EUROPEAN PROJECT RESPONSE to challenges in forensic drugs analyses

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COLLECT, ANALYSE, ORGANIZE, EVALUATE, SHARE – A RESPONSE TO CHALLENGES IN FORENSIC DRUGS ANALYSES

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INTRODUCTION

Over the past few years Europe has seen an unexpected growth in the number, type and availability of new psychoactive substances (NPS). One of the main challenges to effectively respond to new psychoactive substances is the detection of these followed by the correct identification.

NPS are mainly detected in forensic and customs labs. The difficulty lies in identifying them.
To identify new substance reference material (RM) or special techniques are needed. However, RM are mostly not available in real time. Therefore, most of laboratories have to rely on comparison of their results to available MS and/or FTIR spectra in searchable electronic libraries (not updated in real time) or existing information exchange channels and/or open source data.

This work presents the first impressions of a systematic (pro-active) tackling and identification of the new psychoactive substances (NPS) as applied in the frame of EU co-funded ISEC project “RESPONSE". The project is divided into several modules (see Table 1).

Table 1: Main response project modules

Activity	Issue
Collect/purchase NPS	Setting up a robust and efficient system for detection and collection/purchasing of the new compounds appearing on the market .
Analyses	Chemical characterizations – to define core analytical techniques necessary for NPS identification structure elucidation - to provide reliable MS and FTIR spectra.
Evaluation	To establish validation procedures which guarantee scientifically valid results.
Organize data & information	Integration of all analytical data available for the substance in analytical reports and development of databases and tools for implementation of acquired IR and MS spectra in ENFSI DWG databases.
Share	The main project objective is **to share**: - knowledge, information (& spectra) effectively through different communication platforms - project`s identification reference materials" among partner`s laboratories and wider and **- to rise the awareness** among NPS users

SOURCES OF MATERIALS (PURCHASED/SEIZED/COLLECTED)

The main goal of the project is to provide numerous MS and FTIR spectral data on new or recently reported NPS. Several sources of materials will be applied (see Table 2). Undoubtedly certified **reference materials** (CRM) are the most reliable source. The drawback is that CRMs are not available in real time, i.e. (when laboratory detects a new compound). The producers (of CRM’s) are usually several steps - months behind the “gray” internet market.

Thus as the main source of NPS **the internet vendors** of **research chemicals** (**NET-NPS**) are foreseen (the target number of NPS being purchased over the internet is 200-300). The strategy and challenges for finding and purchasing novel NPS over the Internet are described at a separate poster presented at this conference [L. Ask Reitzel et all, Systematical methodology for finding novel NPS (New Psychoactive Substances) over the Internet, EAFS-2015, Prague, 2015]. So far around 50 samples have been purchased form 10 different web pages.

Other important sources have been the Police and Customs **seizures**, samples **collected** by SI NGO (nongovernmental organizations) or seized materials provided from some other forensic laboratories.

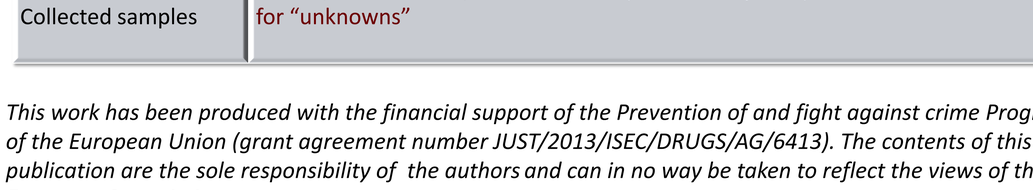

Figure 1: Purchased samples obtained from different web vendors (examples)

CHEMICAL CHARACTERIZATIONS (ANALYTICAL METHODS) FOLLOWED BY THE INTERPRETATION

The selection of methods depends on the sample type and information available about the sample (see Table 2). CRMs are mostly not problematic. The starting point is always GC-MS. If there are no matches in available libraries (NFL in house library, SWGDRUG, NIST, ENFSI, etc), HPLC-TOF is applied to find exact mono-isotopic mass and proposed empirical formula. For newly detected compounds the structure is confirmed or elucidated by NMR. FTIR-ATR is scanned when one compound is detected and FTIR condensed phase when we have mixture of compounds or pills, blotters and similar.

Table 2: Types of materials and analytical methods

Type of materials	Analytical methods
Reference materials	GC-MS, FTIR-ATR, GC-MS-FTIR-(condensed phase) - optional
Test purchases	GC-MS, FTIR-ATR, GC-MS-FTIR-(condensed phase), HPLC-TOF, NMR
Seized samples	GC-MS, FTIR-ATR and/or GC-MS-FTIR-(condensed phase), HPLC-TOF and NMR for “unknowns”
Collected samples	GC-MS, FTIR-ATR and/or GC-MS-FTIR-(condensed phase) and HPLC-TOF and NMR for “unknowns”

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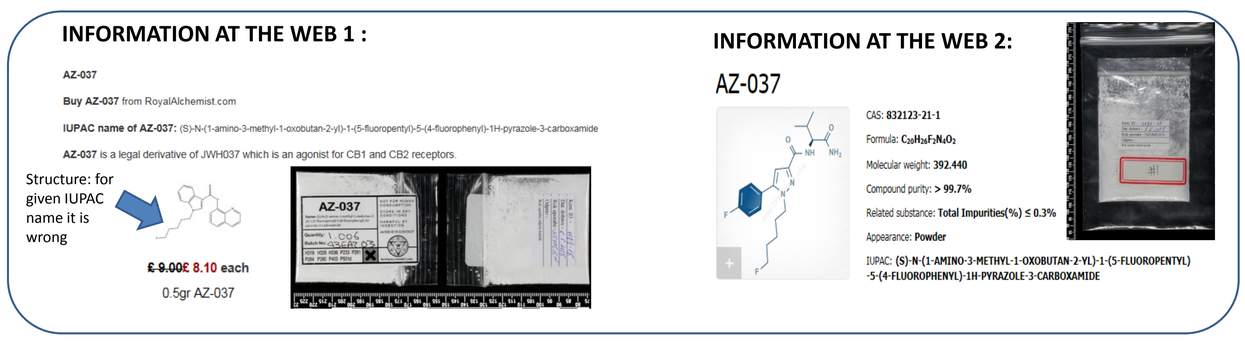
CHARACTERIZATION: example of purchased samples AZ-037 (2 web pages):



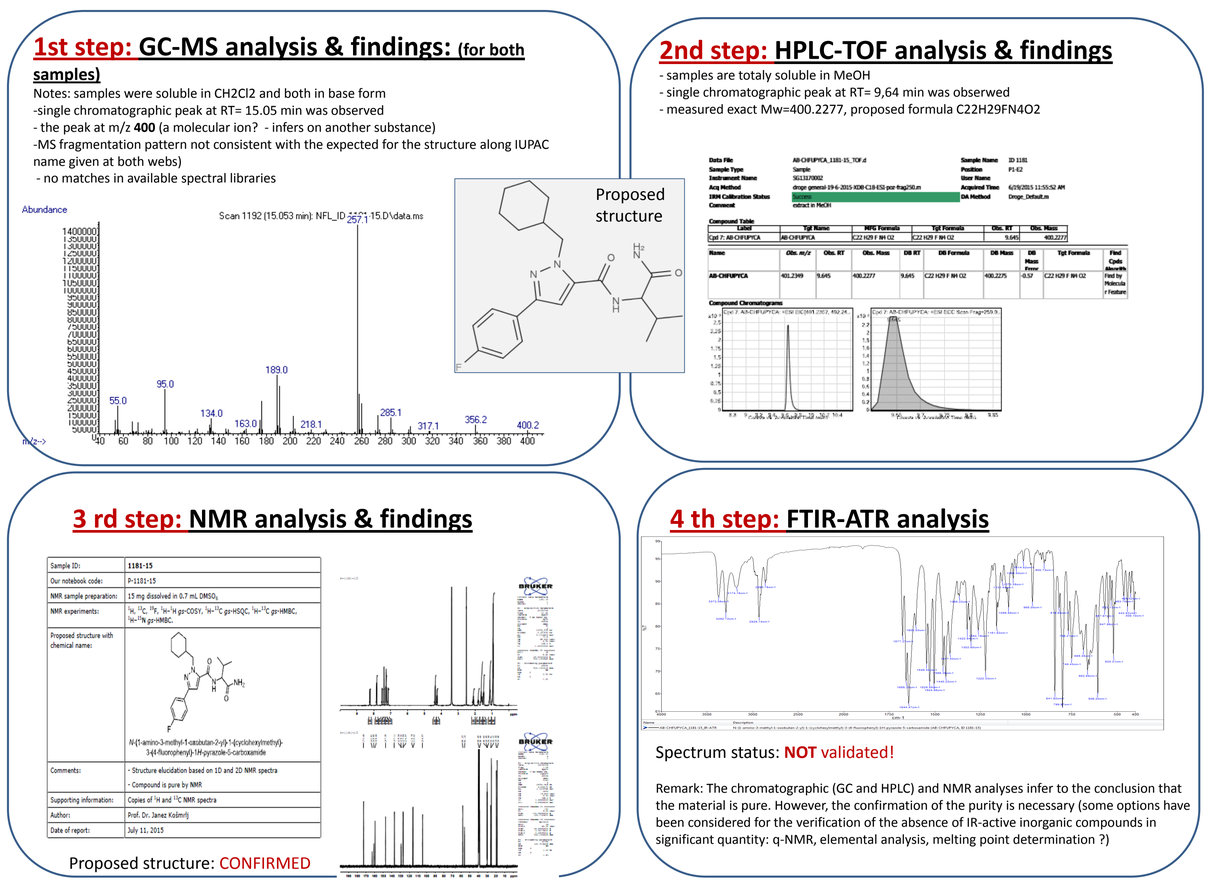


Figure 2: Characterization example

EVALUATION of analytical data:

Validation procedures which guarantee scientifically valid results have been discussed at the first RESPONSE project steering committee meeting. For successful validation all available information shall be taken into account. The most challenging is the validation of analytical data (reliability of spectra) for new compounds, which has not yet been reported. When the structure is elucidated and confirmed by NMR mass spectrum fragmentation pattern has to be checked (by manual interpretation and/or by some supporting computerized tools).

Validation of FTIR-ATR spectra is even more demanding; however in depth explanation is beyond the scope of this poster. The idea of FTIR spectra validation was the comparison of FTIR-ATR vs FTIR condensed phase spectra. It was expected that for pure compounds (in base form) correlation coefficients will be in good agreement (correlation coefficient (based on cosine function) of 0.98 or better was expected. Some preliminary experiments did not confirm the expectations, at least not for all tested compounds (CRMs were applied).

Cross validation and detection of possible errors in the results reported by coordinators laboratory will be enhanced by sharing the collected/purchased samples among other project partners. "Retrospective" validation will be possible when more reports about the same compound from different sources (laboratories) will be available in “data banks”.

ORGANIZE DATA & SHARE THE INFORMATION

The project has the goal to disseminate the results of the project to the widest communities. Therefore, to share gathered analytical information promptly, the public open RESPONSE project web page has recently been launched. Main analytical information on characterized NPS (CRMs, collected, seized) with several search tools is shown (and will be updated continuously) in "Drugs Monographs" section (see Figure 3). In addition the following connections have been enhanced: cooperation with ENFSI-DWG (MS and FTIR spectra libraries managers) and membership in general, EMCDDA, EUROPOL; Slovenian Early warning system including NGOs. At the non-formal level cooperation/communication and information exchange with several other complementary projects (I-SEE, SPICE - profiling, CLEN2SAND) was established as well.

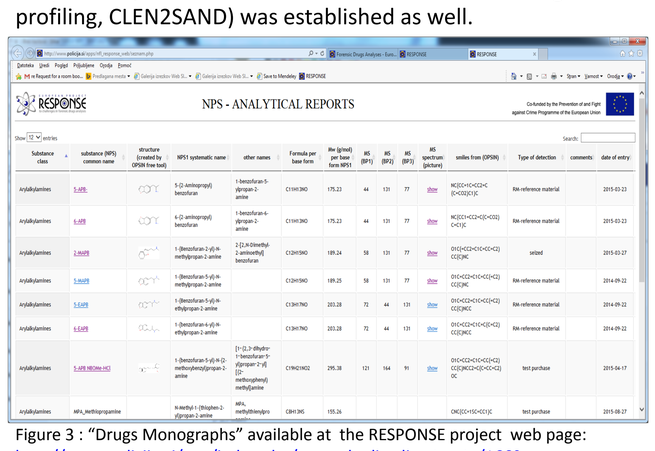


Figure 3 : “Drugs Monographs” available at the RESPONSE project web page: http://www.policija.si/eng/index.php/generalpolicedirectorate/1669

AWARNESS

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. From 50 purchased samples the rate of “false advertised” compounds is at approximately 20%.

This poses a serious health risks for the population of NPS users.

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