



## **BOOK OF ABSTRACTS**

# **NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES**

**University of Novi Sad, Faculty of Technology Novi Sad,**

**Novi Sad, Serbia**

**8 June 2015**



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University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

*The Workshop **New Emergency Approach to the Identification of Targeted Compounds in Environmental Issues** is organised by the Laboratory for Chemical Contaminants and Sustainable Development and the Center of Excellence in Food Safety and Emerging Risks (CEF SER) of the Faculty of Technology University of Novi Sad to addressing the novel environmental approaches to the identification of new emergency contaminants. This event will disseminate the new gathered knowledge created through the realisation of the contracts signed with the Serbian Ministry of Education, Science and Development titled:*

*- Development and application of the advanced chromatographic and spectrometric methods in the analysis of xenobiotics and their degradation pathways in biotic and abiotic matrices*

*and with the Provincial Secretariat for Science and Technological Development titled:*

*- Influence of anthropogenic activities on the urban contamination level: occurrence of xenobiotics in street dust and soil as an indicator of the air pollution, and*

*- Estimation of chemical safety of market basket and population dietary exposure, as well as through the Japan Fellowship S-14034 approved by Japanese Society for the Promotion of Science realised at the University of Kitakyushu. The intention of this event is to make a forum for exchange of research ideas and results concerning the influence of food and the environment on the health.*

*In this way, the event will give the additional value to the environmental issues and strengthen the research capacity of the participating institutions.*

In Novi Sad,  
June 3, 2015

Prof. dr. Biljana Škrbić



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad  
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**PROGRAM**



NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

- 10.30-10.45 Registration
- 10.45-11.00 **Welcome speech**, Prof. Dr. Biljana Škrbić, Head of the Center of Excellence in Food Safety and Emerging Risks (CEF SER) and Laboratory for Chemical Contaminants and Sustainable Development, University of Novi Sad, Faculty of Technology, Serbia
- 11.00-12.00 **Introduction of Japanese culture and landscape**, Prof. Dr. Kiwao Kadokami, University of Kitakyushu, Faculty of Environmental Engineering, Japan
- 12.00-12.45 **Development of AIQS-DB, comprehensive analysis and their application to environmental media**,  
Kiwao Kadokami  
University of Kitakyushu, Faculty of Environmental Engineering, Japan
- 12.45-15.00 Lunch break
- 15.00-15.15 **Occurrence and distribution of multi-class pharmaceuticals in water samples collected from Serbia**,  
Biljana Škrbić<sup>1</sup>, Mira Petrović<sup>2</sup>, Jelena Živančev<sup>1</sup>  
<sup>1</sup>University of Novi Sad, Faculty of Technology Novi Sad, Center of Excellence in Food Safety and Emerging Risks (CEF SER), Serbia  
<sup>2</sup>Institut Català de Recerca de l'Aigua, Girona, Spain
- 15.15-15.30 Coffee break
- 15.30-15.45 **Potential risks and sources of PAHs and PCBs in urban soil**,  
Biljana Škrbić, Đorđe Tadić, Jelena Cvejanov  
University of Novi Sad, Faculty of Technology Novi Sad, Laboratory for Chemical Contaminants and Sustainable Development, Serbia
- 15.45- 16.00 **Occurrence of phthalic acid esters in the environmental matrices**  
Yaqin Ji <sup>1</sup>, Biljana Škrbić<sup>2</sup>, Nataša Đurišić-Mladenović<sup>2</sup>  
<sup>1</sup>Nankai University, College of Environmental Science and Engineering, China  
<sup>2</sup>University of Novi Sad, Faculty of Technology Novi Sad, Laboratory for Chemical Contaminants and Sustainable Development, Serbia
- 16.00-16.15 **Occurrence of polar pesticides, polycyclic aromatic hydrocarbons and selected emerging pollutants in the dissolved water phase of the Danube River**  
Biljana Škrbić<sup>1</sup>, Kiwao Kadokami<sup>2</sup>, Igor Antić<sup>1</sup>, Đorđe Tadić<sup>1</sup>, Csaba Vágvölgyi<sup>3</sup>  
<sup>1</sup> University of Novi Sad, Faculty of Technology Novi Sad, Laboratory for Chemical Contaminants and Sustainable Development, Serbia  
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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

- 16.15-16.30 **Semivolatile and polar organic contaminants in river waters and sediments of the Vojvodina Province**  
Kiwao Kadokami<sup>1</sup>, Biljana Škrbić<sup>2</sup>, Igor Antić<sup>2</sup>, Nataša Đurišić-Mladenović<sup>2</sup>, Nevenka Nikolić<sup>3</sup>  
<sup>1</sup> University of Kitakyushu, Faculty of Environmental Engineering, Japan,  
<sup>2</sup>University of Novi Sad, Faculty of Technology Novi Sad, Laboratory for Chemical Contaminants and Sustainable Development, Serbia  
<sup>3</sup>Public Water Management Company, "Vode Vojvodine" Novi Sad, Serbia
- 16.30-16.45 **A multi-analyte UHPLC-MS/MS method for analysis of 11 principal mycotoxins in different food matrices,**  
Biljana Škrbić, Jelena Živančev, Igor Antić  
University of Novi Sad, Faculty of Technology Novi Sad, Center of Excellence in Food Safety and Emerging Risks (CEF SER), Serbia
- 16.45-17.00 **Risk assessment of Serbian and Chinese population exposure to phthalate esters and heavy elements through rice consumption**  
Yaqin Ji<sup>1</sup>, Biljana Škrbić<sup>2</sup>, Nataša Mrmoš<sup>2</sup>, Jelena Cvejanov<sup>2</sup>  
<sup>1</sup>Nankai University, College of Environmental Science and Engineering, China  
<sup>2</sup>University of Novi Sad, Faculty of Technology Novi Sad, Laboratory for Chemical Contaminants and Sustainable Development, Serbia
- 17.00-17.15 **Development of a simultaneous pressurised-liquid extraction and in-cell clean-up procedure for the determination of PAHs and PCBs in soil samples**  
Biljana Škrbić<sup>1</sup>, Vesna Marinković<sup>2</sup>, Nataša Đurišić-Mladenović<sup>1</sup>, Igor Antić<sup>1</sup>, Đorđe Tadić<sup>1</sup>, Nataša Mrmoš<sup>1</sup>  
<sup>1</sup> University of Novi Sad, Faculty of Technology Novi Sad, Laboratory for Chemical Contaminants and Sustainable Development, Serbia  
<sup>2</sup>Higher education technical school of professional studies, University of Novi Sad, Serbia
- 17.15-17.30 **Financial sustainability of European funded contracts: case study of FP7 CEF SER, IPA LACREMED and IPA BIOXEN**  
Radmila Lukić, Biljana Škrbić  
University of Novi Sad, Faculty of Technology Novi Sad, Serbia





*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

**ABSTRACTS**



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

**Development of AIQS-DB, comprehensive analysis and their application to environmental media**

Kiwao Kadokami

*University of Kitakyushu, Faculty of Environmental Engineering, Japan*

Chemical substances are indispensable materials for modern society. The number and production volume of them therefore have been rapidly increasing. Since some of them however give adverse effects on human health and the ecosystem, a lot of survey has been carried out for clarifying occurrence of them and has found that the environment is polluted by a large number of chemicals. However, the number of chemicals and survey frequency are not insufficient due to limitation of the existing analytical methods, indicating needs of novel comprehensive analytical methods.

The objectives of this study were to develop 1) novel automated identification and quantification system (AIQS) and 2) comprehensive analytical methods using AIQS by which we can simultaneously analyse a large number of chemicals in a short time and with low cost.

At present two types analytical instruments, GC-MS and LC-MS, are usually used for analysing chemical substances due to high sensitivity and selectivity. So we have developed two AIQSs for them. AIQS for GC-MS consists of retention times, mass spectra and calibration curves of nearly 1000 semi-volatile organic compounds (SVOCs). Retention times when analysing samples are predicted with programmed temperature retention index. Difference between actual and predicted RTs is less than 3s, which is the same level as RTs obtained by measuring standards. Slopes of calibration curves are maintained using the designated GC-MS conditions. As a result, correct identification and quantification are performed without the use of standard substances. In AIQS for LC-TOF-MS, RTs, accurate-mass spectra and calibration curves of 300 polar substances are registered. Certain identification of them is done using relative RTs (ratio of analyte's RT to an internal standard's RT) and in-source fragment ions.

Two comprehensive analytical methods have been developed using the developed AIQSs. SVOCs in water samples were extracted liquid-liquid extraction with  $\text{CH}_2\text{Cl}_2$  or tandem solid-phase extraction (SPE) using Sep Pak PS-2 and AC-2 cartridges. Results of recovery tests showed that almost SVOCs except for polar substances were quantitatively extracted. Method detection limits of most substances were less than 10 ng/L. In addition, since 110 substances such as PCBs and organochlorine pesticides were measured by SIM, their MDLs were one-tenth of MDLs obtained by scan mode.

Polar substances in water samples were extracted by SPE with Oasis HLB and Sep Pak AC-2 cartridges. Results of recovery tests showed that almost polar substances



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

except for relatively hydrophobic substances were quantitatively extracted. These results showed that 1170 substances comprised of wide range of physico-chemical properties can be determined by using two comprehensive analytical methods.

As a result of survey of rivers in China, Vietnam and Japan by the both methods, substances detected in three countries were almost the same, indicating that chemical pollution has spread globally by globalization of the economy. Whereas, detected concentrations between three countries were quite different from each other; concentrations in China and Vietnam were much higher than Japan. This difference may result from inappropriate use, management and disposal of chemical substances. From these results, it was confirmed that comprehensive analysis is a useful tool for grasping a whole pollution picture by chemicals



NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

**Occurrence and distribution of multi-class pharmaceuticals in water samples collected from Serbia**

Biljana Škrbić<sup>1</sup>, Mira Petrović<sup>2</sup>, Jelena Živančev<sup>1</sup>

<sup>1</sup>University of Novi Sad, Faculty of Technology Novi Sad, Center of Excellence in Food Safety and Emerging Risks (CEF SER), Serbia

<sup>2</sup>Institut Català de Recerca de l'Aigua, Girona, Spain

Environmental contamination by pharmaceutical active compounds (PhAC) is not a new issue. PhAC have been recognized as emerging pollutants of concern for the last fifteen years but no legal control over their discharge and/or environmental levels has been set up yet. Different classes of PhAC are widespread in the environment including anti-inflammatories, analgesic, blood lipid regulators, antidepressants, antiepileptics, etc. These compounds are designed to have biologic activity and once in the environment they can provoke undesired effects in non-target organisms and become contaminants potentially hazardous, persistent and ubiquitous. For many categories of PhAC, the quantities consumed continue to increase, partly due to the growing demand for drugs to treat ageing-related and chronic diseases. This, jointly with the fact that analytical techniques have improved markedly in the last years to allow the detection of these compounds at ng and sub-ng/L levels in environmental waters has reinforced the interest for them as well as the scientific efforts dedicated to investigate their occurrence and potential impact in the aquatic environment.

There are concerns for both ecosystem and a potential health risk for humans through the consumption of food and water containing pharmaceuticals residues. Thus, analysis of PhAC and data on the environmental fate, behavior and ecological effects of these substances are urgently needed.

In the light of these concerns, the main objectives of the present study were (i) to analyse the presence of 81 selected pharmaceuticals in different types of water collected from Serbia and (ii) to compare the concentrations obtained with those reported in previously conducted studies in order to establish a list of potentially toxic, priority pharmaceuticals to be considered and as well as to adopted in future regulations concerning pharmaceuticals. The obtained data confirmed presence of PhAC in all types of investigated water samples (n=25) i.e. waste, surface, underground and drinking water. Forty seven compounds of 81 drugs were found in four different types of analyzed water, while the highest concentration of 20.1 µg/L was detected for ibuprofen. In all investigated drinking water (n=5) the salicylic acid as a metabolite of acetylsalicylic acid was the most abundant with concentration ranged from 0.3-1.4 ng/L.



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

**Acknowledgement:** The results presented here are obtained within the project “Conceiving wastewater treatment in 2020 – Energetic, environmental and economic challenges (Water\_2020)” and they are part of the contract No 114-451-4567/2013-01 of Secretariat for Science and Technological Development of the Province of Vojvodina.



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

**Potential risks and sources of PAHs and PCBs in urban soil**

Biljana Škrbić, Đorđe Tadić, Jelena Cvejanov

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Organic pollutants are known to accumulate in surface soils as a result of both contaminations from point sources and from long-range aerial transport. Polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) are organic pollutants which have posed high risk to the human health and ecological systems. PAHs are present in soil as a result of anthropogenic and natural processes, while PCBs are not naturally found in the environment. Since there is paucity of comprehensive background data on health and ecological risks and sources of PAHs and PCBs in soil for Serbia, the results obtained in this study bridges this knowledge gap.

Exposure of humans to 16 US EPA PAHs and 6 indicator PCBs in 22 samples of surface soil from 11 locations within the Novi Sad city area were estimated. Exposure can occur via three main pathways: direct oral ingestion of substrate particles; inhalation of soil particles through the mouth and nose; and dermal absorption of pollutants in particles adhered to exposed skin. For PAHs the Total Lifetime Carcinogenic Risk (TLCR) was calculated by summing the cancer risks (CRs) of over the three main exposure pathways. Also, mean risk quotient (m-RQ) was used to assess the ecological risks of target PAHs and PCBs based on recommended soil quality guidelines (SQGs) proposed by the Canadian Council of Ministers of the Environment for 14 PAHs and 6 indicator PCBs, whereas a complementary approach based on PAHs molecular composition analysis was used to identify the dominant sources of PAHs in Novi Sad city area.

The CRs of three exposure pathways decreased in the order: ingestion > dermal contact > inhalation. All health risks were calculated for two age groups, children and adults. The CRs through soil ingestion by children were the greatest. TLCR values for 11 samples for children and for 7 samples for adults were above  $10^{-4}$  which is considered to be of concern, whereas for the rest of the samples TLCR values were within the acceptable range ( $10^{-6}$ - $10^{-4}$ ) at 50% and 68 % of sampling sites for children and adults, respectively. The measured concentrations of PAHs and PCBs were below their SQGs in all samples - for all soil samples m-RQs were below 0.1



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

indicating the negligible risk. Calculated isomeric PAH ratios suggested predominant traffic emission source of pollution at the majority of sampled locations.

**Acknowledgements.** The data presented here were obtained within the project No. 114-451-1148/2014-03 “Influence of anthropogenic activities on the urban contamination level: occurrence of xenobiotics in street dust and soil as an indicator of the air pollution” funded by the Secretariat of the Science and Technological Development of the Autonomous Province of Vojvodina, coordinated by Prof. B. Škrbić.



## NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

### Occurrence of phthalic acid esters in the environmental matrices

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Phthalic acid esters (PAEs) are widely used in plastic, personal care products, building materials, home furnishings, transportation, clothing, food packaging and medical products, etc. World production of PAEs was estimated to exceed 3.5 million tons/year. They can enter into the environment during manufacture, use and disposal. As a result of their widespread applications, large-scale production and substantial threat to human health, PAEs occurrence has attracted extensive attention in global scale in various environmental mediums including soil and dust. Additionally, PAEs presence in soil and dust can cause atmospheric or groundwater contamination by leaching, evaporation and migration.

In this study the levels of six phthalate acid esters: dimethyl phthalate, DMP; diethyl phthalate, DEP; dibutyl phthalate, DBP; butyl benzyl phthalate, BBP; di-(2-ethylhexyl) phthalate, DEHP and dioctyl phthalate, DOP were determined in 30 soil and 30 dust samples collected from 15 locations in Novi Sad, Serbia (railway station, schools, parks, industrial area, beach, and university campus). At each location two samples of soil and of dust were taken: one was taken near traffic road and the other was taken away from the road (e.g. in the schoolyard). Each sample (5 g) was extracted with dichloromethane and then the extract was concentrated and the determination of PAEs was done by using gas chromatograph equipped with mass selective detector (GC-MS).

PAEs were detected in all analyzed soil and dust samples. DEHP and DBP are the most abundant species in both analyzed matrices with average values 0.73 µg/g and 0.07 µg/g in soil, and 1.19 µg/g and 0.07 µg/g in dust, respectively. In all analyzed soil and dust samples the presence of DEHP was higher than 60% of the total PAEs content. The results obtained in this study were in the range of the relevant literature published data.

**Acknowledgement:** The presented results are obtained during the bilateral Chinese-Serbian project "Human exposure assessment to heavy elements, phthalic acid esters and persistent organic pollutants through air, water, dust and food" and supported by Secretariat of the Science and Technological Development of the Autonomous Province of Vojvodina contract No. 114-451-3843/2013-01.





NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

**Occurrence of polar pesticides, polycyclic aromatic hydrocarbons and selected emerging pollutants in the dissolved water phase of the Danube River**

Biljana Škrbić<sup>1</sup>, Kiwao Kadokami<sup>2</sup>, Igor Antić<sup>1</sup>, Đorđe Tadić<sup>1</sup>, Csaba Vágvölgyi<sup>3</sup>

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In this work, an ultra high-pressure liquid chromatography-tandem mass spectrometry (UHPLC-MS/MS) method has been developed for the simultaneous determination of the 3 most consumed pharmaceuticals (carbamazepine, ibuprofen and diclofenac), pesticides (diuron, cypermethrin) and 11 perfluoroalkyl acids, PFAAs (perfluorobutane sulfonic acid, PFBS; perfluorooctane sulfonic acid, PFOS; perfluorooctane sulfonamide, PFOSA; perfluorobutyric acid, PFBA; perfluorohexanoic acid, PEHxA; perfluoroheptanoic acid, PFHpA; perfluorooctanoic acid, PFOA; perfluorononanoic acid, PFNA; perfluorodecanoic acid, PFDA; perfluoroundecanoic acid, PFUnA; perfluorododecanoic acid, PFDoA) in 12 surface water samples (SWS) collected down and up stream of the discharged points and 8 wastewater samples (WWS) discharged directly to the Serbian part of the Danube river. Additionally it was determined the presence of 16 US EPA polycyclic aromatic hydrocarbons by using gas chromatograph equipped with mass selective detector (GC-MS). For all investigated compounds the satisfactory values of recoveries were obtained, while limits of detections and quantifications were in ranges of the literature published values. Among the perfluoroalkyl acids, PFOA and PFHxA were the most abundant compounds in SWS being in the range of 6.31 – 29.15 ng/L and 6.04 – 14.17 ng/L, respectively, while PFOSA was the dominant in the WWS being in the range of 2.36 – 7.38 ng/g. The total concentrations of PAHs in SWS and WWS were in the range of 14.05 – 52.8 ng/L and 87.69 – 1642.6, respectively. Fluorene and naphthalene were detected in all analysed SWS, while fluoranthene, pyrene, benzo(ghi)perylene and naphthalene were detected in all analysed WWS. Diuron was detected in all analysed SWS in concentration range of 1.97 – 3.71 ng/L, while presence of cypermethrin was not detected in any analysed water samples. Among emerging pollutants, detected concentrations were approximately 10 or 100 fold higher in WWS than in SWS, i.e. diclofenac was the most frequently detected in both water matrices in concentration range of 28.27 – 46.18 and 479.8 – 730.8 ng/L for SWS and WWS, respectively, while ibuprofen was detected at the highest



NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

concentration level in WWS. In comparison with published data the concentration levels of PAHs, PFAAs, selected emerging pollutants and polar pesticides were in the range of the literature published data for analysis of selected compounds in surface and wastewater samples.

**Acknowledgement:** The results presented here are obtained within the project co-financed by EU within Hungary-Serbia IPA Cross-border Co-operation programme implemented within the 2007 – 2013 European Union financial framework under the Instrument for Pre-accession Assistance (IPA), *Development of an enzymological (laccase-based) remediation product and technology (LACREMED)*, HU - SRB /1002/214/147, 2012 -2013. Coordinator of the Serbian team (Deputy Research Project Manager): Prof. Dr. Biljana Škrbić and fellowship awarded Prof. Dr. Biljana Škrbić through Japanese Society for the Promotion of Science (S-14034).



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

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*8 June 2015*

**Semivolatile and polar organic contaminants in river waters and sediments of the Vojvodina Province**

Kiwao Kadokami<sup>1</sup>, Biljana Škrbić<sup>2</sup>, Igor Antić<sup>2</sup>, Nataša Đurišić-Mladenović<sup>2</sup>, Nevenka Nikolić<sup>3</sup>

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The presence of organic micropollutants such as pesticides, pharmaceuticals, phthalates, polycyclic aromatic hydrocarbons and other xenobiotics in aquatic ecosystems is a major concern worldwide. In the European Union, there are more than 100000 registered chemicals of which 30000-70000 are in daily use. Thus, it is of utmost importance to have possibility to screen as many as possible chemicals in the environmental matrices, particularly in the aquatic environment, in order to better understand water quality and pollution sources and to allow the implementation of sustainable water use management strategies. Nevertheless, most monitoring studies of trace organic chemicals in the aquatic environment have been focused on a limited number of priority pollutants, because widespread screening for hundreds of organic chemicals in surface water is technically and financially challenging.

Prof. K. Kadokami and his group from University of Kitakyushu developed recently methods that allow automated screening of more than 1000 semivolatile organic compounds (SVOCs), including persistent organic pollutants (POPs) by gas chromatography with mass spectrometry (GC-MS and GC-MS/MS) and of around 300 polar compounds by liquid chromatography-time of flight mass spectrometry (LC-TOF-MS). These methods were also applied for screening of organic micropollutants in river sediment and water samples taken in the Vojvodina Province, Serbia, in September 2014. Sampling was performed on several locations, including spots on the Danube, Tisza, Begej and Krivaja rivers, as well as Danube-Tisa-Danube canal, in the Vojvodina Province in order to allow the first insight into a complete pollution picture of micro-pollutants in water systems of this northern Serbian province.

Water samples for LC-TOF-MS analysis were prepared by solid-phase extraction, SPE (Waters Sep Pak PS-2 and AC-2), while for GC-MS and GC-MS/MS analysis by liquid-liquid extraction. Sediment samples were extracted by accelerated solvent extractor and then cleaned by liquid-liquid extraction and SPE (Silica Sep-Pak Vac).



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

In general, very low number of analyzed pollutants was detected in 10 sediment samples and 20 river water samples. Concerning the GC-MS analysis, in average, 66 and 72 SVOCs were detected in the sediment and water samples, respectively, representing about 7% of all compounds in the developed GC-MS database; there were n-alkanes, polycyclic aromatic hydrocarbons, sterols, domestic and industrial chemicals. The total concentration of all detected SVOCs in sediment ranged 8-109  $\mu\text{g/g}$ , while in the waters 7-114  $\mu\text{g/L}$ ; naturally occurring organic compounds like odd long-chain n-alkanes and sterols were dominant in the samples. Specific PAHs diagnostic ratios indicated the combustion as dominant source of these compounds in the water systems. With respect to the GC-MS/MS analysis of POPs (67 polychlorinated biphenyls (PCBs) and 21 organochlorine pesticides (OCPs)), 33 to 48 compounds were detected in 10 sediment samples, while in the water samples from none to 26 compounds; their total concentrations were rather low, being in the ranges from 2 to 68 ng/g and from not detected to 13.9 ng/L in sediments and waters, respectively. Only 4 polar compounds were detected in water samples by LC-TOF-MS: metformin (in 3 samples), cotinine (in 8 samples), sulfadiazine (in 1 sample), and carbamazepin (in 17 samples) generally in concentrations lower than 0.5  $\mu\text{g/L}$ . Hence, it could be concluded that the results revealed generally low anthropogenic pollution of the samples; nevertheless, the presence of micro-pollutants should be continuously monitored in order to provide further valuable information for refining chemical inventories and technical report support for sustainable water strategies with respect to the xenobiotics.

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015

**A multi-analyte UHPLC-MS/MS method for analysis of 11 principal mycotoxins in different food matrices**

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The occurrence of potentially harmful compounds in food, such as mycotoxins, has meant food safety is one of the main concerns of society. Up to now, more than 400 mycotoxins produced by *Fusarium*, *Penicillium*, *Aspergillus*, and *Claviceps* genera have been identified. The analysis of mycotoxins in food samples requires analytical methods with high selectivity and sensitivity due to the high toxicity of these substances even at very small amounts and the low maximum residue limits established by legislation. In addition to these requirements, the co-occurrence of different mycotoxins in food matrices has prompted the need to develop multi-mycotoxin methodologies capable of detecting a wide range of mycotoxins and metabolites.

In this presentation it is demonstrated the possibility to applied crude extract method for the simultaneous determination of 11 principal mycotoxins in different food matrices including cereals, nuts, dried fruits, biscuit and fig jams by ultra-high performance liquid chromatography coupled to triple quadrupole mass spectrometry (UHPLC-MS/MS). Additionally, implementation of multi-mycotoxin analytical strategy provides a comprehensive data on mycotoxin occurrence in various food items.

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

**Risk assessment of Serbian and Chinese population exposure to phthalate esters and heavy elements through rice consumption**

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Food consumption has been identified as the major pathway of human exposure to potentially toxic elements, compared with other ways of exposure such as inhalation and dermal contact. Human exposure to phthalates mainly occurs via food ingestion and can induce adverse health effects. Contamination of phthalates plasticisers to food has raised concern as some of the phthalates are suspected to be endocrine disruptors. Numerous studies have indicated that for phthalates, the intake of contaminated foods is the most important exposure pathway for the general population. The objective of the present study was to examine the presence of phthalate esters and heavy elements in rice samples collected from Serbian and Chinese market. For this purpose, thirteen collected samples from Serbia (all imported from different countries like: Macedonia, Italia, Bulgaria, Thailand, Vietnam) and China (cultivated in 3 north Chines Province as: Helongjiang, Liaoning, Hebei) were analysed on six phthalate esters (dimethyl phtalate (DMP), diethyl phthalate (DEP), di-n-butyl phthalate (DBP), benzyl butyl phthalate (BBP), di(2-ethyl- hexyl) phthalate (DEHP), di-n-octyl phthalate(DOP)) and ten heavy elements (Pb, As, Cd, Ni, Co, Cr, Cu, Mn, Fe i Sn). Determination of phthalate esters was carried out by gas chromatography (HP-5MS, 30 m x 0,25 mm x 0,25 µm) with mass spectrometry (Agilent Technologies 7890B GC/ 5977MSD), while contents of heavy elements were determined by atomic absorption spectrometry with a graphite furnace (Varian AA240/GTA120 ).

The estimated weekly intakes (EWIs) of potentially toxic elements and phthalates depended on both the concentrations of potentially toxic elements in rice and the associated amount of rice consumption were determined. Also, the target hazard quotients (THQs) and hazard index (HI) were calculated to evaluate the non-carcinogenic health risk from individual and combined potentially toxic elements and phthalates due to rice consumption. All the target hazard quotients of individual elements and phthalates for Serbian samples were lower than one and hazard index (HI) was lower than one as well as, indicating no health risk associated with the



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

intake of these elements and phthalates through consumption of rice. For Chinese samples the target hazard quotients of manganese and DEHP were higher than one. The results of this study will help in understanding the human exposure to toxic elements and phthalate esters.

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

**Development of a simultaneous pressurized-liquid extraction with in-cell clean-up procedure for the determination of PAHs and PCBs in soil samples**

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According to current demands and future perspectives it is necessary to do more analysis in a short period of time. However, extraction techniques (such as Soxhlet extraction, liquid–liquid extraction, ultrasonic extraction) are time-consuming and require a large amount of solvent that is contrary to the principle of so-called "green chemistry". Therefore, in this study a pressurized liquid extraction method (PLE), for the simultaneous extraction and clean-up of polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) in soil was developed.

Soil (10 g) dispersed with diatomaceous earth (2 g) was loaded into the stainless-steel extraction cell (33 ml) above silica gel (2 g) and alumina oxide (3 g) each separated by cellulose filters. Extraction was performed at temperature of 100 °C and two 10 min extraction cycles using a 1:1 (v/v) hexane–dichloromethane mixture gave good extraction efficiency. The method was validated, in terms of its accuracy, precision, and application using soil samples spiked at low concentrations. The accuracy measured as the recoveries of the PAHs and PCBs in the fortified samples ranged from 33.09% to 103.07% and from 106.37 to 132.80, respectively. The precision measured as the relative standard deviation of five replicate samples fortified with studied compounds was within  $\pm 25\%$ . The total concentrations of PAHs in soil were in the range 43.24 – 1568.47 ng/g; while the total concentrations of PCBs were in the range of 0.44 – 141.59 ng/g for surface soil samples. The PLE procedure reduces organic solvent consumption and increases the sample throughput when compared with a traditional stepwise extraction and cleanup procedure. This study demonstrates that the PLE procedure can be applied to complex soil matrices for analysis of PAHs and PCBs for large scale exposure or environmental monitoring studies.

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN  
ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015

*(laccase-based) remediation product and technology (LACREMED), HU - SRB /1002/214/147, 2012 -2013. Coordinator of the Serbian team (Deputy Research Project Manager): Prof. Dr. Biljana Škrbić and fellowship awarded Prof. Dr. Biljana Škrbić through Japanese Society for the Promotion of Science (S-14034).*



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

**Financial sustainability of European funded contracts: case study of FP7 CEF SER, IPA LACREMED and IPA BIOXEN**

Radmila Lukić, Biljana Škrbić

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The group of researchers for chemical contaminants analysis in various matrices coordinated by Prof. Dr. B. Škrbić substantially upgraded the resources of the Faculty of Technology Novi Sad in the domain of the environmental and food chemical safety through FP7 project CEF SER (229629), which lasted 42 months (2009-2012). The total budget of the CEF SER was about 1 million €, enabling reinforcement of the material and human resources of the group and the Faculty itself. Namely, this project enabled installation of advanced, highly sophisticated analytical instruments like ultra high performance liquid chromatograph with triple quadrupole mass spectrometer (UHPLC-MS/MS, Accela-TSQ Vantage, Thermo Fisher Scientific), UHPLC with high resolution mass spectrometer with Orbitrap technology (Accela-Exactive, Thermo Fisher Scientific) and gas chromatograph with mass spectrometer (GC-MS, Agilent 7890B/5977MSD). The price of these instruments was over 700 000 €. With this instruments, the CEF SER Lab became the unique Western Balkan laboratory for chemical contaminants. The instruments installation was followed by the intensive training of the researchers, then by development and validation of the analytical methods for various chemical contaminants determination, and some of these methods have been accredited through involvement of the CEF SER Lab in the Faculty's Laboratory for Food Products Analysis accredited in accordance to SRPS ISO/IEC 17025 standard. Recently, the same research group established the Laboratory for Chemical Contaminants and Sustainable Development, which has been recognized by the Faculty's latest Statute as a special constitutional unit, adding also to the CEF SER sustainability. Additionally, through the CEF SER Lab, several young researchers have been employed, obtaining the unique chance to work at this Lab and learn in the international environment, being also trained by the outstanding EU researchers visiting the CEF SER Lab or at the well-known labs of the EU institutions, with which the CEF SER Lab has collaborated.

The CEF SER capacities have been the basis for contracting more projects on both national and international levels. The projects that followed the CEF SER, in the period from 2010, are: 2 Hungarian-Serbian IPA (Instrument for Pre-accession) Cross-border Co-operation Programme projects co-financed by the EU, 6 bilateral and 1 national projects funded by the Serbian Ministry for Education, Science and Technological Development, 3 projects funded by the Secretariat for Science and Technological Development of the Province of Vojvodina, and the collaboration



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

project with University of Stuttgart. The important upgrades of the material resources have been further made through the Hungary–Serbia IPA projects: LACREMED (HUSRB/1002/214/147, 2012-2013, total budget: 287000 €), and BIOXEN (HUSRB/0901/214/150, 2010-2011, total budget: 248000 €). Hence, the total income of the Faculty of Technology through all these projects has been about 2 million €, with 40000 € being used for the administrative, indirect costs of the Faculty itself, proving unambiguous benefits of the participation in the EU programmes.

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

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*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

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*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

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*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

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*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

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*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

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NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES

University of Novi Sad, Faculty of Technology Novi Sad

8 June 2015

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**NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES**

University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015

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**Projects**

**Project of Provincial Secretariat for Science and Technological Development:**

*Influence of anthropogenic activities on the urban contamination level: occurrence of xenobiotics in street dust and soil as an indicator of the air pollution*, 2015. Coordinator: Prof. Dr. Biljana Škrbić

**Project of Provincial Secretariat for Science and Technological Development:**

*Occurrence of emerging pollutants in the environment and foodstuffs from the Serbian market*, 2014. Coordinator: Prof. Dr. Biljana Škrbić

**Project of Serbian Ministry of Education and Science:** No. 172050, *Development and application of the advanced chromatographic and spectrometric methods in the analysis of xenobiotics and their degradation pathways in biotic and abiotic matrices*, 2011-2015.

Coordinator: Prof. Dr. Biljana Škrbić

**Project of Provincial Secretariat for Science and Technological Development:**

*Estimation of chemical safety of market basket and population dietary exposure*, 2011-2015. Coordinator: Prof. Dr. Biljana Škrbić

**Bilateral project** within Programme of Serbian - Chinese Science and Technology Cooperation, *Human exposure assessment to heavy elements, phthalic acid esters and persistent organic pollutants through air, water, dust and food*, 2013 -2015. Coordinator of the Serbian team: Prof. Dr. Biljana Škrbić

**Project cofinanced by EU within Hungary-Serbia IPA Cross-border Co-operation programme** implemented within the 2007 – 2013 European Union financial framework under the Instrument for Pre-accession Assistance (IPA), *Development of an enzymological (laccase-based) remediation product and technology (LACREMED )*, HU - SRB /1002/214/147, 2012 -2013. Coordinator of the Serbian team (Deputy Research Project Manager): Prof. Dr. Biljana Škrbić

**Bilateral project** within Programme of scientific and technological cooperation between the Republic of Serbia and the Kingdom of Spain, *Advanced chromatographic and mass*



**NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES**

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

*spectrometric techniques in food chemical safety analysis, 2012-2013. Coordinator of the Serbian team: Prof. Dr. Biljana Škrbić*

**COST Action TD 1203 – Food waste valorization for sustainable chemicals, materials and fuels (EUBis), 2012-2016, participation**

**COST Action ES 1202 – Conceiving wastewater treatment in 2020 – Energetic, environmental and economic challenges (Water\_2020), 2012-2016, participation**

**COST Action ES 1403 - New and emerging challenges and opportunities in wastewater reuse (NEREUS), 2014-2018, participation**

**FP7 project No . 229629, CEF SER - Reinforcing research potential in the Laboratory for Chemical Contaminants at the Faculty of Technology towards the establishment of the Center of Excellence in Food Safety and Emerging Risks, 2009-2012. Coordinator: Prof. Dr. Biljana Škrbić**

**Bilateral project within Serbian-Portugal intergovernmental S&T programme, Polycyclic aromatic hydrocarbons and biogenic amines in smoked dry traditionally manufactured meat products from Serbia and Portugal, 2011-2012. Coordinator of the Serbian team: Prof. Dr. Biljana Škrbić**

**Bilateral project within Serbian–Croatian intergovernmental S&T programme, Inorganic and organic pollutants in urban areas, 2011-2012. Coordinator of the Serbian team: Prof. Dr. Biljana Škrbić**

**Project cofinanced by EU within Hungary-Serbia IPA Cross-border Co-operation programme implemented within the 2007 – 2013 European Union financial framework under the Instrument for Pre-accession Assistance (IPA), Development of xenobiotic - degrading bioaugmentation products ( BIOXEN ), HU - SRB /0901/214/150, 2010-2011. Coordinator of the Serbian team (Deputy Project Manager): Prof. Dr. Biljana Škrbić**

**Bilateral project within Serbian– Hungarian intergovernmental S&T programme, Comparison of various analytical and chemometric methods, 2010-2011. Coordinator of the Serbian team: Prof. Dr. Biljana Škrbić**

**Bilateral project within Serbian–Slovenian intergovernmental S&T programme, Heavy metals in the environment as a consequence of the anthropogenic activities , 2010-2011. Coordinator of the Serbian team: Prof. Dr. Biljana Škrbić**

**Project of Serbian Ministry for Science and Technological Development: No. 152001B, Sources identification and correlations amongst the elements and organic compounds in abiotic and biotic matrices: risk analysis and a contribution to the monitoring and improvement of the environmental status, 2008-2010. Coordinator: Prof. Dr. Biljana**





**NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES**

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

Škrbić

*Certificated reference materials – wheat flour and soil certificated contents of polycyclic aromatic hydrocarbons, Carlsberg Srbija d.o.o., 2006/2007. Coordinator: Prof. Dr. Biljana Škrbić*

**Project of Serbian Ministry for Science and Technological Development:** No. BTN-321004B, within National Biotechnology and Agriculture Program: *Baked goods and flour confectioneries with addition of industrial plant seed, 2006-2007.* Coordinator: Prof. Dr. Biljana Škrbić

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**Project of Serbian Ministry for Science** No. 1775, within Basic Research-Chemistry: *Development of methods for identification of chemical residues and contaminants in major food crops, 2002-2005. Coordinator: Prof. Dr. Biljana Škrbić*

**Project of Serbian Ministry for Science and Environment,** No. 02E31: *Development of new technologies for wastegas and wastewater purification and methods for gas and water quality control, 1996-2000. Coordinator: Prof. Dr. Biljana Škrbić*

**Project of Serbian Ministry for Science and Environment,** No. 0935: *Development of the ecologically accepted processes and products in oil-petrochemical industry, 1991-1995. Coordinator: Prof. Dr. Biljana Škrbić*

**President of the Scientific and Organizing Committee of:**

- The 5<sup>th</sup> Danube-Kris-Mures-Tisa (DKMT) Euroregion Conference on Environment and Food Quality, Novi Sad, Serbia and Montenegro, 4-5 September, 2003.
- FP7 CEF SER Симпозијум „Значај преноса научних сазнања“, Технолошки факултет, Нови Сад, 30.11.2009.
- The 1<sup>st</sup> CEF SER Training Course – Capabilities of U-HPLC-MS/MS in Analysis of Contaminants and Pharmaceutical Compounds in Food and the Environment, Novi Sad, Serbia, April 6-8, 2010.
- The 2<sup>nd</sup> CEF SER Training Course - Quality Assurance (QA) and Quality Control (QC) Procedures in Analysis of Contaminants and Pharmaceutical Compounds in Food and the Environment, Novi Sad, Serbia, April 9, 2010.



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad*

*8 June 2015*

- The 1st Center of Excellence for Food Safety and Emerging Risks Workshop, Regional perspective in food safety, Novi Sad, Serbia, September 14, 2010.
- The 12<sup>th</sup> Danube-Kris-Mures-Tisa (DKMT) Euroregion Conference on Food, Environment and Health, Novi Sad, Serbia, September 14-15, 2010.
- The 3<sup>rd</sup> CEF SER Training Course – High resolution mass spectrometry in quantitative analysis and screening of organic contaminants in food and environment, Novi Sad, Serbia, September 16-17, 2010.
- Meeting "BIOXEN-Development of xenobiotic-degrading bioaugmentation", Novi Sad, Serbia, October 26, 2010.
- BIOXEN Training Course - High resolution mass spectrometry of xenobiotics, Novi Sad, Serbia, June 1-3, 2011.
- 2<sup>nd</sup> CEF SER Workshop – Persistent organic pollutants in food and environment, Novi Sad, Serbia, September 8-10, 2011.
- BIOXEN Seminar - Novel approaches for environmental protection, Novi Sad, Serbia, September 8-10, 2011.
- 4<sup>th</sup> CEF SER Training Course - Persistent organic pollutants in food and environment: Risk assessment, Novi Sad, Serbia, November 14-15, 2011.
- 5<sup>th</sup> CEF SER Training Course - Analysis of chemical contaminants in food and the environment, Novi Sad, Serbia, May 7-11, 2012.
- CEF SER Closing event and final training, Novi Sad, Serbia, 30 July 2012.
- LACREMED-Mid-term meeting, Novi Sad, 18. January 2013.
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- Training Course on Chemical Contaminants in the Environment, Novi Sad, Serbia, 21-23 July, 2014.
- Workshop on Valorisation of Vegetable Waste, COST Actions no. TD1203 EUBis, Novi Sad, Serbia, 6-7 August, 2014.



*NEW EMERGENCY APPROACH TO THE IDENTIFICATION OF TARGETED COMPOUNDS IN ENVIRONMENTAL ISSUES*

*University of Novi Sad, Faculty of Technology Novi Sad  
8 June 2015*

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