-Chlordanne	---	0.30	0.30
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.11	0.11
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.11	0.11
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.85	1.85
Sum-Endosulfanes	---	1.85	1.85
Endosulfane-sulphate	<LOD	0.82	0.82
op-DDE	<LOD	<LOD	<LOD
pp-DDE	0.55	3.98	4.53
op-DDD	0.45	6.35	6.80
pp-DDD	1.12	25.41	26.52
op-DDT	<LOD	0.88	0.88

Transect:	JOSS 4		
Sample name:	Filter 4	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
pp-DDT	0.24	4.05	4.29
Sum-DDTtotal	2.36	40.66	43.03
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	<LOD	<LOD	<LOD
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	<LOD	<LOD
Triallate	<LOD	1.27	1.27
Chlorpyriphos	<LOD	108	108
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

Table 116: Final concentration of Pesticides and Chlorinated Flame Retardants in Transect 5 (JOSS 5)

Transect:	JOSS 5		
Sample name:	Filter 5	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	88.50	88.50	88.50
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
<i>Chlorinated Pesticides</i>			
PeCBz	<LOD	7.25	7.25
HCB	<LOD	6.46	6.46
a-HCH	<LOD	136	136
b-HCH	<LOD	3060	3060
g-HCH	<LOD	65.47	65.47
d-HCH	<LOD	7.59	7.59
e-HCH	<LOD	6.29	6.29
Sum-HCHs	---	3276	3276
Heptachlor	<LOD	<LOD	
Heptachlor-exo-epoxide	<LOD	4.16	4.16
Heptachlor-endo-epoxide	<LOD	<LOD	<LOD
Sum-Hetachlorepoxydes	---	4.16	4.16
Aldrin	<LOD	<LOD	
Dieldrin	<LOD	10.79	10.79
Endrin	<LOD	<LOD	<LOD
Isodrin	<LOD	<LOD	<LOD
Sum-Drins	---	10.79	10.79
trans-chlordanne	<LOD	0.24	0.24
cis-chlordanne	<LOD	0.23	0.23
Sum-Chlordanne	---	0.47	0.47
Oxychlordanne	<LOD	<LOD	<LOD
trans-nonachlor	<LOD	0.12	0.12
cis-nonachlor	<LOD	<LOD	<LOD
Sum-nonachlor	---	0.12	0.12
Endosulfane-alpha	<LOD	<LOD	<LOD
Endosulfane-beta	<LOD	1.81	1.81
Sum-Endosulfanes	---	1.81	1.81
Endosulfane-sulphate	<LOD	2.12	2.12
op-DDE	<LOD	1.16	1.16
pp-DDE	2.76	15.55	18.31
op-DDD	1.56	34.67	36.23
pp-DDD	5.83	137	142.95
op-DDT	0.44	1.48	1.92

Transect:	JOSS 5		
Sample name:	Filter 5	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	88.50	88.50	88.50
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
pp-DDT	1.87	4.79	6.65
Sum-DDTtotal	12.46	195	207.23
Methoxychlor	<LOD	<LOD	<LOD
Mirex	<LOD	<LOD	<LOD
<i>Other pesticides:</i>			
HCBD	<LOD	13.35	13.35
Dichlorvos	<LOD	<LOD	<LOD
Trifluralin	<LOD	0.28	0.28
Triallate	<LOD	4.27	4.27
Chlorpyriphos	1.20	76.31	77.52
Chlorfenvinphos	<LOD	<LOD	<LOD
Dicofol	<LOD	<LOD	<LOD
Cypermethrins	<LOD	<LOD	<LOD
<i>Chlorinated Flame Retardants:</i>			
Choran 542	<LOD	<LOD	<LOD
syn-Decchlorane Plus	<LOD	<LOD	<LOD
anti-Decchlorane Plus	<LOD	<LOD	<LOD

### 6.3.3. Triazines

Table 117: Final concentration of Triazines in Transect 1 (JOSS 1)

Transect:	JOSS 1		
Sample name:	Filter 1	Sum Cell 1 and 2	Sum Filter and Cells
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:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	3.70	3.70
Atrazine	0.009	29.39	29.40
Terbutylazine	<LOD	1.52	1.52
Sum triazines	0.009	34.61	34.62

Table 118: Final concentration of Triazines detected in Transect 2 (JOSS 2)

Transect:	JOSS 2		
Sample name:	Filter 2	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.90	90.90	90.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	4.39	4.39
Atrazine	0.022	35.10	35.12
Terbutylazine	<LOD	1.28	1.28
Sum triazines	0.022	40.78	40.80

Table 119: Final concentration of Triazines in Transect 3 (JOSS 3)

Transect:	JOSS 3		
Sample name:	Filter 3	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	87.90	87.90	87.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	5.00	5.00
Atrazine	0.034	37.85	37.88
Terbutylazine	<LOD	1.37	1.37
Sum triazines	0.034	44.22	44.26

Table 120: Final concentration of Triazines in Transect 4 (JOSS 4)

Transect:	JOSS 4		
Sample name:	Filter 4	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	3.46	3.46
Atrazine	0.019	28.16	28.18
Terbutylazine	<LOD	1.71	1.71
Sum triazines	0.019	33.33	33.35

Table 121: Final concentration of Triazine in Transect 5 (JOSS 5)

Transect:	JOSS 5		
Sample name:	Filter 5	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	88.50	88.50	88.50
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	ng/L	ng/L	ng/L
Simazine	<LOD	2.97	2.97
Atrazine	0.021	25.10	25.12
Terbutylazine	<LOD	2.07	2.07
Sum triazines	0.021	30.14	30.16

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

Table 122: Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 1 (JOSS 1)

Transect:	JOSS 1		
Sample name:	Filter 1	Sum Cell 1 and 2	Sum Filter and Cells
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:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Phenanthrene	<LOD	543	543
Anthracene	<LOD	33.72	33.72
Fluoranthene	9.89	29.16	39.05
Pyrene	<LOD	133	133
Benz(a)anthracene	2.39	4.23	6.62
Chrysene	6.61	49.43	56.04
Sum Benzo(b,j,k)fluoranthene	9.91	17.16	27.07
Benzo(e)pyrene	4.36	6.74	11.10
Benzo(a)pyrene	4.72	1.50	6.22
Perylene	2.30	1.03	3.33
Indeno(123-cd)pyrene	4.27	1.68	5.95
Benzo(ghi)perylene	7.69	1.94	9.63
Dibenz(ah)anthracene	2.49	1.05	3.54
Coronene	6.16	0.72	6.89
BHT	<LOD	<LOD	<LOD
EHMC	<LOD	<LOD	<LOD

Table 123: Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 2 (JOSS 2)

Transect:	JOSS 2		
Sample name:	Filter 2	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.90	90.90	90.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Phenanthrene	<LOD	361	361
Anthracene	<LOD	29.37	29.37
Fluoranthene	10.29	30.66	40.95
Pyrene	<LOD	120	120
Benz(a)anthracene	<LOD	2.43	2.43
Chrysene	<LOD	43.09	43.09
Sum Benzo(b,j,k)fluoranthene	5.92	17.04	22.96
Benzo(e)pyrene	1.86	4.92	6.78
Benzo(a)pyrene	1.35	0.77	2.12
Perylene	0.75	0.77	1.52
Indeno(123-cd)pyrene	2.17	3.60	5.77
Benzo(ghi)perylene	3.46	1.86	5.32
Dibenz(ah)anthracene	0.89	1.26	2.15
Coronene	3.77	<LOD	3.77
BHT	<LOD	<LOD	<LOD
EHMC	77.17	93.25	170

Table 124: Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 3 (JOSS 3)

Transect:	JOSS 3		
Sample name:	Filter 3	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	87.90	87.90	87.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Phenanthrene	44.27	381	426
Anthracene	13.87	30.16	44.03
Fluoranthene	12.79	67.86	80.65
Pyrene	<LOD	127.36	127
Benz(a)anthracene	2.28	<LOD	2.28
Chrysene	8.38	35.13	43.50
Sum Benzo(b,j,k)fluoranthene	11.53	15.75	27.28
Benzo(e)pyrene	5.22	4.23	9.45
Benzo(a)pyrene	3.34	1.31	4.65
Perylene	1.19	0.43	1.62
Indeno(123-cd)pyrene	4.90	2.15	7.06
Benzo(ghi)perylene	7.07	3.66	10.73
Dibenz(ah)anthracene	2.20	<LOD	2.20
Coronene	6.06	1.64	7.70
BHT	<LOD	76.06	76
EHMC	96.11	187	283

Table 125: Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 4 (JOSS 4)

Transect:	JOSS 4		
Sample name:	Filter 4	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Phenanthrene	< LOD	463	463
Anthracene	< LOD	26.55	26.55
Fluoranthene	< LOD	16.81	16.81
Pyrene	< LOD	116.38	116
Benz(a)anthracene	< LOD	< LOD	< LOD
Chrysene	< LOD	30.66	30.66
Sum Benzo(b,j,k)fluoranthene	5.08	9.10	14.18
Benzo(e)pyrene	1.98	1.78	3.77
Benzo(a)pyrene	1.40	< LOD	1.40
Perylene	0.93	0.51	1.44
Indeno(123-cd)pyrene	1.64	0.97	2.61
Benzo(ghi)perylene	3.47	1.51	4.97
Dibenz(ah)anthracene	0.90	0.76	1.66
Coronene	2.61	< LOD	2.61
BHT	< LOD	< LOD	< LOD
EHMC	74.56	85.10	160

Table 126: Final concentration of Polycyclic Aromatics Hydrocarbons (PAHs), EHMC and BHT in Transect 5 (JOSS 5)

Transect:	JOSS 5		
Sample name:	Filter 5	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	88.50	88.50	88.50
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
Phenanthrene	35.98	543	579
Anthracene	< LOD	45.08	45.08
Fluoranthene	19.08	52.50	71.58
Pyrene	24.99	128	153
Benz(a)anthracene	6.33	4.09	10.43
Chrysene	13.92	42.90	56.82
Sum Benzo(b,j,k)fluoranthene	18.86	10.67	29.52
Benzo(e)pyrene	9.40	7.10	16.50
Benzo(a)pyrene	8.54	1.42	9.96
Perylene	119	61.30	180
Indeno(123-cd)pyrene	6.60	1.15	7.76
Benzo(ghi)perylene	15.48	3.21	18.69
Dibenz(ah)anthracene	4.80	1.15	5.95
Coronene	14.78	1.55	16.33
BHT	< LOD	< LOD	< LOD
EHMC	74.46	198	273

### 6.3.5. Polychlorinated Biphenyls (EC-7 PCBs)

Table 127: Final concentration of EC-7 Polychlorinated Biphenyls in Transect 1 (JOSS 1)

Transect:	JOSS 1		
Sample name:	Filter 1	Sum Cell 1 and 2	Sum Filter and Cells
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:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	< LOD	1.51	1.51
PCB 52	< LOD	0.80	0.80
PCB 101	< LOD	0.67	0.67
PCB 118	< LOD	0.51	0.51
PCB 138	< LOD	0.62	0.62
PCB 153	< LOD	0.88	0.88
PCB 180	< LOD	0.34	0.34
Sum EC-7 PCBs	---	5.34	5.34

Table 128: Final concentration of EC-7 Polychlorinated Biphenyls in Transect 2 (JOSS 2)

Transect:	JOSS 2		
Sample name:	Filter 2	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.90	90.90	90.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	< LOD	0.83	0.83
PCB 52	< LOD	0.46	0.46
PCB 101	< LOD	0.38	0.38
PCB 118	< LOD	0.25	0.25
PCB 138	< LOD	< LOD	< LOD
PCB 153	< LOD	< LOD	< LOD
PCB 180	< LOD	< LOD	< LOD
Sum EC-7 PCBs	---	1.93	1.93

Table 129: Final concentration of EC-7 Polychlorinated Biphenyls in Transect 3 (JOSS 3)

Transect:	JOSS 3		
Sample name:	Filter 3	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	87.90	87.90	87.90
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	< LOD	1.33	1.33
PCB 52	< LOD	0.61	0.61
PCB 101	< LOD	0.39	0.39
PCB 118	< LOD	< LOD	< LOD
PCB 138	< LOD	< LOD	< LOD
PCB 153	< LOD	< LOD	< LOD
PCB 180	< LOD	< LOD	< LOD
Sum EC-7 PCBs	---	2.33	2.33

Table 130: Final concentration of EC-7 Polychlorinated Biphenyls in Transect 4 (JOSS 4)

Transect:	JOSS 4		
Sample name:	Filter 4	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	90.00	90.00	90.00
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	< LOD	1.58	1.58
PCB 52	< LOD	1.27	1.27
PCB 101	< LOD	0.62	0.62
PCB 118	< LOD	< LOD	< LOD
PCB 138	< LOD	< LOD	< LOD
PCB 153	< LOD	< LOD	< LOD
PCB 180	< LOD	< LOD	< LOD
Sum EC-7 PCBs	---	5.74	5.74

Table 131: Final concentration of EC-7 Polychlorinated Biphenyls in Transect 5 (JOSS 5)

Transect:	JOSS 5		
Sample name:	Filter 5	Sum Cell 1 and 2	Sum Filter and Cells
Type of sample:	LV-TS Black Sea water	LV-TS Black Sea water	LV-TS Black Sea water
Volume sampled (L):	88.50	88.50	88.50
Sampling period:	2017	2017	2017
Matrix/Phase:	Suspended Particulate Matter	Dissolved	Whole Water
Concentration	pg/L	pg/L	pg/L
EC-7			
PCB 28	0.60	10.07	10.66
PCB 52	0.45	7.54	7.99
PCB 101	0.71	4.98	5.69
PCB 118	0.83	4.39	5.22
PCB 138	1.32	2.91	4.23
PCB 153	1.43	3.74	5.17
PCB 180	0.48	0.61	1.09
Sum EC-7 PCBs	5.81	34.25	40.06

## **7. Conclusions**

Two different sampling methodologies were successfully applied during EMBLAS II cruise for a total of 49 samples collected, 104 substances analysed (35 enclosed in the priority list of WFD) and about 4266 final individual results.

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:

The coastal samples from Ukraine and Georgia collected in the HDPE containers showed very low recoveries, so the methodological LODs and LOQs obtained for the 20L spot samples must be considered to be at least 5 times higher and their positive findings should be considered as indicative only.

- While some absolute recoveries of standards in samples collected by stainless steel tanks have been 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).
- Very good sampling efficiency for all detectable compounds was obtained with extra large sampling device even sampling up to 600L (Large Volume Transect Sampling).
- Large Volume Transect Sampling allows to reach the most stringent and challenging EOS for Heptachlor with LOD 0.1 pg/L (EQS: 0.2 pg/L).
- The Large Volume Transect sampling gets close to the required LOD for Heptachlor-epoxide but remains slightly to high (LOD 0.29 pg/L vs. EQS: 0.2 pg/L). It allows to reach the required performance if the sampling volume is sufficient high. Splitting of the samples for different increases LODs.
- High concentrations of Triazines, in particular Simazine and 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, Acenaphthene and Fluorene) have been analysed but not reported, as the methodology resulted to be not suitable for them.
- Low molecular weight of HCBD and his high volatility makes the methodology not suitable for HCBD analysis. The concentration of HCBD must be considered as indicative only.
- For the interpretation of elevated concentrations of EHMC, a sunscreen agent, 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.

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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	PCBs	Polychlorinated Biphenyls
CAD	Collision Gas	PPG	Polypropylene glycol
CUR	Curtain Gas	PS	Priority substances
CRM	Certified reference material	QC	Quality control sample
CXP	Collision Cell Exit Potential	R <sup>2</sup>	Coefficient of determination
DG	Directorate-General	RT	Retention time
E1	Estrone	SD	Standard deviation
E2	17 $\beta$ -estradiol	S/N	Signal to Noise
EE2	17 $\alpha$ -ethinyl estradiol	SPE	Solid-phase extraction
EC	European Commission	TEM	Temperature
EHMC	2-Ethylhexyl-methoxycinnamate	UHPLC	Ultra-high-pressure liquid chromatography
EI	Electron Impact	WFD	Water Framework Directive
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		
IES	Institute for Environment and Sustainability		
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		

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## JRC Mission

As the science and knowledge service of the European Commission, the Joint Research Centre's mission is to support EU policies with independent evidence throughout the whole policy cycle.



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