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On-site identification of psychoactive drugs by portable Raman spectroscopy during drug-checking service in electronic music events

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Identification of psychoactive drugs by portable Raman instrument during on-field drug checking

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Identification of psychoactive drugs by portable Raman instrument during ~~on-~~field drug ~~checking~~

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Abstract

Introduction. In the last decade, hundreds of New Psychoactive Substances have burst into the marketplace, making both the scientific community and ~~users~~ lacking of adequate information about their diffusion and effects. In this scenario, drug-checking services have been recently proposed as effective tools for harm reduction policies and ~~to generate~~ a global picture of the circulating drugs in different geographical areas.

Methods. In this paper, we report the results obtained by the analysis of 472 alleged drugs, tested during 27 night events within the first formal implementation of drug checking in Italy, by means of a portable Raman device.

Results. Illicit substances were detected in 304 samples. Findings included MDMA (106 samples), ketamine (87 samples), cocaine (51 samples), amphetamine (47 samples), methamphetamine (2 samples), heroin (2 samples) and NPS (9 samples). Two samples were identified as precursors of psychoactive substances. A result linked to a non-controlled substance was displayed with 38 samples. Finally, 128 samples resulted as inconclusive when tested on-field. Among these, in 68 cases the user allowed us to sample a small part to perform a delayed laboratory analysis by GC-MS or LC-MS/MS.

Discussion and Conclusions. Drug checking by Raman proved effective in the identification of psychoactive drugs including NPS and ~~to explore~~ the drug ~~distribution found~~ in various recreational settings at different times. The ~~on~~ field testing activity revealed the presence of several NPS in the nightlife scenario, often in replacement of traditional illicit drugs, thus posing a high overdose risk and a life-threatening situation.

Keywords: drug checking; Raman; NPS; harm reduction

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Introduction

Nightlife plays an essential role in the personal growth of the youth and allows easy social interactions for people of any age around the world. Although nightlife is commonly associated with celebration, festivals and a sense of group identity, it also provides the setting for risk taking and experimentation, especially regarding the consumption of alcohol and drugs [1]. While the drug scenario has remained basically unchanged through the 20th century, the first two decades of the new millennium are facing the emergence of a new phenomenon, identified with the "NPS" acronym worldwide. As a matter of fact, hundreds of New Psychoactive Substances (NPS) have burst into the marketplace in recent years, making both the scientific community and users lacking of adequate information about the effects of these new drugs. Synthetic cathinones (a.k.a. "bath salts") raise particular concern because some of these drugs (e.g. mephedrone, methylone) are trafficked as replacements for ecstasy [2,3], but they entail unpredictable and often unknown adverse effects. Likewise, it occurs that also other classes of "traditional drugs", e.g. hallucinogens or heroin, are replaced or added with new designer compounds, making many drug users unintentionally or unknowingly using synthetic NPS. Even when NPS are intentionally purchased on-line, substantial risk exists that they are mislabeled, either because they contain chemical analogues of the ordered drug (e.g. 25B/C-NBOMe instead of 25I-NBOMe, pentedrone instead of 3,4-DMMC), or because the active principle differs from what was advertised on the website [4].

As an intervention of harm reduction within this context of drug use, a pill testing/drug checking service has been recently introduced in the nightlife scenario. For many users, drug checking is often the first point of contact with the social support system. Drug checking can be completed in a drug counseling center and also onsite, e.g. at parties, raves, and festivals [5]. Pioneer reports of drug-, pill-, and substance-testing have been published in the recent years, describing the identification of the active principle, particularly NPS, in different contexts [6-11]. Even though some limitations of drug checking have been recently highlighted [12], it is hardly disputable that

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testing drugs before they are consumed involves three primary advantages: (i) adverse effects (including overdose) can be avoided by the consumer; (ii) institutions in charge of the problem (such as hospitals and testing laboratories) and public health authorities are made aware when a new substance breaks into the market; and, (iii) a global picture of the circulating drugs is generated, with respect to the appearance time and the different geographical areas [13].

The most common analytical techniques used for drug checking include Thin Layer Chromatography (TLC), Gas Chromatography-Mass Spectrometry (GC-MS), Liquid Chromatography (LC), Raman Spectroscopy, colorimetric tests, Infrared Spectroscopy (IR) and Nuclear Magnetic Resonance (NMR). Most of these analytical techniques offer adequate performances: high specificity, good sensitivity, versatility with different matrices, quantitative analysis and comprehensive libraries. Unfortunately, most of them are not portable on field and require extensive operators' training. Moreover, they involve some handling of the analyzed material and its destruction, with consequent legal hurdles to overcome.

~~Aim~~ of our study was to identify the drugs purchased and commonly used by partygoers and music festivals attendants, using a Raman-based portable instrument. Raman spectroscopy is commonly used in chemistry to provide a structural fingerprint by which molecules can be identified [14]. In most cases, sample preparation is minimal or unnecessary, allowing for the non-destructive *in situ* analysis of tablets, powders, and liquids. This is particularly important with regard to the speed of analysis, prevention of sample contamination, and preservation of evidential material [15]. Moreover, the analysis can be performed through the drug-containing envelope, avoiding any contact with the operator, ~~together with its legal implications~~. Over the past decade, there have been numerous reports detailing the use of Raman spectroscopy to screen for drugs [16-20], and more recently a very few exploratory studies investigated the identification of NPS [21-25]. The identification of NPS aimed to risk assessment and drug control poses a great analytical challenge, due to the wide number of NPS already identified, the presence of adulterants in them, or the

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presence of NPS themselves as adulterants added to traditional drugs (e.g. MDMA), and the continued emergence of new and unknown chemical substances [25].

In this paper, we report the results obtained by the analysis of 472 alleged drugs, tested during 27 night events within the first formal implementation of drug checking in Italy, by means of a portable Raman device. The hazard caused by the unaware intake of drugs will be also highlighted.

Experimental

Drug checking procedure

All samples were tested during electronic dance music festivals, rave parties, GOA parties and street parades in the Italian territory, during 2016 and 2017. The drug-checking protocol consisted of the following steps: i) the substance to test is voluntarily taken by the user (or by someone on his/her behalf) to the drug checking service. The alleged drug is not actually handled by any social workers or technicians. The person itself requesting drug checking will insert a small amount of the compound in a plastic bag and he/she will take it back after the analysis is completed, with no need of dispose it; ii) a picture of the substances is taken; iii) rapid analysis with the portable Raman instrument is performed; iv) if the active principle or the main component is identified, the user is immediately informed about the result; v) in case a NPS is identified, a general warning accessible to all the participants of the event is released; vi) if the analysis result is "inconclusive", so allegedly no evidence of the presence of a prohibited substance is obtained, a small sample of it is collected and transferred to the lab for deferred GC-MS or LC-MS analysis; vii) a final report is published on a specific website; viii) in case a NPS is identified, a report is prepared and sent to the National Early Warning System.

Raman Instrumentation

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On-site drug checking was performed using a ThermoScientific TruNarc® portable Raman analyzer, running library version v1.6, equipped with a 785-nm Class IIIB laser at 250mW. Raman spectra in the interval 300-1800 cm^{-1} were recorded. The identification of the substances was performed by comparing the spectrum of the unknown compound with those present in a proprietary library containing traditional drugs, NPS, cutting agents, and precursors. When the compound is identified, the result is directly shown on the instrument display, without need of a connected PC. The results are color-coded to highlight four circumstances: (i) the instrument identifies one or more controlled substances (alarm result, red color); (ii) the instrument identifies one precursor used in the manufacturing of illegal drugs (warning result, orange color); (iii) the instrument does not identify any controlled substances, but recognizes a cutting agent present in its library (clear result, green color); (iv) the instrument does not identify any of the controlled substances, precursors, or cutting agents present in its library (inconclusive result, grey color).

GC-MS and LC-MS

Sample preparation and screening analysis for unknown substances were performed using a previously published method [26]. Briefly, a 6890N GC apparatus (Agilent Technologies, Milan, Italy) equipped with a 17-m fused-silica capillary column (J&W Scientific HP-5), of 0.2-mm inner diameter and 0.33-µm film thickness. Helium was employed as the carrier gas at a constant pressure of 23.24 psi. The GC oven temperature was set at 90°C for 1 min and then raised to 180°C with a 30°C/min heating rate. The oven temperature was maintained at 180°C for 7 min and then raised to 315°C with a 15°C/min heating rate. The GC injector and transfer line were maintained at 280°C. Full scan spectra in the interval 40-650 amu were acquired using a 5975 inert mass-selective detector (Agilent Technologies, Milan, Italy) operating in the EI mode at 70 eV. The qualitative identification of underivatized compounds was initially performed by comparing the full scan spectra obtained with those recorded in updated spectra libraries (PMWTox2, SWGDRUG version

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2.5, AAFS2012, CaymanSpectraLib), and then by comparing the retention times and relative abundances of the diagnostic ions obtained from a reference standard.

Targeted analysis for selected NPS was performed using a previously published method used for hair analysis [27]. Briefly, the analyses were performed using an Agilent 1290 Infinity LC system (Agilent, Palo Alto, CA, USA), interfaced to a QTRAP® 4500 mass spectrometer (AB Sciex, Darmstadt, Germany) equipped with an electrospray Turbo Ion source operated in the positive ion mode. A Zorbax Eclipse Plus C18 RRHD column (100 mm × 2.1 mm, 1.8 µm), protected by a C18 pre-column, was used for the separation of the target analytes. The column oven was maintained at 45°C and the elution solvents were water/formic acid 5 mM (solvent A) and acetonitrile/methanol 80:20 plus formic acid 5 mM (solvent B). After an initial isocratic elution at 95% A for 0.5 min, the mobile phase composition was varied by a linear gradient (A:B; v/v) from 95:5 to 45:55 in 2.5 min; then isocratic elution at 55% B was maintained for 0.5 min. The flow rate was 0.5 mL/min and the total run time was 5.5 min including re-equilibration at the initial conditions before each injection. MS/MS detection was executed in the selected reaction monitoring (SRM) mode.

Results and discussion

Altogether, 472 samples were analyzed using the ThermoScientific TruNarc® portable Raman analyzer, mostly powders, crystals, and pills. A summarized description of all results is presented in Figure 1. Illicit substances (alarm result) were detected in 304 samples (64.4%). Positive findings included MDMA (106 samples), ketamine (87 samples), cocaine (51 samples), amphetamine (47 samples), methamphetamine (2 samples), heroin (2 samples) and NPS (9 samples; 3%). Among NPS, the instrument identified mephedrone (2 samples), methylone, 4-fluoroamphetamine (4-FA), 2,5-dimethoxy-4-chloroamphetamine (DOC), 4-methylethcathinone (4-MEC), mexedrone, methoxyphenidine, and a mixture 4-FA/methylone. Furthermore, two samples were identified as precursors of psychoactive substances (warning result). Specifically, one was recognized as norephedrine and one as pseudoephedrine. For all traditional drugs, the result matched the

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anticipation offered by the subject who volunteered to drug-check the substance he or she was about to use. Even though a correlation with other independent methods of analysis for all samples would have been beneficial, we decided to not carry any illicit drug to the laboratory.

A result indicating a non-controlled substance (clear result) was displayed with 38 samples (8.0% of the total). The identified compounds included caffeine (10 samples), dypirone (3 samples), lidocaine, procaine, baking soda, calcium carbonate, cellulose, corn starch, lactose, epsom salt, polyethylene, mannitol and sodium sulfate. When caffeine or a cocaine cutting agent (e.g. mannitol) was identified, two analogous scenarios are plausible: either a fake drug was packaged, or the active substance (cocaine or speed, i.e., amphetamine) was diluted in a predominant amount of cutting agent, so that the illicit substance was masked in the analysis. On the other hand, the identification of licit drugs with psychoactive effects (e.g. dypirone, which was sold as MDMA during an event), still poses a significant health concern, because the user is not aware of the real composition of the substance he or she is taking. Taking into account all the alarm, clear, and warning results, a total of 344 samples (72.9%) was identified by the TruNarc, generally in less than 2 minutes per sample. In about 3 cases out of 4, the offered drug checking service proved to yield prompt answer to the subjects willing to test their alleged drugs.

The remaining 128 samples tested (27.1%) produced an inconclusive result as none of the controlled substances, precursors, or cutting agents present in the TruNarc library matched the experimental spectrum. For 68 of them, the user allowed us to sample a small part of the dose to perform a delayed laboratory analysis by GC-MS or LC-MS/MS. Notably, no law infringement was committed in the sampling, as no occurrence of illicit drugs had been evidenced. A summary of the results is presented in Table 1. In 29 cases (43%), a traditional drug was identified, including amphetamine, MDMA, heroin, or LSD. In 18 cases (26%), adulterants/diluents or licit pharmaceuticals drugs were detected, including caffeine, acetaminophen, dypirone, modafinil, lidocaine, metronidazole and oxycodone. Three more samples resulted to not contain any active principle. The remaining 18 samples were found to contain a NPS. The active principle found were

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5-MeO-MiPT (5-Methoxy-N-methyl-N-isopropyltryptamine; 4 cases), 2-CI (2,5-Dimethoxy-4-iodophenethylamine; 2 cases), MXE (Methoxetamine; 2 cases), 25B-NBOMe (2 cases), 2-CB (2,5-dimethoxy-4-bromophenethylamine), 4-AcO-MET (4-Acetoxy-N-ethyl-N-methyltryptamine), 4-FA, 25I-NBOMe, DOC, DOM (2,5-Dimethoxy-4-methylamphetamine) and DMT (N,N-Dimethyltryptamine). Overall, 27 samples out of 472 (5.7%) proved to contain at least one NPS. The possible reasons why the TruNarc was unable to identify the NPS in 18 cases out of 27 were either (i) their low amount/concentration, (ii) the high fluorescence of the main component, corresponding to the active principle itself or some excipient/additive/filler, (iii) the lack of their Raman spectrum in the current library. While the third issue can be easily handled by updating the library periodically, to keep pace with the introduction of NPS into the black market (or at least their analytical characterization), the first two limitations appear not to be easily overcome, since they require further hardware or software improvement. In particular, an important challenge when using Raman spectroscopy is the interference of fluorescence, which can eventually mask the Raman signal completely and result in a significant amount of background noise. Fluorescence is encountered particularly with plant-based narcotics, and substances that are pigmented with an array of colors, insomuch that drugs like heroin and illicit tablets that contain pigments and binders are challenging, and results from plant material like marijuana are impossible to generate [28]. Changing the operating wavelength can surely be a future development [24,28], basically designing systems at higher excitation wavelengths (e.g. from 785 to 1064 nm), in order to minimize fluorescence. However, more Raman scattering will occur with the more energetic excitation wavelength (i.e. at 785 nm) [29].

Finally, we observed that the presence of NPS in the tested samples was often in disagreement with the user's expectation (see Table 2). The replacement of traditional drugs with NPS is a well-known phenomenon. In previous studies [30-32], we combined surveys and hair analysis to demonstrate that MDMA users were often taking NPS without being aware. Most of the times, alleged extasy crystals or pills, believed to contain only MDMA, were composed either entirely or partially by

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some NPS. In the present study, 15 samples out of 121 (i.e. 1 out of 9) expected to be MDMA turned out to be an unexpected NPS. While some were simply "fake drugs" with none active principle in it and consequently represent a minor danger, other were containing some NPS. In this study, we found that also hallucinogens are no longer containing LSD. While in the common belief a "hallucinogen" is always an acid, nowadays it is actually very likely that instead a NPS, and particularly a compound of the NBOMe series, is often spotted on the blot. This general situation, in which a drug would be used without knowing the real effects, represent a further risk to the health of drug users.

Conclusions

The present study has double value, one of which is merely technical and the other essentially social. For the first time, the portable Raman-based ThermoScientific TruNarc® instrument was used for an extended on-field drug checking investigation, i.e. without prosecuting commitment. Drug checking by Raman spectroscopy proved effective in the identification of several psychoactive drugs, including NPS, and to explore the drug distribution found in various recreational settings at different times. In particular, portable Raman instrumentation demonstrated several advantages within these contexts, because it allows the direct sample analysis through water, glass, and plastic bags, avoiding direct contact with the substance, and it is non-destructive, non-invasive, and fast. Moreover, it does not require specific facilities and power supply. All these features proved essential for the fulfillment of our project's goal, which aimed to NPS qualitative identification. For the future, the regular update of instrument's library with spectra collected from the most recent NPS remains an essential requirement.

This project also represented the first formal implementation of a drug-checking activity in the Italian territory. The on-field testing activity revealed the presence of several NPS in the nightlife scenario, often in replacement of the traditional illicit drugs. Since several of these substances are potentially more toxic than the usual recreational drugs, their intake poses a high overdose risk and a life-threatening situation, especially for unaware users. With this said, the identification of a

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particular substance (either suspected or unsuspected) does not necessarily equate to harm, given that drug use and individual health risks are also influenced by various other factors, such as social contexts, route of administration and dose. Therefore, drug checking is a service that has to be integrated in outreach interventions or in services that offer drug prevention or harm reduction advices and counseling. In conclusion, in order to pursue a real harm reduction policy in different nightlife contexts, we envision that national governments would authorize and coordinate the work of local organizations able to offer efficient and comprehensive drug-checking services with the medical staff appointed to provide counseling and emergency intervention.

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Conflict of interest

None to declare

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381

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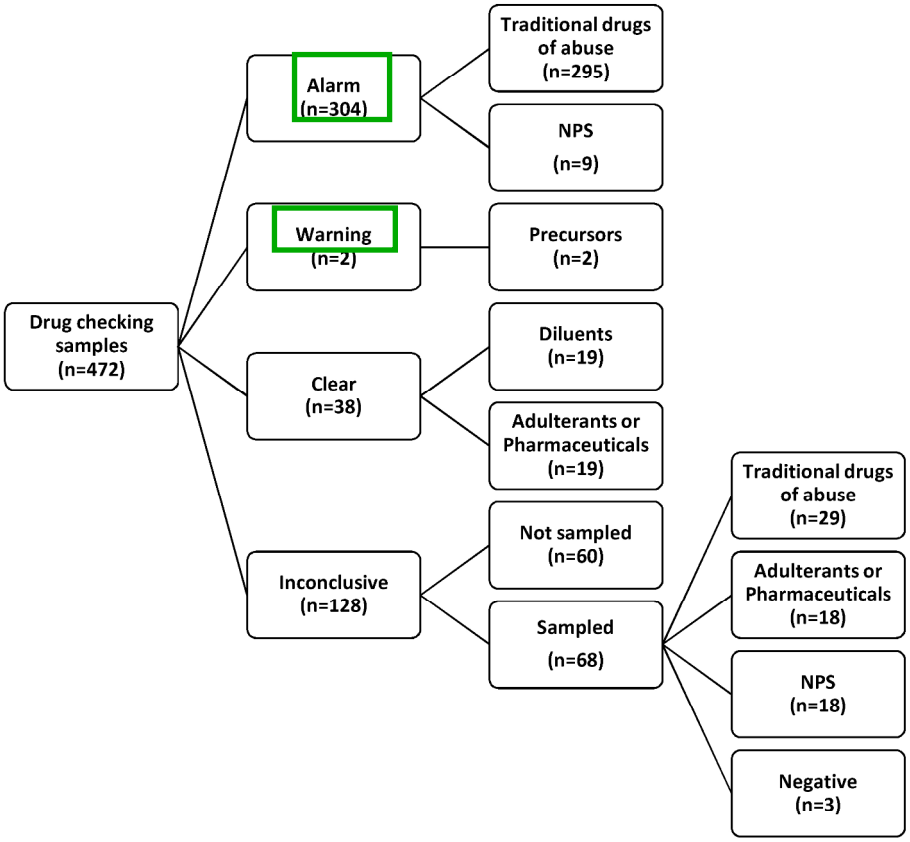

Figure 1 Summary of findings during the drug-checking activity

266x251mm (300 x 300 DPI)

Table 1. GC-MS and LC-MS analysis for samples with inconclusive result after drug checking by TruNarc®

Substance	Number of cases	Notes
Amphetamine (AMP)	11	
MDMA	9	
Heroin	4	
LSD	3	Too fluorescent for Raman detection
Opium	1	Too fluorescent for Raman detection
Cocaine	1	Found in traces
Total traditional drugs	**29 (42.6%)**	
Caffeine	8	Found alone or in combination with traces of AMP
Lidocaine	2	
Modafinil	2	Not present in TruNarc library
Acetaminophen	1	
Dipyrone	1	
Metronidazole	1	Not present in TruNarc library
Levomepromazine	1	Not present in TruNarc library
Buprenorphine	1	In combination with lidocaine
Oxycodone	1	
Total adulterants/diluents pharmaceuticals	**18 (26.5%)**	
5-MeO-MiPT	4	Not present in TruNarc library
2C-I	2	Blot samples
MXE	2	
25B-NBOMe	2	Blot samples
25I-NBOMe	1	
4-AcO-MET	1	Not present in TruNarc library
4-FA	1	
2C-B	1	
DOC	1	Blot sample
DOM	1	Blot sample
DMT	1	Dry herb
Pentylone	1	
Total NPS	**18 (26.5%)**	
Negative samples	**3 (4.4%)**	
TOTAL SAMPLES	**68 (100%)**	

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Table 2. Cases of discordant result between user declaration and instrumental analysis outcome

Expected drug	Found drug	Samples (n)
MDMA	Amphetamine	3
MDMA	Heroin	1
MDMA	Buprenorphine + Lidocaine	1
MDMA	Metronidazole	1
MDMA	Levomepromazine	1
MDMA	Lidocaine	2
MDMA	Baking soda	1
MDMA	Dipyrone	1
MDMA	5-MeO-MiPT	2
MDMA	Methylone	1
MDMA	None	1
Amphetamine	Caffeine	7
Amphetamine	Methylone + 4FA	1
Amphetamine	Modafinil	1
Amphetamine + Mescaline	5-MeO-MiPT	1
Amphetamine	MDMA	1
Ketamine	Cocaine	1
Ketamine	Pseudoephedrine	1
LSD	25I-NBOMe	1
LSA	25B-NBOMe	1
Mescaline + 2C-B (aka "Solaris")	25I-NBOMe	1
Psilocybin	DOC	1
Tryptamine	4-AcO-MET	1
PMA	Ketamine	1
Mescaline	2C-B	1
Unknown (generic "Legal High")	4-FA	1
Bk-2CB	Pentylone	1
Generic NBOMe	2C-I	2
DMT	Dipyrone	1
	TOTAL	**40**

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