November 2006

IRON GATES SEDIMENT EVALUATION - ROMANIA

Final Report
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**ABBREVIATIONS**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>DRB</td>
<td>Danube River Basin</td>
</tr>
<tr>
<td>DRP</td>
<td>Danube Regional Project</td>
</tr>
<tr>
<td>EG</td>
<td>Expert Group</td>
</tr>
<tr>
<td>EU</td>
<td>European Union</td>
</tr>
<tr>
<td>EU WFD</td>
<td>EU Water Framework Directive</td>
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<tr>
<td>GEF</td>
<td>Global Environment Facility</td>
</tr>
<tr>
<td>ICPDR</td>
<td>International Commission for the Protection of the Danube River</td>
</tr>
<tr>
<td>UNDP</td>
<td>United Nations Development Programme</td>
</tr>
<tr>
<td>WB</td>
<td>World Bank</td>
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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);
- 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;

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

<table>
<thead>
<tr>
<th>River kilometer on the Danube River</th>
<th>Name of sampling site</th>
</tr>
</thead>
<tbody>
<tr>
<td>1071.0</td>
<td>Bazias/Banatska Palanka</td>
</tr>
<tr>
<td>1059.0</td>
<td>Veliko Gradiste</td>
</tr>
<tr>
<td>1044.5</td>
<td>Moldova Veche</td>
</tr>
<tr>
<td>1040.0</td>
<td>Iron Gates Reservoir Golubac/Koroin</td>
</tr>
<tr>
<td>999.0</td>
<td>Greben</td>
</tr>
<tr>
<td>996.0</td>
<td>Milanovac</td>
</tr>
<tr>
<td>969.5</td>
<td>Plavisevita</td>
</tr>
<tr>
<td>959.5</td>
<td>Ieselnita</td>
</tr>
<tr>
<td>956.0</td>
<td>Iron Gates Reservoir Tekija/Orsova</td>
</tr>
<tr>
<td>952.0</td>
<td>Ada Kale</td>
</tr>
<tr>
<td>947.2</td>
<td>Upstream Iron Gates I Dam</td>
</tr>
<tr>
<td>943.0</td>
<td>Iron Gates I Dam</td>
</tr>
<tr>
<td>934.0</td>
<td>Kladovo</td>
</tr>
<tr>
<td>924.0</td>
<td>Vrbica/Simijan</td>
</tr>
<tr>
<td>867.0</td>
<td>Upstream Iron Gate II</td>
</tr>
<tr>
<td>849.0</td>
<td>Upstream Timok Gruia/Radujevac</td>
</tr>
<tr>
<td>834.0</td>
<td>Pristol/NovoSelo</td>
</tr>
</tbody>
</table>
Table 2: List of sampling locations in the available data sets

<table>
<thead>
<tr>
<th>No</th>
<th>Sampling location</th>
<th>River km</th>
<th>Location in profile</th>
<th>Longitude</th>
<th>Latitude</th>
<th>Year of available data set</th>
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<td>″</td>
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<tr>
<td>2</td>
<td>Veliko Gradiste</td>
<td>1059</td>
<td>L</td>
<td>n.a.</td>
<td>n.a.</td>
<td>n.a.</td>
</tr>
<tr>
<td>3</td>
<td>Moldova Veche</td>
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<td>L</td>
<td>n.a.</td>
<td>n.a.</td>
<td>n.a.</td>
</tr>
<tr>
<td>4</td>
<td>Iron Gates Reservoir, Golubac/Koronin</td>
<td>1040</td>
<td>L, R</td>
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<td>40</td>
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<tr>
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<td>n.a.</td>
<td>n.a.</td>
<td>n.a.</td>
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<td>6</td>
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<tr>
<td>7</td>
<td>Plavisevita</td>
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<td>n.a.</td>
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<td>10</td>
<td>Ada Kale</td>
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<td>n.a.</td>
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<td>11</td>
<td>Upstream Iron Gates I Dam</td>
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<td>Kladovo</td>
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<td>n.a.</td>
<td>n.a.</td>
<td>n.a.</td>
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<td>14</td>
<td>Simijan/Vrbica</td>
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<td>L, R</td>
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<td>Upstream Iron Gate II</td>
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<td>n.a.</td>
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<td>n.a.</td>
</tr>
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</table>

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

<table>
<thead>
<tr>
<th>Group of determinands</th>
<th>Determinand</th>
<th>rkm 1071</th>
<th>rkm 1059</th>
<th>rkm 1045</th>
<th>rkm 1040</th>
<th>rkm 999</th>
<th>rkm 996</th>
<th>rkm 969.5</th>
<th>rkm 959.5</th>
<th>rkm 956</th>
<th>rkm 952</th>
<th>rkm 947.2</th>
<th>rkm 943</th>
<th>rkm 934</th>
<th>rkm 924</th>
<th>rkm 867</th>
<th>rkm 849</th>
<th>rkm 834</th>
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<td><strong>WFD Priority Substances (PS)</strong></td>
<td>Anthracene</td>
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<td>4</td>
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<td>Brominated diphenylethers (Pentabromodiphenylether)</td>
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<td>5</td>
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<td>Lead and its compounds</td>
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<td>Nickel and its compounds</td>
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<td>4-(para)-Nonylphenol</td>
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<td>(para-terc-Octylphenol)</td>
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<td>Pentachlorobenzene</td>
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<tr>
<td><strong>Other PAHs</strong></td>
<td>Benzo(a)pyrene</td>
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<td>4</td>
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<td>Benzo(b)fluoranthene</td>
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<td>4</td>
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<tr>
<td></td>
<td>Benzo(g,h,i)perylenane</td>
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<tr>
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<td>Benzo(k)fluoranthene</td>
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<tr>
<td>Total number of data on each section</td>
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<td>10</td>
<td>60</td>
<td>10</td>
<td>51</td>
<td>10</td>
<td>10</td>
<td>79</td>
<td>10</td>
<td>10</td>
<td>51</td>
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<td>60</td>
<td>56</td>
<td>79</td>
<td>79</td>
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<tr>
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<td>320</td>
<td>56</td>
<td>10</td>
<td>237</td>
<td>10</td>
<td>51</td>
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<td>10</td>
<td>10</td>
<td>51</td>
<td>51</td>
<td>212</td>
<td>61</td>
<td>267</td>
<td>247</td>
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</tbody>
</table>
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 fulfill 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 bioavailability. 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, Pentachlorophenol, 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**

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

+ 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**

<table>
<thead>
<tr>
<th>Date</th>
<th>Schedule</th>
</tr>
</thead>
<tbody>
<tr>
<td>11 September 2006</td>
<td>Departure: Belgrade</td>
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<tr>
<td></td>
<td>Arrival: Veliko Gradiste</td>
</tr>
<tr>
<td></td>
<td>Sampling at sites No. 1-3</td>
</tr>
<tr>
<td>12 September 2006</td>
<td>Departure: Veliko Gradiste</td>
</tr>
<tr>
<td></td>
<td>Arrival: Donji Milanovac</td>
</tr>
<tr>
<td></td>
<td>Sampling at sites No. 4-6</td>
</tr>
<tr>
<td>13 September 2006</td>
<td>Departure: Donji Milanovac</td>
</tr>
<tr>
<td></td>
<td>Arrival: Kladovo</td>
</tr>
<tr>
<td></td>
<td>Sampling at sites No. 7-10</td>
</tr>
</tbody>
</table>
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
Objectives

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. Pentachlorophenol 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>
<thead>
<tr>
<th>Determinand</th>
<th>Sample preparation</th>
<th>Analytical Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Nitrogen</td>
<td>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>Freeze dried sample is extracted in n-hexan</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>Microwave digester – ETHOS 1600 – method EPA 3051</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>Microwave digester – ETHOS 1600 – method EPA 3051</td>
<td>AAS with graphite furnace according to ISO 15586</td>
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<tr>
<td>Hg</td>
<td>Microwave digester – ETHOS 1600 – method EPA 3051</td>
<td>AAS with graphite furnace and cold vapour technique according to SR EN 1483: 2003</td>
</tr>
<tr>
<td>As</td>
<td>Microwave digester – ETHOS 1600 – method EPA 3051</td>
<td>AAS with graphite furnace and hydride technique</td>
</tr>
<tr>
<td>Organo-chlorine Pesticides</td>
<td>Ultrasonic bath and dichloro-methane</td>
<td>GC-MS HEWLETT – PACKARD 5890; carrier gas: Helium; column: HP 5MS (cross-linked 5% Phenyl-Methyl Silicone)</td>
</tr>
</tbody>
</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

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## ANNEX 2

### RESULTS OF ANALYSIS: HEAVY METALS

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### ANNEX 3

**RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (PESTICIDES & PCBS)**

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<th>No</th>
<th>Sample code</th>
<th>LOD = 0.001 mg/kg</th>
<th>Aldrin</th>
<th>Dieldrin</th>
<th>Endrin</th>
<th>pp'-DDT</th>
<th>PCB 28+31</th>
<th>PCB 52</th>
<th>PCB 101</th>
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ICIM / POPESCU, HAMCHEVICI
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### ANNEX 4
### RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (PAHS – PART 1)

**LOD = 0.001 mg/kg**

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<th>Acenaphthene</th>
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### RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (PAHS – PART 2)

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<th>No.</th>
<th>Sample code</th>
<th>Chrycene</th>
<th>Benzo(b)fluoranthene</th>
<th>Benzo(k)fluoranthene</th>
<th>Benzo(a)pyrene</th>
<th>Indeno(1,2,3-cd)pyrene</th>
<th>Dibenz(a,h)anthracene</th>
<th>Benzo(g,h,i)perylene</th>
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<td>Benzo(a)pyrene</td>
<td>Indeno(1,2,3-c,d)pyrene</td>
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<td>0.017</td>
<td>0.033</td>
<td>0.048</td>
<td>0.059</td>
<td>0.007</td>
</tr>
<tr>
<td>31</td>
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<td>0.056</td>
<td>0.024</td>
<td>0.040</td>
<td>0.059</td>
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<td>0.057</td>
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<td>0.056</td>
<td>0.002</td>
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<td>0.021</td>
<td>0.044</td>
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<td>0.032</td>
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<td>0.018</td>
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<td>0.050</td>
<td>0.002</td>
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<td>0.023</td>
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<td>0.026</td>
<td>0.049</td>
<td>0.002</td>
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<td>0.057</td>
<td>0.003</td>
<td>0.035</td>
</tr>
<tr>
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<td>0.019</td>
<td>0.004</td>
<td>0.008</td>
<td>0.019</td>
<td>&lt; LOD</td>
<td>0.002</td>
</tr>
<tr>
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<td>0.007</td>
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<td>0.007</td>
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<td>0.021</td>
<td>0.007</td>
<td>0.010</td>
<td>0.022</td>
<td>&lt; LOD</td>
<td>0.015</td>
</tr>
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<td>0.028</td>
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</tr>
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<td>0.016</td>
<td>0.015</td>
<td>0.027</td>
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<td>0.039</td>
<td>0.019</td>
<td>0.019</td>
<td>0.032</td>
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<td>0.023</td>
</tr>
<tr>
<td>51</td>
<td>core-924-R 40-50 cm - RO</td>
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<td>0.037</td>
<td>0.015</td>
<td>0.012</td>
<td>0.027</td>
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<td>0.019</td>
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<td>52</td>
<td>core-924-R 50-60 cm - RO</td>
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<td>0.038</td>
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<td>0.014</td>
<td>0.033</td>
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</tr>
<tr>
<td>54</td>
<td>core-924-R 70-80 cm - RO</td>
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<td>0.063</td>
<td>0.018</td>
<td>0.027</td>
<td>0.049</td>
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### ANNEX 5
### RESULTS OF ANALYSIS: ORGANIC MICROPOLLUTANTS (OCTYLPHENOL AND NONYLPHENOL)

<table>
<thead>
<tr>
<th>No</th>
<th>Sample code</th>
<th>Location</th>
<th>Nonylphenol (mg/kg)</th>
<th>Octylphenol (mg/kg)</th>
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<tbody>
<tr>
<td>1</td>
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<td>Bazias</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
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<td>2</td>
<td>3-1072-R-RO</td>
<td>Bazias</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
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<td>Veliko Gradiste / Belobresca</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
<td>4</td>
<td>4-1061-R-RO</td>
<td>Veliko Gradiste / Belobresca</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
<td>5</td>
<td>5-1040-L-RO</td>
<td>Golubac / Koronin</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
<td>6</td>
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<td>Golubac / Koronin</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
<td>7</td>
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<td>Dobra Lubcova</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
<td>8</td>
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<td>Dobra Lubcova</td>
<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
<tr>
<td>9</td>
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<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
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<td>&lt; LOD</td>
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<td>&lt; LOD</td>
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<td>&lt; LOD</td>
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<td>&lt; LOD</td>
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<td>&lt; LOD</td>
</tr>
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<td>&lt; LOD</td>
<td>&lt; LOD</td>
</tr>
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<td>&lt; LOD</td>
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<td>&lt; LOD</td>
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<td>Octylphenol</td>
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<td>---------------</td>
<td>-------------------</td>
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<td>-------------</td>
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## ANNEX 6

### RESULTS OF ANALYSIS: EXTRACTABLE PETROLEUM HYDROCARBONS

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<td>Golubac / Koronin</td>
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<td>3.018</td>
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<tr>
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<td>core-991-L 70-74 cm - RO</td>
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<td>5.476</td>
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<tr>
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<td>5.802</td>
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