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PROJECT

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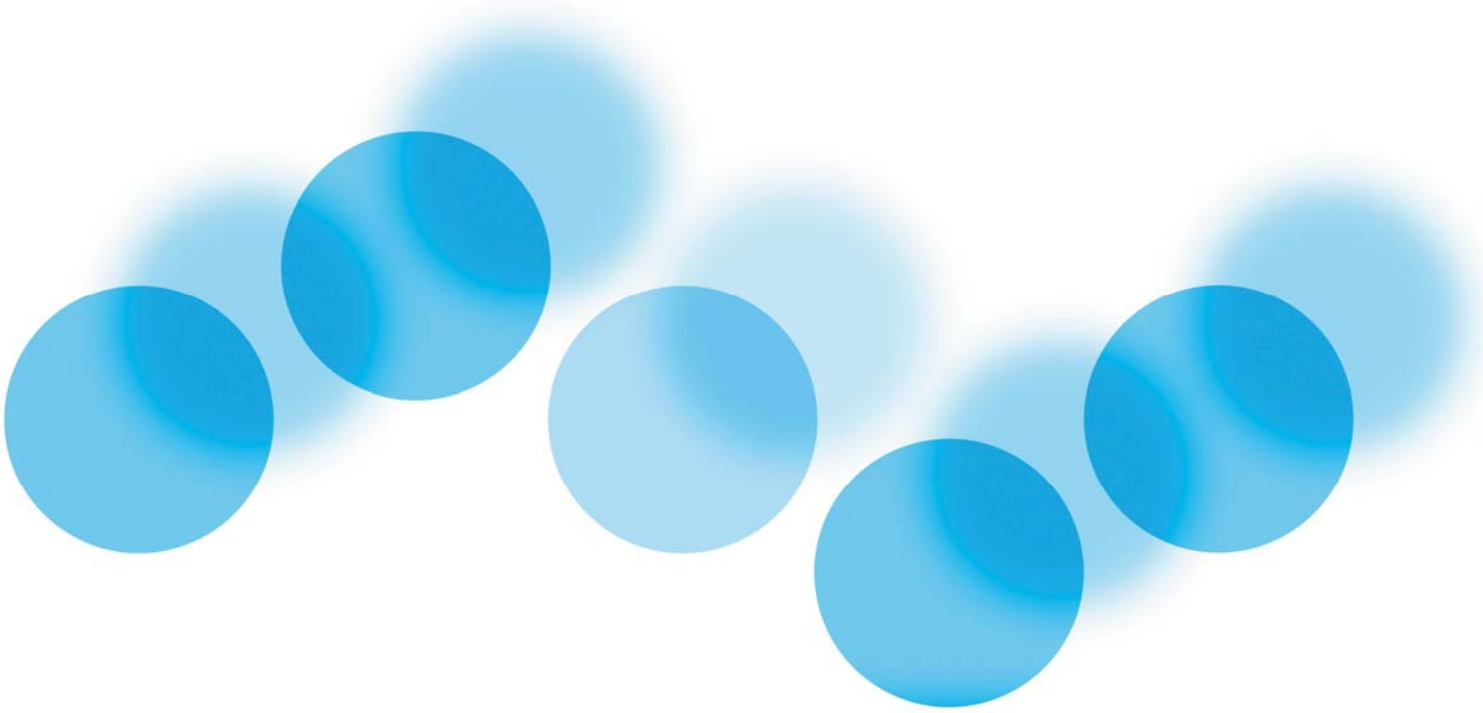
IRON GATES SEDIMENT EVALUATION - ROMANIA

Final Report



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ABBREVIATIONS

DRB	Danube River Basin
DRP	Danube Regional Project
EG	Expert Group
EU	European Union
EU WFD	EU Water Framework Directive
GEF	Global Environment Facility
ICPDR	International Commission for the Protection of the Danube River
UNDP	United Nations Development Programme
WB	World Bank

1. INTRODUCTION

This report presents the final report of the work performed by the Romania team from National Research and Development Institute for Environmental Protection - ICIM Bucharest, as part of the consortium designated for accomplishment of "Iron Gate Sediments Evaluation" project, within the UNDP/GEF Danube Regional Project - the group of small contracts for services.

2. OBJECTIVES

According to TORs, the main general objective of the project is to assess the sediment quality in the Iron Gates Reservoir and based on those results to prepare initial recommendations for future protection of the Danube River and Black Sea.

2.1. Specific objectives

Out of the specific objectives outlined in the TORs, the following ones have been dealt with in the previous stage of the project:

2.1.1. Collecting and reviewing the existing data and information on present situation

As it was specified in the Inception Report and further in the Technical Report, the national team collected existing and available data on sediment quality from the sites located in the Iron Gate Reservoir area. The sources of sediment quality data (as well as essential issues concerning the Quality Control/Quality Assurance, where available) are below briefly presented.

2.1.1.1. Description of existing data sources

EROS 2000 PROJECT – for data from year 1995

This study was the output of a research contract between the European Community (PHARE Programme) and the Romanian Centre of Marine Geology and Geo-Ecology regarding the eutrophication and contamination state of the Danube River.

During the EROS Danube River Cruise (June 1995) bottom sediments were collected by grabs and corers and the samples were analysed for grain size distribution, chemistry (major compounds, heavy metals) and mineralogy (clay and heavy minerals).

The samples from the core were collected for measuring the variations in heavy metals and oxides contents.

The Technical Report on Iron Gates contains also some information on the grain size distribution analyses.

ECOTOXICOLOGICAL STUDY CONCERNING THE DANUBE RIVER POLLUTION RESULTING FROM THE EVENTS OF YUGOSLAVIA - for data from year 1999

This study consisted of a chemical and biological survey along the border between Romania and FRY (as it was in that time) in order to assess the impact produced by relevant persistent pollutants potentially released during the conflict in Yugoslavia, on the Danube River's ecosystems.

As far as concerns sediment quality investigation, samples were taken in July 1999, from 9 sections along the Danube (Romanian and Yugoslavian border) between rkm 1071 - 834; sampling sites from each section were at various distances from left bank and on the main stream.

TRANS-NATIONAL MONITORING NETWORK - NATIONAL DATA BASE for data from 2000

As it was agreed within the former MLIM – Expert Group of ICPDR, for this monitoring programme, sediment samples are taken and analysed according to Standard Operating Procedures (SOPs) applied within the TNMN. These SOPs are applied as well in all TNMN National and Regional Reference Laboratories from the Danubian countries.

JOINT DANUBE SURVEY (JDS) – for data from 2001

Sediment samples were taken from the left and right banks of the river either with a sampling net or with the grab sampler in 98 stations within the whole catchment area out of which 13 stations were in the Iron Gate reservoir. Sediment sampling was followed by on-board grain size fractionation with wet sieving for obtaining the less-than 63 µm fraction for laboratory analysis.

Determination of heavy metals

For heavy metals analysis sediment samples were pretreated according to ISO/DIS 11464:1992 – "*Soil Quality. Pre-treatment of samples for physico - chemical analyses*".

Determination of aluminium, cadmium, chromium, copper, iron, lead, nickel, manganese and zinc in the extracts was carried out by ICP-AES (Perkin Elmer OPTIMA 2000 DV) according to ISO 11885. Arsenic and mercury were determined by flow injection-graphite furnace-AAS using the hydride/cold vapour principle (Perkin Elmer 4100 ZL with FIAS 200).

Determination of organic pollutants

Petroleum hydrocarbons were analysed during JDS by using different analytical methods, including GC-FID for total petroleum hydrocarbons (TPH), TPH was determined by using UV absorption and fluorescence procedures, as well as GC/MS analysis of Polycyclic Aromatic Hydrocarbons (PAHs).

The JDS list of determinants included the following:

- five organochlorine compounds (Lindane, Hexachlorobenzene, Hexachlorobutadiene, Pentachlorobenzene, pp'-DDT);
- seven polychlorinated biphenyls (PCB 25, 52, 101, 118, 153, 138, 180).
- Polycyclic Aromatic Hydrocarbons (PAHs)
- Nonylphenol (4-para-nonylphenol) and Octylphenol (para-tert-octylphenol)
- Organotin compounds (tributyltin cation – TBT).

AQUATERRA INTEGRATED PROJECT – for data from 2004

The *Aquaterra Danube Survey* (August – September 2004) provided additional results and data concerning the concentration of selected chemical compounds in sediments and suspended matters.

Sampling sites were selected out from the sites investigated during the Joint Danube Survey and were selected a number of 30 stations on the Danube River.

Sediment samples were taken from both left and right banks of the river, using a sampling net and a grab sampler. Sediment sampling was followed by on-board grain size fractionation with wet sieving for obtaining the less-than 63 µm fraction for laboratory analysis of selected determinants.

Sediment samples were sampled and handled according to the appropriate Standard Operations Procedures and Standards Methods, as it follows:

- ISO 5667-3:1985: Preservation and handling of samples;
- ISO 5667-6:1990: Sampling of rivers and streams;

- ISO 5667-12:1990: Sampling of bottom sediments;
- ISO 5667-15:1991: Preservation and handling of sludge and sediment samples;
- ISO 5667-17:1991: Guidance on sampling of suspended matters.

With each series of samples were analysed certified reference materials. In total, 5 certified reference materials (1640 Trace Elements in Natural Water (NIST, USA); TMDA – 54.3 a fortified calibration standard for trace elements (NWRI, Canada); Sewage sludge BCR 144 R (IRMM, Belgium); Soil LGC 6138 (GB); Sediment GBW 067306 – China) were used in order to assure quality control of measurements.

Following appropriate dilution to fit the calibration range, the elements were determined in digests of suspended matters, bottom sediments and certified reference materials as follows:

- Cd, Cu, Fe, Mn, Ni, Pb and Zn by flame atomic absorption spectroscopy according to ISO 8288/1986 (Cd, Cu, Ni, Pb and Zn) and Cr according to EPA/600/4-79/020, 1983;
- As by hydride generation technique of atomic absorption spectroscopy ;
- Hg by cold vapour technique of atomic fluorescence spectroscopy according to EN 13506/2001;
- Al by inductively coupled plasma mass spectroscopy according to ISO 17294-2/2003.

2.1.1.2. Considerations on existing data and information

- The sampling sites and river sections for which data were available in above mentioned sources are listed in **Table 1**;
- The geographical coordinates (where possible) and the time period for the available data are specified in **Table 2**;
- The groups of determinands as well as determinands from each group for which data were available are presented in **Table 3**;
- The approximate number of data (as number of entries) collected from the above mentioned sources (according to Table 3) is (around) 1800.

Table 1: Sampling sites in the studied area for which data were available

River kilometer on the Danube River	Name of sampling site
1071.0	Bazias/Banatska Palanka
1059.0	Veliko Gradiste
1044.5	Moldova Veche
1040.0	Iron Gates Reservoir Golubac/Koronin
999.0	Greben
996.0	Milanovac
969,5	Plavisevita
959.5	Ieselnita
956.0	Iron Gates Reservoir Tekija/Orsova
952.0	Ada Kale
947.2	Upstream Iron Gates I Dam
943.0	Iron Gates I Dam
934.0	Kladovo
924.0	Vrbica/Simijan
867.0	Upstream Iron Gate II
849.0	Upstream Timok Gruia/Radujevac
834.0	Pristol/NovoSelo

Table 2: List of sampling locations in the available data sets

No	Sampling location	River km	Location in profile	Longitude			Latitude			Year of available data set
				°	′	″	°	′	″	
1	Bazias	1071	L, R	21	23	31.0	44	48	15.2	1999, 2000, 2001, 2002, 2004
2	Veliko Gradiste	1059	L	n.a.[1]	n.a.	n.a.	n.a.	n.a.	n.a.	1999
3	Moldova Veche	1044.5	L							1995
4	Iron Gates Reservoir Golubac/Koronin	1040	L, R	21	40	45.7	44	40	37.5	2001, 2004
5	Greben	999	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1995
6	Milanovac	996	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1999
7	Plavisevita	969.5	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1995
8	Ieselnita	959.5	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1995
9	Iron Gates Reservoir Orsova/Tekija	956	L, R	22	24'	31.5	44	42	08.2	1999, 2001, 2004
10	Ada Kale	952	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1995
11	Upstream Iron Gates I Dam	947.2	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1995
12	Iron Gates I Dam	943	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1999
13	Kladovo	934	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1999
14	Simijan/Vrbica	924	L, R	22	42	55.9	44	36	10.8	2001, 2004
15	Upstream Iron Gate II	867	L	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1999
16	Upstream Timok Gruia/Radujevac	849	L, R	22	41	24.0	44	14	55.7	1999, 2001, 2004
17	Pristol/NovoSelo	834	L, R	22	48	25.0	44	09	44.0	1999, 2000, 2001, 2002, 2004

[1] not available

Table 3: Determinands for which data were available for the river stretch rkm 1071 – rkm 834 and number of entries for each determinand

		Danube River km (rkm)																
Group of determinands	**Determinand**	**1071**	**1059**	**1045**	**1040**	**999**	**996**	**969.5**	**959.5**	**956**	**952**	**947.2**	**943**	**934**	**924**	**867**	**849**	**834**
WFD Priority Substances (PS)	Anthracene	5	1		4		1			4			1	1	4	1	5	4
	Brominated diphenylethers (Pentabromodiphenylether)	2			2					1					2		2	2
	Cadmium and its compounds	13	2	1	5	1	1	1	1	3	1	1	1	1	4	2	6	9
	Di (2-ethylhexyl)phthalate (DEHP)	4			4					3					4		4	3
	Fluoranthene	5	1		4		1			4			1	1	4	1	5	4
	Hexachlorobenzene	4			4					3					4		4	3
	Hexachlorobutadiene	4			4					3					4		4	3
	Lindane (gamma-hexachlorocyclohexane)	5	1		4		1			4			1	1	4	1	5	4
	Lead and its compounds	13	2	1	5	1	1	1	1	4	1	1	1	1	4	2	6	9
	Mercury and its compounds	4		1	5	1		1	1	3	1	1			4	1	4	3
	Naphthalene	5	1		4		1			4			1	1	4	1	5	4
	Nikel and its compounds	9		1	5	1		1	1	3	1	1			4	1	4	6
	4-(para)-Nonylphenol	4			4					3					4		4	3
	(para-terc-Octylphenol)	4			4					3					4		4	3
	Pentachlorobenzene	4			4					3					4		4	3
	Pentachlorophenol	4			4					4					4		4	3
Other PAHs	Benzo(a)pyrene	5	1		4		1			4			1	1	4	1	5	4
	Benzo(b)fluoranthene	5	1		4		1			4			1	1	4	1	5	4
	Benzo(g,h,i)perylene	5	1		4		1			4			1	1	4	1	5	4
	Benzo(k)fluoranthene	5	1		4		1			4			1	1	4	1	5	4
	Indeno(1,2,3-c,d)pyrene	5	1		4		1			4			1	1	4	1	5	4
	Crysene	5	1		4		1			4			1	1	4	1	5	4
	Dibenzo(a,h)anthracene	5	1		4		1			4			1	1	4	1	5	4

		Danube River km (rkm)																
Group of determinands	**Determinand**	**1071**	**1059**	**1045**	**1040**	**999**	**996**	**969.5**	**959.5**	**956**	**952**	**947.2**	**943**	**934**	**924**	**867**	**849**	**834**
	Acenaphthylene	5	1		4		1			4			1	1	4	1	5	4
	Acenaphthene	5	1		4		1			4			1	1	4	1	5	4
	Fluorene	5	1		4		1			4			1	1	4	1	5	4
	Pyrene	5	1		4		1			4			1	1	4	1	5	4
	Benz(a)anthracene	5	1		4		1			4			1	1	4	1	5	4
	Phenanthrene	5	1		4		1			4			1	1	4	1	5	4
Organochlorine pesticides	α-HCH	1	1				1			1			1	1		1	1	1
	β-HCH	1	1				1			1			1	1		1	1	1
	δ-HCH	1	1				1			1			1	1		1	1	1
	Aldrine	1	1				1			1			1	1		1	1	1
	Endrine	1	1				1			1			1	1		1	1	1
	Dieldrin	1	1				1			1			1	1		1	1	1
	Endrin aldehyde	1	1				1			1			1	1		1	1	1
	Endosulphan I	1	1				1			1			1	1		1	1	1
	Endosulphan II	1	1				1			1			1	1		1	1	1
	Heptachlor	1	1				1			1			1	1		1	1	1
	Heptachlor epoxid	1	1				1			1			1	1		1	1	1
	Endosulphan Sulphate	1	1				1			1			1	1		1	1	1
	4,4' DDE	1	1				1			1			1	1		1	1	1
	4,4' DDD	1	1				1			1			1	1		1	1	1
	4,4' DDT	5	1		4		1			4			1	1	4	1	5	4
Other WFD PS	Bisphenol	2			2					1					1		1	1
	Tributyltin Compounds	2			2					1					2		2	2
PCBs	PCB 18 (2,2',5 trichlorbiphenyl)	1	1				1			1			1	1		1	1	1
	PCB 31 (2,4',5-trichlorbiphenyl)	3	1		4		1			3			1	1	4	1	3	2

		Danube River km (rkm)																
Group of determinands	**Determinand**	**1071**	**1059**	**1045**	**1040**	**999**	**996**	**969.5**	**959.5**	**956**	**952**	**947.2**	**943**	**934**	**924**	**867**	**849**	**834**
	PCB 28 (2,4,4'-trichlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 44 (2,2',3,5' tetrachlorbiphenyl)	4	1				1			1			1	1		1	1	1
	PCB 52 (2,2',5,5'-tetrachlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 101 (2,2',4,5,5'-pentachlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 137 (2,2',3,4,4',5' hexachlorbiphenyl)	1	1				1			1			1	1		1	1	1
	PCB 118 (2,3',4,4',5 pentachlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 153 (2,2',4,4',5,5' hexachlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 138 (2,2',3,4,4',5 hexachlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 170 (2,2',3,3',4,4',5 heptachlorbiphenyl)	3	1		4		1			3			1	1	1	1	3	2
	PCB 180 (2,2',3,4,4',5,5' heptachlorbiphenyl)	5	1		4		1			4			1	1	4	1	5	4
	PCB 194 (2,2',3,3',4,4',5,5' octachlorbiphenyl)	1	1				1			1			1	1		1	1	1
	PCB 209 (decachlorbiphenyl)	1	1				1			1			1	1		1	1	1
	PCB 77 (3,3',4,4' tetrachlorbiphenyl)	2			4					2					2		2	2
	PCB 105 (2,3,3',4,4' pentachlorbiphenyl)	1			4					2					2		2	2
	PCB 126 (3,3',4,4',5 pentachlorbiphenyl)	2			4					2					2		2	2

		Danube River km (rkm)																
Group of determinands	**Determinand**	**1071**	**1059**	**1045**	**1040**	**999**	**996**	**969.5**	**959.5**	**956**	**952**	**947.2**	**943**	**934**	**924**	**867**	**849**	**834**
	PCB 128 (2,2',3,3',4,4' hexachlorbiphenyl)	2			4					2					2		2	2
	PCB 156 (2,3,3',4,4',5 hexachlorbiphenyl)	2			4					2					2		2	2
	PCB 169 (3,3',4,4',5,5' hexachlorbiphenyl)	2			4					2					2		2	2
Other Heavy Metals	Cu	13	2	1	5	1	1	1	1	4	1	1	1	1	4	2	6	9
	Cr	13	2	1	5	1	1	1	1	4	1	1	1	1	4	2	6	9
	Zn	13	2	1	5	1	1	1	1	4	1	1	1	1	4	2	6	9
	Fe	6			4					3					4		4	4
	Mn	10		1	5	1		1	1	3	1	1			4	1	4	7
	Al	4			4					3					4		4	4
	Vn			1		1		1	1		1	1				1		
	As	5		1	5	1		1	1	3	1	1			4	1	4	3
Aggregate variables	TEM	4			4					3					4		4	3
	Petroleum Hydrocarbons	3			2					1					2		2	3
	TOC	4			4					4					4		4	2
Nutrients	Organic N	2			2					2					2		2	2
	Total N	2			2					2					2		2	1
	Total P	4			4					3					4		4	3
Total number of data on each section		79	51	10	60	10	51	10	10	79	10	10	51	51	60	56	79	79
TOTAL number of data	**1827**	**320**	**56**	**10**	**237**	**10**	**51**	**10**	**10**	**214**	**10**	**10**	**51**	**51**	**212**	**61**	**267**	**247**

2.1.2. Assessment and reviewing the existing data and information

Pollutants in sediment constitute a major factor in the way in which ecological or chemical water status is influenced. Assessment of sediment quality is not always an easy task to fulfil given the fact that there is not one "best" method available. The key issue in quality sediment evaluation is the "***crossing approach***": certain pollutants negatively impact the ecological status by their hazardous character. On the other hand, the real of contaminants is mainly determined by their biovailability. Sometimes, strongly and irreversibly sediment-bound sediment pollutants are hazardous but their risk is negligible. Moreover, even if a sediment contaminant becomes available by various factors – bioturbation or a flooding event – still there is not a direct relationship with impact to chemical or ecological status. Therefore, it is rather difficult to estimate whether or not a certain level of pollution will adversely influence chemical or ecological water quality.

Expert judgment in assessment of sediment quality provides a simultaneous application of three tailor-made solutions, commonly referred to as the ***Triad-approach***:

- ***Chemical analysis*** – in order to determine concentrations of selected, hazardous chemicals and then the levels are checked against the quality standards or guidelines.
- ***Bioassays*** – to test the toxic effects of contaminated sediments on living organisms.
- ***Field inventory*** – to investigate the long-term impact on sediment biota.

2.1.2.1. Technical approach in assessing the Iron Gates sediment quality

The technical report took into account the "chemical analysis" method by groups of determinands and depending on the existing pre-defined standards and guidelines and based on a number of Sediment Quality Standards and Guidelines the assessment was done for the types of pollutants identified also in the previous projects. Based on the data processing done and the discussions of the results it was concluded a certain level of pollution associated to sediments and based on the gaps identified as well the new data necessary for better assessment were recommended as part of the field trip sampling campaign planned and executed in September.

For the technical report, four sets of values were used, both as national quality standards and international quality guidelines – the Romanian, the Canadian, the Dutch and the North-American guidelines (EPA) - briefly described below:

National (Romanian) Environmental Quality Standards are legislated by *Order 161/2006 for Surface Water Quality Classification for establishing the Ecological Status of Water Bodies*. Four groups of determinands, containing a total number of 44 compounds, are selected for chemical investigation of sediments (fraction less than 63 μm). For each contaminant one value is set as Quality Standard.

Canadian Sediment Quality Guidelines are numerical concentrations or narrative statements that are set with the intention to protect all forms of aquatic life and all aspects of their aquatic life cycles during an indefinite period of exposure to substances associated with bed sediments. The levels on which the sediment evaluation has been carried out were:

- The Threshold Effect Level (TEL) - also the same as Interim Sediment Quality Guidelines ISQG - represents the concentration below which adverse biological effects are expected to occur rarely;

- The Probable Effect Level (PEL) that defines the level above which adverse effects are expected to occur frequently; unless otherwise specified, SQGs refer to the total concentration of the substance in surface sediments (i.e., the upper few centimeters) on a dry weight basis (e.g., mg/kg or µg/kg dry weight).

By deriving TEL and PEL, three ranges of chemical concentrations are consistently defined:

- ***the minimal effect range*** (values below TEL) within which adverse effects *rarely* occur;
- ***the possible effect range*** (values between TEL and PEL) within which adverse effects *occasionally* occur;
- ***the probable effect range*** (values above PEL) within which adverse effects *frequently* occur.

EPA Sediment Quality Guidelines consist of two sets of values:

- Sediment quality guidelines that reflect ***threshold*** effect concentration (TEC); is the level below which harmful effects are unlikely to be observed
- Sediment quality guidelines that reflect ***probable*** effect concentration (PEC); is the level above which harmful effects are likely to be observed

Dutch approach for sediment quality: The Dutch Environmental Quality Objectives are specified in terms of a target value that is set at a negligible concentration, usually 1/100th of the maximum permissible concentration. If the negligible level is lower than the background concentration, the target value is set to that level. Limit values were only set for the upper sediment layer, in direct contact with water.

2.1.2.2. Main conclusions drawn from the Technical Report

Based on the present evaluation of results regarding sediments quality in the Iron Gates area, the following conclusions can be drawn:

- The existing sources of raw data were heterogeneous as concerns the order of magnitude of pollutants concentration.
- The reliability of data could not be consistently proved in the case of data from 1999. Therefore, compliance of these data with various Sediment Quality Guidelines has a small degree of correlation with the compliance of data from years 2000s and after.
- Next sampling step should take into account core sediments samples in the studied stretch in order to evaluate historic contamination and to compare the results with previous investigations.
- The current data should be further correlated with previous data concerning certain target pollutants existing in suspended solids phase (data from 2001 and 2004).
- There is a strong need for further investigations especially in the case of Priority Substances recommended within the Water Framework Directive or other xenobiotics, which are included in the Lists of Hazardous and Dangerous Hazardous Substances.
- A more comprehensive approach of the assessment of sediment quality should be adopted and applied in terms of looking at actual risks or impact of the contamination rather than checking whether quality standards and guidelines are exceeded and in doing this the eco-toxicological tests can be applied.

2.2. Specific objectives dealt with in the present stage

According with the TORs project, the last phase's specific objectives are the following:

- Undertaking sampling and analysis as agreed by the overall project team
- Proposing further monitoring programmes.

2.2.1. Description of the field trip and sampling activities performed

Taking into account the results from the Technical Report and in order to keep the data continuity, it was agreed that the following quality determinands would be analysed in sediment samples collected during the field campaign: organic nitrogen and total phosphorous, heavy metals (Mercury, Cadmium, Lead, Nickel, Chromium, Arsenic, Copper, Zinc), organic micro-pollutants (DDT, Lindane, Aldrin, Endrin, Dieldrin), Nonylphenol, Octylphenol, Pentachlorphenol, Di(2-ethylhexyl)phthalate, PAHs, PCBs), Extractable Petroleum Hydrocarbons and Particle Size Distribution.

The surveyed stretch was comprised between Upstream Velika Morava (river km 1107) and Mala Vrbica / Simian (river km 924). The sampling sites as well as the type of sediment samples from the each sampling site are presented in the **Table 4**.

Table 4: **Sampling sites and samples type during the Iron Gate Survey**

No.	Name of sampling site	km	Grab sample[+]		Core sample[++]	
			Left	Right	Left	Right
1	Upstream of Velika Morava	1107	+	+		
2	Downstream of Velika Morava	1097	+	+		
-	Ram / Stara Palanka	1077				+
3	Banatska Palanka / Bazias	1072	+	+		
4	Veliko Gradiste / Belobresca	1061	+	+		
5	Golubac / Coronini	1040	+	+		
6	Dobra / Lubcova	1022	+	+		
7	Donji Milanovac	991	+	+	+	+
8	Dubova	971	-	+		
9	Orsova	956	+	+	+	+
10	Mala Vrbica / Simian	924	+	+		+

[+] upper 10 cm layer of sediment

[++]vertical profile (10 cm deep layers, max. 1 m deep profile)

The sampling period of time and the sampling schedule are presented in **Table 5**.

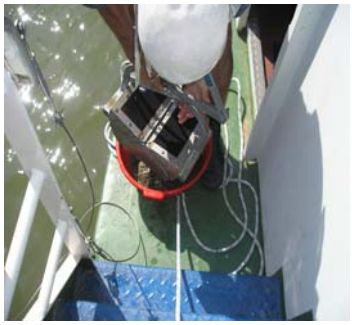
Table 5: Sampling time and schedule

Date	Schedule
11 September 2006	Departure: Belgrade
	Arrival: Veliko Gradiste
	Sampling at sites No. 1-3
12 September 2006	Departure: Veliko Gradiste
	Arrival: Donji Milanovac
	Sampling at sites No. 4-6
13 September 2006	Departure: Donji Milanovac
	Arrival: Kladovo
	Sampling at sites No. 7-10

2.2.1.1. Technical conditions of the sediment sampling

Grab sediment samples were taken using an Ekman dredge from both left and right banks of the river, at each sampling site, except for site no 8 (Dubova, river km 971 where no sediment was found on the left side. Grab sampling was followed by wet sieving in order to obtain the less-than 63 µm fraction for chemical analysis in the lab. Core sampling was carried out using an Eijkelkamp core sampler and samples were taken from the right side at river km 1077 (Stara Palanka – Ram) and at rkm 924 (Vrbica / Simijan) and from the both sides of the river at sampling sites no 7 and 9 (Donji Milanovac, river km 991 and Orsova, river km 956 respectively. The core sample was then divided into 10 centimetres slices for further analysis in the lab – see Fig. 1.

Figure 1: Practical aspects of grab and core sediment sampling



Grab sediment sampling and core sediment sampling



Wet sieving of the grab sediment samples; labelling of grab sediment samples; common discussions between sampling sites

Core sediment sampling

Dividing core sediment samples into 10 centimetres slices

2.2.1.2. Sample analysis

According to the proposed and agreed list, the following determinands were analysed by ICIM: Total Phosphorus, Organic Nitrogen, Heavy Metals (Mercury, Cadmium, Lead, Nickel, Chromium, Arsenic, Copper, Zinc), Extractable Petroleum Hydrocarbons, Organo-chlorine Pesticides (DDT, Lindane, Aldrin, Endrin, Dieldrin), Nonylphenol, Octylphenol, PAHs and selected PCBs. Pentachlorphenol and Di(2-ethylhexyl)phthalate were not analysed because of the lack of the suitable equipment. In Table 6 a short description of the samples preparation and the analytical methods that were used in the ICIM's laboratory is shown:

Table 6: Description of the analytical methods for sediment samples analysis from the Iron Gate Survey

<table>
<tr><th>Determinand</th><th>Sample preparation</th><th>Analytical Method</th></tr>
<tr><td>Organic Nitrogen</td><td rowspan="2">Freeze dried sample is oxidized by a mixture of sulphuric acid and hydrogen peroxide (HACH Digesdahl Method)</td><td>Determination of N-Ammonium according ISO 7150/1-1984 by spectrometric measurement at 700 nm of the blue compound formed by reaction of ammonium with salicylate and hypochlorite ions in the presence of sodium nitroprusside)</td></tr>
<tr><td>Total Phosphorous</td><td>Determination of P-orthophosphate according to ISO 6878/1-1996 by spectrometric measurement at 650 nm of the molybdenum blue complex formed by reduction with ascorbic acid of the antimony phosphomolybdate complex</td></tr>
<tr><td>Petroleum Hydrocarbons</td><td>Freeze dried sample is extracted in n-hexan</td><td>Determination of petroleum hydrocarbons by molecular absorption spectroscopy in UV at 225 nm</td></tr>
<tr><td>Cr, Cu, Cd, Ni, Pb, Zn,</td><td rowspan="3">microwave digester – ETHOS 1600 – method EPA 3051</td><td>AAS with graphite furnace according to ISO 15586</td></tr>
<tr><td>Hg</td><td>AAS with graphite furnace and cold vapour technique according to SR EN 1483: 2003</td></tr>
<tr><td>As</td><td>AAS with graphite furnace and hydride technique</td></tr>
<tr><td>Organo-chlorine Pesticides</td><td rowspan="4">Ultrasonic bath and dichloro-methane</td><td rowspan="4">GC-MS HEWLETT – PACKARD 5890; carrier gas: Helium; column: HP 5MS (cross-linked 5% Phenyl-Methyl Silicone)</td></tr>
<tr><td>PCBs, PAHs</td></tr>
<tr><td>Octylphenol</td></tr>
<tr><td>Nonylphenol</td></tr>
</table>

The results of the performed analysis are presented in Annex 1 of this final report.

2.2.1.3. Results evaluation

The evaluation of the sediment quality based on both pre-existing data and information and on data emerged from the sampling campaign performed in September 2006 is going to be done based on the final agreed data set reported by the three laboratories involved.

In accordance with the agreement made between the DRP leader, the International Expert Co-coordinator (VITUKI) and the working teams of the two countries involved (Serbia and Romania), the final processing of the newly produced data and the already collected data (including the final interpretation of the results) will be done by VITUKI.

2.2.2. Proposing further monitoring programmes

Many previous cases showed that sediment investigations were designed to be descriptive studies presenting temporal and spatial distributions of contaminants. This approach - also applied in some above mentioned studies related to the Iron Gates sediment evaluation – was mainly focused on assessing the compliance of the determined concentrations of selected contaminants with the pre-defined quality standards (if available). Once the exceedence of a certain quality standard is checked, this assessment method has to be completed by taking into consideration the actual risk and/or impact of contaminated sediment given the fact that contaminants in sediment may strongly impact the ecological or chemical water quality status. Hence, a further monitoring programme for the Iron Gates reservoir area shall continue with:

- Monitoring of WFD priority hazardous substances that have a strong preference to stick to sediment (such as hydrophobic organic compounds);
- Given the WFD objective related to "non-deterioration" sediment quality, monitoring of the above mentioned substances should be applied both in terms of spatial and trend monitoring. Spatial monitoring is necessary in order to evaluate the extent in which a certain contaminant is spread over a studied area and probably to detect its source based on available emission data. Trend monitoring should be carried out in order to evaluate the temporal pattern over a long time period. This type of programme shall include also the study of deeper sediment layers (core sediment samples at the same spots as the ones from the previous studies) in order to reflect the historic contamination;
- Along the Iron Gates area, the spatial monitoring shall include at least the sampling sites from the studies that have been taken into account in this evaluation (data from 2001 and 2004) and that have been included in the list of the sampling campaign from September 2006;
- The present monitoring programme (hazard assessment method as the first circle of the Triad-approach) might be complementary followed by the evaluation the toxic effect of sediment on organisms using bioassays and by impact assessment using field inventory (assessment of taxonomic composition and abundance of benthic invertebrate fauna).
- The frequency of the Iron Gates sediment monitoring should be established based on a common agreement among the stakeholders involved and based on technical criteria such as: present information on sediment quality compliance with EQSs, sedimentation rate and existing or further identification of new anthropogenic pressures.

ANNEXES

ANNEX 1	Results of analysis: Organic Nitrogen and Total phosphorous in sediment samples
ANNEX 2	Results of analysis: Heavy Metals
ANNEX 3	Results of analysis: Organic Micropollutants (Pesticides and PCBs)
ANNEX 4	Results of analysis: Organic Micropollutants (PAHs)
ANNEX 5	Results of analysis: Organic Micropollutants (Octylphenol and Nonylphenol)
ANNEX 6	Results of analysis: Extractable Petroleum Hydrocarbons

ANNEX 1

RESULTS OF ANALYSIS: ORGANIC NITROGEN AND TOTAL PHOSPHOROUS

No.	Sample code	Location	Organic Nitrogen	Total Phosphorous
			mg/kg	
1	3-1072-L-RO	Bazias	3037	1106
2	3-1072-R-RO	Bazias	2859	1218
3	4-1061-L-RO	Veliko Gradiste / Belobresca	2898	1080
4	4-1061-R-RO	Veliko Gradiste / Belobresca	2769	985
5	5-1040-L-RO	Golubac / Koronin	2154	985
6	5-1040-R-RO	Golubac / Koronin	3067	1095
7	6-1022-L-RO	Dobra Lubcova	2968	1012
8	6-1022-R-RO	Dobra Lubcova	2988	893
9	7-991-L-RO	Donji Milanovac	2720	980
10	7-991-R-RO	Donji Milanovac	2670	1289
11	core-991-L 0-10 cm - RO	Donji Milanovac - L	2710	1124
12	core-991-L 10-20 cm - RO		2888	1067
13	core-991-L 20-30 cm - RO		2362	893
14	core-991-L 30-40 cm - RO		1806	980
15	core-991-L 40-50 cm - RO		2303	1289
16	core-991-L 50-60 cm - RO		2998	948
17	core-991-L 60-70 cm - RO		2521	913
18	core-991-L 70-74 cm - RO		3643	1043
19	core-991-R 0-10 cm - RO	Donji Milanovac - R	2660	1084
20	core-991-R 10-20 cm - RO		2739	960
21	core-991-R 20-30 cm - RO		4040	1003
22	core-991-R 30-40 cm - RO		2670	1078
23	core-991-R 40-50 cm - RO		2462	1049
24	core-991-R 50-60 cm - RO		1171	719
25	core-991-R 60-67 cm - RO		1925	709
26	8-971-R-RO	Dubova	1737	715
27	9-956-L-RO	Tekija / Orsova	2511	996
28	9-956-R-RO	Tekija / Orsova	2372	928
29	core-956-L 0-10 cm - RO	Tekija / Orsova - L	2332	926
30	core-956-L 10-20 cm - RO		1667	753
31	core-956-L 20-30 cm - RO		2591	1059
32	core-956-L 30-40 cm - RO		2809	1065
33	core-956-L 40-50 cm - RO		2749	1126
34	core-956-L 50-60 cm - RO		2670	1116
35	core-956-L 60-70 cm - RO		2759	1222
36	core-956-L 70-78 cm - RO		2323	932
37	core-956-R 0-10 cm - RO	Tekija / Orsova - R	2561	1149
38	core-956-R 10-20 cm - RO		2918	1017
39	core-956-R 20-30 cm - RO		2362	928
40	core-956-R 30-40 cm - RO		2720	1142

No.	Sample code	Location	Organic Nitrogen	Total Phosphorous
			mg/kg	
41	core-956-R 40-50 cm - RO		3037	1059
42	core-956-R 50-60 cm - RO		2700	1045
43	core-956-R 60-70 cm - RO		2988	1177
44	core-956-R 70-82 cm - RO		2809	1161
45	10-924-L-RO	Mala Vrbica / Simian	2938	1021
46	10-924-R-RO	Mala Vrbica / Simian	2759	1088
47	core-924-R 0-10 cm - RO	Mala Vrbica / Simian - R	2789	923
48	core-924-R 10-20 cm - RO		3414	1037
49	core-924-R 20-30 cm - RO		3067	940
50	core-924-R 30-40 cm - RO		2759	1110
51	core-924-R 40-50 cm - RO		4953	1098
52	core-924-R 50-60 cm - RO		3732	1142
53	core-924-R 60-70 cm - RO		3980	1183
54	core-924-R 70-80 cm - RO		2928	1471

ANNEX 2

RESULTS OF ANALYSIS: HEAVY METALS

No.	Sample code	Location	Hg	Cr	Pb	Ni	Cu	Cd	Zn	As
			mg/kg				mg/kg			
1	3-1072-L-RO	Bazias	0.671	88.86	192.77	41.11	88.36	0.652	226.30	17.52
2	3-1072-R-RO	Bazias	0.400	111.70	148.35	70.19	103.29	1.161	289.78	7.78
3	4-1061-L-RO	Veliko Gradiste / Belobresca	5.567	94.65	358.46	61.83	76.09	0.723	269.72	5.66
4	4-1061-R-RO	Veliko Gradiste / Belobresca	0.517	154.98	276.42	116.07	88.13	1.327	309.96	8.06
5	5-1040-L-RO	Golubac / Koronin	0.357	78.08	195.50	59.52	335.96	0.736	281.12	15.67
6	5-1040-R-RO	Golubac / Koronin	0.423	125.22	279.62	80.78	85.99	1.356	325.26	10.57
7	6-1022-L-RO	Dobra Lubcova	0.456	142.12	319.83	52.81	72.49	1.208	310.26	8.08
8	6-1022-R-RO	Dobra Lubcova	0.533	91.58	199.24	67.15	103.26	0.652	238.84	6.17
9	7-991-L-RO	Donji Milanovac	4.703	114.85	221.12	88.09	99.56	0.722	304.00	8.12
10	7-991-R-RO	Donji Milanovac	0.402	92.18	184.80	51.16	55.10	1.313	311.52	6.33
11	core-991-L 0-10 cm - RO	Donji Milanovac - L	0.372	107.07	125.87	63.15	99.16	1.193	313.09	7.39
12	core-991-L 10-20 cm - RO		0.382	167.28	240.16	77.36	92.52	1.165	301.97	7.38
13	core-991-L 20-30 cm - RO		1.151	115.08	215.17	89.83	117.23	4.864	310.81	8.42
14	core-991-L 30-40 cm - RO		0.462	70.72	399.32	67.54	95.26	1.196	291.58	12.54
15	core-991-L 40-50 cm - RO		0.390	75.26	174.60	59.87	77.67	1.459	336.78	15.71
16	core-991-L 50-60 cm - RO		0.427	73.90	161.32	94.85	109.40	1.108	282.26	11.73
17	core-991-L 60-70 cm - RO		0.418	120.48	320.40	124.10	186.01	1.444	355.77	13.53
18	core-991-L 70-74 cm - RO		0.441	102.99	221.04	64.79	164.61	1.468	363.51	16.08
19	core-991-R 0-10 cm - RO	Donji Milanovac - R	0.201	152.45	169.19	68.21	62.46	0.559	204.11	5.24
20	core-991-R 10-20 cm - RO		0.365	213.31	168.69	83.77	143.03	1.074	269.28	6.58
21	core-991-R 20-30 cm - RO		0.427	96.38	473.40	87.49	107.61	1.267	337.95	13.40
22	core-991-R 30-40 cm - RO		0.887	204.21	201.03	111.49	181.21	1.949	542.27	31.42
23	core-991-R 40-50 cm - RO		0.864	160.44	277.32	136.96	158.55	1.796	469.72	24.37
24	core-991-R 50-60 cm - RO		1.108	158.39	213.48	83.21	171.18	1.916	562.29	25.73
25	core-991-R 60-67 cm - RO		0.467	177.29	223.71	98.21	133.51	1.187	315.14	23.11
26	8-971-R-RO	Dubova	0.405	103.30	395.06	71.77	145.33	1.247	338.40	5.78

No.	Sample code	Location	Hg	Cr	Pb	Ni	Cu	Cd	Zn	As
			mg/kg				mg/kg			
27	9-956-L-RO	Tekija / Orsova	0.366	109.22	204.46	92.24	90.13	1.326	336.10	7.40
28	9-956-R-RO	Tekija / Orsova	0.403	130.11	623.09	85.72	126.49	1.423	341.34	7.09
29	core-956-L 0-10 cm - RO	Tekija / Orsova - L	0.451	158.82	142.22	86.51	84.68	1.316	356.14	23.74
30	core-956-L 10-20 cm - RO		0.785	150.69	335.71	89.58	130.64	1.967	519.72	24.04
31	core-956-L 20-30 cm - RO		0.410	143.29	531.45	87.97	179.87	1.557	370.72	30.44
32	core-956-L 30-40 cm - RO		0.556	153.76	381.74	81.59	104.25	1.641	376.14	23.04
33	core-956-L 40-50 cm - RO		0.500	173.90	171.26	102.22	98.80	1.323	371.90	11.50
34	core-956-L 50-60 cm - RO		0.406	108.60	265.37	63.22	104.35	1.398	336.49	11.94
35	core-956-L 60-70 cm - RO		0.858	163.58	511.58	98.74	143.83	1.797	520.17	27.14
36	core-956-L 70-78 cm - RO		0.717	168.48	488.59	111.21	154.47	1.666	418.30	29.02
37	core-956-R 0-10 cm - RO	Tekija / Orsova - R	0.356	397.00	315.12	96.46	116.90	0.753	346.55	18.18
38	core-956-R 10-20 cm - RO		0.444	146.79	271.62	102.47	195.44	1.339	407.33	24.76
39	core-956-R 20-30 cm - RO		0.440	224.22	668.31	181.68	167.92	1.401	358.11	18.66
40	core-956-R 30-40 cm - RO		0.513	147.60	279.98	104.53	132.17	1.450	382.76	22.39
41	core-956-R 40-50 cm - RO		0.461	129.84	294.23	94.10	138.79	1.873	539.76	21.11
42	core-956-R 50-60 cm - RO		0.725	190.07	420.91	95.92	154.87	2.176	523.78	18.28
43	core-956-R 60-70 cm - RO		0.651	106.88	337.16	104.31	185.52	1.361	500.48	19.40
44	core-956-R 70-82 cm - RO		1.086	174.68	214.41	120.47	178.34	1.943	622.20	24.31
45	10-924-L-RO	Mala Vrbica / Simian	0.465	127.37	224.39	87.67	211.37	1.357	332.54	7.37
46	10-924-R-RO	Mala Vrbica / Simian	0.479	123.50	116.43	62.98	58.11	0.478	164.27	6.97
47	core-924-R 0-10 cm - RO	Mala Vrbica / Simian - R	0.375	138.29	382.10	155.81	125.61	1.146	323.30	16.61
48	core-924-R 10-20 cm - RO		0.375	129.80	262.68	96.80	103.22	1.152	314.22	12.09
49	core-924-R 20-30 cm - RO		0.290	409.24	262.52	116.20	114.85	1.177	318.00	12.00
50	core-924-R 30-40 cm - RO		0.462	145.72	222.50	160.10	391.52	1.548	371.28	7.35
51	core-924-R 40-50 cm - RO		0.516	146.81	423.30	81.68	155.82	1.207	326.26	14.07
52	core-924-R 50-60 cm - RO		0.445	167.65	238.18	111.39	460.91	1.103	364.02	15.90
53	core-924-R 60-70 cm - RO		0.569	106.84	572.92	87.43	124.75	0.626	285.82	13.80
54	core-924-R 70-80 cm - RO		0.622	124.35	357.02	345.99	141.93	1.125	353.10	14.35

ANNEX 3
RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (PESTICIDES & PCBS)

No	LOD = 0.001 mg/kg	Aldrin	Dieldrin	Endrin	pp'-DDT	PCB 28+31	PCB 52	PCB 101	PCB 118	PCB 138	PCB 153	PCB 180
	Sample code	**mg/kg**										
1	3-1072-L-RO	0.0037	< LOD	0.0110	0.0070	< LOD	0.0018	< LOD	< LOD	< LOD	< LOD	< LOD
2	3-1072-R-RO	0.0100	< LOD	0.0050	0.0065	< LOD	0.0017	< LOD	< LOD	< LOD	< LOD	< LOD
3	4-1061-L-RO	0.0110	< LOD	0.0050	0.0080	< LOD	0.0018	< LOD	< LOD	< LOD	< LOD	< LOD
4	4-1061-R-RO	0.0080	< LOD	0.0120	0.0045	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
5	5-1040-L-RO	0.0110	< LOD	0.0030	0.0049	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
6	5-1040-R-RO	0.0110	< LOD	0.0160	0.0150	0.0012	< LOD	< LOD	< LOD	< LOD	< LOD	0.0016
7	6-1022-L-RO	0.0170	< LOD	0.0160	0.0100	0.0012	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
8	6-1022-R-RO	0.0120	< LOD	0.0050	0.0230	< LOD	0.0016	< LOD	< LOD	< LOD	< LOD	< LOD
9	7-991-L-RO	0.0160	< LOD	0.0120	0.0430	< LOD	0.0018	< LOD	< LOD	< LOD	< LOD	< LOD
10	7-991-R-RO	0.0130	0.0030	0.0060	0.0290	< LOD	0.0019	< LOD	< LOD	< LOD	< LOD	< LOD
11	core-991-L 0-10 cm - RO	0.0110	< LOD	0.0160	0.0140	< LOD	0.0020	< LOD	0.0012	0.0020	< LOD	< LOD
12	core-991-L 10-20 cm - RO	0.0050	< LOD	0.0160	0.0050	< LOD	0.0013	< LOD	< LOD	< LOD	< LOD	< LOD
13	core-991-L 20-30 cm - RO	0.0090	< LOD	0.0070	0.0140	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
14	core-991-L 30-40 cm - RO	0.0049	< LOD	0.0030	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
15	core-991-L 40-50 cm - RO	0.0050	< LOD	0.0060	0.0110	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
16	core-991-L 50-60 cm - RO	0.0050	< LOD	0.0070	0.0050	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
17	core-991-L 60-70 cm - RO	0.0045	< LOD	0.0050	0.0060	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
18	core-991-L 70-74 cm - RO	0.0030	< LOD	0.0020	0.0050	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
19	core-991-R 0-10 cm - RO	0.0046	< LOD	0.0030	0.0040	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
20	core-991-R 10-20 cm - RO	0.0060	< LOD	0.0160	0.0080	< LOD	0.0017	< LOD	< LOD	< LOD	< LOD	< LOD
21	core-991-R 20-30 cm - RO	0.0090	< LOD	0.0070	0.0090	< LOD	0.0012	< LOD	< LOD	< LOD	< LOD	< LOD
22	core-991-R 30-40 cm - RO	0.0060	< LOD	< LOD	0.0040	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
23	core-991-R 40-50 cm - RO	0.0048	< LOD	0.0040	0.0080	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
24	core-991-R 50-60 cm - RO	0.0046	< LOD	0.0020	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
25	core-991-R 60-67 cm - RO	0.0030	< LOD	0.0030	0.0060	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
26	8-971-R-RO	0.0190	0.0040	0.0290	0.0330	< LOD	0.0023	< LOD	< LOD	< LOD	< LOD	0.0020
27	9-956-L-RO	0.0056	< LOD	0.0080	0.0180	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD

No	LOD = 0.001 mg/kg	Aldrin	Dieldrin	Endrin	pp'-DDT	PCB 28+31	PCB 52	PCB 101	PCB 118	PCB 138	PCB 153	PCB 180
	Sample code	**mg/kg**										
28	9-956-R-RO	0.0130	< LOD	0.0030	0.0240	< LOD	0.0015	< LOD	< LOD	< LOD	< LOD	< LOD
29	core-956-L 0-10 cm - RO	0.0040	< LOD	0.0090	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
30	core-956-L 10-20 cm - RO	0.0040	< LOD	0.0080	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
31	core-956-L 20-30 cm - RO	0.0070	< LOD	0.0050	0.0110	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
32	core-956-L 30-40 cm - RO	0.0080	< LOD	0.0070	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
33	core-956-L 40-50 cm - RO	0.0030	< LOD	0.0070	0.0050	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
34	core-956-L 50-60 cm - RO	0.0080	< LOD	0.0050	0.0090	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
35	core-956-L 60-70 cm - RO	0.0050	< LOD	0.0070	0.0100	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
36	core-956-L 70-78 cm - RO	0.0040	< LOD	0.0080	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
37	core-956-R 0-10 cm - RO	0.0060	< LOD	0.0060	0.0070	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD	< LOD
38	core-956-R 10-20 cm - RO	0.0060	< LOD	0.0070	0.0060	< LOD	< LOD	< LOD	< LOD	0.0018	< LOD	< LOD
39	core-956-R 20-30 cm - RO	0.0060	< LOD	0.0080	0.0090	< LOD	< LOD	< LOD	< LOD	0.0015	< LOD	< LOD
40	core-956-R 30-40 cm - RO	0.0050	< LOD	0.0130	0.0120	< LOD	< LOD	< LOD	< LOD	0.0018	< LOD	< LOD
41	core-956-R 40-50 cm - RO	0.0160	< LOD	0.0040	0.0100	< LOD	< LOD	< LOD	< LOD	0.0020	< LOD	< LOD
42	core-956-R 50-60 cm - RO	0.0050	< LOD	0.0040	0.0080	< LOD	< LOD	< LOD	< LOD	0.0015	< LOD	< LOD
43	core-956-R 60-70 cm - RO	0.0060	< LOD	0.0080	0.0120	< LOD	< LOD	< LOD	< LOD	0.0020	< LOD	< LOD
44	core-956-R 70-82 cm - RO	0.0080	< LOD	0.0100	0.0080	< LOD	< LOD	< LOD	< LOD	0.0020	< LOD	< LOD
45	10-924-L-RO	0.0120	< LOD	0.0120	0.0460	< LOD	0.0020	< LOD	< LOD	0.0028	< LOD	< LOD
46	10-924-R-RO	0.0048	< LOD	0.0110	0.0330	< LOD	< LOD	< LOD	< LOD	0.0020	< LOD	< LOD
47	core-924-R 0-10 cm - RO	0.0050	< LOD	0.0120	0.0110	< LOD	< LOD	< LOD	< LOD	0.0014	< LOD	< LOD
48	core-924-R 10-20 cm - RO	0.0030	< LOD	0.0100	0.0090	< LOD	< LOD	< LOD	< LOD	0.0017	< LOD	< LOD
49	core-924-R 20-30 cm - RO	0.0030	< LOD	0.0080	0.0060	< LOD	< LOD	< LOD	< LOD	0.0020	< LOD	< LOD
50	core-924-R 30-40 cm - RO	0.0040	< LOD	0.0070	0.0040	< LOD	< LOD	< LOD	< LOD	0.0013	< LOD	< LOD
51	core-924-R 40-50 cm - RO	0.0050	< LOD	0.0070	0.0040	< LOD	< LOD	< LOD	< LOD	0.0020	< LOD	< LOD
52	core-924-R 50-60 cm - RO	0.0030	< LOD	0.0070	0.0080	< LOD	< LOD	< LOD	< LOD	0.0030	< LOD	< LOD
53	core-924-R 60-70 cm - RO	0.0030	< LOD	0.0050	0.0060	< LOD	< LOD	< LOD	< LOD	0.0013	< LOD	< LOD
54	core-924-R 70-80 cm - RO	0.0020	< LOD	0.0030	0.0030	< LOD	< LOD	< LOD	< LOD	0.0015	< LOD	< LOD

ANNEX 4
RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (PAHS – PART 1)

	LOD = 0.001 mg/kg	Naphtalene	Acenapthylene	Acenaphthene	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Benzo(a)anthracene
No.	Sample code	*mg/kg*							
1	3-1072-L-RO	0.003	0.003	< LOD	0.023	0.002	0.030	0.033	0.018
2	3-1072-R-RO	0.003	0.003	< LOD	0.020	0.002	0.024	0.028	0.017
3	4-1061-L-RO	0.007	0.003	0.002	0.016	0.002	0.028	0.032	0.015
4	4-1061-R-RO	0.004	0.002	< LOD	0.012	0.002	0.023	0.029	0.012
5	5-1040-L-RO	0.003	0.003	< LOD	0.017	< LOD	0.029	0.034	0.014
6	5-1040-R-RO	0.003	0.002	< LOD	0.013	< LOD	0.027	0.031	0.014
7	6-1022-L-RO	0.003	0.003	< LOD	0.020	< LOD	0.050	0.077	0.015
8	6-1022-R-RO	0.004	0.003	< LOD	0.022	0.003	0.048	0.071	0.017
9	7-991-L-RO	0.003	0.003	< LOD	0.023	0.003	0.040	0.053	0.020
10	7-991-R-RO	0.006	0.002	0.002	0.015	0.003	0.035	0.036	0.016
11	core-991-L 0-10 cm - RO	0.002	0.004	< LOD	0.038	0.006	0.061	0.086	0.022
12	core-991-L 10-20 cm - RO	0.002	0.004	< LOD	0.034	0.003	0.063	0.089	0.024
13	core-991-L 20-30 cm - RO	0.003	0.005	0.002	0.028	0.003	0.067	0.093	0.026
14	core-991-L 30-40 cm - RO	0.005	0.006	0.002	0.029	0.002	0.074	0.108	0.028
15	core-991-L 40-50 cm - RO	0.005	0.007	0.003	0.038	0.004	0.083	0.112	0.032
16	core-991-L 50-60 cm - RO	0.004	0.008	0.003	0.047	0.006	0.091	0.122	0.034
17	core-991-L 60-70 cm - RO	0.002	0.008	0.004	0.063	0.011	0.116	0.136	0.041
18	core-991-L 70-74 cm - RO	0.003	0.008	0.005	0.065	0.011	0.119	0.138	0.041
19	core-991-R 0-10 cm - RO	< LOD	0.002	< LOD	0.016	0.002	0.031	0.041	0.008
20	core-991-R 10-20 cm - RO	0.002	0.003	< LOD	0.016	0.003	0.036	0.048	0.009
21	core-991-R 20-30 cm - RO	0.002	0.003	0.002	0.018	0.004	0.036	0.050	0.011
22	core-991-R 30-40 cm - RO	0.003	0.004	0.002	0.019	0.004	0.047	0.062	0.016
23	core-991-R 40-50 cm - RO	0.007	0.006	0.003	0.023	0.007	0.067	0.088	0.024
24	core-991-R 50-60 cm - RO	0.004	0.008	0.003	0.022	0.002	0.078	0.096	0.031
25	core-991-R 60-67 cm - RO	0.003	0.008	0.004	0.024	< LOD	0.098	0.113	0.039

	LOD = 0.001 mg/kg	Naphtalene	Acenapthylene	Acenaphthene	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Benzo(a)anthracene
No.	Sample code	*mg/kg*							
26	8-971-R-RO	0.007	0.005	< LOD	0.024	0.006	0.053	0.077	0.010
27	9-956-L-RO	0.002	0.003	< LOD	0.028	0.005	0.045	0.068	0.013
28	9-956-R-RO	0.007	0.005	0.002	0.032	0.007	0.051	0.101	0.013
29	core-956-L 0-10 cm - RO	0.010	0.006	0.002	0.024	0.003	0.083	0.110	0.018
30	core-956-L 10-20 cm - RO	0.016	0.006	0.003	0.027	0.003	0.092	0.131	0.019
31	core-956-L 20-30 cm - RO	0.028	0.007	0.003	0.030	0.002	0.106	0.153	0.022
32	core-956-L 30-40 cm - RO	0.024	0.007	0.004	0.035	0.006	0.102	0.159	0.020
33	core-956-L 40-50 cm - RO	0.016	0.008	0.004	0.043	0.012	0.101	0.168	0.019
34	core-956-L 50-60 cm - RO	0.016	0.008	0.005	0.044	0.013	0.093	0.133	0.018
35	core-956-L 60-70 cm - RO	0.017	0.009	0.005	0.042	0.008	0.096	0.126	0.022
36	core-956-L 70-78 cm - RO	0.019	0.009	0.006	0.038	0.005	0.095	0.117	0.024
37	core-956-R 0-10 cm - RO	0.004	0.006	0.004	0.031	0.002	0.074	0.119	0.015
38	core-956-R 10-20 cm - RO	0.003	0.006	0.004	0.031	0.003	0.082	0.120	0.013
39	core-956-R 20-30 cm - RO	0.020	0.006	0.004	0.033	0.006	0.091	0.123	0.011
40	core-956-R 30-40 cm - RO	0.020	0.006	0.003	0.038	0.006	0.099	0.131	0.014
41	core-956-R 40-50 cm - RO	0.022	0.007	0.003	0.052	0.008	0.127	0.148	0.022
42	core-956-R 50-60 cm - RO	0.021	0.007	0.004	0.048	0.006	0.116	0.142	0.021
43	core-956-R 60-70 cm - RO	0.015	0.008	0.006	0.049	0.006	0.109	0.144	0.027
44	core-956-R 70-82 cm - RO	0.011	0.012	0.009	0.055	0.007	0.112	0.148	0.036
45	10-924-L-RO	0.005	0.005	0.002	0.031	0.003	0.028	0.082	0.007
46	10-924-R-RO	0.004	0.002	< LOD	0.022	0.003	0.034	0.047	0.006
47	core-924-R 0-10 cm - RO	0.009	0.008	0.003	0.038	0.004	0.044	0.083	0.015
48	core-924-R 10-20 cm - RO	0.006	0.007	0.003	0.033	< LOD	0.042	0.052	0.018
49	core-924-R 20-30 cm - RO	0.008	0.007	0.003	0.030	< LOD	0.045	0.059	0.018
50	core-924-R 30-40 cm - RO	0.012	0.007	0.003	0.031	0.002	0.052	0.071	0.019
51	core-924-R 40-50 cm - RO	0.013	0.006	0.004	0.029	0.002	0.044	0.068	0.020
52	core-924-R 50-60 cm - RO	0.016	0.007	0.004	0.032	0.002	0.052	0.081	0.023
53	core-924-R 60-70 cm - RO	0.021	0.013	0.004	0.041	0.004	0.077	0.119	0.019
54	core-924-R 70-80 cm - RO	0.014	0.018	0.006	0.058	0.003	0.086	0.106	0.029

RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (PAHS – PART 2)

	LOD = 0.001 mg/kg	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenz(a,h)anthracene	Benzo(g,h,i)perylene
No.	Sample code	*mg/kg*						
1	3-1072-L-RO	0.022	0.026	0.006	0.009	0.019	0.003	0.012
2	3-1072-R-RO	0.022	0.024	0.005	0.010	0.016	< LOD	0.011
3	4-1061-L-RO	0.022	0.021	0.008	0.009	0.018	0.002	0.010
4	4-1061-R-RO	0.021	0.018	0.007	0.007	0.018	< LOD	0.010
5	5-1040-L-RO	0.019	0.020	0.005	0.008	0.019	0.002	0.010
6	5-1040-R-RO	0.025	0.015	0.005	0.006	0.014	< LOD	0.009
7	6-1022-L-RO	0.02	0.034	0.009	0.012	0.022	0.002	0.013
8	6-1022-R-RO	0.016	0.026	0.010	0.011	0.025	0.002	0.014
9	7-991-L-RO	0.033	0.036	0.004	0.009	0.031	0.002	0.015
10	7-991-R-RO	0.023	0.034	0.008	0.011	0.037	0.005	0.019
11	core-991-L 0-10 cm - RO	0.033	0.035	0.011	0.024	0.044	0.003	0.022
12	core-991-L 10-20 cm - RO	0.041	0.039	0.006	0.013	0.048	0.003	0.024
13	core-991-L 20-30 cm - RO	0.041	0.040	0.008	0.017	0.032	0.003	0.019
14	core-991-L 30-40 cm - RO	0.043	0.040	0.008	0.020	0.023	0.004	0.018
15	core-991-L 40-50 cm - RO	0.048	0.046	0.011	0.021	0.029	0.004	0.022
16	core-991-L 50-60 cm - RO	0.057	0.051	0.016	0.024	0.037	0.006	0.028
17	core-991-L 60-70 cm - RO	0.062	0.058	0.021	0.028	0.046	0.008	0.033
18	core-991-L 70-74 cm - RO	0.062	0.057	0.019	0.026	0.042	0.008	0.032
19	core-991-R 0-10 cm - RO	0.015	0.020	0.003	0.007	0.017	< LOD	0.009
20	core-991-R 10-20 cm - RO	0.018	0.024	0.003	0.009	0.023	0.002	0.012
21	core-991-R 20-30 cm - RO	0.021	0.019	0.007	0.011	0.024	0.002	0.013
22	core-991-R 30-40 cm - RO	0.029	0.023	0.009	0.016	0.022	0.003	0.015
23	core-991-R 40-50 cm - RO	0.041	0.031	0.013	0.021	0.021	0.003	0.018
24	core-991-R 50-60 cm - RO	0.047	0.036	0.016	0.022	0.026	0.004	0.021
25	core-991-R 60-67 cm - RO	0.051	0.043	0.019	0.025	0.031	0.007	0.028
26	8-971-R-RO	0.019	0.035	0.010	0.013	0.025	0.002	0.012

	LOD = 0.001 mg/kg	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenz(a,h)anthracene	Benzo(g,h,i)perylene
No.	Sample code	*mg/kg*						
27	9-956-L-RO	0.024	0.033	0.008	0.020	0.026	< LOD	0.018
28	9-956-R-RO	0.023	0.044	0.015	0.018	0.044	< LOD.	0.026
29	core-956-L 0-10 cm - RO	0.024	0.030	0.014	0.021	0.044	0.006	0.027
30	core-956-L 10-20 cm - RO	0.028	0.039	0.017	0.033	0.048	0.060	0.032
31	core-956-L 20-30 cm - RO	0.034	0.056	0.024	0.040	0.059	0.007	0.037
32	core-956-L 30-40 cm - RO	0.03	0.052	0.020	0.019	0.057	0.003	0.036
33	core-956-L 40-50 cm - RO	0.027	0.048	0.019	0.034	0.056	0.002	0.034
34	core-956-L 50-60 cm - RO	0.03	0.054	0.021	0.037	0.062	0.004	0.041
35	core-956-L 60-70 cm - RO	0.32	0.056	0.022	0.033	0.057	0.002	0.036
36	core-956-L 70-78 cm - RO	0.035	0.063	0.024	0.031	0.054	0.002	0.034
37	core-956-R 0-10 cm - RO	0.026	0.034	0.015	0.021	0.044	0.003	0.032
38	core-956-R 10-20 cm - RO	0.019	0.034	0.009	0.018	0.038	0.004	0.023
39	core-956-R 20-30 cm - RO	0.015	0.036	0.008	0.014	0.034	0.004	0.022
40	core-956-R 30-40 cm - RO	0.021	0.042	0.013	0.036	0.040	0.002	0.027
41	core-956-R 40-50 cm - RO	0.034	0.048	0.021	0.027	0.050	0.002	0.033
42	core-956-R 50-60 cm - RO	0.032	0.044	0.019	0.023	0.046	0.002	0.032
43	core-956-R 60-70 cm - RO	0.037	0.051	0.018	0.026	0.049	0.002	0.032
44	core-956-R 70-82 cm - RO	0.048	0.068	0.018	0.034	0.057	0.003	0.035
45	10-924-L-RO	0.021	0.019	0.004	0.008	0.019	0.002	0.011
46	10-924-R-RO	0.012	0.013	0.003	0.007	0.011	< LOD	0.007
47	core-924-R 0-10 cm - RO	0.026	0.021	0.007	0.010	0.022	< LOD	0.016
48	core-924-R 10-20 cm - RO	0.029	0.028	0.014	0.013	0.022	< LOD	0.015
49	core-924-R 20-30 cm - RO	0.028	0.033	0.016	0.015	0.027	< LOD	0.017
50	core-924-R 30-40 cm - RO	0.032	0.039	0.019	0.019	0.032	< LOD	0.023
51	core-924-R 40-50 cm - RO	0.034	0.037	0.015	0.012	0.027	< LOD	0.019
52	core-924-R 50-60 cm - RO	0.038	0.038	0.012	0.010	0.026	< LOD	0.018
53	core-924-R 60-70 cm - RO	0.036	0.043	0.019	0.014	0.033	< LOD	0.015
54	core-924-R 70-80 cm - RO	0.049	0.063	0.018	0.027	0.049	0.003	0.028

ANNEX 5

RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (OCTYLPHENOL AND NONYLPHENOL)

No	LOD = 0.001 mg/kg Sample code	Location	Nonylphenol mg/kg	Octylphenol mg/kg
1	3-1072-L-RO	Bazias	< LOD	< LOD
2	3-1072-R-RO	Bazias	< LOD	< LOD
3	4-1061-L-RO	Veliko Gradiste / Belobresca	< LOD	< LOD
4	4-1061-R-RO	Veliko Gradiste / Belobresca	< LOD	< LOD
5	5-1040-L-RO	Golubac / Koronin	< LOD	< LOD
6	5-1040-R-RO	Golubac / Koronin	< LOD	< LOD
7	6-1022-L-RO	Dobra Lubcova	< LOD	< LOD
8	6-1022-R-RO	Dobra Lubcova	< LOD	< LOD
9	7-991-L-RO	Donji Milanovac	< LOD	< LOD
10	7-991-R-RO	Donji Milanovac	< LOD	< LOD
11	core-991-L 0-10 cm - RO	Donji Milanovac - L	< LOD	< LOD
12	core-991-L 10-20 cm - RO		< LOD	< LOD
13	core-991-L 20-30 cm - RO		< LOD	< LOD
14	core-991-L 30-40 cm - RO		< LOD	< LOD
15	core-991-L 40-50 cm - RO		< LOD	< LOD
16	core-991-L 50-60 cm - RO		< LOD	< LOD
17	core-991-L 60-70 cm - RO		< LOD	< LOD
18	core-991-L 70-74 cm - RO		< LOD	< LOD
19	core-991-R 0-10 cm - RO	Donji Milanovac - R	< LOD	< LOD
20	core-991-R 10-20 cm - RO		< LOD	< LOD
21	core-991-R 20-30 cm - RO		< LOD	< LOD
22	core-991-R 30-40 cm - RO		< LOD	< LOD
23	core-991-R 40-50 cm - RO		< LOD	< LOD
24	core-991-R 50-60 cm - RO		< LOD	< LOD
25	core-991-R 60-67 cm - RO		< LOD	< LOD
26	8-971-R-RO	Dubova	< LOD	< LOD
27	9-956-L-RO	Tekija / Orsova	< LOD	< LOD
28	9-956-R-RO	Tekija / Orsova	< LOD	< LOD
29	core-956-L 0-10 cm - RO	Tekija / Orsova - L	< LOD	< LOD
30	core-956-L 10-20 cm - RO		< LOD	< LOD
31	core-956-L 20-30 cm - RO		< LOD	< LOD
32	core-956-L 30-40 cm - RO		< LOD	< LOD
33	core-956-L 40-50 cm - RO		< LOD	< LOD
34	core-956-L 50-60 cm - RO		< LOD	< LOD
35	core-956-L 60-70 cm - RO		< LOD	< LOD
36	core-956-L 70-78 cm - RO		< LOD	< LOD
37	core-956-R 0-10 cm - RO	Tekija / Orsova - R	< LOD	< LOD
38	core-956-R 10-20 cm - RO		< LOD	< LOD
39	core-956-R 20-30 cm - RO		< LOD	< LOD

No	LOD = 0.001 mg/kg Sample code	Location	Nonylphenol mg/kg	Octylphenol mg/kg
40	core-956-R 30-40 cm - RO		< LOD	< LOD
41	core-956-R 40-50 cm - RO		< LOD	< LOD
42	core-956-R 50-60 cm - RO		< LOD	< LOD
43	core-956-R 60-70 cm - RO		< LOD	< LOD
44	core-956-R 70-82 cm - RO		< LOD	< LOD
45	10-924-L-RO	Mala Vrbica / Simian	< LOD	< LOD
46	10-924-R-RO	Mala Vrbica / Simian	< LOD	< LOD
47	core-924-R 0-10 cm - RO	Mala Vrbica / Simian - R	< LOD	< LOD
48	core-924-R 10-20 cm - RO		< LOD	< LOD
49	core-924-R 20-30 cm - RO		< LOD	< LOD
50	core-924-R 30-40 cm - RO		< LOD	< LOD
51	core-924-R 40-50 cm - RO		< LOD	< LOD
52	core-924-R 50-60 cm - RO		< LOD	< LOD
53	core-924-R 60-70 cm - RO		< LOD	< LOD
54	core-924-R 70-80 cm - RO		< LOD	< LOD

ANNEX 6

RESULTS OF ANALYSIS: EXTRACTABLE PETROLEUM HYDROCARBONS

No	Sample code	Location	Petroleum Hydrocarbons
			mg/kg
1	3-1072-L-RO	Bazias	3.673
2	3-1072-R-RO	Bazias	3.916
3	4-1061-L-RO	Veliko Gradiste / Belobresca	3.462
4	4-1061-R-RO	Veliko Gradiste / Belobresca	4.013
5	5-1040-L-RO	Golubac / Koronin	4.192
6	5-1040-R-RO	Golubac / Koronin	3.279
7	6-1022-L-RO	Dobra Lubcova	10.650
8	6-1022-R-RO	Dobra Lubcova	4.235
9	7-991-L-RO	Donji Milanovac	2.530
10	7-991-R-RO	Donji Milanovac	4.706
11	core-991-L 0-10 cm - RO	Donji Milanovac - L	2.576
12	core-991-L 10-20 cm - RO		2.944
13	core-991-L 20-30 cm - RO		3.603
14	core-991-L 30-40 cm - RO		5.941
15	core-991-L 40-50 cm - RO		12.050
16	core-991-L 50-60 cm - RO		3.679
17	core-991-L 60-70 cm - RO		3.018
18	core-991-L 70-74 cm - RO		5.476
19	core-991-R 0-10 cm - RO	Donji Milanovac - R	5.802
20	core-991-R 10-20 cm - RO		7.914
21	core-991-R 20-30 cm - RO		12.099
22	core-991-R 30-40 cm - RO		6.645
23	core-991-R 40-50 cm - RO		5.328
24	core-991-R 50-60 cm - RO		5.837
25	core-991-R 60-67 cm - RO		4.293
26	8-971-R-RO	Dubova	4.865
27	9-956-L-RO	Tekija / Orsova	10.812
28	9-956-R-RO	Tekija / Orsova	11.393
29	core-956-L 0-10 cm - RO	Tekija / Orsova - L	16.824
30	core-956-L 10-20 cm - RO		11.696
31	core-956-L 20-30 cm - RO		9.519
32	core-956-L 30-40 cm - RO		5.701
33	core-956-L 40-50 cm - RO		11.584
34	core-956-L 50-60 cm - RO		9.425
35	core-956-L 60-70 cm - RO		9.202
36	core-956-L 70-78 cm - RO		10.367
37	core-956-R 0-10 cm - RO	Tekija / Orsova - R	7.920
38	core-956-R 10-20 cm - RO		11.577
39	core-956-R 20-30 cm - RO		6.322
40	core-956-R 30-40 cm - RO		4.727

No	Sample code	Location	Petroleum Hydrocarbons
			mg/kg
41	core-956-R 40-50 cm - RO		7.186
42	core-956-R 50-60 cm - RO		5.614
43	core-956-R 60-70 cm - RO		5.552
44	core-956-R 70-82 cm - RO		10.876
45	10-924-L-RO	Mala Vrbica / Simian	10.778
46	10-924-R-RO	Mala Vrbica / Simian	9.171
47	core-924-R 0-10 cm - RO	Mala Vrbica / Simian - R	3.216
48	core-924-R 10-20 cm - RO		7.629
49	core-924-R 20-30 cm - RO		18.339
50	core-924-R 30-40 cm - RO		16.378
51	core-924-R 40-50 cm - RO		10.005
52	core-924-R 50-60 cm - RO		20.011
53	core-924-R 60-70 cm - RO		18.046
54	core-924-R 70-80 cm - RO		25.092