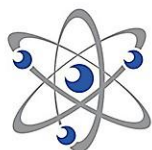




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EUROPEAN PROJECT
RESPONSE
to challenges in forensic drugs analyses

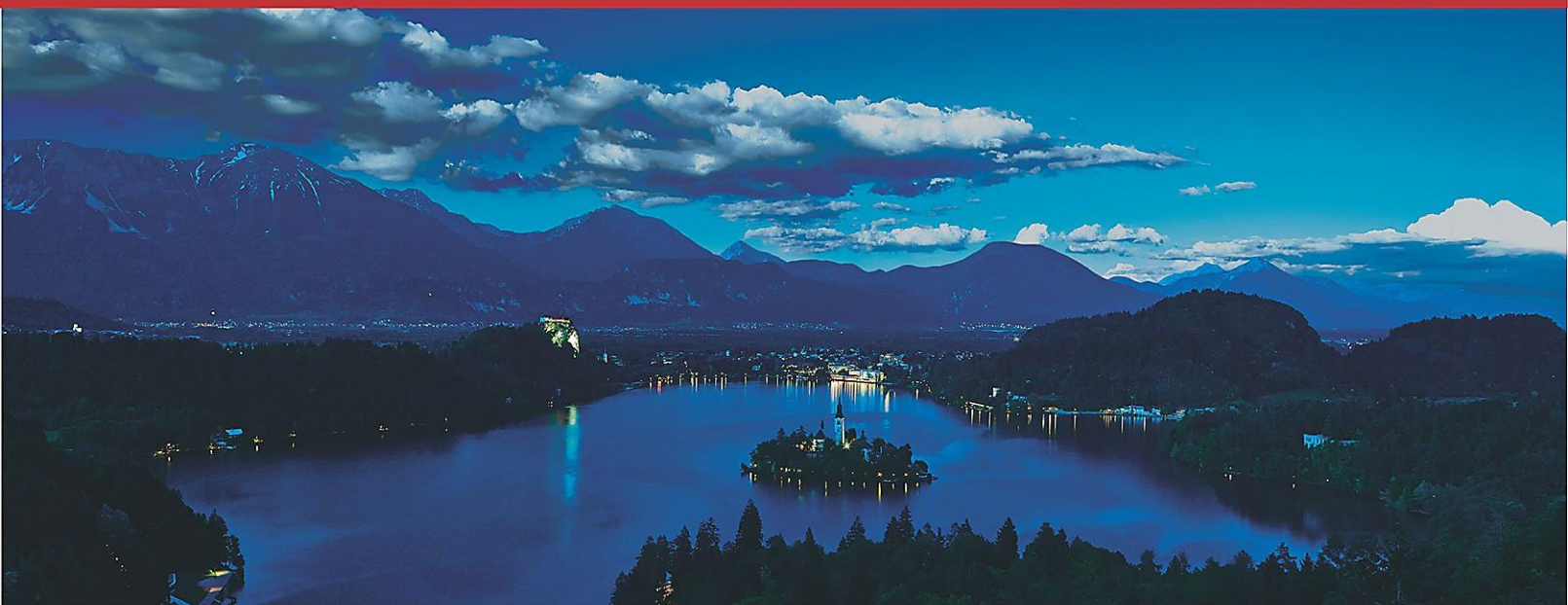


ENFSI DRUGS WORKING GROUP **22nd ENFSI-DWG Meeting**



Hosted by the Slovenian National Forensic Laboratory

PROGRAMME AND BOOK OF ABSTRACTS



May 10th – 12th, 2016, Bled, Slovenia

22nd ENFSI DRUGS WORKING GROUP MEETING

Bled, SLOVENIA, May 2016

Conference Chairperson

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International Programme Subcommittee

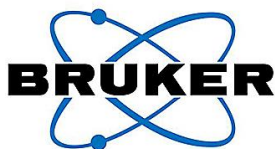
Dr. Nataša Radosavljevič, The National Criminal-Technical Center, Belgrade, Serbia (chair)
Dr. John Power, Forensic Science Ireland, Dublin, Ireland
Dr. Sonja Klemenc, National forensic Laboratory, Slovenia (host)

Local Organizing Committee

National forensic Laboratory - Chemistry department, Ljubljana, Slovenija.

Dr. Sonja Klemenc (sonja.klemenc@policija.si)
Dr. Katja Benčina (katja.bencina@policija.si)

Sponsors of the event are kindly acknowledged for
their contributions!



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PROGRAMME

Monday, May 9th 2016

- 9:00-16:00 RESPONSE project Steering Committee meeting
Chair: Sonja Klemenc, National Forensic Laboratory (NFL), Ljubljana, Slovenia
- 9:00-16:00 DWG Steering Committee meeting
Chair: Udo Zerell, Bundeskriminalamt (BKA), Wiesbaden, Germany
- 16:00-17:00 RESPONSE project Steering Committee /DWG Steering Committee meeting
Chair: Udo Zerell, Bundeskriminalamt (BKA), Wiesbaden, Germany

Tuesday, May 10th 2016

- 9:00-12:00 Chemometrics subcommittee meeting
Chair: Michael Bovens, Forensic Science Institute Zurich, Switzerland
- 9:00-10:00 RESPONSE project /CLEN2SAND/EMCDDA representatives meeting
Chair: Sonja Klemenc, National Forensic Laboratory (NFL), Slovenia

11.00 – 13:15 **Registration** /Get-together/ Uploading of presentations / Posters / *Sponsors Exhibition*

13:30 **Welcome and opening of the meeting** by Dr. Dorian Kežan, director of the National Forensic Laboratory (NFL), Slovenia

Welcome and practical issues
Udo Zerell, Bundeskriminalamt (BKA), Wiesbaden, Germany and Sonja Klemenc, National Forensic Laboratory (NFL), Slovenia

14:00 **Session I**
Chair: John Power, Forensic Science Ireland, Dublin, Ireland

O1 – EU co-funded project »response to new challenges in forensic drug analyses« - objectives and some selected results - ^aSonja Klemenc, ^bJanez Košmrlj; ^aNational Forensic Laboratory, Slovenia
^bUniversity Ljubljana, Faculty of Chemistry and Chemical Technology, Ljubljana, Slovenia (30 min)

O2 – The EU-project 'SPICE-profiling' (ISEC 2013) - general aims and results of an in depth study on the synthetic cannabinoid MDMB-CHMICA - ^aMichael Pütz^a, Sascha Münster-Müller^a, Nicole Scheid^a, Volker Auwärter^b, Laurence Dujourdy^c, ^aFederal Criminal Police Office (BKA), Forensic Science Institute, Toxicology, Germany, ^bUniversity Medical Center, Institute of Forensic Medicine, Freiburg, Germany, ^cNational Police Science Institute (INPS), France (15 min)

O3 – Detected NPS cases by Forensic Toxicology Unit of University of Florence in the frame of the European I-SEE Project - ^aFabio Vaiano, Giovanni Serpelloni, Valeria Catalani, Elisabetta Bertol, U.R.I.To.N., Forensic Toxicology Division, Department of Health Science, University of Florence, Italy (15 min)

15:15-15:45 *Coffee Break & Sponsors Exhibition/ Late registrations*

Session I (continued)

Chair: Ulla Maija Laakkonen, National Bureau of Investigation Forensic Laboratory (NBIFL), Vantaa, Finland

O4 – Sharing of data for rapid identification and detection of new psychoactive substances (NPS) in Europe- ^aGuillou Claude, Reniero Fabiano, Holland Margaret, Lobo Pereira Vicente Joana, Chassaigne Hubert, Kolar Kamil, Tirendi Salvatore, Joint Research Centre of European Commission, Institute for Health and Consumer Protection (IHCP), Italy (15 min)

O5 – EU co-funded project RESPONSE: New psychoactive substances database - ^aSonja Klemenc and Borut Jesenko, National Forensic Laboratory, Slovenia (15 min)

O6 –The revision of the ENFSI qualitative sampling guidelines - ^aHugh Coyle, Forensic Science Ireland, Dublin, Ireland, Ireland (15 min)

16:30 **Session II**
Poster session

P1 – NFIDENT- Reliable drug analyses within a day - ^aA. Sprong, S. Verheij (projectleader), R. Walinga, R.-J. Raterink, K. Still, Netherlands Forensic Institute, Netherlands

- P2** – Illegal production of Legal Highs in the legal laboratory “The new Czech A. Shulgin” - [Michael Roman](#), Lukáš Franěk, Ludmila Komorousová, Institute of Criminalistics Prague, Czech Republic
- P3** – Application of automated sample preparation for drug analysis - Eszter Holló-Sitkei, [Tamás Csesztregi](#), Hungarian Institute for Forensic Sciences, Budapest, Hungary
- P4** – Qualitative determination of residual solvents in cocaine samples by static headspace-GC-MS (development of cocaine profiling methodology)- [Tomaž Gostič](#), National forensic laboratory, Slovenia
- P5** – Cannabis preparations for "self medication" - chemical characterizations of some recently seized products - [Rajko Koren](#), Katja Benčina, National forensic laboratory Slovenia
- P6** – Heroin profiling - methodology development and first impressions – Mojca Janežič, [Katja Benčina](#), National forensic laboratory, Slovenia
- P7** – Novel NPS - test purchases over the internet - what is inside the package? – [Sonja Klemenc](#), [Mojca Janežič](#) and Bojana Koštrun, National forensic laboratory, Slovenia
- P8** – The role of the National forensic laboratory in the Slovenian EWS and wider – RESPONSE and I-SEE projects - [Sonja Klemenc](#), National forensic laboratory, Slovenia
- P9** - Getting Ahead of the Game: Complete Analysis of New Psychoactive Substances, [N. Tonge](#), P. Gierth, C. Stapleton, J. Smith, C. Barrow, A. Gibbs, and A. Codina, Bruker, UK

17:15 – end flexible	Session III Chair: Natalie Meert, Nationaal instituut for criminalistics and criminology, Brussel, Belgium Short presentations "Bring your case / Bring your own slides"
19:30	<i>Social time (welcome dinner)</i>

Wednesday, May 11th 2016

9.00	Session IV Chair: Fraser Johnston, Key Forensic Services Ltd, University of Warwick Science Park, United Kingdom
	O7 – Drug identification by UPLC-QTOF-MS - Petur W. Dalsgaard , Lotte A. Reitzel, Irene B. Müller, University of Copenhagen, Denmark (invited speaker - 30 min)
	O8 – NPS data hub for NMR and other analytical data - Torsten Schoenberger , Bundeskriminalamt (BKA), Wiesbaden, Germany (15 min)
	O9 – In-house Reference Material Certification Project - István Balatoni , Hungarian Institute for Forensic Sciences, Hungary (15 min)
10:15	<i>Group photo</i>
10.40-11:10	<i>Coffee Break & Sponsors Exhibition</i>
	Session IV (continue) Chair: Annette Sprong, Netherlands Forensic Institute, The Hague, The Netherlands
	O10 – A Metabolomics Study of Retrospective Forensic Data from Whole Blood Samples of Humans Exposed to 3,4-Methylenedioxymethamphetamine: A New Approach for Identifying Drug Metabolites and Changes in Metabolism Related to Drug Consumption - Kirstine L. Nielsen, Rasmus Telving, Mette F. Andreassen, Jørgen B. Hasselstrøm, and Mogens Johannsen , Department of Forensic Medicine, Aarhus University, Denmark (15 min)
	O11 – An update on some novel by-products formed during the synthesis of amphetamine via the APAAN to P2P Leuckart route - John D. Power ^a and Pierce V. Kavanagh ^b , ^a Forensic Science Ireland, Dublin, Ireland, ^b Department of Pharmacology and Therapeutics, Trinity College Dublin, Ireland (15 min)
	O12 – Alpha-PVP as an active component of herbal highs - Roman Stanaszek , Bogumiła Byrska, Dariusz Zuba, Institute of Forensic Research, Kraków, Poland (15 min)
	O13 –The Use of Solid Phase GC-IRD for Forensic Drug Analysis - Stephanie Fisher , Dani Instruments, Inc, Marlborough, United States (SPONSOR`S PRESENTATION - 15 min)
12:30-14:00	<i>Lunch</i>

Session V**Chair:** Nataša Radosavljević-Stevanović, The National Criminal-Technical Center, Belgrade, Serbia

O14 – DWG Profiling Subcommittee: overview of activities - Natacha Gentile^a, Fabrice Besacier^b, Pierre Esseiva^a, Filip Van Durme^c, ^aUNIL, Ecole des Sciences Criminelles, Switzerland, ^bINPS, France, ^cNICC, Brussels, Belgium (15 min)

O15 – Results of proficiency test 2015- Tamás Csesztregi, Hungarian Institute for Forensic Sciences, Budapest, Hungary (15 min)

O16 – Subcommittee Chemometrics - Status Report 2016 - Michael Bovens^a, Sami Huhtala^b, Ivo Alberink^c, Björn Ahrens^d, ^aForensic Science Institute Zurich, Switzerland, ^bNational Bureau of Investigation, Vantaa, Finland, ^cNetherlands Forensic Institute, The Hague, The Netherlands, ^dBundeskriminalamt, Wiesbaden, Germany (15 min)

O17 – Communication subcommittee: status for DWG on members website - Lotte Reitzel, University of Copenhagen, Faculty of Health Sciences, Department of Forensic Medicine, Denmark (15 min)

15:30-
16:00 *Coffee Break & Sponsors Exhibition*

Session V (continue)**Chair:** Irene Breum Müller, Section of Forensic Chemistry, Department of Forensic Medicins, Faculty of Health Sciences, University of Copenhagen, Denmark

O18 – Overview of DWG QA subcommittee activities - N. Meert^a, Ulla-Maija Laakkonen^b, Tamás Csesztregia^c, Julia Nagy^c and Peter Mell^d, ^aNational Institute for Criminalistics and Criminology, Brussels, Belgium, ^b NBI, Forensic laboratory, Vantaa, Finland, ^c Hungarian Institute for Forensic Sciences, Budapest, Hungary, ^d Institute for Expert Services, Budapest, Hungary (15 min)

O19 – Update from the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) and the Organization of the Scientific Area Committees (OSAC) - Sandra Rodriguez-Cruz, Drugs Enforcement Administration, United States (20 min)

O20 – New psychoactive substances in Europe – diversity of the market and associated challenges - Rachel Christie, European Monitoring Centre for Drugs and Drug Addiction, Lisbon, Portugal_ (15 min)

O21 – Proposal of joint efforts towards a European Cloud spectroscopy system for identification of new drugs - Guillou Claude, Joint Research Centre of European Commission, Institute for Health and Consumer Protection (IHCP), Ispira, Italy (15 min)

17: 30 *End of Day 2*

19:00 *Social programme and dinner (in Grand Hotel Toplice)*

Thursday, May 12th 2016

9.00

Session VI**Chair:** Fabrice Besacier, National Scientific Police Institute, Lyon, France

O22 – Research institute of forensic sciences and criminology (RIFSC) "drugs" sector - Stefan Yosifov, Research Institute of Forensic Sciences, Sofia, Bulgaria (15 min)

O23 – Legislative approach on new psychoactive substances (NPS) in Cyprus - later changes. results of ISEC project on NPS - Maria Afxentiou, Lefkia Panayiotidou, Popi Kanari, Forensic Science and Toxicology Department, State General Laboratory, Nicosia, Cyprus (15 min)

O24 – Blotting paper identification process – recalculating – Eran Fenigstein, Simcha Shimron, Ehud (Udi) Wolf, Analytical lab, DIFS, Israel police HQ, Jerusalem, Israel (15 min)

O25 – The Stability of the Physical Features of 'Ecstasy' Tablets - Mario Mifsud^a, Sue Jickells^b, Janet Mifsud, Kim Wolff, ^aForensic Laboratory Services, Malta, ^bFaculty of Science, University of East Anglia, UK, Clinical Pharmacology & Therapeutics, Faculty of Medicine & Surgery, University of Malta, Malta, ^d Institute of Pharmaceutical Science, UK (15 min)

10.30 –
11:00 *Coffee Break & Sponsors Exhibition*

11.00	Session VI (continued) Chair: Maria Afxentiou, State General Laboratory, Nicosia, Cyprus
	O26 – Drug Prevention in Berlin - <u>Nadia El-Khadra-Kluth</u> , LKA Berlin, Germany (15 min)
	Announcement of the 23rd ENFSI-DWG in 2017, Sweden - <u>Åsa Klasén</u> , Swedish National Forensic Centre, Sweden (10 min)
	Evaluation form to be completed (15 min)
11.50	Closing of the Scientific Meeting
12:00-14:00	<i>Lunch</i>
14:00	Session VII: DWG Business Meeting (one member per laboratory) Chair: Udo Zerell, Bundeskriminalamt (BKA), Wiesbaden, Germany
	<u>Agenda Business Meeting</u> 1. Welcome and opening remarks 2. Acceptance of agenda 3. Minutes of 21st ENFSI DWG Meeting in Dublin, 2015 4. ENFSI Joint Meeting 2015 5. Revised guidelines on sampling of illicit drugs for qualitative analysis 6. Cooperation with Society of Toxicological and Forensic Chemistry in Germany 7. Finances 8. Cooperation with NIST/OSAC (USA) 9. Networking within DWG 10. Membership changes 11. Subcommittees 12. Elections 13. Host and time of next annual meeting 14. Any other business 15. Closing of the Business Meeting
14:00	RESPONSE project SC meeting (in parallel session with business meeting) Chair: Sonja Klemenc, National Forensic Laboratory (NFL), Slovenia
16:30	DWG Steering Committee & SWGDRUG and EMCDDA meeting Chair: Irene Breum Müller, Institute of Forensic Medicine, University of Copenhagen, Denmark
16:30 - 20:00	Social programme (<u>optional - not included in the registration fee</u>)* Guided excursion to Bled Castle or Bled Island Departure: from Golf hotel/ Price 50 includes an English speaking guide, entrance fees, transfer by boat or bus, late meal (dinner) see more at meeting web page http://www.enfsidwg2016.com/programme/

Friday, May 13th 2016

9:00-11:00	DWG Steering Committee meeting
9:00-	Any other meeting (optional)

ABSTRACTS ORAL PRESENTATIONS

O1: EU CO-FUNDED PROJECT »RESPONSE TO NEW CHALLENGES IN FORENSIC DRUG ANALYSES« - OBJECTIVES AND SOME SELECTED RESULTS

Sonja Klemenc^a and Janez Košmrlj^b

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The RESPONSE project [1] addresses two specific topics of forensic drugs investigations:

a) New psychoactive substances (NPS) where unexpected growth in the number and type of NPSs advertised and sold over the Internet at affordable prices, lack of the availability of reference materials (RM) and specific reliable spectra databases MS and FTIR are the main challenges. Within its "NPS" activities the project has created FTIR database (tool), drafted guidelines on FTIR data acquisition and interpretation (joined documents with ENFSI-DWG). Numerous (MS, FTIR-ATR and FTIR-condensed phase) spectral data have been provided to different data repositories and beside this "identification reference materials" (84 compounds, so far) have been shared with project partners and wider.

Several types of NPS materials have been in focus: reference materials (CRM), samples from internet - test purchases, from the police and customs seizures and collected from other sources.

Presentation will cover:

- the strategies, challenges and experiences gained through the test purchasing over the internet
- the analytical strategies of identifying "unknowns" by means of HPLC-TOF, NMR, GC-MS, FTIR and some supplemental methods
- interpretation challenges (and some possible pitfalls)
- the problem and the project solution (public open database [2]) on how to share gained analytical data and knowledge efficiently (in real time)
- the importance of networking (with other projects, scientific communities and policy makers).

b) Drugs profiling where the main problem is the recognized gap between customers (law enforcement, judicial, etc.) needs and labs capacities and staff competencies. Two approaches have been applied: *mentor based* (development of profiling methodologies in NFL) and *workshop assisted* learning to enhance the understanding of general profiling concepts. Profiling matters will be mainly covered by posters and some other presentations in the frame of the same meeting.

Acknowledgement: The presenting author wishes to acknowledge the staff of Chemistry department NFL: Tomaž Gostič, Andreja Hiti Vidic, Mojca Janežič, Bojana Koštrun, Brigita Nemeč, Katja Benčina, Rajko Koren, Tomaž Premuš and Ksenija Jurca for samples preparations, hundreds of analyses and interpretations. Many thanks also to all project partners and other contributions to the project.



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Notes:

O2: THE EU-PROJECT 'SPICE-PROFILING' (ISEC 2013) - GENERAL AIMS AND RESULTS OF AN IN DEPTH STUDY ON THE SYNTHETIC CANNABINOID MDMA-CHMICA

Michael Pütz^a, Sascha Münster-Müller^a, Nicole Scheid^a, Volker Auwärter^b, Laurence Dujourdy^c

^aFederal Criminal Police Office (BKA), Forensic Science Institute, Toxicology, 65173 Wiesbaden, Germany; ^bUniversity Medical Center, Institute of Forensic Medicine, 79104 Freiburg, Germany; ^cNational Police Science Institute (INPS), 69134 Ecully Cedex, France

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Introduction: The project SPICE-profiling, funded within EU's ISEC 2013 programme (JUST/2013/ISEC/DRUGS/AG/ISEC/4000006421) develops integrated and innovative approaches tackling the phenomenon of new psychoactive substances (NPS). The project builds on the results of the finalized SPICE (JUST/2009/DPIP/AG/0948) and SPICE IIplus (JUST/2011-2012/DPIP/AG/4000003597) projects which were focused on health risks and prevention issues related to NPS.

Core activities of the project SPICE-profiling employ and analytically characterize NPS-samples from internet test purchases, laboratory syntheses, police and customs seizures as an information pool delivering details about the manufacturing procedures, required key chemicals and chemical relations between different products. Comparative analysis is based on quantitative assessment of characteristic synthesis impurities by UHPLC-MS/MS and of carbon, nitrogen and hydrogen stable-isotope-ratios by EA-IRMS and GC-IRMS, assisted by multivariate data analysis.

Project results for a range of recently seized 'Spice'-products and seized powder samples containing the particularly hazardous cannabimimetic aminoalkylindole MDMA-CHMICA (methyl-(S)-2-(1-(cyclohexylmethyl)-1H-indole-3-carboxamido)-3,3-dimethylbutanoate) are presented.

Methods: More than 120 samples of 'Spice'-products of different brands from test purchases in various internet shops and from police and customs seizures were extracted and submitted to preparative flash column chromatography (Büchi X50). Pure MDMA-CHMICA was isolated from the herbal matrices for subsequent stable-isotope-ratio analysis (Thermo EA- and GC-IRMS systems) and combined fractions of synthesis impurities submitted to profiling via UHPLC-MS/MS (Bruker AmaZon Speed). Data processing and multivariate data analysis were conducted via ProfileAnalysis (Bruker) and UnscramblerX (Camo) software.

Results and Discussion: MDMA-CHMICA was separated from synthesis impurities and structure elucidation of key impurities performed by MSⁿ, HR-MS and NMR experiments. GC-IRMS was successfully applied to assess reliable carbon, nitrogen and hydrogen isotope-ratio data of MDMA-CHMICA. A chemometric model based on a combination of chromatographic/mass spectrometric impurity signatures and stable-isotope-ratio data was implemented to establish links between samples from different seizures and internet shops.

Conclusion: Identification of synthesis impurities and profiling of cannabimimetics in Spice-products by state-of-the-art MS and IRMS techniques is an important factor to stay on track with the ongoing introduction of NPS to the worldwide illicit drug market.

Notes:

03: DETECTED NPS CASES BY FORENSIC TOXICOLOGY UNIT OF UNIVERSITY OF FLORENCE IN THE FRAME OF THE EUROPEAN I-SEE PROJECT

Fabio Vaiano, Giovanni Serpelloni, Valeria Catalani, Elisabetta Bertol

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Identification and quantification of New Psychoactive Substances (NPS), in biological and non-biological samples, represent a hard challenge for forensic toxicologists. NPS are increasingly emerging on illegal drug market. Forensic Toxicology Division (FTD) of University of Florence founded an Innovative Research Unit (U.R.I.To.N., Unit of Research and Innovation in Forensic Toxicology and Neuroscience of Addiction) focused on NPS issue working also as Coordinator of the European I-SEE Project (JUST/2013/ISEC/DRUGS/AG/6426). Its main goals are: to strengthen NPS information exchange between Italy and South East Europe (Slovenia and Croatia); to support the development and consolidation of national Early Warning System networks; to create a joint mechanism for information and good practice exchange and mutual learning; to increase information flow towards Law Enforcement and health professional about NPS. Here we describe the NPS detection cases analyzed by the FTD in seized material, in vivo and in post-mortem samples. A new screening method in liquid chromatography-tandem mass spectrometry (LC-MS/MS) was built and fully validated for fast and sensitive detection of 69 compounds (28 synthetic cannabinoids, 19 synthetic cathinones, 5 phenetilamines, 5 amphetamines, 3 indanes, 2 piperazines, 2 tryptamines, 2 phencyclidine, methoxetamine, ketamine and nor-ketamine). Over the last few months several NPS detection cases were brought up. In seized material were found: 3-MMC, 4-FA, penthedrone, methoxethamine, AB-FUBINACA, 5-MAPB+bk-2C-B+an under investigation substance (likely a synthesis intermediate). In vivo: JWH-073, MDPV, AM-694, AB-FUBINACA, 3-MMC+4-MEC, 3-MeO-PCP. Postmortem analysis: mephedrone, Methylone+MDMA (and its metabolite)+ketamine (and its metabolite). Our experience about NPS detection in seized material, in vivo and in postmortem samples is a clear sign that their spread is real and worrisome. Analytical methods, as the one here described, are effective tools to face this phenomenon. The European I-SEE Project could be a starting point in planning valid counteractions against NPS.

Notes:

O4: SHARING OF DATA FOR RAPID IDENTIFICATION AND DETECTION OF NEW PSYCHOACTIVE SUBSTANCES (NPS) IN EUROPE

Guillou Claude, Reniero Fabiano, Holland Margaret, Lobo Pereira Vicente Joana, Chassaing Hubert, Kolar Kamil, Tirendi Salvatore.

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New psychoactive substances (NPS) are a serious public health threat. Their identification is a challenge for their increasing number and diversity and for the lack of standards. In the past two years almost two new substances were discovered every week. The European Monitoring Centre for Drugs and Drug Addiction (EMCDDA) is now monitoring more than 560 NPS [1]. NPS are generally imported from non-EU countries, with 'false declaration' of identity. The project CLEN2SAND of the Joint Research Centre (JRC) of the European Commission and of DG TAXUD aims at establishing efficient approaches for fast identification of unknown chemicals, NPS, medicines and also plant extracts. The JRC is providing scientific support with a focus on the following tasks:

- Set-up an electronic analytical repository of NPS
- Test fast screening techniques
- Support European law enforcement laboratories with JRC's analytical facilities and expertise
- Establish harmonised methods for identification of chemical structures
- Dissemination of the scientific methods and best practices

This work also aims at facilitating the sharing of data and knowledge between various organisations (forensic, customs, public health) and is undertaken in collaboration with the EMCDDA. The lecture will present the progress, the main results achieved and the lessons learnt:

- Analysis of more than 100 seizures, among which identification of 20 new substances, and of about 250 purchased samples [2]
- Edition of methods for analysis and interpretation of chemical structures (NMR, HR-MS...)
- Use of chemoinformatic tools
- Interoperability and sharing of data (eg. with Project RESPONSE and EMCDDA's EDND)
- Spectra for handheld portable devices

In conclusion, working groups for further needs (e.g. harmonisation of interpretation, quantification with NMR, etc...) will be suggested.

[1] European Monitoring Centre for Drugs and Drug Addiction and Europol (2016), EU Drug Markets Report: In-Depth Analysis

[2] J. Lobo Vicente et al., Forensic Sci. Int. 265, 107-115 (2016)

Notes:

O5: EU CO-FUNDED PROJECT RESPONSE: NEW PSYCHOACTIVE SUBSTANCES DATABASE

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Functionality of the public opened RESPONSE project "Drugs monographs" database [1] will be presented and explained. One can access database on the RESPONSE project website [2].

In the framework of the RESPONSE project numerous new compounds have been characterized by means of several analytical methods (GC-MS, FTIR-ATR, GC-MS-IR-condensed phase, HPLC-TOF, Ion chromatography, NMR and some other complementary techniques, when needed). To share gathered analytical information on NPS effectively, which means almost in real time and even before the databases updates are available, the public opened RESPONSE project "Drugs monographs" database was developed, tested and launched on web at the end of 2015.

This innovative approach and tool enhanced and speeded up the information exchange which is one of the most critical points, especially if one has in mind the speed of novel NPSs appearance. The free access database is also a good approach for additional cross-validation of data provided by the project (data can be checked world-wide).

Several contributors of samples (National Forensic Centre, Sweden; SPL Portugal; Forensic Science Institute Zurich, Switzerland; Institute of forensic medicine, University of Freiburg, Germany; Hungarian Institute for Forensic Science; INPS Lyon, France; Estonian Forensic Science Institute) already improved the quality of database as well as the speed of its growing.

In March 2016 (after a non-formal agreement) four CLEN2SAND project reports kindly provided by the European Commission Joint Research Centre (JRC), DG TAXUD and the CLEN have been implemented into database. Collaboration between forensic and customs communities has been strongly enhanced.

Furthermore, the Slovenian National Forensic Laboratory (which coordinates the RESPONSE project) is also a partner in the I-SEE project [3] from the same ISEC call. It was agreed that I-SEE project chemical characterization data or joined reports (when applicable) from both projects will be published in the "Drugs Monographs" database as well.



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[1] "Drugs monographs" database: http://www.policija.si/apps/nfl_response_web/seznam.php

[2] RESPONSE project website: <http://www.policija.si/eng/index.php/generalpolicedirectorate/1669-nfl-page-response>

[3] I-SEE project web site: <http://www.dss.unifi.it/vp-107-i-see.html?newlang=eng>

Notes:

O6: THE REVISION OF THE ENFSI QUALITATIVE SAMPLING GUIDELINES

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The original sampling document produced by ENFSI DWG describes a number of sampling methods, from arbitrary methods to methods with a statistical background. The document focused on sampling in cases where large numbers of relatively homogeneous material are available. During the last few years the document has undergone a revision thanks to the work done by Sonja Klemenc and Hugh Coyle, with external review of their draft by Prof. Colin Aitken, University of Glasgow. This revision will be brought to the Business meeting for approval and agreement to publish. Hugh Coyle will present the main points that were updated in this revision.

Notes:

O7: DRUG IDENTIFICATION BY UPLC-QTOF-MS

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Analysis of drugs in tablets and powders from Police and forensic investigations can be performed on a variety of techniques such as Gas Chromatography-Mass Spectrometry (GC-MS), High Performance Liquid Chromatography – Diode Array Detection (HPLC-DAD), Nuclear Magnetic Resonance (NMR), infrared (IR) and Raman spectroscopy.

Ultra Performance Liquid Chromatography coupled to Quadrupole Time-of-Flight Mass Spectrometry (UPLC-QTOF-MS) with Data-Independent Acquisition (DIA) offers an alternative way to determine known or new drugs in tablets and powders.

We have developed a simple clean-up step where 50 mg of tablet or powder is dissolved in methanol, filtered and diluted by a factor of 50 to an end concentration of 2-200 mg/L of a drug (if the tablet or powder contained 1-100% of that drug). 0.3 µL is injected in the UPLC-TOF-MS system that uses a 15 min gradient. The drugs can be identified by Target or Non-target analysis. The difference between the two analysis types is the amount of information in the library. The Target library contains structures, retention times, and exact mass of fragments, while the Non-target library only contains structures. The Target analysis is easy to use while the Non-target analysis relies on predicted in-silico fragments.

Notes:

O8: NPS DATA HUB FOR NMR AND OTHER ANALYTICAL DATA

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A data hub for New Psychoactive Substances (NPS) is in the process of being established by the BKA in cooperation DEA and NIST (USA). It is primarily intended for (platform independent) NMR data. However, all kind of other analytical data can be added as well.

The data hub will be accessible for all official forensic laboratories. The mode of operation is Wikipedia-like. Data can be uploaded by laboratories all over the world. A witnessing tool is implemented in order to ensure mutual data validation.

Data can be easily up- and downloaded by drag and drop. Chemical structures and meta data, such as names and molecular weights, are automatically registered and visualized by the data hub. Internet catalogues from chemical suppliers are used to read out meta data. Other smart internet sources are used too, e.g. for the creation of IUPAC names for given structures.

An annotation tool allows to highlight certain points or to give remarks directly on the analytical data, which is also automatically visualized in the data hub window.

The platform presented cannot only be used for data sharing. It would be an awesome tool for reporting the newest emerging drugs and for establishing an up-to-date common platform for the nomenclature of new substances.

Notes:

O9: IN-HOUSE REFERENCE MATERIAL CERTIFICATION PROJECT

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In Hungary, new psychoactive substances (NPSs) scheduled in generic ban containing four groups of species and an additional list contains named molecules. Since January 2014, manufacture, supply and even possession of NPS are punishable according to the Criminal Code, and the category of the penalty is depends on the amount of pure substance. Couple of scheduled illicit drugs has individual limit of quantity, but the limit of active compounds in case of all sorts of NPSs is consistently 10 grams. This fact obliges the forensic chemists to exact and precise quantification in case of not only scheduled illicit drugs but of NPSs too. However, quantitative measurements by chromatographic techniques need reference materials certified for quantitative analysis. Those are expensive – and even not available – and their procurement is often difficult, not only of quantitative, but also qualitative reference materials. Furthermore, the diversity of NPSs and their often just brief existence on the illicit market might make the high-priced, valuable reference materials facilely outdated.

Approximately one and a half years ago, an in-house reference material certification project has been started at the Hungarian Institute for Forensic Sciences Drug & Arson Investigation Department for alleviating of this sensitive problem. Usually, parts of seized evidences or in house-synthesized compounds become in-house reference materials after purification (such as column chromatography, recrystallization and salt modification) and complex analytical characterization by GC-MS, ATR-FTIR, NMR, HPLC and occasionally melting point measurement and XRF. Beyond the qualitative certification, the homogeneity test and quantitative NMR technique makes possible to certify in-house quantitative reference materials.

The in-house reference material certification project gave a dynamic, quick and suitable solution for the qualitative and quantitative analysis of NPSs and other illicit drugs.

Notes:

O10: A METABOLOMICS STUDY OF RETROSPECTIVE FORENSIC DATA FROM WHOLE BLOOD SAMPLES OF HUMANS EXPOSED TO 3,4-METHYLENEDIOXYMETHAMPHETAMINE: A NEW APPROACH FOR IDENTIFYING DRUG METABOLITES AND CHANGES IN METABOLISM RELATED TO DRUG CONSUMPTION

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The illicit drug 3,4-methylenedioxyamphetamine (MDMA) has profound physiological cerebral, cardiac, and hepatic effects that are reflected in the blood. Screening of blood for MDMA and other narcotics are routinely performed in forensics analysis using ultra-performance liquid chromatography with high-resolution time-of-flight mass spectrometry (UPLC-HR-TOFMS). The aim of this study was to investigate whether such UPLC-HR-TOFMS data collected over a two-year period could be used for untargeted metabolomics to determine MDMA metabolites as well as endogenous changes related to drug response and toxicology. Whole blood samples from living Danish drivers' positive for MDMA in different concentrations were compared to negative control samples using various statistical methods. The untargeted identification of known MDMA metabolites was used to validate the methods. The results further revealed changes of several acylcarnitines, adenosine monophosphate, adenosine, inosine, thiomorpholine 3-carboxylate, tryptophan, S-adenosyl-l-homocysteine (SAH), and lysophosphatidylcholine (lysoPC) species in response to MDMA. These endogenous metabolites could be implicated in an increased energy demand and mechanisms related to the serotonergic syndrome as well as drug induced neurotoxicity. The findings showed that it was possible to extract meaningful results from retrospective UPLC-HR-TOFMS screening data for metabolic profiling in relation to drug metabolism, endogenous physiological effects, and toxicology [1].

[1] K.L. Nielsen, R. Telving, M.F. Andreasen, J.B. Hasselstrøm, and M. Johannsen, *J. Proteome Res.* **2016**, *15*, 619-627.

Notes:

O11: AN UPDATE ON SOME NOVEL BY-PRODUCTS FORMED DURING THE SYNTHESIS OF AMPHETAMINE VIA THE APAAN TO P2P LEUCKART ROUTE

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International controls on the supply of phenyl-2-propanone (P2P, BMK) has resulted in illicit manufacturers of amphetamine type drugs exploring other means of obtaining the desired starting materials. Seizures of alpha-phenylacetoacetonitrile (APAAN) have increased dramatically in recent years along with a reduction in seizures of P2P. The by-products formed from the synthetic route of APAAN to (P2P, BMK) will be explored. The emphasis in this presentation is to explore any differences in by-product formation depending on which acid was used to perform the hydrolysis of APAAN to P2P. If no clean up or refinement of the P2P from APAAN takes place, then, it is possible that these APAAN hydrolysis by-products will be seen in the profile of any amphetamine produced from APAAN derived P2P.

By-products from clandestine synthesis will most likely be found amongst the waste products of any clandestine lab site under investigation rather than with the desired product. Therefore an increased understanding of these by-products will be useful to clandestine laboratory investigations.

Notes:

O12: ALPHA-PVP AS AN ACTIVE COMPONENT OF HERBAL HIGHS

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Alpha-Pyrrolidynovalerophenone (α -PVP) is a synthetic derivative of cathinone. It has been one of the most popular new psychoactive substances (NPSs) present on the drug market in Poland in recent years. On 1 July 2015 α -PVP became controlled. Two illicit production sites synthesising this substance were seized in Southern Poland. Both cases were linked to local groups of 'football hooligans'. The first production facility, where brephedrone was also manufactured, was seized in July 2013 in Chorzów. Approximately 50 kg of α -PVP was produced in this site, which was destined both for the domestic market and to be exported. The other synthetic drug production facility, dismantled in October 2014 in Kraków, also produced brephedrone and NEB (N-ethylbuphedrone). The amount of α -PVP and brephedrone seized was 4.5 kg. Location of production sites may be related to the frequency with which α -PVP was found in drug cases especially in Southern regions.

Reported doses of α -PVP were 15 - 300 mg. The routes of administration of the drug include: oral, insufflation, injection and smoking (inhalation).

Unexpectedly, we dealt with a great number of herbal samples which turned out to contain α -PVP. Seized products which were received by our Laboratory were first screened by GC-MS. Quantification of α -PVP was performed by UPLC-PDA.

In relatively many herbal samples (201) α -PVP was found as the only psychoactive component: in samples of mass less than 0.4 g - mean concentration was 9.7 %, in 0,4 - 0.5 g samples: 25.1 % and in samples of mass above 0.5 g samples: 29.4 %. Mean amount of α -PVP was: 39, 128, 160 mg, respectively. In 21 samples α -PVP was present in the mixture with MPHP. In such samples α -PVP concentrations were: in 0.35 g samples: 7.3 %, in 0.50 g samples also varied: 4.1 %. Mean amount of PVP was: 26 and 20 mg, respectively.

Notes:

O13: THE USE OF SOLID PHASE GC-IRD FOR FORENSIC DRUG ANALYSIS

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A direct deposition infrared spectrometer coupled with a gas chromatograph (GC-IRD) provides a reasonable alternative/complimentary technique to traditional instrumentation. Forensic exhibits come in a multitude of forms, including residues, liquids, powders and other matrices routinely observed by the forensic drug chemist. The GC-IRD has demonstrated the ability to differentiate positional isomers of designer drugs of abuse such as the isomers of 3,4-methylenedioxymethamphetamine (MDMA), synthetic cannabinoids, NBOMe and other phenylethylamine type compounds. The instrument can also be utilized as a second technique/second sampling resulting in more robust data. The GC-IRD utilizing solid phase deposition is a capable instrument in the analysis of compounds commonly encountered in the forensic drug chemistry laboratory. Discussion will include presentation of data as well as QA/QC and operational suggestions.

Notes:

O14: DWG PROFILING SUBCOMMITTEE: OVERVIEW OF ACTIVITIES

Natacha Gentile^a, Fabrice Besacier^b, Pierre Esseiva^a, Filip Van Durme^c

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The presentation will cover the activities done by the profiling sub-committee in 2015: holding a profiling seminar in the frame of the RESPONSE project and sending a survey on profiling practices.

A feedback on the profiling seminar will be given as well as a brief overview on the survey.

Discussion of the future of the profiling subcommittee.

Notes:

O15: RESULTS OF PROFICIENCY TEST 2015

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The qualitative and quantitative results (z-scores and En numbers) of ENFSI DWG Proficiency Test 2015 will be presented in this lecture. In current year, a preliminary stability test was completed besides the standard PT procedure. Short discussion on quantitative NMR technique will also be presented because of increasing number of submitted results provided by this method.

Notes:

O16: SUBCOMMITTEE CHEMOMETRICS - STATUS REPORT 2016

Michael Bovens^a, Sami Huhtala^b, Ivo Alberink^c, Björn Ahrens^d

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Objective of the subcommittee is to compile a software based working tool and a step by step guideline to enable a forensic chemist to evaluate a dataset by multivariate analysis.

Beside a classification or discrimination of a sample to a group, the user will be able to give these results the correct interpretation, knowing the strengths and weakness of the applied method.

The project has been proposed to be performed under MP 2016. Given its acceptance a final product can be awaited end of 2019.

Notes:

O17: COMMUNICATION SUBCOMMITTEE: STATUS FOR DWG ON MEMBERS WEBSITE

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One year ago, the new members website - intranet.ensfiweb.eu - was launched. I will give a status and show some examples of what kind of information you can find.

Notes:

O18: OVERVIEW OF DWG QA SUBCOMMITTEE ACTIVITIES

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Since the last DWG meeting the QA subcommittee has been working on one main project, the establishment of a database for IR-spectra of new psychotropic substances: Revision of old spectra and validation of new spectra were performed. Three IR supporting documents “issues in identification of substances by FTIR”, “how to submit spectra for the ENFSI DWG IR-library” and “verification process of IR-spectra in ENFSI DWG IR-library” were prepared with support of the Response project.

In November 2015 a subcommittee member was the DWG representative on the QCC meeting in Tallinn.

The DWG PT guidelines were not updated since they are in agreement with the new QCC PT document.

According to QCC the annual update of the accreditation status of the member laboratories is no longer necessary and this activity is closed.

Notes:

O19: UPDATE FROM THE SCIENTIFIC WORKING GROUP FOR THE ANALYSIS OF SEIZED DRUGS (SWGDRUG) AND THE ORGANIZATION OF THE SCIENTIFIC AREA COMMITTEES (OSAC)

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This presentation will summarize recent activities from the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) and the Organization of Scientific Area Committees (OSAC).

The SWGDRUG core committee is revising Part IIIB and Part IVB of the SWGDRUG Recommendations. These sections pertain to the categorization of analytical techniques, and the validation of analytical methods, respectively. The development of new instrumental technologies and their introduction to seized-drug laboratories have brought up the need to clarify the categorization of techniques into A, B or C groups. Revisions to SWGDRUG Recommendations Part IIIB will not involve changes in these categories, but will focus on providing guidance regarding the different modes of operation and variations within a technique and how their categorization could be affected.

Revisions to Part IVB incorporate clarifications on the performance characteristics that should be evaluated during the validation of qualitative and quantitative methods. Also, examples of method validation schemes for routinely used techniques, like color test, gas chromatography – mass spectrometry (GC-MS) and infrared (IR) spectroscopy will also be provided.

SWGDRUG committee members have also developed Supplemental Document 6 (SD-6) titled 'Examples of Measurement Uncertainty for Net Weight and Count Extrapolations'. This document will assist seized-drug analysts by providing step-by-step examples for estimating uncertainty for scenarios where the net weight of an exhibit is obtained via extrapolation or when the total count of a dosage unit exhibit needs to be extrapolated. SD-6 is currently undergoing a 60-day public comment period closing on May 23, 2016.

This presentation will also provide an overview of OSAC and the process for development and approval of documentary standards. Recent activities of the OSAC Seized Drugs subcommittee will also be discussed.

Notes:

O20: NEW PSYCHOACTIVE SUBSTANCES IN EUROPE - DIVERSITY OF THE MARKET AND ASSOCIATED CHALLENGES

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Abstract is not available.

Notes:

O21: PROPOSAL OF JOINT EFFORTS TOWARDS A EUROPEAN CLOUD SPECTROSCOPY SYSTEM FOR IDENTIFICATION OF NEW DRUGS

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Law enforcement, forensic and customs laboratories are facing challenges for identification, detection and monitoring of new psychoactive substances (NPS).

Ideally a collective “cloud spectroscopy system” developed across various organisations and countries would help the work of these laboratories for these tasks.

This would enable the sharing and access to chemical and spectroscopic data on new substance within short laps of time after a new compound has been identified for the first time.

On the other hand the identification of new chemicals is time-demanding and requires highly sophisticated analytical instrumentation such as nuclear magnetic resonance and high resolution mass spectrometry.

Although these analytical techniques are very powerful and can also be used beyond the scope of chemical identification of the main substance, for instance for identification of impurities, profiling and also for quantitative purposes (e.g. qNMR), they also require the competence of well trained experts for operating the instrumentation and for interpretation of the data.

In our experience the interpretation of data (especially for NMR and HR-MS) by experts constitutes a “bottleneck”, is time consuming and could also be improved regarding traceability and validation aspects.

The proposal of common working group of experts, for examining and discussing the possibilities for harmonization and sharing of best practices and knowledge in these techniques could therefore be considered across different networks and organizations. This should also facilitate long-term interoperability of data management systems as well as enhancing quality and reliability of data for such a “European Cloud spectroscopy system”.

This proposal has to be considered in the context of the rapid emergence in number and diversity of NPS which, in order to be globally efficient, needs to be addressed with a collaborative and collective scientific approach across and with the contribution of various public national and international organizations.

Notes:

O22: RESEARCH INSTITUTE OF FORENSIC SCIENCES AND CRIMINOLOGY (RIFSC) "DRUGS" SECTOR

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Presentation of the Research Institute of Forensic Sciences (RIFSC) –Bulgaria, structure and tasks. The network of the Bulgarian Drugs Labs. Role and responsibility of the “Drugs” –Sector as a separate unit in the RIFSC. Workflow in the “Drugs” –Sector- analytical approach in drugs identification. Quantitative and qualitative examination. Short statistic in respect of type of the drugs. Challenges and projects for future development of the “Drugs”-sector.

Notes:

O23: LEGISLATIVE APPROACH ON NEW PSYCHOACTIVE SUBSTANCES (NPS) IN CYPRUS - LATER CHANGES, RESULTS OF ISEC PROJECT ON NPS

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The international community as well as many EU Member States, in an effort to protect public health, have tried a wide range of legislative approaches to control the NPS market.

On International and European level in order to determine whether a new psychoactive substance should be controlled, an integrated risk assessment is required. This approach, while is well documented, takes a long time to be completed. On the other hand, generic legislation approach, which is implemented in Cyprus as well as in some other countries, does not need an extended risk assessment and acts preventively on controlling New Psychoactive Substances.

New Psychoactive Substances have been appearing in Cyprus since 2009. Through these years many National Law changes took place. At the beginning, not yet knowing the "NPS trend", lists of prohibited substances (Schedule I and Schedule II) of Narcotic Drugs and Psychotropic Substances Law (L.29/1977) were extended (until 2011). Because of the growth of the market over the past few years, as well as the serious harms of NPS, a generic approach was developed by the State General Laboratory and implemented in 2011 including nine groups of molecules, one at Schedule I and eight at Schedule II.

The last change was made in 3/2016 and NPS's were categorized in twenty groups according to their chemical structure and harmfulness. Five of them fall to Schedule I (fentanyl, tryptamines, phenethylamines, phenylpropylamines, benzofury) of Cyprus National law and fifteen fall to Schedule II (benzoyl, naphthoyl and phenylacetyl indoles, naphthoyl pyrroles, indanes, indenenes, cyclohexyl phenols, cathinones, benzylpiperazines, indole and indazole aldehydes, indole and indazole carboxylic acids as well as indole and indazole carboxamides and cyclohexanamines).

Meanwhile, the ISEC project JUST/2013/ISEC/DRUGS/AG/6411: "New psychoactive substances (NPS): Building knowledge and evidence-based training, through research" in which State General Laboratory is partner, is about obtaining information of the prevalence of NPS in Cyprus and the "Darknet market". Some of the results will be also presented.

Notes:

O24: BLOTTING PAPER IDENTIFICATION PROCESS – RECALCULATING

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The identification process of blotting paper suspected as illicit drug stamps has at least two main aspects: the need to ID and measure the active agent (qualitatively and quantitatively) and the visual description of the supporting agent – the perforated blotting papers. In this work we will try to look at exhibits suspected as LSD or NBoMe stamps in two different forensic points of view: First, the technical point of view which focuses on the general shapes of the exhibits, the imprints, the perforation tool marks, the possibilities of the active agent to spread throughout the paper, the signatory of the added substance solution on the paper and the options to transform substances between the paper layers. Second, from the scientific point of view (i.e. the eyes of the producer), which examines the physical and the chemical characters of the blotting paper that make them the common platform for psychedelic drugs units, the impact of the perforation on the homogeneity of the active agent on the paper, zoom in into the blotting process etc. We will then return to re-inspect the international guidelines for blotting paper sampling according to the conclusions of the first 2 steps.

Notes:

O25: THE STABILITY OF THE PHYSICAL FEATURES OF 'ECSTASY' TABLETS

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Background: The stability of the physical features mass, diameter, thickness, volume and hardness of 'ecstasy' tablets are important if these are used to link samples of tablets for intelligence purposes. In order to explore the stability of these features testing were conducted on samples from a batch of tablets to determine how the relative humidity (RH) and temperature may affect these characteristics. Storage conditions for 'ecstasy' tablets were recommended based on the findings of these investigations.

Method: Eight different samples of white 'ecstasy' tablets with versace logo were used. Four samples of tablets (n = 10 each) were stored at a RH of 33 ±1% and another four samples at a RH of 75 ±1% and temperatures of 5, 15, 25, 35 or 40°C respectively. The physical characteristics of each tablet in the eight samples were measured over a 16 week period.

Results: Significant increase in the mean diameter, thickness and volume of tablets were recorded when these were stored at 75% RH during the 16 week period regardless of temperature. Significant decrease in the mean hardness of tablets was noted after 16 weeks when tablets were stored at 75% RH. The mean mass, diameter, thickness and volume increased more significantly in samples of tablets stored at 75% RH and 40°C when compared to 33% RH and 35°C.

Conclusion: The measurable features of 'ecstasy' tablets, when stored under different RH and temperatures were found to change, mostly when stored at high RH (75%) RH, and a temperature of 40°C. The least changes in the measurable features occurred when tablets were stored at 33% RH and at temperatures between 15 and 35°C. Thus it is recommended that batches of 'ecstasy' should be stored at low RH (≈ 25%) and temperature (≈ 25 °C) if used for intelligence purposes.

Notes:

O26: DRUG PREVENTION IN BERLIN

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Prevention is a theme, on which several institutions work on.

There is a strict division between drug prevention and prevention in cases of addiction.

The competence for legal drug prevention is placed at the police.

For prevention in case of addiction exist in Berlin several institutions, some of them are private, others are state owned.

But the police in Berlin does not only work on legal drug prevention, they also care about all types of prevention in conjunction with violence.

In my talk I give you an overview about the work of Berlin police on legal drug prevention especially in schools, and my part of this education.

Notes:

ABSTRACTS POSTER PRESENTATIONS

P1: NFIDENT- RELIABLE DRUG ANALYSES WITHIN A DAY

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In the Netherlands the National Police, the Public Prosecutor and the NFI felt the need to accelerate the process of identifying the most common drugs. NFIDENT makes it possible to handle the small cases ('user amounts') much faster with less time and effort.

NFIDENT is part of the Innovation Program "Remote Forensics" of the NFI: Connecting the lab to the scene. In a co-creation project with the National Police and the Public Prosecutor we made a process to accelerate drug analysis for the 'big four': cocaine, heroin, MDMA and amphetamine. This is about 80% of the samples the NFI get for regular identification.

Analysis of these four most prevalent drugs is performed by police officers with a mobile GC-MS. The data of the GC-MS is sent to the NFI and interpreted and reported by a reporting officer of the NFI.

Within one day identification is made possible, without transportation of the material. The NFI maintains the knowledge and developments of the drugsmarket for intelligence purposes.

Proof of concept, validation steps and pilots with two police districts are successfully completed. The first case has already been nominated for court and the suspect is convicted.

Notes:

P2: ILLEGAL PRODUCTION OF LEGAL HIGHS IN THE LEGAL LABORATORY "THE NEW CZECH A. SHULGIN"

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In September 2015 the Institute of Criminalistics Prague was asked to execute an expert evidence to an unusual case. The case was concerned by illegal production of Mephedrone, Ephedrone and Metaqualone. These substances were clandestinely produced by an employee with university education which worked in a legal chemical and pharmaceutical laboratory. This company aimed at development, production, and marketing of generic active ingredients.

The Mephedrone was produced by Friedel-Crafts acylation of toluene with propionylchloride under anhydrous aluminium chloride catalysis. The product of this reaction is 4'-methylpropiofenone and by-product 2'-methylpropiofenone. Bromination of 4'-methylpropiofenone and subsequent reaction with methylamine yielded final product Mephedrone. From by-product was produced 2-MMC by analogy. 53 g of the final product was obtained with this reaction. Final product was in nearly pharmaceutical purity with the traces of 2-MMC.

The Ephedrone was produced by bromination of propiofenone and reaction with methylamine. Almost 1547 g of the final reaction mixture was obtained, which contained 9,5 % base.

The Metaqualone was produced by reaction anthranilic acid with o-toluidine in presence of acetic anhydride, P₂O₅ and POCl₃. 180 g of the final product was obtained. This product was not quantified but according to qualitative analysis this product had no impurities.

All substances are prohibited in the Czech Republic.

Notes:

P3: APPLICATION OF AUTOMATED SAMPLE PREPARATION FOR DRUG ANALYSIS

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The number of drug samples has doubled during the last five years at the Hungarian Institute for Forensic Sciences. To improve our laboratory capacity and quality assurance a customised robotic system had been developed for routine sample preparation.

The robotic system measures the weights of test tubes before and after sampling, calculates the weights of the samples and the required amounts of solvent. The automated system carries out the extraction, dilution, filtration of the samples. After the crimping, the vials are transferred automatically to the sample tray of the chromatographic system. The automated procedure is integrated into the barcode based evidence identification system of the laboratory. Barcodes are used to identify the test tubes and vials in the sample preparation process and afterwards, during the whole instrumental analysis.

The major part of seizures consists of herbal materials (cannabis or materials impregnated with new psychoactive substances), hence the first method introduced for the automated system was the sample preparation of "herbals" for routine GC-MS identification.

The robotic sample preparation is preferred to manual process because of reliable automated data transfer, computer generated reports, adjustable target concentrations and the traceability of the whole process by barcodes from the seized materials to the evaluation of the results.

Notes:

P4: QUALITATIVE DETERMINATION OF RESIDUAL SOLVENTS IN COCAINE SAMPLES BY STATIC HEADSPACE-GC-MS (DEVELOPMENT OF COCAINE PROFILING METHODOLOGY)

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A method for the determination of volatile compounds, utilized in illicit cocaine production, was developed. Static headspace analyses were performed on a GC-MS Model 7890-5975C (Agilent Technologies), equipped with a MPS 2 robotic sampler with a heated headspace syringe (Gerstel). The critical method parameters were optimised as well their effects evaluated.

Samples of cocaine (hydrochloride) were obtained by 53 "unlinked" and 7 "linked" seizures by the Slovenian Police in the period from October 2014 to February 2016. Over 60 volatile compounds were detected. Most of them were identified as residuals solvents, occluded in the crystal matrix of the final cocaine product. The rest identified compounds were most likely by products-artifacts, formed during the processing of illicit cocaine (methyl benzoate, chloromethane, chloroethane, etc.) and other impurities (e.g. fatty acid esters). Identified compounds were encountered in different amounts and frequency of appearance. The detection limits of typical compounds from chemically different groups between 0,0022-0,1600 ppm were determined on a w/w basis for 100 mg cocaine base.

Additionally, the method has been established for estimating if cocaine samples are linked or not. Approx. 40 volatile compounds were preliminarily selected as variables for profiling methodology. The first correlations of cocaine samples are obtained.

Keywords: cocaine, static headspace-GC-MS, occluded compounds analysis

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Notes:

P5: CANNABIS PREPARATIONS FOR "SELF MEDICATION" - CHEMICAL CHARACTERIZATIONS OF SOME RECENTLY SEIZED PRODUCTS

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Cannabis is one of the oldest cultivated and versatile plants. Even in the past it was used to attain fibers, building material, for self-medication and also as recreational drug.

Δ^9 -tetrahydrokanabinol (THC) and other cannabinoids, may have potential in the treatment of pain, nausea, epilepsy, obesity, wasting disease, addiction, autoimmune disorders, and other conditions.

In the recent years a growing number of cannabis products for self-medication were seized in Slovenia such as tinctures, oils, suppositories, creams and honey.

Analyses of the composition of those products were performed by GC-MS, HPLC and Headspace GC-MS.

Notes:

P6: HEROIN PROFILING - METHODOLOGY DEVELOPMENT AND FIRST IMPRESSIONS

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Profiling of heroin samples for study trafficking and distribution networks is commonly performed by dealing with the characterization of main heroin alkaloids, e.g. diacetylmorphine, meconine, acetylcodeine, acetylthebaol, 6-monoacetylmorphine, papaverine and noscapine. Several GC methods with derivatization to avoid problems associated with transacetylation were already developed, mostly by using flame ionization detector.

In National Forensic Laboratory we used GC-MS method for heroin profiling. The heroin samples were derivatized with MSTFA prior the analysis. The specific target and qualifier ions were selected for the main heroin alkaloids to measure the area of the peaks.

For successful assessment of links between heroin samples with different diacetylmorphine concentration, samples were prepared by weighing homogenized powder containing approximately 1,2 mg of diacetylmorphine base, which is equivalent for 15 mg of 8% heroin concentration. The concentration threshold above 8% was established through validation of the GC MS method. We observed non-linear behavior of GC-MS method, which was not mentioned in previous studies of heroin profiling methods.

Similarity (dissimilarity) among samples was evaluated by multivariate analysis using Pearson correlation. To achieve similar magnitude of target variables for Pearson correlation, each area of main heroin alkaloids was pretreated with a standard deviation, calculated from areas of particular alkaloid through long-term measurements.

To evaluate the discriminative power of the method the distribution of inter and intra variability was investigated.

Intravariability distribution was evaluated by measuring the similarity value (in our case the Pearson correlation) between pairs of samples from the same seizure. 100 measurements have been calculated for evaluating the intravariability of heroin.

Intervariability was evaluated by measuring Pearson value between pairs of samples selected from different seizures in the period from 2014 to 2015. 70 real samples from 70 different seizures were analyzed.

We determined the thresholds for linked and non-linked samples.

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Notes:

P7: NOVEL NPS - TEST PURCHASES OVER THE INTERNET - WHAT IS INSIDE THE PACKAGE?

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What we have learned from the test purchases in general?

The test purchases performed in the frame of the RESPONSE project so far, show that drugs users can never be sure of what they get when buying from internet vendors. The rate of the "false advertised" compounds is at approximately 20% - 30%. This poses a serious health risks for the population of NPS users. Therefore all activities which can raise the awareness are welcomed and should be enhanced at all levels (national and international EWS stakeholders are the key actors).

Analytical strategies and the results of some "false advertised" compounds will be presented.

One of the options with regard to harm reduction are also enhanced activities and capacities for the support of "anonymous testing" of samples provided by users [1].

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[1] The role of the National forensic laboratory in the Slovenian EWS and wider – RESPONSE and I-SEE projects - Sonja Klemenc, National forensic laboratory, Slovenia, ENFSI-DWG meeting, Bled, Slovenia, May 10th-12th, 2016 [see poster P8].

Notes:

P8: THE ROLE OF THE NATIONAL FORENSIC LABORATORY IN THE SLOVENIAN EWS AND WIDER - RESPONSE AND I-SEE PROJECTS

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The poster will present the role and activities of the Slovenian National Forensic Laboratory (NFL) in the Slovenian Early warning system. Some monitoring results and the results of common activities (case examples) obtained in the framework of two complementary; currently running EU co-funded projects will be presented:

- a) RESPONSE - Collect, Analyse, Organize, Evaluate, Share – A Response to Challenges in Forensic Drugs Analyses (Short Project Title – Response), Grant agreement no: JUST/ 2013/ISEC/DRUGS/AG/6413]; see: <http://www.policija.si/eng/index.php/generalpolicedirectorate/1669>
- b) European project “I-SEE” for strengthening information exchange between Italy and South East Europe neighbouring countries on New Psychoactive Substances, Grant agreement no: JUST/2013/ISEC/DRUGS/AG/6426]; see more at <http://www.dss.unifi.it/vp-107-i-see.html>



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Notes:

P9 : GETTING AHEAD OF THE GAME: COMPLETE ANALYSIS OF NEW PSYCHOACTIVE SUBSTANCES

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The world of recreational drugs has dramatically changed in recent years with new psychoactive substances (NPS) emerging every year (> 300 legal highs since 2009 and 97 in 2003)[1]. NPS, also called legal highs, are produced and formulated under non-regulated conditions and therefore constitute an unpredictable risk for consumer's health [2,3].

The wide spread and popularity of synthetic NPS are nowadays also affecting the course of justice when dealing with cases of illegal drug use. Some of the legal substances have analogues with a high degree of structural similarity to their corresponding illegal entities, for example there is the need to distinguish between regioisomers. Therefore, the analytical data that accompanies drug cases must be able to discern between those compounds with certainty.

In this work we propose and demonstrate the utility of NMR to complement IR and MS data for the unequivocal structural identification of illicit drugs and legal highs.

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