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N-MOC-MDMA a novel "masked" variant found by Argentina Federal Police

Abstract

NSP encompass both the appearance of new substances and the variation of other classic ones, among which are N-substituted derivatives of MDMA with substituents that function as a protective group or 'mask' in order to evade legal controls. Two different unrelated seizures were examined, physical characteristics and presentation indicating they would be destined directly for consumer use. Samples were analyzed with PE Clarus 600 - 600T GC-MSD. In both cases, the amphetamine derivative N-MOC-MDMA (N-methoxycarbonyl-3,4-methylenedioxymethamphetamine) was identified as a minor component mixed with MDMA, with ephedrine also present in the second one. Identification was carried out from theoretical deconvolution with the support of predictive software and SWGDRUG's v. 3.10 spectral library matching. Obtained results are congruent in N-MOC-MDMA identification according to revised bibliography. Bibliographic background indicates N-MOC-MDMA, among others, as a possible prodrug made with the aim of eluding controls for its distribution, and subsequent reconversion to the typical drug in a simple form. This hypothesis could be supported by the mixture identified in the samples, a consequence of a possible inefficient conversion. Detection of this new "masked"-type MDMA in Argentina demonstrates the need for its inclusion in the local lists of prohibited substances.

Keywords: N-MOC-MDMA, masked MDMA, MDMA protecting groups, PFA

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Abbreviations: N-MOC-MDMA, N-methoxycarbonyl-3,4-methylenedioxymethamphetamine;MDMA, 3,4-methylenedi oxymethamphetamine; NPS, new psychoactive substances; GC-MSD, gas chromatography with mass spectroscopy; ATS, amphetamine type stimulants

Introduction

New Psychoactive Substances (NPS) are in constant evolution, either because of newly developed drugs or the modification of already known ones, with the main objective of evading drug regulations and possibly augmenting addiction among users.[1] Within this group we find Amphetamine Type Stimulants (ATS), MDMA (3,4-methylenedioxymethamphetamine) being its most prominent representative. The organic synthesis uses amine protective groups for legitimate purposes and these include fluorenylmethyl oxycarbonyl chloride (FMOC-Cl), trichloroethyl chloroformate (Troc), tertiary butoxycarbonyl (t-BOC), p-toluenesulfonyl (p-tosyl), among others.[2] Changes in the production process can become evident in routine analyzes since changes can be found in the chromatographic profile of a substance.[3] Concealment methods cannot only be limited to physical or odor-related maneuvers, but chemical tactics have been a constant since changes in the synthesis routes from various precursors, as well as the use of protective masks or groups that make detection impossible or difficult.[4]

As opposed to what happens with other traditional drugs, ATS can be produced virtually anywhere and in relatively simple operating conditions. Their illegal production is carried out from legal precursors that can be obtained illegally, such as safrole, isosafrole and piperonal, used in the production of fragrances and flavorings. As well as other substances with limited legal applications such as 3,4-methylenedioxyphenyl-2-propanone (MDP-2-P) or piperonyl methyl ketone (PMK).[5] From the international and national control of these precursors, the illicit production had to look for other alternatives.[2]

Collins et al.[6] identified and characterized N-tert-butoxycarbonyl-3,4-methylenedioxymethylamphetamine (t-BOC-MDMA) in a seizureof alleged hair products. This substance was converted in acidic medium to MDMA, so it wasthen proposed to explore the possibility of it being an *in vivo* pro-drug, by experimenting with a simulated gastric juice. From this experience, it has been concluded that t-BOC-MDMA could slowly be transformed and absorbed as MDMA during the digestive process.

This is indicative of the use of "masked" substances or new precursors, which may becirculating in the illicit market and which, due to their structural transformation, are not detected by routine tests, thus evading legal controls.[2] This represents a great challenge for forensic science since it is necessary to be able to identify these "masks" and the underlying substances, especially since the addition of protective groups to the ATS that give rise to these "masked" substances turns out to be simple, fast and with good yields, although not its deprotection.[7]

t-BOC-MDMA was the first masked compound to be detected. Since 2015, several cases havebeen reported in Australia, New Zealand and the Netherlands and later in 2017 masked methamphetamine and MDA (3,4-methylenedioxyamphetamine) were also found, making evident the development of this chemical technique.[8]. In 2016 in Germany, a mixture of amorphous silicon dioxide mixed with t-BOC-MDMA was detected.[9] The t-BOC-MDMA, after dilute acid reflux, the original amine is obtained with good yield, while with the p-tosyl group it is not so easily cleaved.[2]

In recent years, there have been reports in Argentina about the dismantling of several illicit laboratories destined to pill production. Even with local consumption being low compared to other more readily available psychoactive substances (Comisión Interamericana para el Control del Abuso de Drogas (CICAD), 2019, 215),[10] there is indication of a rise in the annual prevalence of MDMA´s consumption, as reported by the United Nations Office on Drug and Crime.[11]

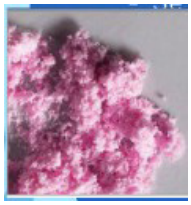
The División Laboratorio Químico of the Argentine Federal Police receives samples from judicial cases to be analyzed within the framework of the national law on narcotics, Law 23.737. The samples come from different parts of the country, although they are mainly focused on the City of Buenos Aires and adjacent areas, where drug crime is high. Regarding the volume of samples received, they can be dosage units ready for consumption as well as large quantities obtained from seizures in clandestine laboratories or shipments destined for trafficking. When a narcotic substance is found in the seizure material, it must be confirmed by instrumental techniques. The technique mainly used, in our Laboratory, is Gas Chromatography with Mass Spectroscopy (GC-MSD).

The work carried out focuses on the identification of a new "masked" substance, N-methoxycarbonyl-3,4-methylenedioxymethamphetamine (N-MOC-MDMA) found in two unrelated seizures where the examination of its physical characteristics and presentation, indicated that they were intended directly for the consumer.

Materials and methods

The Chemical Laboratory Division of the Argentine Federal Police receives seizures samples of different origins and varied dimensions. In this particular case, two seizures of different geographical origins were received, one of them from the City of Buenos Aires and the other from the suburbs (Table 1).

Table 1 Origin and physical characteristics of substances. (A)Suburbs. (B) Buenos Aires city

Origin	Physical characteristics	
Suburbs (A)	Pink Powder (0,06 g)	
Buenos Aires City (B)	Mushroom-shaped tablet (41 tablets)	

The samples were subjected to screening by thin plate chromatography (TLC Silcagel F254) with different running systems TL, TB, TAD and TE.[12]

The samples are derived to the instrumental area to continue with their analysis. Sample preparation is performed and the internal standard (dodecane solution 4mg/mL) is added. Instrumental analysis was carried out on a Perkin Elmer GC System Clarus 600 Gas Chromatograph interfaced with a PERKIN ELMER CLARUS 600T Mass Selective Detector (MSD). A 30 m×0.25 mm × 0.25µm PE-5MS column was employed using helium (5.0) carrier gas in the constant flow rate mode (1.0mL/min). Injection port temperature was 280 °C and the MSD interface temperature was 280 °C. The oven temperature program was 90 °C (hold for 5 min), ramped at 25 °C/min to 250 °C. Total run time 17min. Injections (1µL) were made in split mode (50:1). Electron ionisation (70 eV) was used, and a mass range of m/z 45 to 450 was scanned.

Results and discussion

The screening was performed by TLC, for both samples, the development of colored macules and Rf's not exact with the ATS controls used were observed.

The pink powder sample, obtained from seizures in the suburbs, presented three chromatographic peaks corresponding to MDMA (main component), Ephedrine/Pseudoephedrine, and N-MOC-MDMA, as a minor compound (Figure 1). While the pills obtained in the seizures of the City of Buenos Aires, shaped like a mushroom, presented two chromatographic peaks corresponding to MDMA, as the main compound, and N-MOC-MDMA, as a secondary compound (Figure 2). In both chromatograms the peak corresponding to the internal standard is also observed.

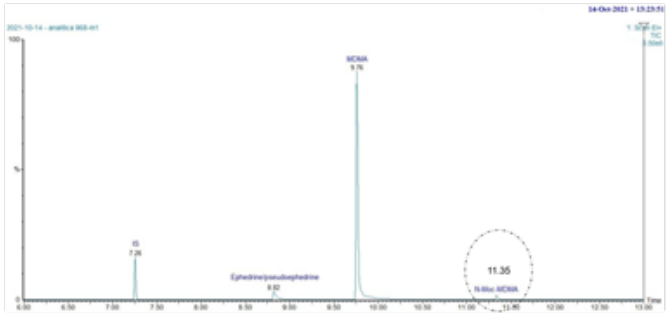


Figure 1 Chromatogram of the pink powder, obtained from the seizures in the suburbs. At time 11.35 the peak of N-MOC-MDMA is shown.

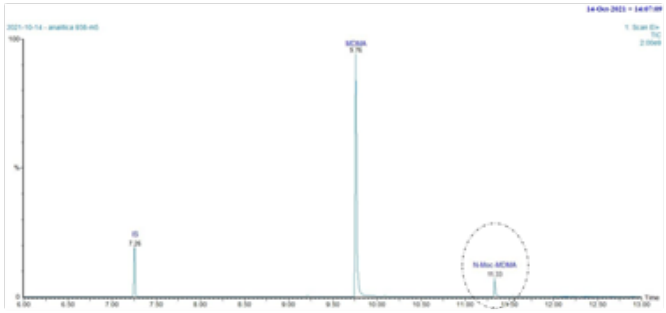


Figure 2 Chromatogram of a mushroom-shaped tablet, obtained from the seizures in the City of Buenos Aires. At time 11.33 the peak of N-MOC-MDMA is shown.

For the retention time assigned to N-MOC.MDMA, both chromatograms were analyzed in full scan. The *m/z* fragments were studied by theoretical deconvolution and with the support of predictive software and SWGDRUG's v. 3.10 spectral library matching.

The appearance of a single chromatographic peak in the GC-MSD analysis corresponding to N-MOC-MDMA is consistent with the thermal stability of MDMA N-methoxycarbonyl, so it can be characterized by this instrumental technique. This characteristic cannot be extrapolated to other substances such as ephedrine and pseudoephedrine.[8]

It is evident that the discovery of this "mask" is a warning regarding the evolution of the evasion maneuvers that drug trafficking constantly uses and allows the ability to adapt. Once this protective group is unmasked, they will surely use another.[2] Therefore, it is extremely important that forensic laboratories update themselves in this regardand assume the commitment to find specific and sensitive techniques to be able to detect this type of substance as well as new possible precursors.

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Conclusions

Concealment maneuvers by drug traffickers have evolved based on the ability of police forces to detect illicit substances. In recent years, masking tactics have become more common, mainly due to the difficulty in detecting them, since through classic screening analyzes they can evade legal controls.

From the instrumental analysis by GC-MSD, m/z fragments were obtained (Figure 3A) matching the SWGDRUG V. 3.10 spectral library (Figure 3B) and with the results published by Xu et al[7] for N- MOC-MDMA.

Figure 3 Mass spectrum for N-MOC-MDMA. A). Mass spectrum of both samples

B) Mass spectrum of SWGDRUG's v. 3.10 spectral library.

Bibliographic background indicates N-MOC-MDMA, among others, as a possible prodrug made with the aim of eluding controls for its distribution, and subsequent reconversion to the typical drug in a simple form. This hypothesis could be supported by the mixture identified in the samples, a consequence of a possible inefficient conversion. Detection of this new "masked"-type MDMA in Argentina demonstrates the need for its inclusion in the local lists of prohibited substances.

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Conflicts of interest

The authors declare that there are no conflicts of interest.

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