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Multi-residue determination of 10 selected new psychoactive substances in wastewater samples by liquid chromatography–tandem mass spectrometry

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ABSTRACT

New psychoactive substances (NPSs) have become increasingly popular in recent years. The analysis of these substances in influent wastewater (IWW) can be used to track their use in communities. In addition, an evaluation of the amount of NPSs released to the aquatic environment can be performed through the analysis of effluent wastewater (EWW). This study presents the development, validation and application of an analytical methodology, based on solid phase extraction (SPE) and liquid chromatography-tandem mass spectrometry (LC-MS/MS), for the determination of 10 NPSs in IWW and EWW. Synthetic cannabinoids, cathinones, piperazines and pyrrolidophenones are included among the target analytes. To the authors' knowledge, it is the first time that eight out of these substances (4'-methyl-pyrrolidinobutyrophenone (MPPP), a-pyrrolidinopentiophenone (a-PVP), 2-[(1S,3R)-3-hydroxycyclohexyl]-5-(2-methyl-2-octanyl) phenol (CP47,497), (1-naphthyl(1-pentyl-1H-indol-3-yl) methanone (JWH-018), (1-butyl-1H-indol-3-yl)(1-naphthyl) methanone (JWH-073), (4-ethyl-1-naphthyl)(1-pentyl-1H-indol-3-yl) methanone (JWH-210), (4-methyl-1-naphthyl) (1-pentyl-1H-indol-3-yl) methanone (JWH-122) and 2-(2-methoxyphenyl)-1-(1-pentyl-1H-indol-3-yl) ethanone (JWH-250)) are investigated in wastewater. The optimized conditions for the analysis of this set of compounds included a SPE clean-up step using a polymeric sorbent and the use of a pentafluorophenyl (PFP) chromatographic column. Despite the broad range of physicochemical properties of the analytes the method allowed acceptable absolute recoveries (40–109%) for all the studied compounds at different levels of concentration. Low method limits of detection (MLODs) were achieved, ranging between 0.3 and 10 ng/L except for BZP and CP47,497 (20 and 23 ng/L, respectively), allowing a reliable and accurate quantification of the analytes. The method was successfully applied to the analysis of IWW and EWW samples from five wastewater treatment plants (WWTPs) located in Santorini Island (a highly touristic resort in Greece). Four out of 10 compounds (a-PVP, CP47,497, JWH-122 and JWH-210) were detected at least in one sample, being the first evidence of their presence in wastewater. CP47,497 was the most ubiquitous and abundant compound, showing concentrations up to 634 ng/L in some cases.

1. Introduction

In recent years, a high number of new substances, commonly named as new psychoactive substances (NPSs), have been appeared on the market. According to the definition provided by EMCDDA, NPSs are new narcotic or psychotropic drugs, in pure form or in preparation, that are not controlled by the 1961 United Nations Single Convention on Narcotic Drugs or the 1971 United Nations Convention on Psychotropic Substances, but which may pose a public health threat comparable to that posed by substances listed in these conventions (Council Decision 2005/387/JHA) [1]. NPSs comprise a broad range of substances, including synthetic cannabinoids, cathinones, piperazines, tryptamines, amphetamine derivatives or pyrrolidinophenones. These compounds are often presented under "innocent" appearances (house scents, herbal mixtures, bath salts, or incenses) and have found a wide and efficient distribution mechanism through the "e-commerce" or specialized shops. These products claim to contain only "non-illegal" compounds and consequently have no limitations in their commercial distribution, although they exhibit important psychoactive effects [2,3]. The purpose of these substances is mainly to simulate the effects of common drugs of abuse,

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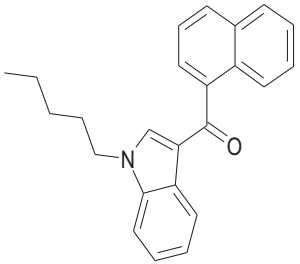
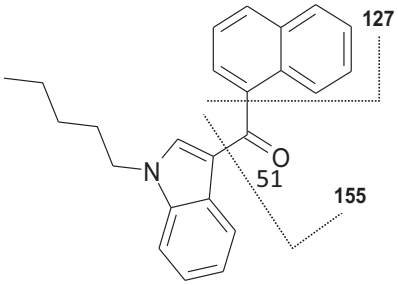
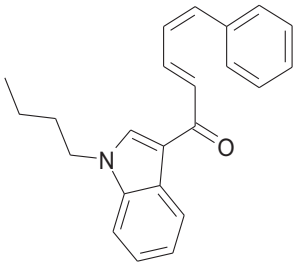
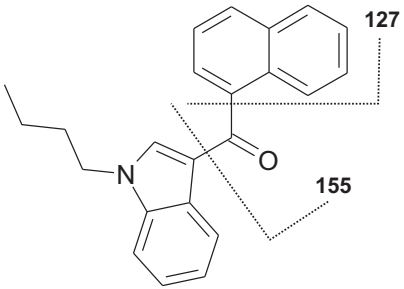
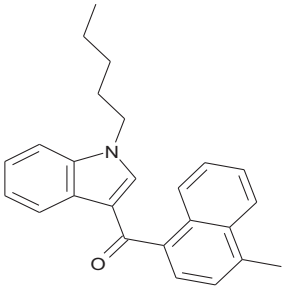
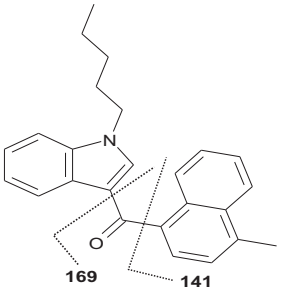
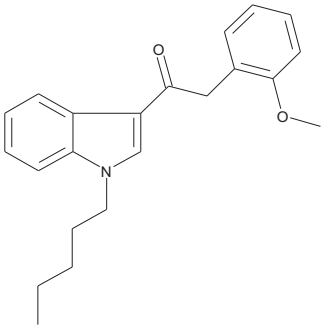
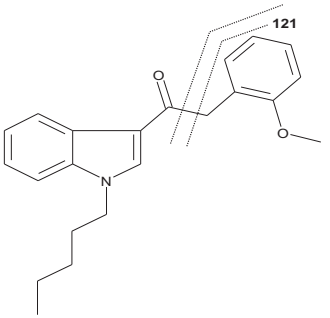
Table 1
Selected new psychoactive substances (NPSs), experimental ESI-MS/MS parameters, proposed product ions and predicted log *P* values.

Target compound[a]	CAS number	Chemical structure	Precursor ion	Retention time (min) ($n=6$)	Internal standard	Transition	Tube lens (V)	Collision energy (eV)	Proposed product ions	log *P*[b]
1-Naphthyl (1-pentyl-1H-indol-3-yl) methanone (**JWH-018**)	209414-07-3		$[M+H]^+$	16.68 ± 0.30	JWH-018-d9	342 > 155 342 > 127	82	25 44		6.51
1-Naphthyl (1-butyl-1H-indol-3-yl) methanone (**JWH-073**)	208987-48-8		$[M+H]^+$	15.99 ± 0.25	JWH-018-d9	328 > 155 328 > 127	76	25 43		6.07
(4-Methyl-1-naphthyl) (1-pentyl-1H-indol-3-yl) methanone (**JWH-122**)	619294-47-2		$[M+H]^+$	17.22 ± 0.15	JWH-018-d9	356 > 169 356 > 141	95	25 38		7.03
2-(2-Methoxyphenyl)-1-(1-pentyl-1H-indol-3-yl) ethanone(**JWH-250**)	864445-43-2		$[M+H]^+$	15.55 ± 0.20	JWH-018-d9	336 > 121 336 > 91	79	20 35		5.30
(4-Ethyl-1-naphthyl)(1-	824959-		$[M+H]^+$	17.43 ± 0.25	JWH-018-d9	370 > 183	96	25 24		7.47